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Demonstration Plan

Field Measurement Technologies for Total Petroleum Hydrocarbons in Soil

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Field Measurement Technologies for Total Petroleum Hydrocarbons in Soil

Prepared by

Tetra Tech EM Inc.
Chicago, Illinois

Contract No. 68-C5-0037

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Concurrence Signatures

The primary purpose of the demonstration is to evaluate innovative field measurement technologies for total petroleum hydrocarbons in soil based on their performance and cost as compared to a conventional, off-site laboratory analytical method. The demonstration will take place under the sponsorship of the U.S. Environmental Protection Agency (EPA) Superfund Innovative Technology Evaluation Program.

This document is intended to ensure that all aspects of the demonstration are documented and scientifically sound and that operational procedures are conducted in accordance with quality assurance and quality control specifications and health and safety regulations.

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Foreword

The U.S. Environmental Protection Agency (EPA) is charged by Congress with protecting the nation's natural resources. Under the mandate of national environmental laws, the agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. To meet this mandate, the EPA's Office of Research and Development provides data and scientific support that can be used to solve environmental problems, build the scientific knowledge base needed to manage ecological resources wisely, understand how pollutants affect public health, and prevent or reduce environmental risks.

The National Exposure Research Laboratory is the agency's center for investigation of technical and management approaches for identifying and quantifying risks to human health and the environment. Goals of the laboratory's research program are to (1) develop and evaluate methods and technologies for characterizing and monitoring air, soil, and water; (2) support regulatory and policy decisions; and (3) provide the scientific support needed to ensure effective implementation of environmental regulations and strategies.

The EPA's Superfund Innovative Technology Evaluation (SITE) Program evaluates technologies designed for characterization and remediation of contaminated Superfund and Resource Conservation and Recovery Act sites. The SITE Program was created to provide reliable cost and performance data in order to speed acceptance and use of innovative remediation, characterization, and monitoring technologies by the regulatory and user community.

Effective monitoring and measurement technologies are needed to assess the degree of contamination at a site, provide data that can be used to determine the risk to public health or the environment, and monitor the success or failure of a remediation process. One component of the EPA SITE Program, the Monitoring and Measurement Technology Program, demonstrates and evaluates innovative technologies to meet these needs.

Candidate technologies can originate within the federal government or the private sector. Through the SITE Program, developers are given the opportunity to conduct a rigorous demonstration of their technologies under actual field conditions. By completing the demonstration and distributing the results, the agency establishes a baseline for acceptance and use of these technologies. The Monitoring and Measurement Technology Program is managed by the Office of Research and Development's Environmental Sciences Division in Las Vegas, Nevada.

Gary Foley, Ph.D.
Director
National Exposure Research Laboratory
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Abstract

The demonstration of innovative field measurement devices for total petroleum hydrocarbons (TPH) in soil is being conducted under the U.S. Environmental Protection Agency (EPA) Superfund Innovative Technology Evaluation Program in June 2000 at the Navy Base Ventura County site in Port Hueneme, California. The primary purpose of the demonstration is to evaluate innovative field measurement devices for TPH in soil based on their performance and cost as compared to a conventional, off-site laboratory analytical method. The seven field measurement devices listed below will be demonstrated.

- CHEMetrics, Inc.'s, RemediAid™ Total Petroleum Hydrocarbon Starter Kit
- Wilks Enterprise, Inc.'s, Infracal® TOG/TPH Analyzer, Models CVH and HATR-T
- Horiba Instruments, Incorporated's, OCMA-350 Oil Content Analyzer
- Dexsil® Corporation's PetroFLAG™ Hydrocarbon Test Kit for Soil
- Environmental Systems Corporation's Synchronous Scanning Luminoscope
- siteLAB® Corporation's Analytical Test Kit UVF-3100A
- Strategic Diagnostics, Inc.'s, EnSys Petro Test System

This demonstration plan describes the procedures that will be used to verify the performance and cost of each field measurement device. The plan incorporates the quality assurance and quality control elements needed to generate data of sufficient quality to document each device's performance and cost. A separate innovative technology verification report (ITVR) will be prepared for each device. The ITVRs will present the demonstration findings associated with the demonstration objectives.

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Abbreviations, Acronyms, and Symbols

>	Greater than
$	Greater than or equal to
<	Less than
#	Less than or equal to
±	Plus or minus
8	Wavelength
µg	Microgram
µL	Microliter
µm	Micrometer
%C	Percent completeness
%R	Percent recovery
AC	Alternating current
ACGIH	American Conference of Governmental Industrial Hygienists
AEHS	Association for Environmental Health and Sciences
AFB	Air Force Base
AIHA	American Industrial Hygiene Association
$AlCl_3$	Aluminum chloride
API	American Petroleum Institute
ASTM	American Society for Testing and Materials
AZUR	AZUR Environmental Ltd
bgs	Below ground surface
BTEX	Benzene, toluene, ethylbenzene, and xylene
BVC	Base Ventura County
C	Carbon
C_6H_6	Benzene
Catalyst	Catalyst Information Resources, L.L.C.
CCV	Continuing calibration verification
CFC	Chlorofluorocarbon
CFR	*Code of Federal Regulations*
CH_2Cl_2	Dichloromethane
CHEMetrics	CHEMetrics, Inc.
CLP	Contract Laboratory Program
cm	Centimeter
dBA	Decibel as measured on the A-weighted scale
DC	Direct current
DER	Data evaluation report
Dexsil®	Dexsil® Corporation
DOT	U.S. Department of Transportation

DRO	Diesel range organics
EDD	Electronic data deliverable
EDRO	Extended diesel range organics
ELISA	Enzyme-linked immunosorbent assay
EPA	U.S. Environmental Protection Agency
EPH	Extractable petroleum hydrocarbon
ERA	Environmental Resource Associates
ESC	Environmental Systems Corporation
ESLI	End-of-service-life indicator
FFA	Fuel Farm Area
FID	Flame ionization detector
GC	Gas chromatograph
GRO	Gasoline range organics
HEPA	High-efficiency particulate
Horiba	Horiba Instruments, Incorporated
HSR	Health and safety representative
IATA	International Air Transport Association
ICV	Initial calibration verification
ID	Inside diameter
IDLH	Immediately dangerous to life or health
IDW	Investigation-derived waste
Ig	Immunoglobulin
ITVR	Innovative technology verification report
kg	Kilogram
L	Liter
LCS	Laboratory control sample
LCSD	Laboratory control sample duplicate
LEL	Lower explosive limit
Luminoscope	Synchronous Scanning Luminoscope
m	Meter
MCAWW	“Methods for Chemical Analysis of Water and Wastes”
MDL	Method detection limit
mg	Milligram
min	Minute
mL	Milliliter
mm	Millimeter
MMT	Monitoring and Measurement Technology
mRem/hr	Millirem per hour
MS	Matrix spike
MSD	Matrix spike duplicate
MSDS	Material safety data sheet
MTBE	Methyl-tert-butyl ether
NA	Not applicable
NDIR	Nondispersive infrared
NERL	National Exposure Research Laboratory
NEX	Naval Exchange
ng	Nanogram
NIOSH	National Institute for Occupational Safety and Health
NIST	National Institute for Standards and Technology
nm	Nanometer

NRMRL	National Risk Management Research Laboratory
OCMA-350	OCMA-350 Oil Content Analyzer
ORD	Office of Research and Development
OSHA	Occupational Safety and Health Administration
OSWER	Office of Solid Waste and Emergency Response
PAH	Polynuclear aromatic hydrocarbon
PbSe	Lead selenium
PC	Petroleum company
PCB	Polychlorinated biphenyl
PE	Performance evaluation
PEG	Polyethylene glycol
PEL	Permissible exposure limit
PetroFLAG™ test kit	PetroFLAG™ Hydrocarbon Test Kit for Soil
PHC	Petroleum hydrocarbon
PID	Photoionization detector
ppb	Part per billion
PPE	Personal protective equipment
ppm	Part per million
PRA	Phytoremediation Area
PRO	Petroleum range organics
QA	Quality assurance
QC	Quality control
R^2	Square of the correlation coefficient
RemediAid™ starter kit	RemediAid™ Total Petroleum Hydrocarbon Starter Kit
RPD	Relative percent difference
RSD	Relative standard deviation
SCBA	Self-contained breathing apparatus
SDI	Strategic Diagnostics, Inc.
SFT	Slop Fill Tank
SITE	Superfund Innovative Technology Evaluation
siteLAB®	siteLAB® Corporation
SSC	Site safety coordinator
STL Tampa East	Severn Trent Laboratories in Tampa, Florida
SW-846	“Test Methods for Evaluating Solid Waste”
SWP	Safe work practice
Tetra Tech	Tetra Tech EM Inc.
TLD	Thermoluminescence detector
TLV	Threshold limit value
TMB	Tetramethylbenzidine
TPH	Total petroleum hydrocarbons
TSA	Technical system audit
USCS	Unified Soil Classification System
UST	Underground storage tank
UVF-3100A	siteLAB® Analytical Test Kit UVF-3100A
V	Volume
v/v	Volume per volume
VOA	Volatile organic analysis
VOC	Volatile organic compound
VPH	Volatile petroleum hydrocarbon
Wilks	Wilks Enterprise, Inc.

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Tetra Tech EM Inc. acknowledges the support of the following individuals in preparing this document: Dr. Stephen Billets and Mr. George Brilis of the U.S. Environmental Protection Agency's (EPA) National Exposure Research Laboratory (NERL); Mr. Ernest Lory of Navy Base Ventura County; Ms. Amy Whitley of Kelly Air Force Base; Mr. Jay Simonds of Handex of Indiana; Mr. Henry Castaneda of CHEMetrics, Inc.; Ms. Sandy Rintoul of Wilks Enterprise, Inc.; Mr. Jim Vance of Horiba Instruments, Incorporated; Dr. Ted B. Lynn of Dexsil® Corporation; Dr. George Hyfantis of Environmental Systems Corporation; Mr. Stephen Greason of siteLAB® Corporation; and Mr. Joseph Dautlick of Strategic Diagnostics, Inc.

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Executive Summary

Performance verification of innovative environmental technologies is an integral part of the regulatory and research mission of the U.S. Environmental Protection Agency (EPA). The Superfund Innovative Technology Evaluation (SITE) Program was established by the EPA Office of Solid Waste and Emergency Response and Office of Research and Development under the Superfund Amendments and Reauthorization Act of 1986. The program is designed to meet three primary objectives: (1) identify and remove obstacles to the development and commercial use of innovative technologies, (2) demonstrate promising innovative technologies and gather reliable performance and cost information to support site characterization and cleanup activities, and (3) develop procedures and policies that encourage use of innovative technologies at Superfund sites as well as other waste sites or commercial facilities. The intent of a SITE demonstration is to obtain representative, high-quality performance and cost data on innovative technologies so that potential users can assess a given technology's suitability for a specific application.

The demonstration of innovative field measurement devices for total petroleum hydrocarbons (TPH) in soil is to be conducted under the SITE Program in June 2000 at the Navy Base Ventura County site in Port Hueneme, California. The demonstration is being conducted under the Monitoring and Measurement Technology Program, which is administered by the Environmental Sciences Division of the EPA National Exposure Research Laboratory in Las Vegas, Nevada. The primary purpose of the demonstration is to evaluate innovative field measurement devices for TPH in soil based on comparison of their performance and cost to those of a conventional, off-site laboratory analytical method.

The following seven field measurement devices will be demonstrated:

- CHEMetrics, Inc.'s, RemediAid™ Total Petroleum Hydrocarbon Starter Kit
- Wilks Enterprise, Inc.'s, Infracal® TOG/TPH Analyzer, Models CVH and HATR-T
- Horiba Instruments, Incorporated's, OCMA-350 Oil Content Analyzer
- Dexsil® Corporation's PetroFLAG™ Hydrocarbon Test Kit for Soil
- Environmental Systems Corporation's Synchronous Scanning Luminoscope
- siteLAB® Corporation's Analytical Test Kit UVF-3100A
- Strategic Diagnostics, Inc.'s, EnSys Petro Test System

The performance and cost of each device will be compared to those of a conventional, off-site laboratory analytical method—that is, a reference method. The performance and cost of one device will not be compared to those of another device. The reference method that will be used for the demonstration is "Test Methods for Evaluating Solid Waste" (SW-846) Method 8015B (modified). A separate innovative technology verification report (ITVR) will be prepared for each device.

The demonstration has both primary and secondary objectives. The primary objectives are critical to the technology evaluations and require use of quantitative results to draw conclusions regarding technology performance. The secondary objectives pertain to information that is useful but do not necessarily require use of quantitative results to draw conclusions regarding technology performance. The primary objectives for the demonstration of the individual field measurement devices are as follows:

P1. Determine the method detection limit

P2. Evaluate the accuracy and precision of TPH measurement for a variety of contaminated soil samples

P3. Evaluate the effect of interferents on TPH measurement

P4. Evaluate the effect of soil moisture content on TPH measurement

P5. Measure the time required for TPH measurement

P6. Estimate costs associated with TPH measurement

The secondary objectives for the demonstration of the individual field measurement devices are as follows:

S1. Document the skills and training required to properly operate the device

S2. Document health and safety concerns associated with operating the device

S3. Document the portability of the device

S4. Evaluate the device's durability based on its materials of construction and engineering design

S5. Document the availability of the device and associated spare parts

To address the demonstration objectives, both environmental and performance evaluation (PE) samples will be analyzed during the demonstration. The environmental samples will be collected from five areas contaminated with gasoline, diesel, lubricating oil, or other petroleum products, and the PE samples will be obtained from a commercial provider. Collectively, the environmental and PE samples will have the range of physical (sand, silt, and clay) and chemical (petroleum hydrocarbon type and concentration) characteristics necessary to properly evaluate the field measurement devices.

Upon completion of the demonstration, field measurement device and reference method results will be compared to evaluate the performance and associated cost of each device. The ITVRs for the seven devices are scheduled for completion in October 2001.

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Chapter 1
Introduction

The U.S. Environmental Protection Agency (EPA) Office of Research and Development (ORD) National Exposure Research Laboratory (NERL) has contracted with Tetra Tech EM Inc. (Tetra Tech) to conduct a demonstration of innovative field measurement devices for total petroleum hydrocarbons (TPH) in soil. The demonstration is being conducted as part of the EPA Superfund Innovative Technology Evaluation (SITE) Monitoring and Measurement Technology (MMT) Program in June 2000 at Port Hueneme in California. The purpose of this demonstration is to obtain reliable performance and cost data on the devices in order to provide (1) potential users with a better understanding of the devices' performance and operating costs under well-defined field conditions and (2) the device developers with documented results that will help them promote acceptance and use of their devices.

This demonstration plan describes the procedures that will be used to verify the performance and associated cost of each field measurement device. The plan also incorporates the quality assurance and quality control (QA/QC) elements needed to generate data of sufficient quality to document each device's performance and cost. This plan has been prepared using the NERL's "A Guidance Manual for the Preparation of Site Characterization and Monitoring Technology Demonstration Plans" (EPA 1996a) and in accordance with the EPA National Risk Management Research Laboratory's (NRMRL) "Quality Assurance Project Plan Requirements for Applied Research Projects" (EPA 1998).

This demonstration plan describes the SITE Program, the scope of the demonstration, and the components and definition of TPH (Chapter 1); the seven innovative TPH field measurement devices that will be demonstrated and the technologies that they are based on (Chapter 2); the three demonstration sites (Chapter 3); the demonstration approach (Chapter 4); the reference method and laboratory that will be used during the demonstration (Chapter 5); the demonstration organization and responsibilities (Chapter 6); the field sampling procedures (Chapter 7); the calibration requirements and sample management procedures for the devices (Chapter 8); the laboratory sample preparation and analytical methods, calibration requirements, and sample management procedures (Chapter 9); the QA/QC procedures (Chapter 10); the audits and associated corrective actions (Chapter 11); the data management procedures (Chapter 12); the health and safety procedures (Chapter 13); and the references used to prepare this demonstration plan (Chapter 14).

1.1 Description of SITE Program

Performance verification of innovative environmental technologies is an integral part of the regulatory and research mission of the EPA. The SITE Program was established by the EPA Office of Solid Waste and Emergency Response (OSWER) and ORD under the Superfund Amendments and Reauthorization Act of 1986. The overall goal of the SITE Program is to conduct performance verification studies and to promote the acceptance of innovative technologies that may be used to achieve long-term protection of human health and the environment. The program is designed to meet three primary objectives: (1) identify and remove obstacles to the development and commercial use of innovative technologies, (2) demonstrate promising innovative technologies and gather reliable performance and cost information to support site characterization and cleanup activities, and (3) develop procedures and policies that encourage use of innovative technologies at Superfund sites as well as at other waste sites or commercial facilities.

The intent of a SITE demonstration is to obtain representative, high-quality performance and cost data on one or more innovative technologies so that potential users can assess a given technology's suitability for a specific application. The SITE Program includes the following elements:

- **MMT Program**—Evaluates technologies that sample, detect, monitor, or measure hazardous and toxic substances. These technologies are expected to provide better, faster, or more cost-effective methods for producing real-time data during site characterization and remediation studies than do conventional technologies.

- **Remediation Technology Program**—Conducts demonstrations of innovative treatment technologies to provide reliable performance, cost, and applicability data for site cleanups.

- **Technology Transfer Program**—Provides and disseminates technical information in the form of updates, brochures, and other publications that promote the SITE Program and participating technologies. The Technology Transfer Program also offers technical assistance, training, and workshops to support the technologies. A significant number of these activities are performed by EPA's Technology Innovation Office.

The TPH field measurement device demonstration is being conducted as part of the MMT Program, which provides developers of innovative hazardous waste sampling, monitoring, and measurement technologies with an opportunity to demonstrate their devices' performance under actual field conditions. These devices may be used to sample, detect, monitor, or measure hazardous and toxic substances in water, soil, soil gas, and sediment. The technologies include chemical sensors for in situ (in place) measurements, groundwater samplers, soil and sediment samplers, soil gas samplers, field-portable analytical equipment, and other systems that support field sampling or data acquisition and analysis.

The MMT Program promotes acceptance of technologies that can be used to (1) accurately assess the degree of contamination at a site, (2) provide data to evaluate potential effects on human health and the environment, (3) apply data to assist in selecting the most appropriate cleanup action, and (4) monitor the effectiveness of a remediation process. The program places a high priority on innovative technologies that provide more cost-effective, faster, or safer methods for producing real-time or near-real-time data than do conventional, laboratory-based technologies. These innovative technologies are demonstrated under field conditions, and the results are compiled, evaluated, published, and disseminated by the ORD. The primary objectives of the MMT Program are as follows:

- Test and verify the performance of field sampling and analytical technologies that enhance sampling, monitoring, and site characterization capabilities
- Identify performance attributes of innovative technologies to address field sampling, monitoring, and characterization problems in a more cost-effective and efficient manner
- Prepare protocols, guidelines, methods, and other technical publications that enhance acceptance of these technologies for routine use

The MMT Program is administered by the Environmental Sciences Division of the NERL in Las Vegas, Nevada. The NERL is the EPA's center for investigation of technical and management approaches for identifying and quantifying risks to human health and the environment. The NERL's mission components include (1) developing and evaluating methods and technologies for sampling, monitoring, and characterizing water, air, soil, and sediment; (2) supporting regulatory and policy decisions; and (3) providing the technical support needed to ensure effective implementation of environmental regulations and strategies. By demonstrating selected innovative field measurement devices for TPH in soil, the MMT Program is supporting the development and evaluation of methods and technologies for field measurement of TPH concentrations in a variety of soil types.

The MMT Program's technology verification process is designed to conduct demonstrations that will generate high-quality data so that potential users have reliable information regarding the device performance and cost. Four steps are inherent in the process: (1) needs identification and technology selection, (2) demonstration planning and implementation, (3) report preparation, and (4) information distribution.

The first step of the technology verification process begins with identifying technology needs of the EPA and regulated community. The EPA regional offices, the U.S. Department of Energy, the U.S. Department of Defense, industry, and state environmental regulatory agencies are asked to identify technology needs for sampling, measurement, and monitoring of environmental media. Once a need is identified, a search is conducted to identify suitable technologies that will address the need. The technology search and identification process consists of examining industry and trade publications, attending related conferences, exploring leads from technology developers and industry experts, and reviewing responses to *Commerce Business Daily* announcements. Selection of technologies for field testing includes evaluation of the candidate technologies based on several criteria. A suitable technology for field testing

- Is designed for use in the field
- Is applicable to a variety of environmentally contaminated sites
- Has potential for solving problems that current methods cannot satisfactorily address
- Has estimated costs that are lower than those of conventional methods
- Is likely to achieve better results than current methods in areas such as data quality and turnaround time
- Uses techniques that are easier or safer than current methods
- Is commercially available

Once candidate technologies are identified, their developers are asked to participate in a developer conference. This conference gives the developers an opportunity to describe their technologies' performance and to learn about the MMT Program.

The second step of the technology verification process is to plan and implement a demonstration that will generate high-quality data to assist potential users in selecting a technology. Demonstration planning activities include a predemonstration sampling and analysis investigation that assesses existing conditions at the proposed demonstration site or sites. The objectives of the predemonstration investigation are to (1) confirm available information on applicable physical, chemical, and biological characteristics of contaminated media at the sites to justify selection of site areas for the demonstration; (2) provide the technology developers with an opportunity to evaluate the areas, analyze representative samples, and identify logistical requirements; (3) assess the overall logistical requirements for conducting the demonstration; and (4) provide the reference laboratory involved with an opportunity to identify any matrix-specific analytical problems associated with the contaminated media and to propose appropriate solutions. Information generated through the predemonstration investigation is used to develop the final demonstration design and sampling and analysis procedures.

Demonstration planning activities also include preparation of a demonstration plan that describes the procedures to be used to verify the performance and cost of each innovative technology. The demonstration plan incorporates information generated during the predemonstration investigation as well as input from technology developers, demonstration site representatives, and technical peer reviewers. The demonstration plan also incorporates the QA/QC elements needed to produce data of sufficient quality to document the performance and cost of each technology.

During the demonstration, each innovative technology is evaluated independently and, when possible and appropriate, is compared to a reference technology. The performance and cost of one innovative technology are not compared to those of another technology evaluated in the demonstration. Rather, demonstration data are used to evaluate the performance, cost, advantages, limitations, and field applicability of each technology.

As part of the third step of the technology verification process, the EPA publishes a verification statement and a detailed evaluation of each technology in an innovative technology verification report (ITVR). To ensure its quality, the ITVR is published only after comments from the technology developer and external peer reviewers are satisfactorily addressed. All demonstration data used to evaluate each innovative technology are summarized in a data evaluation report (DER) that constitutes a complete record of the demonstration. The DER is not published as an EPA document, but an unpublished copy may be obtained from the EPA project manager.

The fourth step of the verification process is to distribute demonstration information. To benefit technology developers and potential technology users, the EPA distributes demonstration bulletins and ITVRs through direct mailings, at conferences, and on the Internet. ITVRs and additional information on the SITE Program are available on the EPA ORD web site (http://www.epa.gov/ORD/SITE).

1.2 Scope of Demonstration

The purpose of the demonstration is to evaluate innovative field measurement devices for TPH in soil in order to provide (1) potential users with a better understanding of each device's performance and cost under well-defined field conditions and (2) developers with documented results that will assist them in promoting acceptance and use of their devices.

Chapter 2 describes the field measurement devices that will be evaluated during the demonstration. Because TPH is a "method-defined parameter," the performance results for each device will be compared to the results obtained using an off-site laboratory measurement method—that is, an approved reference method. Details on the selection of the reference method and laboratory are provided in Chapter 5.

The demonstration has both primary and secondary objectives. Primary objectives are critical to the technology verification and required use of quantitative results. Secondary objectives pertain to information that is useful but will not necessarily require the use of quantitative results. Both the primary and secondary objectives are presented in Chapter 4.

To meet the demonstration objectives, samples will be collected from five individual areas at three sites. The first site is referred to as the Navy Base Ventura County (BVC) site; is located in Port Hueneme, California; and contains three sampling areas. The Navy BVC site lies in EPA Region 9. The second site is referred to as the Kelly Air Force Base (AFB) site; is located in San Antonio, Texas; and contains one sampling area. The Kelly AFB site lies in EPA Region 6. The third site is referred to as the petroleum company (PC) site, is located in north-central Indiana, and contains one sampling area. The PC site lies in EPA Region 5.

In preparation for the demonstration, a predemonstration sampling and analysis investigation was completed at the three sites in January 2000. The purpose of this investigation was to assess whether the sites and sampling areas were appropriate for evaluating the field measurement devices based on the demonstration objectives. Demonstration field activities are scheduled to occur between June 5 and 18, 2000. Draft ITVRs will be available for peer and developer review in March 2001, and final ITVRs will be submitted to the EPA in October 2001.

1.3 Components and Definition of TPH

To understand the term "TPH," it is necessary to understand the composition of petroleum and its products. This section briefly describes the composition of petroleum and its products and defines TPH from a measurement standpoint. The organic compounds containing only hydrogen and carbon that are present in petroleum and its derivatives are collectively referred to as petroleum hydrocarbons (PHC). Therefore, in this demonstration plan, the term "PHC" is used to identify sample constituents, and the term "TPH" is used to identify analyses performed and the associated results (for example, TPH concentrations).

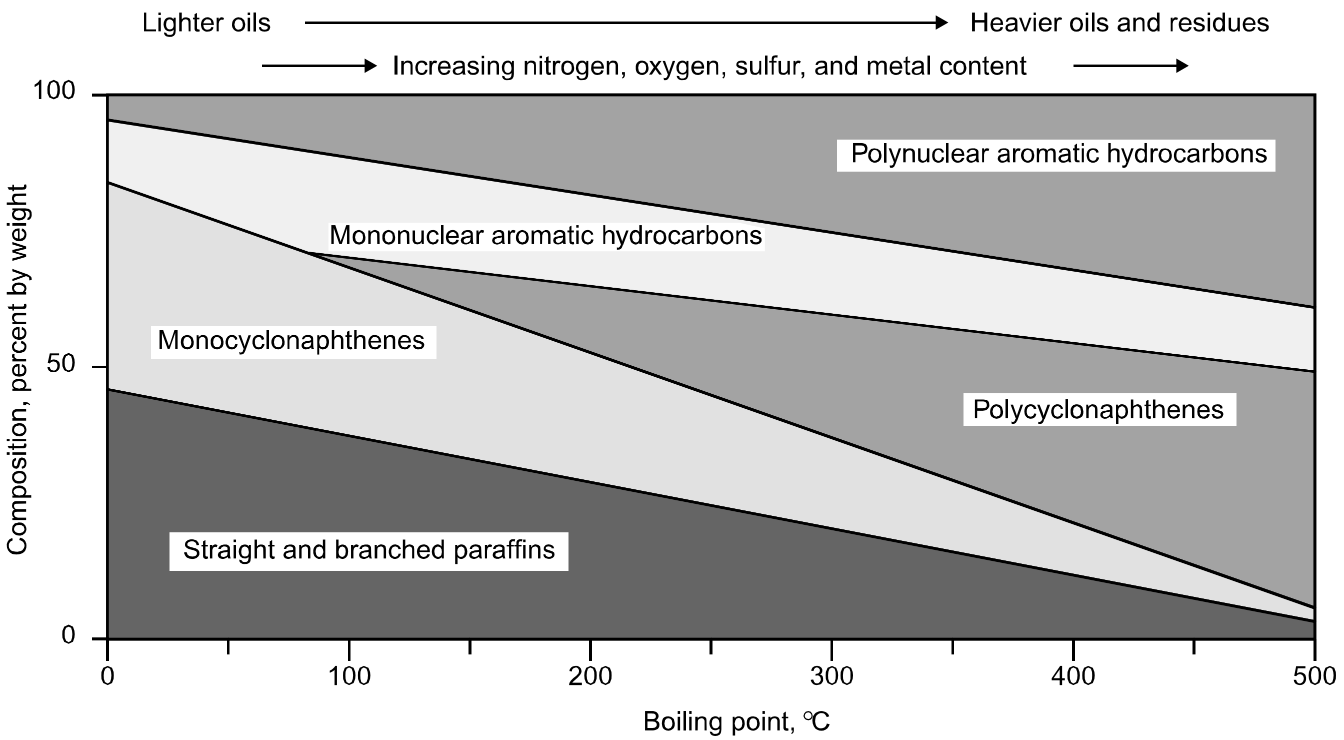
1.3.1 Composition of Petroleum and Its Products

Petroleum is essentially a mixture of gaseous, liquid, and solid hydrocarbons that occur in sedimentary rock deposits. On the molecular level, petroleum is a complex mixture of hydrocarbons; organic compounds of sulfur, nitrogen, and oxygen; and compounds containing metallic constituents, particularly vanadium, nickel, iron, and copper. Based on the limited data available, the elemental composition of petroleum appears to vary over a relatively narrow range: 83 to 87 percent carbon, 10 to 14 percent hydrogen, 0.05 to 6 percent sulfur, 0.1 to 2 percent nitrogen, and 0.05 to 1.5 percent oxygen. Metals are present in petroleum at concentrations of up to 0.1 percent (Speight 1991).

Petroleum in the crude state (crude oil) is a mineral resource, but when refined, it provides liquid fuels, solvents, lubricants, and many other marketable products. The hydrocarbon components of crude oil include paraffinic, naphthenic, and aromatic groups. Paraffins (alkanes) are saturated, aliphatic hydrocarbons with straight or branched chains but without any ring structure. Naphthenes are saturated, aliphatic hydrocarbons containing one or more rings, each of which may have one or more paraffinic side chains (alicyclic hydrocarbons). Aromatic hydrocarbons contain one or more aromatic nuclei, such as benzene, naphthalene, and phenanthrene ring systems, that may be linked with (substituted) naphthenic rings or paraffinic side chains. In crude oil, the relationship among the three primary groups of hydrocarbon components is a result of hydrogen gain or loss between any two groups. Another class of compounds that is present in petroleum products such as automobile gasoline but rarely in crude oil is known as olefins. Olefins (alkenes) are unsaturated, aliphatic hydrocarbons with straight or branched chains but without any ring structure.

The distribution of paraffins, naphthenes, and aromatic hydrocarbons depends on the source of crude oil. For example, Pennsylvania crude oil contains high levels of paraffins (about 50 percent), whereas Borneo crude oil contains less than 1 percent paraffins. As shown in Figure 1-1, the proportion of straight or branched paraffins decreases with increasing molecular weight or boiling point fraction for a given crude oil; however, this is not true for naphthenes or aromatic hydrocarbons. The proportion of monocyclonaphthenes decreases with increasing molecular weight or boiling point fraction, whereas the opposite is true for polycyclonaphthenes and polynuclear aromatic hydrocarbons (PAH); the proportion of mononuclear aromatic hydrocarbons appears to be independent of molecular weight or boiling point fraction.

Various petroleum products consisting of carbon and hydrogen are formed when crude oil is subjected to distillation and other processes in a refinery. Processing of crude oil results in petroleum products with trace quantities of metals and organic compounds that contain nitrogen, sulfur, and oxygen. These products include liquefied petroleum gas, gasoline, naphthas, kerosene, fuel oils, lubricating oils, coke, waxes, and asphalt. Of these products, gasoline,



Source: Speight 1991

Figure 1-1. Distribution of various petroleum hydrocarbon types throughout boiling point range of crude oil.

naphthas, kerosene, fuel oils, and lubricating oils are liquids and may be present at petroleum-contaminated sites. Except for gasoline and some naphthas, these products are made primarily by collecting particular boiling point fractions of crude oil from a distillation column. Because this classification of petroleum products is based on boiling point and not on chemical composition, the composition of these products, including the ratio of aliphatic to aromatic hydrocarbons, varies depending on the source of crude oil. In addition, specific information (such as boiling points and carbon ranges) for different petroleum products, varies slightly depending on the source of the information. Commonly encountered forms and blends of petroleum products are briefly described below. The descriptions are primarily based on information in books written by Speight (1991) and Gary and Handwerk (1993). Additional information is provided by Dryoff (1993).

1.3.1.1 Gasoline

Gasoline is a major exception to the boiling point classification described above because “straight-run gasoline” (gasoline directly recovered from a distillation column) is only a small fraction of the blended gasoline that is commercially available as fuel. Commercially available gasolines are complex mixtures of hydrocarbons that boil below 180 °C or at most 200 °C and that contain hydrocarbons with 4 to 12 carbon atoms per molecule. Of the commercially available gasolines, aviation gasoline has a narrower boiling range (38 to 170 °C) than automobile gasoline (-1 to 200 °C). In addition, aviation gasoline may contain high levels of paraffins (50 to 60 percent), moderate levels of naphthenes (20 to 30 percent), a low level of aromatic hydrocarbons (10 percent), and no olefins, whereas automobile gasoline may contain up to 30 percent olefins and up to 40 percent aromatic hydrocarbons.

Gasoline composition can vary widely depending on the source of crude oil. In addition, gasoline composition varies from region to region because of consumer needs for gasoline with a high octane rating to prevent engine "knocking." Moreover, EPA regulations regarding the vapor pressure of gasoline, the chemicals used to produce a high octane rating, and cleaner-burning fuels have affected gasoline composition. For example, when use of tetraethyl lead to produce gasoline with a high octane rating was banned by the EPA, oxygenated fuels came into existence. Production of these fuels included addition of methyl-tert-butyl ether (MTBE), ethanol, and other oxygenates. Use of oxygenated fuels also results in reduction of air pollutant emissions (for example, carbon monoxide and nitrogen oxides).

1.3.1.2 Naphthas

"Naphtha" is a generic term applied to petroleum solvents. Under standardized distillation conditions, at least 10 percent of naphthas should distill below 175 °C, and at least 95 percent of naphthas should distill below 240 °C. Naphthas can be both aliphatic and aromatic and contain hydrocarbons with 6 to 12 carbon atoms per molecule. Depending on the intended use of a naphtha, it may be free of aromatic hydrocarbons (to make it odor-free) and sulfur (to make it less toxic). Many forms of naphthas are commercially available, including Varnish Makers' and Painters' naphthas (Types I and II), mineral spirits (Types I through IV), and aromatic naphthas (Types I and II). Stoddard solvent, a commonly used dry cleaning solvent, is an example of an aliphatic naphtha.

1.3.1.3 Kerosene

Kerosene is a straight-run petroleum fraction that has a boiling point range of 205 to 260 °C. Kerosene typically contains hydrocarbons with 12 or more carbon atoms per molecule. Because of its use as an indoor fuel, kerosene must be free of aromatic and unsaturated hydrocarbons as well as sulfur compounds.

1.3.1.4 Jet Fuels

Jet fuels, which are also known as aircraft turbine fuels, are manufactured by blending gasoline, naphtha, and kerosene in varying proportions. Therefore, jet fuels may contain a carbon range that covers gasoline through kerosene. Jet fuels are used in both military and commercial aircraft. Some examples of jet fuels include Type A, Type A-1, Type B, JP-4, JP-5, and JP-8. The aromatic hydrocarbon content of these fuels ranges from 20 to 25 percent. The military jet fuel JP-4 has a wide boiling point range (65 to 290 °C), whereas commercial jet fuels, including JP-5 and Types A and A-1, have a narrower boiling point range (175 to 290 °C) because of safety considerations. Increasing concerns over combat hazards associated with JP-4 jet fuel led to development of JP-8 jet fuel, which has a flash point of 38 °C and a boiling point range of 165 to 275 °C. JP-8 jet fuel contains hydrocarbons with 9 to 15 carbon atoms per molecule. Type B jet fuel has a boiling point range of 55 to 230 °C and a carbon range of 5 to 13 atoms per molecule. A new specification is currently being developed by the American Society for Testing and Materials (ASTM) for Type B jet fuel.

1.3.1.5 Fuel Oils

Fuel oils are divided into two classes: distillates and residuals. No. 1 and 2 fuel oils are distillates and include kerosene, diesel, and home heating oil. No. 4, 5, and 6 fuel oils are residuals or black oils, and they all contain crude distillation tower bottoms (tar) to which cutter stocks (semirefined or refined distillates) have been added. No. 4 fuel oil contains the most cutter stock, and No. 6 fuel oil contains the least.

Commonly available fuel oils include No. 1, 2, 4, 5, and 6. The boiling points, viscosities, and densities of these fuel oils increase with increasing number designation. The boiling point ranges for No. 1, 2, and 4 fuel oils are about 180 to 320, 175 to 340, and 150 to 480 °C, respectively. No. 1 and 2 fuel oils contain hydrocarbons with 10 to 22 carbon atoms per molecule; the carbon range for No. 4 fuel oil is 22 to 40 atoms per molecule. No. 5 and 6 fuel oils have a boiling point range of 150 to 540 °C but differ in the amounts of residue they contain: No. 5 fuel oil contains a small amount of residue, whereas No. 6 fuel oil contains a large amount. No. 5 and 6 fuel oils contain hydrocarbons with 28 to 90 carbon atoms per molecule. Fuel oils typically contain about 60 percent aliphatic hydrocarbons and 40 percent aromatic hydrocarbons.

1.3.1.6 Diesel

Diesel is primarily used to operate motor vehicle and railroad diesel engines. Automobile diesel is available in two grades: No. 1 and 2. No. 1 diesel has a boiling point range of 180 to 320 °C and a cetane number above 50. The cetane number is similar to the octane number of gasoline; a higher number corresponds to less knocking. No. 2 diesel is very similar to No. 2 fuel oil. No. 2 diesel has a boiling point range of 175 to 340 °C and a minimum cetane number of 52. No. 1 diesel is used in high-speed engines such as truck and bus engines, whereas No. 2 diesel is used in other diesel engines. Railroad diesel is similar to No. 2 diesel but has a higher boiling point (up to 370 °C) and lower cetane number (40 to 45). The ratio of aliphatic to aromatic hydrocarbons in diesel is about 5. The carbon range for hydrocarbons present in diesel is 10 to 28 atoms per molecule.

1.3.1.7 Lubricating Oils

Lubricating oils can be distinguished from other crude oil fractions by their high boiling points (greater than 400 °C) and viscosities. Materials suitable for production of lubricating oils are composed principally of hydrocarbons containing 25 to 35 or even 40 carbon atoms per molecule, whereas residual stocks may contain hydrocarbons with 50 to 60 or more (up to 80 or so) carbon atoms per molecule. Because it is difficult to isolate hydrocarbons from the lubricant fraction of petroleum, aliphatic to aromatic hydrocarbon ratios are not well documented for lubricating oils. However, these ratios are expected to be comparable to those of the source crude oil.

1.3.2 Measurement of TPH

As described in Section 1.3.1, the composition of petroleum and its products is complex and variable, which complicates TPH measurement. The measurement of TPH in soil is further complicated by weathering effects. When a petroleum product is released to soil, the product's composition immediately begins to change. The components with lower boiling points are volatilized, the more water-soluble components migrate to groundwater, and biodegradation can affect many other components. Within a short period, the contamination remaining in soil may have only some characteristics in common with the parent product.

This section provides a historical perspective on TPH measurement, reviews current options for TPH measurement in soil, and discusses the definition of TPH that was used for the demonstration.

1.3.2.1 Historical Perspective

Most environmental measurements are focused on identifying and quantifying a particular trace element (such as lead) or organic compound (such as benzene). However, for some "method-defined" parameters, the particular substance being measured may yield different results depending on the measurement method used. Examples of such parameters include oil and grease and surfactants. Perhaps the most problematic of the method-defined parameters is TPH. TPH arose as a parameter for wastewater analyses in the 1960s because of petroleum industry concerns that the original "oil and grease" analytical method, which is gravimetric in nature, might inaccurately characterize petroleum industry wastewaters that contained naturally occurring vegetable oils and greases along with PHCs.

These naturally occurring materials are typically long-chain fatty acids (for example, oleic acid, the major component of olive oil).

Originally, TPH was defined as any material extracted with a particular solvent that is not adsorbed by the silica gel used to remove fatty acids and that is not lost when the solvent is evaporated. Although this definition covers most of the components of petroleum products, it includes many other organic compounds as well, including chlorinated solvents, pesticides, and other synthetic organic chemicals. Furthermore, because of the evaporation step in the gravimetric analytical method, the definition excludes most of the petroleum-derived compounds in gasoline that are volatile in nature. For these reasons, an infrared analytical method was developed to measure TPH. In this method, a calibration standard consisting of three components is analyzed at a wavelength of 3.41 micrometers (µm), which corresponds to an aliphatic CH_2 hydrocarbon stretch. As shown in Table 1-1, the calibration standard is designed to mimic a petroleum product having a relative distribution of aliphatic and aromatic compounds as well as a certain percentage of aliphatic CH_2 hydrocarbons. The infrared analytical method indicates that any compound that is extracted by the solvent, is not absorbed by silica gel, and contains a CH_2 bond is a PHC. Both the gravimetric and infrared analytical methods include a silica gel fractionation step to remove polar, biogenic compounds such as fatty acids, but this cleanup step can also remove some petroleum degradation products that are polar in nature.

Table 1-1. Summary of Calibration Information for Infrared Analytical Method

Standard Constituent	Constituent Type	Portion of Constituent in Standard (percent by volume)	Number of Carbon Atoms				Portion of Aliphatic CH_2 in Standard Constituent (percent by weight)
			Aliphatic			Aromatic	
			CH_3	CH_2	CH	CH	
Hexadecane	Straight-chain aliphatic	37.5	2	14	0	0	91
Isooctane	Branched-chain aliphatic	37.5	5	1	1	0	14
Chlorobenzene	Aromatic	25	0	0	0	5	0
Average							35

In the 1980s, because of the change in focus from wastewater analyses to characterization of hazardous waste sites that contained contaminated soil, many parties began to adapt the existing wastewater analytical methods for application to soil. Unfortunately, the term “TPH” was in common use, as many states had adopted this term (and the wastewater analytical methods) for cleanup activities at underground storage tank (UST) sites. Despite efforts by the American Petroleum Institute (API) and others to establish new analyte names (for example, gasoline range organics [GRO] and diesel range organics [DRO]), “TPH” is still present in many state regulations as a somewhat ill-defined term, and most state programs still have cleanup criteria for TPH.

1.3.2.2 Current Options for TPH Measurement in Soil

Three widely used technologies measure some form of TPH in soil to some degree. These technologies were used as starting points in deciding how to define TPH for the demonstration. The three technologies and the analytes measured are summarized in Table 1-2.

Table 1-2. Current Technologies for TPH Measurement

Technology	What Is Measured	What Is Not Measured
Gravimetry	All analytes removed from the sample by the extraction solvent that are not volatilized	Volatiles; very polar organics
Infrared	All analytes removed from the sample by the extraction solvent that contain an aliphatic CH_2 stretch	Benzene, naphthalene, and other aromatic hydrocarbons with no aliphatic group attached; very polar organics
Gas chromatograph/flame ionization detector	All analytes removed from the sample by the extraction solvent that can be chromatographed and that respond to the detector	Very polar organics; compounds with high molecular weights or high boiling points

Of the three technologies, gravimetry and infrared are discussed in Section 1.3.2.1. The third technology, the gas chromatograph/flame ionization detector (GC/FID), came into use because of the documented shortcomings of the other two technologies. The GC/FID had long been used in the petroleum refining industry as a product QC tool to determine the boiling point distribution of pure petroleum products. In the 1980s, environmental laboratories began to apply this technology along with sample preparation methods developed for soil samples to measure PHCs at environmental levels (Zilis, McDevitt, and Parr 1988). GC/FID methods measure all organic compounds that are extracted by the solvent and that can be chromatographed. However, because of method limitations, the very volatile portion of gasoline compounds containing four or five carbon atoms per molecule is not addressed by GC/FID methods; therefore, 100 percent recovery cannot be achieved for pure gasoline. This omission is not considered significant because these low-boiling-point aliphatic compounds (1) are not expected to be present in environmental samples (because of volatilization) and (2) pose less environmental risk than the aromatic hydrocarbons in gasoline.

The primary limitation of GC/FID methods relates to the extraction solvent used. The solvent should not interfere with the analysis, but to achieve environmental levels of detection (in the low milligram per kilogram [mg/kg] range) for soil, some concentration of the extract is needed because the sensitivity of the FID is in the nanogram (ng) range. This limitation has resulted in three basic approaches for GC/FID analyses for GRO, DRO, and PHCs.

For GRO analysis, a GC/FID method was developed as part of research sponsored by API and was the subject of an interlaboratory validation study (API 1994); the method was first published in 1990. In this method, GRO is defined as the sum of the organic compounds in the boiling point range of 60 to 170 °C, and the method uses a synthetic calibration standard as both a window-defining mix and a quantitation standard. The GRO method was specifically incorporated into the EPA's "Test Methods for Evaluating Solid Waste" (SW-846) Method 8015B in 1996 (EPA 1996). The GRO method uses the purge-and-trap technique for sample preparation, effectively limiting the TPH components to the volatile compounds only.

For DRO analysis, a GC/FID method was developed under the sponsorship of API as a companion to the GRO method and was interlaboratory-validated in 1994. In the DRO method, DRO is defined as the sum of the organic compounds in the boiling point range of 170 to 430 °C. As in the GRO method, a synthetic calibration standard is used for quantitation. The DRO method was also incorporated into SW-846 Method 8015B in 1996. The technology used in the DRO method can measure hydrocarbons with boiling points up to 540 °C. However, the hydrocarbons with boiling points in the range of 430 to 540 °C are specifically excluded from SW-846 Method 8015B so as not to include the higher-boiling-point petroleum products. The DRO method uses a solvent extraction and concentration step, effectively limiting the method to nonvolatile hydrocarbons.

For PHC analysis, a GC/FID method was developed by Shell Oil Company (now Equilon Enterprises). This method was interlaboratory-validated along with the GRO and DRO methods in an API study in 1994. The PHC method originally defined PHCs as the sum of the compounds in the boiling point range of about 70 to 400 °C, but it now defines PHCs as the sum of the compounds in the boiling point range of 70 to 490 °C. The method provides options for instrument calibration, including use of synthetic standards, but it recommends use of products similar to the contaminants present at the site of concern. The PHC method has not been specifically incorporated into SW-846; however, the method has been used as the basis for the TPH methods in several states, including Massachusetts, Washington, and Texas. The PHC method uses solvent microextraction and thus has a higher detection limit than the GRO and DRO methods. The PHC method also begins peak integration after elution of the solvent peak for n-pentane. Thus, this method probably cannot measure some volatile compounds (for example, 2-methyl pentane and MTBE) that are measured using the GRO method.

1.3.2.3 Definition of TPH

It is not possible to establish a definition of TPH that would include crude oil and its refined products and exclude other organic compounds. Rather, the TPH definition selected for the demonstration is intended to

- Include compounds that are PHCs, such as paraffins, naphthenes, and aromatic hydrocarbons
- Include, to the extent possible, the major petroleum products (gasoline, naphthas, kerosene, jet fuels, fuel oils, diesel, and lubricating oils)

- Have little inherent bias based on the composition of an individual manufacturer’s product
- Have little inherent bias based on the relative concentrations of aliphatic and aromatic hydrocarbons present
- Include much of the volatile portion of gasoline, including all weathered gasoline
- Include MTBE
- Exclude crude oil residuals beyond the extended diesel organic (EDRO) range
- Exclude nonpetroleum organic compounds (for example, chlorinated solvents, pesticides, polychlorinated biphenyls [PCB], and naturally occurring oils and greases)
- Allow TPH measurement using a widely accepted method
- Reflect accepted TPH measurement practice in many states

Several states, including Massachusetts, Alaska, Louisiana, and North Carolina, have implemented or are planning to implement a TPH contamination cleanup approach based on the aliphatic and aromatic hydrocarbon fractions of TPH. The action levels for the aromatic hydrocarbon fraction are more stringent than those for the aliphatic hydrocarbon fraction. The approach used in these states involves performing a sample fractionation procedure and two analyses to determine the aliphatic and aromatic hydrocarbon concentrations in a sample. However, in most applications of this approach, only a few samples are subjected to the determination of aliphatic and aromatic hydrocarbon concentrations because of the cost associated with performing sample cleanup and two analyses.

For the demonstration, TPH is not defined based on the aliphatic and aromatic hydrocarbon fractions because

- Such a definition is used in only a few states.
- Variations exist among the sample fractionation and analysis procedures used in different states.
- The repeatability and versatility of sample fractionation and analysis procedures are not well documented.
- In some states, TPH-based action levels are still used.
- The associated analytical costs are high.

As stated in Section 1.3.2.2, analytical methods currently available for measurement of TPH each exclude some portion of TPH and are unable to measure TPH alone while excluding all other organic compounds, thus making TPH a method-defined parameter. After consideration of all the information presented above, the GRO and DRO analytical methods were selected for TPH measurement for the demonstration. However, because of the general interest in higher-boiling-point petroleum products, the integration range of the DRO method was extended to include compounds with boiling points up to 540 °C. Thus, for the demonstration, the TPH concentration is the sum of all organic compounds that have boiling points between 60 and 540 °C and that can be chromatographed, or the sum of the results obtained using the GRO and DRO methods. This approach accounts for most gasoline, including MTBE, and virtually all other petroleum products and excludes a portion (25 to 50 percent) of the heavy lubricating oils. Thus, TPH measurement for the demonstration includes PHCs as well as some organic compounds that are not PHCs. More specifically, TPH measurement does not exclude nonpetroleum organic compounds such as chlorinated solvents, other synthetic organic chemicals such as pesticides and PCBs, and naturally occurring oils and greases. A silica gel fractionation step used to remove polar, biogenic compounds such as fatty acids in some GC/FID methods is not included in the sample preparation step because, according to the State of California, this step can also remove some petroleum degradation products that are also polar in nature (California Environmental Protection Agency 1999). The step-by-step approach used to select the reference method for the demonstration and the project-specific procedures implemented for soil sample preparation and analysis using the reference method are detailed in Chapter 5.

Chapter 2
Innovative Technology and Field Measurement Device Descriptions

This chapter describes the seven innovative TPH field measurement devices that will be demonstrated and the technologies upon which they are based. Table 2-1 identifies the technologies, devices, and device developers.

Table 2-1. Summary of Technologies, Measurement Devices, and Device Developers

Technology	Measurement Device	Measurement Device Developer
Friedel-Crafts alkylation reaction and colorimetry	RemediAid™ Total Petroleum Hydrocarbon Starter Kit	CHEMetrics, Inc., and AZUR Environmental Ltd
Infrared analysis	Infracal® TOG/TPH Analyzer, Models CVH and HATR-T	Wilks Enterprise, Inc.
	OCMA-350 Oil Content Analyzer	Horiba Instruments, Incorporated
Emulsion turbidimetry	PetroFLAG™ Hydrocarbon Test Kit for Soil	Dexsil® Corporation
Ultraviolet fluorescence spectroscopy	Synchronous Scanning Luminoscope	Environmental Systems Corporation
	siteLAB® Analytical Test Kit UVF-3100A	siteLAB® Corporation
Immunoassay and colorimetry	EnSys Petro Test System	Strategic Diagnostics, Inc.

The performance results generated during the demonstration of each device will be compared to the results obtained using a modified, off-site laboratory measurement method—that is, a reference method. For the demonstration, the reference method for measuring TPH is based on SW-846 Method 8015B (EPA 1996). First, soil samples will be extracted using (1) SW-846 Methods 5030B and 5035 for GRO and (2) SW-846 Method 3540C for extended diesel range organics (EDRO), as appropriate. The extracts will then be analyzed for GRO and EDRO using SW-846 Method 8015B (modified). The GRO and EDRO concentrations thus obtained will be summed to estimate the TPH concentration. Chapter 9 further discusses the SW-846 methods that will be used for the demonstration.

Section 2.1 describes the technologies upon which the devices are based, and Section 2.2 describes the devices that will be demonstrated. The technology and device descriptions presented in this chapter will be used to evaluate the developers' field activities during the demonstration.

2.1 Technology Descriptions

This section describes the technologies upon which the field measurement devices are based. In general, TPH measurement by these devices involves extraction of PHCs in soil using an appropriate solvent followed by measurement of the TPH concentration in the extract using an optical method. The extraction solvent is selected such that it will not interfere with the optical measurement of TPH in the extract. Some of the devices use light in the visible wavelength range, and others use light outside the visible wavelength range (for example, infrared). The wavelength range that each device uses is illustrated in Figure 2-1. Use of visible light to measure TPH concentrations requires development of a color whose intensity is a measure of TPH concentration. For measurements that do not us visible light, color development is not required.

The optical measurements made by the devices involve absorbance, reflectance, or fluorescence. In general, the optical measurement for a soil extract is compared to a calibration curve in order to determine the TPH concentration. Calibration curves are developed by (1) using a series of calibration standards selected based on the type of PHCs being measured at a site or (2) establishing a correlation between off-site laboratory measurements and field measurements for selected, site-specific soil samples.

The technology descriptions presented below are not intended to provide complete operating procedures for measuring TPH concentrations in soil using the devices. For example, soil sample extraction procedures are not discussed in this section because the soil extraction step is common to all the devices, although different solvents may be used for extraction. Detailed operating procedures for the devices, including soil extraction procedures, are presented in Section 2.2.

2.1.1 Friedel-Crafts Alkylation Reaction and Colorimetry

TPH measurement in soil using the RemediAid™ Total Petroleum Hydrocarbon Starter Kit (RemediAid™ starter kit) is based on a combination of the Friedel-Crafts alkylation reaction and colorimetry. Collectively, these two technologies are suitable for measuring aromatic hydrocarbons independent of their carbon range. These technologies are described below.

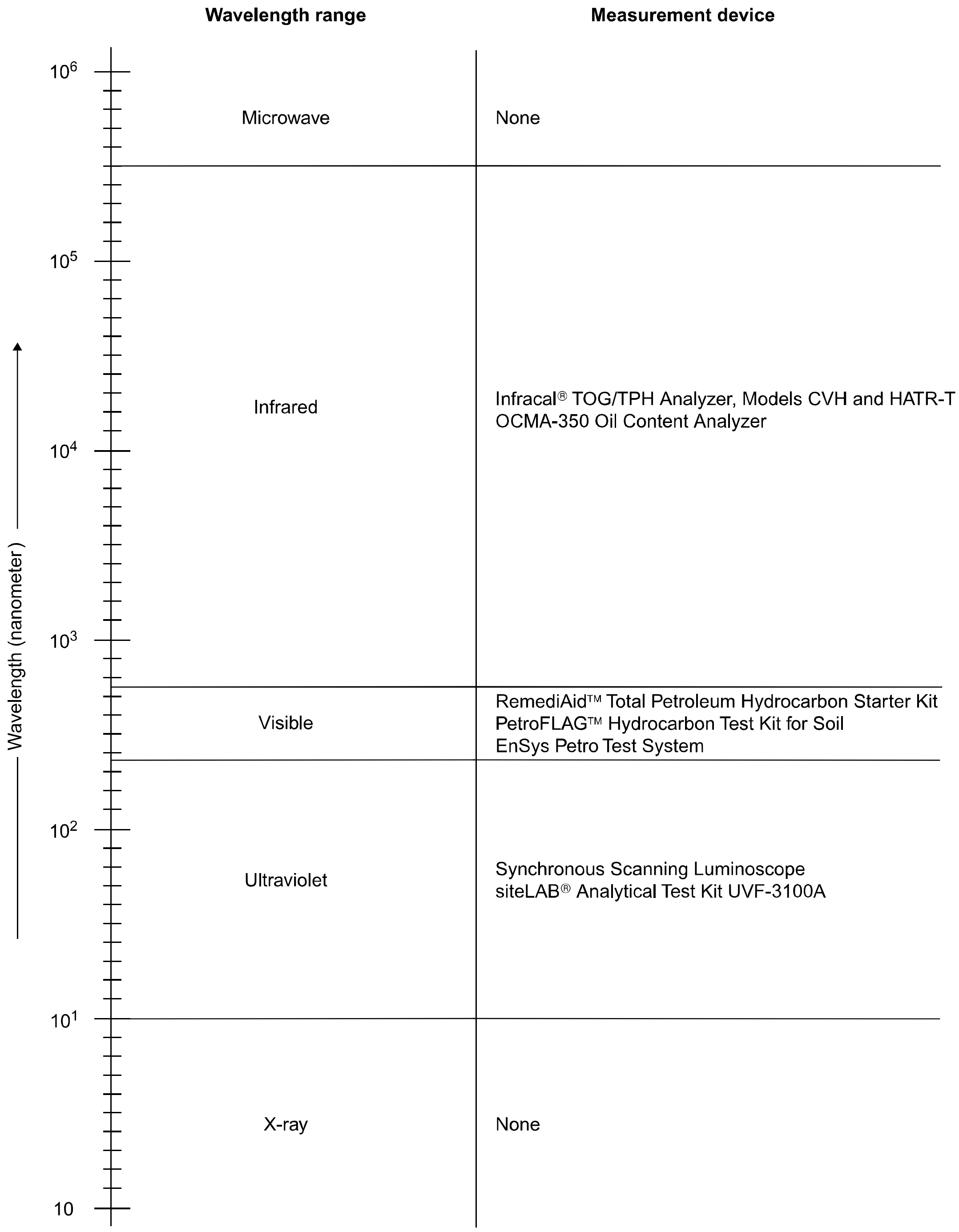


Figure 2-1. Wavelength range used by each measurement device.

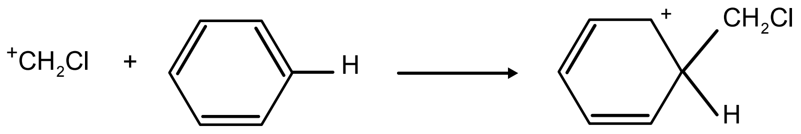
2.1.1.1 Friedel-Crafts Alkylation Reaction

The Friedel-Crafts alkylation reaction involves reaction of an alkyl halide, such as dichloromethane (CH_2Cl_2), with an aromatic hydrocarbon, such as benzene (C_6H_6), in the presence of a solid-phase metal halide catalyst, such as anhydrous aluminum chloride ($AlCl_3$) (Fox 1994).

The first step in the reaction is the metal halide, anhydrous $AlCl_3$, reacting with the alkyl halide, CH_2Cl_2, as shown in Equation 2-1. An alkyl halide is a molecule that contains at least one carbon-chlorine bond. The metal halide polarizes the carbon-chlorine bond or bonds of the alkyl halide, causing the positively charged carbocation (${}^+CH_2Cl$) and negatively charged metal halide ions to separate. This separation results in an intermediate (${}^+CH_2Cl$), which is a positively charged ion whose charge resides on the carbon atom.

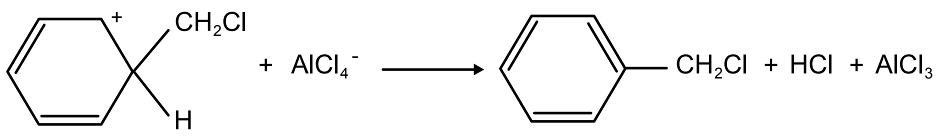
$$CH_2Cl_2 + AlCl_3 \text{ X } {}^+CH_2Cl + AlCl_4^- \qquad (2\text{-}1)$$

In the second step of the reaction, the carbocation attaches to the aromatic hydrocarbon, C_6H_6, producing an intermediate as shown in Equation 2-2.



(2-2)

Equation 2-2 shows one possible structure of the intermediate. The positive charge, like the aromatic double bonds, may be on several of the ring carbon atoms. In the third step of the reaction, this sharing of the charge stabilizes the intermediate and gives it time to react with an $AlCl_4^-$ ion as shown in Equation 2-3. This reaction regenerates the catalyst (anhydrous $AlCl_3$) and forms a colored reaction product (a hydrocarbon derivative) that can absorb light in the visible range of the electromagnetic spectrum. The colored reaction product remains bound to the solid-phase metal halide and settles to the bottom of the reaction mixture.



(2-3)

The concentration of the aromatic hydrocarbon in the reaction mixture is determined by comparing the intensity of the colored reaction product with photographs of standards (color charts) or by using a reflectance spectrophotometer that can measure the concentration of the colored reaction product in the visible range of the electromagnetic spectrum. The intensity of the color produced is directly proportional to the concentration of the aromatic hydrocarbon present.

The RemediAid™ starter kit is based on a modified version of the Friedel-Crafts alkylation reaction. The modified version has the same reaction steps as the classical Friedel-Crafts alkylation reaction described above except that the colored reaction product is not bound to the solid-phase metal halide but remains in the liquid phase of the reaction mixture. This effect is achieved by using the alkyl halide in amounts exceeding the stoichiometry. The TPH concentration in the reaction mixture is determined by comparing the intensity of the colored reaction product with color charts or by using an absorbance spectrophotometer. Color measurement and concentration estimation are further discussed in Section 2.1.1.2.

2.1.1.2 Colorimetry

Colorimetry is a technique by which the intensity of color is assessed using visual or spectrophotometric means. Use of a spectrophotometer is preferred over visual assessment of color charts because the spectrophotometer provides a more accurate and precise measurement and does not rely on a person's skill in interpreting color charts. A reflectance spectrophotometer measures the intensity of light reflected from solid particles in a reaction mixture, and an absorbance spectrophotometer measures the intensity of light that passes through the liquid portion of a reaction mixture. For the classical Friedel-Crafts alkylation reaction (Equations 2-1 through 2-3), a reflectance spectrophotometer is used because the colored reaction product is bound to a solid-phase metal halide. The RemediAid™ starter kit uses an absorbance spectrophotometer because the colored reaction product is present in the liquid phase. Therefore, this section describes colorimetry using an absorbance spectrophotometer.

When a spectrophotometer is used in the visible wavelength range, the reaction mixture is placed in a glass or quartz cuvette that is then inserted into the spectrophotometer and covered with an opaque light shield. A beam of visible light is then passed through the reaction mixture. The wavelength of the light entering the reaction mixture is initially selected by performing a series of absorbance measurements over a range of wavelengths; the selected wavelength generally provides maximum absorbance and allows target compound measurement over a wide concentration range.

Some of the light is absorbed by the chemicals in the reaction mixture, and the rest of the light passes through. Absorbance, which is defined as the logarithm of the ratio of the radiant power of the light source to that of the light that passes through the reaction mixture, is measured by a photoelectric detector in the spectrophotometer (Fritz and Schenk 1987). Absorbance can be calculated using Equation 2-4.

$$A = \log (I_0/I) \tag{2-4}$$

where

A = Absorbance
I_0 = Intensity of light source
I = Intensity of light that passes through the reaction mixture

Therefore, the intensity of the light that passes through the reaction mixture is inversely proportional to the concentration of target compounds in the reaction mixture, or the intensity of the light absorbed by the reaction mixture is directly proportional to the concentration of target compounds in the reaction mixture.

According to Beer-Lambert's law, Equation 2-4 may be expressed as shown in Equation 2-5.

$$A = 0bc \tag{2-5}$$

where

A = Absorbance
0 = Molar absorptivity (centimeter per mole per liter)
b = Light path length (centimeter)
c = Concentration of absorbing species (mole per liter)

Thus, according to Beer-Lambert's law, the absorbance of a chemical species is directly proportional to the concentration of the absorbing chemical species and the path length of the light passing through the reaction mixture. In Equation 2-5, the molar absorptivity is a proportionality constant, which is a characteristic of the absorbing species and changes as the wavelength changes. Therefore, Beer-Lambert's law applies only to monochromatic light (light of one wavelength).

After the absorbance of the reaction mixture is measured, the TPH concentration is determined by comparing the absorbance reading for the reaction mixture to absorbance values for a series of reference standards, which are plotted on a calibration curve.

2.1.2 Infrared Analysis

TPH measurement in soil using the Infracal® TOG/TPH Analyzer and the OCMA-350 Oil Content Analyzer (OCMA-350) is based on infrared analysis. This technology is suitable for measuring aromatic and aliphatic hydrocarbons independent of their carbon range.

Each infrared field measurement device contains a nondispersive infrared (NDIR) spectrophotometer equipped with an infrared radiation source and a filter to isolate the desired wavelength. The NDIR spectrophotometers offer several advantages over conventional, scanning infrared spectrophotometers. A scanning infrared spectrophotometer takes about 1 to 3 minutes to scan a sample and has moderate sensitivity and stability, whereas an NDIR spectrophotometer can achieve a stable reading in about 5 seconds and has greater sensitivity and stability.

The general procedure for infrared analysis of a sample extract for TPH involves the same principles as are described in Section 2.1.1.2 except that light in the infrared wavelength range is used instead of visible light. Measurement of PHCs using infrared analysis involves absorbance measurement because the carbon-hydrogen bonds in the hydrocarbons absorb infrared light. During infrared analysis, absorbances associated with CH, CH_2, and CH_3 configurations are measured at a wavelength of about 3,400 nanometers (nm). Specifically, infrared devices that operate in the 3,380- to 3,500-nm wavelength range should be able to measure CH (3,380 nm), CH_2 (3,420 nm), and CH_3 (3,500 nm) configurations (Simard and others 1951). The absorbance of a sample extract thus measured is directly proportional to the concentration of PHCs present in the extract in accordance with Beer-Lambert's law (see Section 2.1.1.2).

2.1.3 Emulsion Turbidimetry

TPH measurement in soil using the PetroFLAG™ Hydrocarbon Test Kit for Soil (PetroFLAG™ test kit) is based on emulsion turbidimetry. This technology is suitable for measuring aromatic and aliphatic hydrocarbons in the C_8 through C_{36} carbon range.

Turbidimetry may be described as measurement of the attenuation, or loss in intensity, of a light beam as the beam passes through a solution with particles large enough to scatter the light. Emulsion turbidimetry involves measurement of attenuation of light by an emulsion (in an emulsion, one liquid is stably dispersed in a second, immiscible liquid). A direct relationship that follows Beer-Lambert's law exists between the amount of light attenuated and the concentration of the emulsion (McGraw-Hill 1984).

For emulsion turbidimetry, a sample extract is added to a vial containing an aqueous, polar developer solution. The developer solution acts as an emulsifier, causing the aromatic and aliphatic hydrocarbons in solution to precipitate out and form uniformly sized micelles. Micelles are electrically charged, colloidal particles composed of aggregates of large molecules that are stable for some time. The vial containing the resulting emulsion is placed in a turbidimeter. A turbidimeter is similar to the reflectance spectrophotometer described in Section 2.1.1.2 except that the spectrophotometer measures the amount of light reflected by a solution and the turbidimeter measures the amount of light scattered by an emulsion. In the turbidimeter, light at a wavelength of 585 nm is passed through the emulsion, and the amount of light scattered by the emulsion at a 90-degree angle is measured. A wavelength of 585 nm is typically used because the maximum amount of light is scattered by the emulsion at this wavelength. The TPH concentration in the emulsion is then determined by either comparing the turbidity reading for the emulsion to that of a reference standard or a standard calibration curve.

2.1.4 Ultraviolet Fluorescence Spectroscopy

TPH measurement in soil using the Synchronous Scanning Luminoscope (Luminoscope) and the siteLAB® Analytical Test Kit UVF-3100A (UVF-3100A) is based on ultraviolet fluorescence spectroscopy. This technology is suitable for measuring aromatic hydrocarbons independent of their carbon range.

When a sample extract containing both aromatic and aliphatic hydrocarbons is exposed to ultraviolet light, only the aromatic hydrocarbons are excited. The aromatic hydrocarbons then emit light at specific wavelengths that are longer than the excitation wavelength. The intensity and wavelength of the light emitted can be measured, and correlated to the aromatic hydrocarbon concentration in the sample extract. Figure 2-2 shows a schematic of the general process of measurement using ultraviolet fluorescence spectroscopy. The excitation and emission optics may consist of optical lenses that are used to focus light on an optical filter or monochromator. A monochromator is a series of optical filters that reduce a multiple-wavelength light beam to a single-wavelength beam.

In ultraviolet fluorescence spectroscopy, a multiple-wavelength lamp that emits light in the ultraviolet range is used as a light source. Figure 2-1 shows ultraviolet light in relation to the electromagnetic spectrum. The ultraviolet light is directed through the excitation optics. The resulting focused light energy from the excitation optics is used to irradiate the sample extract under analysis. Some of the light energy is absorbed by the molecules of the aromatic hydrocarbons in the sample extract, resulting in excitation of those molecules.

When the excited molecules return to a stable state by losing energy, the energy emitted has longer wavelengths than those of the energy absorbed by the molecules. The emission optics are placed at a 90-degree angle to the excitation optics, and the longer-wavelength energy emitted by the molecules passes through the emission optics and is detected by a photomultiplier tube. The photomultiplier tube detects and amplifies the energy and converts it into an electrical signal that is used to determine the intensity of the light energy emitted. The emission optics and photomultiplier tube are placed at a 90-degree angle to the light source in order to minimize the light source interference detected by the photomultiplier tube.

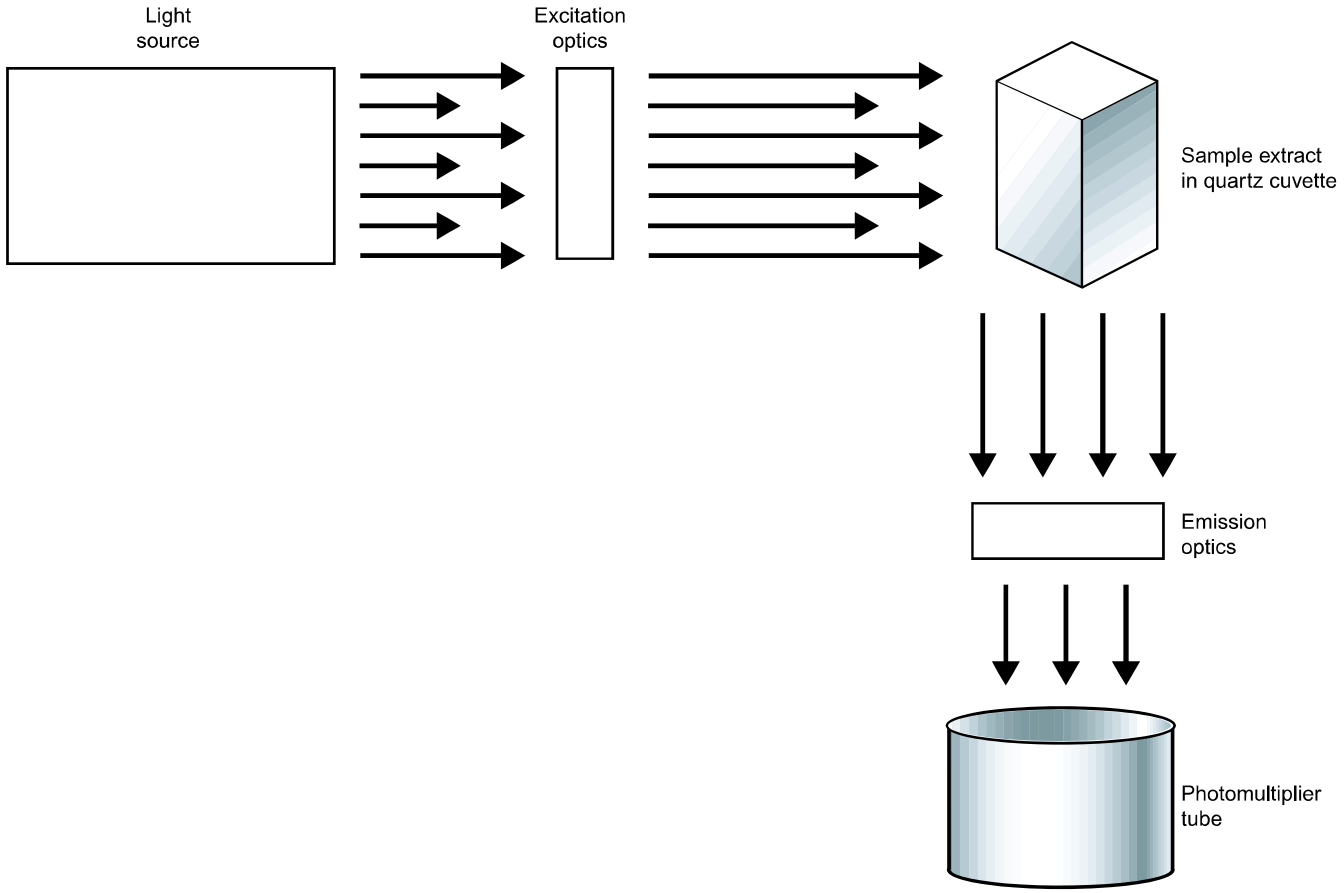


Figure 2-2. Schematic of ultraviolet fluorescence spectroscopy.

A spectrum of fluorescence intensity versus emission wavelength is generated and evaluated to determine whether any of the peaks correspond to known groups of hydrocarbons. To determine the relationship between the fluorescence intensity and the aromatic hydrocarbon concentration of a sample extract, a calibration curve can be generated using site-specific TPH, GRO, or EDRO concentrations or known standards selected based on the type of PHCs being measured at a site.

2.1.5 Immunoassay and Colorimetry

TPH measurement in soil using the EnSys Petro Test System is based on a combination of immunoassay and colorimetry. This combination of technologies is suitable for measuring a large portion of the aromatic hydrocarbons and a few aliphatic hydrocarbons in the C_6 through C_{22} carbon range. Immunoassay and colorimetry are described below.

2.1.5.1 Immunoassay

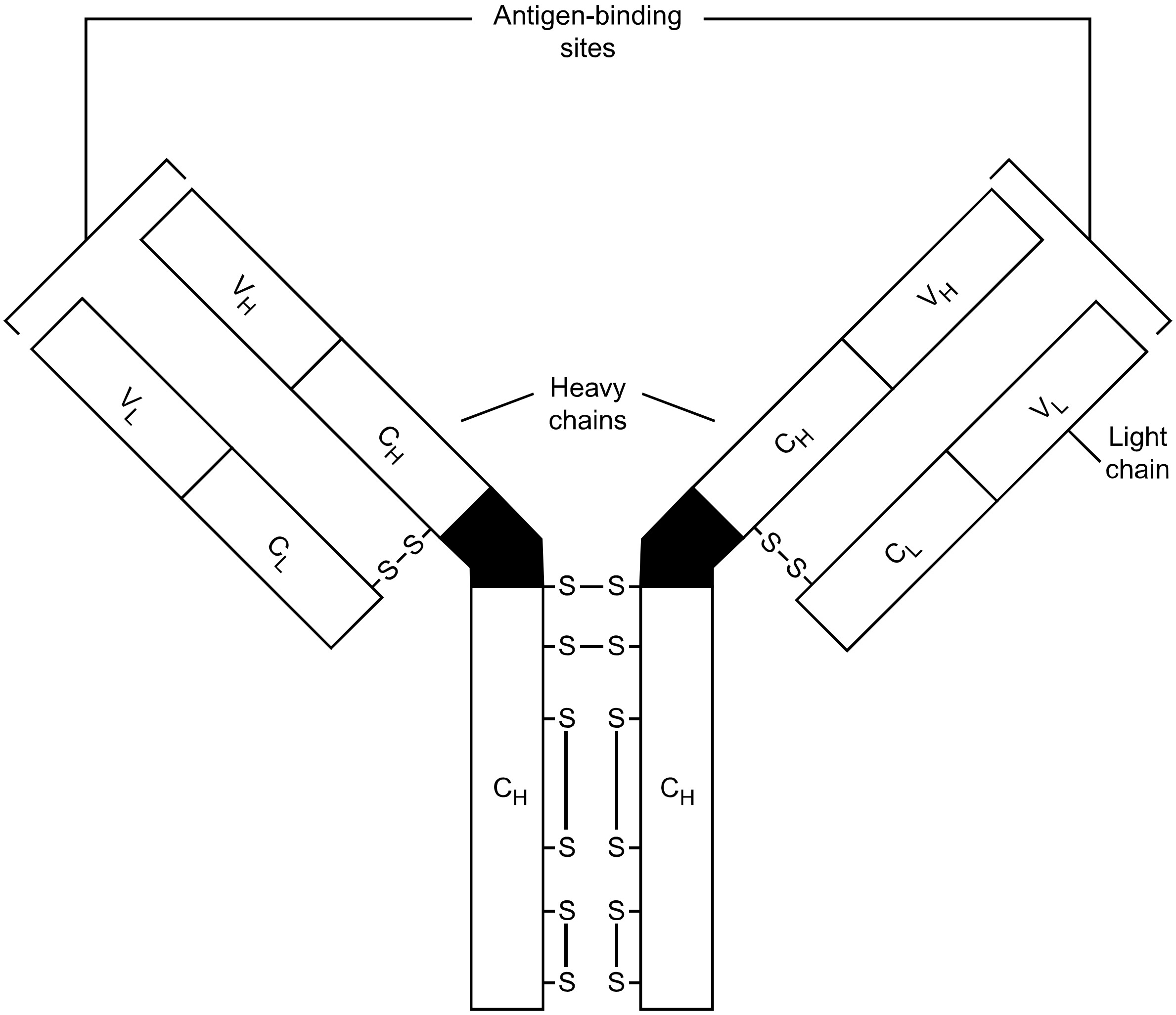
Immunoassay is a technique for measuring a target compound's concentration using biologically engineered antibodies. Antibodies are a class of proteins known as immunoglobulins that are produced by the immune system of animals in response to a foreign substance (an antigen). The antibodies produced can bind with the antigen that stimulated their production. Specifically, antibodies are produced in response to localized, reactive sites called antigenic determinants on the surface of the antigen. Antigenic determinants consist of amino acid sequences (Rittenburg 1990). Because an antigen may possess more than one type of antigenic determinant, more than one type of antibody may be produced by the immune system. In general, the antibodies produced are structured in such a way that they selectively bind to the antigenic determinants on the antigen that stimulated their production, resulting in formation of an antibody-antigen complex.

Five major classes of antibodies (immunoglobulin [Ig] A, IgD, IgE, IgG, and IgM) are produced by the immune system. IgG is the most common type of antibody used in immunoassay (Rittenburg 1990). IgG is a Y-shaped molecule consisting of two identical heavy polypeptide chains and two identical light polypeptide chains bound together by disulfide bonds. Both the heavy and light chains have variable and constant regions. The variable regions at the ends of the two arms of the Y-shaped antibody form areas called antigen-binding sites; therefore, two antigen-binding sites are present on each antibody. The general structure of the IgG antibody is shown in Figure 2-3.

The dimensions and contours of antigen-binding sites are determined by the sequence of amino acids in the variable regions of the antibody. On a single antibody molecule, the two binding sites have identical variable regions. As a result, the two binding sites have identical specificity for a particular antigenic determinant (Rittenburg 1990). However, the binding sites of antibodies produced in response to different antigenic determinants are not the same.

The binding affinity between an antibody and antigen is determined by (1) the sequence of amino acids in the variable regions of the antibody, (2) the structure and location of the antigenic determinant on the antigen, and (3) the attractive forces that stabilize the antibody-antigen complex. The attractive forces include a combination of hydrogen bonds, hydrophobic bonds, coulombic interaction, and van der Waals forces (Rittenburg 1990). The closer the antigenic determinant is to the antigen-binding site on the antibody, the higher the binding affinity.

Immunoassays employ either polyclonal or monoclonal antibodies. Because an antigen generally contains more than one type of antigenic determinant, more than one type of antibody may be produced in the immune response. Therefore, the antibodies produced are not identical and are called polyclonal antibodies. Because polyclonal antibodies are not identical, they will, as a group, exhibit varied specificities and binding affinities for antigenic



Notes:

-S-S- = Disulfide bond
C = Constant region
H = Heavy polypeptide chain
L = Light polypeptide chain
V = Variable region

Figure 2-3. Immunoglobulin G antibody structure and locations of antigen-binding sites.

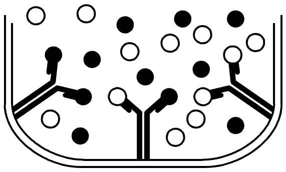
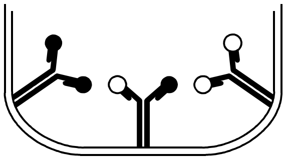
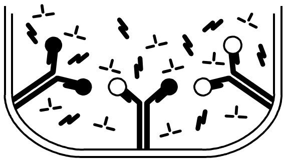
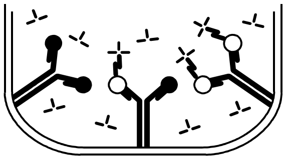
determinants. Monoclonal antibodies are produced by isolating those antibodies produced in response to one type of antigenic determinant. As a result, monoclonal antibodies are structurally identical and exhibit the same specificities and binding affinities for the antigenic determinant that stimulated their production.

Although an antibody has a particular specificity and binding affinity for the antigenic determinant that produced the antibody, cross-reactivity with other compounds may occur. For example, cross-reactivity may occur when the antigenic determinant that stimulated the antibody's production is present in other compounds (SDI 2000). Cross-reactivity may also occur with other compounds that possess structurally similar antigenic determinants (Rittenburg 1990).

Immunoassay effectiveness is primarily a function of (1) the specificities and binding affinities of the polyclonal or monoclonal antibodies used and (2) whether one compound or a group of compounds is being measured. For example, cross-reactivity will result in false positives when only one compound is being measured. However, cross-reactivity is desirable when a group of compounds, such as PHCs, is being measured. Whether polyclonal or monoclonal antibodies are better suited for measuring PHCs depends on the individual antibodies used; for example, highly cross-reactive, monoclonal antibodies can be as effective as less cross-reactive, polyclonal antibodies.

The EnSys Petro Test System is based on a type of immunoassay called enzyme-linked immunosorbent assay (ELISA). ELISA uses either polyclonal or monoclonal antibodies adsorbed to the inside wall of a test tube in order to facilitate separation of target compounds from nontarget compounds during a washing step. In ELISA, an enzyme conjugate solution is used to produce color whose intensity is inversely proportional to the total concentration of PHCs in a sample extract. ELISA involves the following three steps: (1) enzyme conjugate and sample extract addition, (2) washing, and (3) color development. These steps are described below and are illustrated in Figure 2-4. The intensity of the color produced during color development is measured using standard colorimetric principles as described in Section 2.1.5.2.

Enzyme Conjugate and Sample Extract Addition. As a first step, an enzyme conjugate solution is added to the soil sample extract. An enzyme conjugate is an enzyme bound to a target compound. The antigen used to initiate antibody production is also used as the target compound portion of the enzyme conjugate. The enzyme portion of the enzyme conjugate plays its role in ELISA during the color development step; the enzyme typically used in ELISA is horseradish peroxidase. The reaction mixture containing the sample extract and enzyme conjugate solution is added to an antibody-coated test tube. Because both the sample extract target compound and the enzyme conjugate can bind with the antibodies, the sample extract target compound and the enzyme conjugate compete for the antigen-binding sites on the antibodies. The sample extract target compound and the enzyme conjugate bind to the antibodies in direct proportion to their relative concentrations in the reaction mixture. For example, the greater the ratio of the sample extract target compound concentration to the enzyme conjugate concentration, the greater the proportion of antigen-binding sites that are occupied by the sample extract target compound.

Step	Schematic	Description
Enzyme conjugate and sample extract addition		A reaction mixture containing the sample extract and enzyme conjugate solution is added to an antibody-coated test tube. The sample extract target compound and enzyme conjugate compete for antigen-binding sites.
Washing		The unbound sample extract target compound and enzyme conjugate are removed from the test tube.
Color development		A substrate and chromogen are added to the test tube.
		The substrate and chromogen react with the enzyme in the enzyme conjugate to produce color. The lower the color intensity, the higher the sample extract target compound concentration.

Notes:

- Antibody
- Sample extract target compound
- Enzyme conjugate
- Substrate
- Chromogen

Figure 2-4. Enzyme-linked immunosorbent assay.

Washing. The sample extract target compound and the enzyme conjugate that are bound to the antibodies are separated from the unbound sample extract target compound and enzyme conjugate by emptying the reaction mixture from the test tube and washing the test tube with potable water.

Color Development. A substrate, such as hydrogen peroxide, and a chromogen, such as tetramethylbenzidine or orthophenylenediamine, are then added to the test tube in order to produce color when they react with the enzyme in the enzyme conjugate. For example, the enzyme horseradish peroxidase reacts with the hydrogen peroxide to release a proton, which in turn reduces the tetramethylbenzidine or orthophenylenediamine to form the colored product. After a specified period of time, color development in the test tube is terminated using a stopping solution such as hydrochloric acid. The amount of color formed is directly proportional to the amount of enzyme conjugate bound to the antibodies. Because the sample extract target compound competes with the enzyme conjugate for antigen-binding sites, ELISA results in formation of color in the test tube whose intensity is inversely proportional to the concentration of the sample extract target compound; for example, less color indicates a higher concentration of the sample extract target compound.

2.1.5.2 Colorimetry

After completion of color development, the concentration of PHCs in the sample extract is determined using colorimetry. During colorimetry, the intensity of the color is assessed by measuring the absorbance of the colored reaction mixture using a differential spectrophotometer. The differential spectrophotometer is a double-beam instrument in which two equivalent beams of light are produced within the visible range of the electromagnetic spectrum. One beam passes through the colored reaction mixture developed using the sample extract, while the other beam passes through a colored reaction mixture developed using a reference standard. The spectrophotometer measures the difference in absorbance between the two colored reaction mixtures. A positive reading on the spectrophotometer indicates that the concentration of PHCs in the sample extract is less than that in the reference standard. Similarly, a negative reading on the spectrophotometer indicates that the concentration of PHCs in the sample extract is greater than that in the reference standard.

2.2 Field Measurement Device Descriptions

This section describes the seven innovative TPH field measurement devices that will be demonstrated. Field measurement devices may be categorized as quantitative, semiquantitative, and qualitative. These categories are explained below.

- A **quantitative measurement device** measures TPH concentrations ranging from its reporting limit through its linear range. The measurement result is reported as a single, numerical value that has an established precision and accuracy.
- A **semiquantitative measurement device** measures TPH concentrations above its reporting limit. The measurement result may be reported as a concentration range with lower and upper limits.
- A **qualitative measurement device** indicates the presence or absence of PHCs above or below a specified value (for example, the reporting limit or an action level).

Each of the seven devices that will be demonstrated produces either quantitative or semiquantitative results. The device descriptions presented in Sections 2.2.1 through 2.2.7 identify the type of results produced by each device. Performance data included in the device descriptions were provided by the device developers.

2.2.1 RemediAid™ Starter Kit

The RemediAid™ starter kit, a quantitative test kit developed by CHEMetrics, Inc. (CHEMetrics), and AZUR Environmental Ltd (AZUR) in conjunction with Shell Research Ltd. and manufactured by CHEMetrics, is based on a combination of the Friedel-Crafts alkylation reaction and colorimetry discussed in Section 2.1.1. The kit has been commercially available since 1998. This section describes the kit, presents its operating procedure, and discusses its advantages and limitations.

2.2.1.1 Device Description

As stated in Section 2.1.1, the Friedel-Crafts alkylation reaction involves reaction of an alkyl halide with an aromatic compound in the presence of a metal halide. The RemediAid™ starter kit uses CH_2Cl_2 as both the alkyl halide and extraction solvent and uses anhydrous $AlCl_3$ as the metal halide. When excessive CH_2Cl_2 is used, the colored reaction product to be measured remains in the liquid phase. According to the developers of the kit, because the presence of chlorinated solvents in the sample extract may result in false positive results, a premeasured volume of CH_2Cl_2 is included with the kit in a sealed, single-use, double-point ampule. A known volume of CH_2Cl_2 is used so that this value can be incorporated into the sample extract concentration calculation as described in the kit operating procedure (see Section 2.2.1.2). Anhydrous $AlCl_3$ is used because it is the most sensitive metal halide and it provided the most accurate recoveries for various types of hydrocarbons during laboratory tests performed by CHEMetrics and AZUR. As described in Section 2.1.1.2, the kit uses an absorbance LED-based photometer and measures sample extract absorbance using visible light of a 430-nm wavelength.

According to CHEMetrics and AZUR, the RemediAid™ starter kit responds to all hydrocarbon products as long as they contain aromatic hydrocarbons. The kit can respond to aromatic hydrocarbons independent of their carbon range.

For optimum performance, the spectrophotometer should be used in environments with a temperature range of 0 to 50 °C and with a maximum relative humidity of 95 percent, and it should not be stored at temperatures greater than 32 °C. The kit does not require any other special storage conditions because its chemicals are vacuum-sealed and are therefore not susceptible to degradation.

According to CHEMetrics and AZUR, the method detection limit (MDL), precision, and accuracy that can be achieved with the RemediAid™ starter kit vary depending on the reactivity of the hydrocarbons being measured. No information is available on the MDL, precision, and accuracy for soil sample extracts. However, assuming that a sample extract does not require dilution before analysis, the following MDL, precision, and accuracy ranges generally apply to the kit: MDLs ranging from 2.0 mg/L for weathered gasoline to 10 mg/L for heavy oil, precision values ranging from 2.0 mg/L for weathered gasoline to plus or minus (±) 11.0 mg/L for heavy oil, and accuracy values ranging from -4.8 mg/L for weathered gasoline to + 31.3 mg/L for heavy oil.

A kit user must first purchase the RemediAid™ starter kit and may then purchase replenishment kits thereafter. Table 2-2 lists the components of the RemediAid™ starter kit and the replenishment kit. The RemediAid™ starter kit includes enough supplies to perform 8 soil analyses, and the replenishment kit includes enough supplies to perform 16 more soil analyses.

The components of the RemediAid™ starter kit are packaged in a carrying case that is 13.75 inches long, 15.5 inches wide, and 4.5 inches deep. The replenishment kit components are packaged in a box that is 9.25 inches long, 10.25 inches wide, and 4.5 inches deep. A kit user needs to provide disposable gloves, safety glasses, and a disposal pipet or syringe capable of measuring 5 mL. The photometer operates on one 9-volt battery; weighs 0.43 pound; and is 6.0 inches long, 2.4 inches wide, and 1.25 inches deep.

The RemediAid™ starter kit (Model No. TPH0001) can be purchased for $800, and the replenishment kit (Model No. TPH0002) can be purchased for $240. Kit components may also be purchased individually from the developers. The RemediAid™ starter and replenishment kits are not available for rental.

According to CHEMetrics, one technician can perform 16 analyses in about 1 hour using the kit. All kit reagents are premeasured and contained in vacuum-sealed ampules. Only one technician is required to perform analyses using the RemediAid™ starter kit. The kit is designed to be used by those with basic wet chemistry skills. CHEMetrics provides technical support over the telephone at no additional cost.

According to the developers, the RemediAid™ starter kit is innovative because the colored reaction product remains in the liquid phase, which allows measurement of color intensity using a portable absorbance spectrophotometer. According to the developers, portable versions of reflectance spectrophotometers are not commercially available, making interpretation of a solid colored reaction product impossible in the field. All chemicals supplied as part of the RemediAid™ starter and replenishment kits are vacuum-sealed, which minimizes user contact with reagents and eliminates the need for pipetting and measuring skills, thus minimizing the possibility of user error.

Table 2-2. RemediAid™ Starter and Replenishment Kit Components

Starter Kit Components

- Battery-powered balance (9-volt battery included)
- Battery-powered timer (AAA battery included)
- Battery-powered, portable photometer (9-volt battery included)
- 8 double-tipped ampules containing 20 milliliters each of dichloromethane
- 8 vacuum-sealed ampules containing anhydrous aluminum chloride and filtering columns
- Anhydrous sodium sulfate (50 grams)
- 8 extraction cleanup tubes and caps containing Florisil
- 8 reaction tubes and caps containing sodium sulfate
- 8 small, silicone ampule caps
- 8 weighing boats
- Tip-breaking tool
- Light shield
- Ampule rack that holds 36 ampules
- Reaction tube plug/snapper
- Spatula
- Reagent blank ampule
- Test procedure manual and simplified instruction card
- Material Safety Data Sheets
- Carrying case

Replenishment Kit Components

- 16 double-tipped ampules containing 20 milliliters each of dichloromethane
- 16 vacuum-sealed ampules containing anhydrous aluminum chloride and filtering columns
- 16 extraction cleanup tubes and caps containing Florisil
- 16 reaction tubes and caps containing sodium sulfate
- 16 weighing boats

2.2.1.2 Operating Procedure

Measuring TPH in soil using the RemediAid™ starter kit involves the following three steps: (1) extraction/extraction clean-up, (2) color development, and (3) color measurement. These three steps are detailed below. Calibration procedures and QC checks required for the device are presented in Chapter 8.

Step 1 - Extraction

1. Measure 5 grams of soil sample.

2. Transfer the soil sample to the reaction tube containing anhydrous sodium sulfate, a drying agent for removing moisture from the soil sample. Cap the tube, and shake it briefly to obtain a uniform, free-flowing mixture. If the sample still appears to be wet or does not form a granular, uniform mixture, add more anhydrous sodium sulfate, and shake the tube vigorously until a uniform, free-flowing mixture is obtained. If necessary, use a spatula to break up clumps of wet soil.

3. Hold the double-tipped ampule containing 20 milliliters (mL) of solvent (CH_2Cl_2) over the reaction tube, and snip off the top end of the ampule using the green, tip-breaking tool.

4. Carefully invert the ampule over the reaction tube. Snip off the other end of the ampule to allow the solvent to flow freely into the reaction tube. Cap the reaction tube, and shake it vigorously for 3 minutes.

5. Let the reaction tube stand undisturbed for 2 minutes, and allow the soil to settle to the bottom of the tube.

6. Decant the extract to an extraction clean-up tube containing 2.0 grams of Florisil, using care not to transfer any soil to the extraction clean-up tube. Florisil is an activated magnesium silicate used as a polar adsorbent to minimize interference from natural organic material (for example, humic substances). Cap this reaction tube, and shake it for 1 minute. Allow the Florisil to settle for approximately 2 minutes.

7. Remove the cap from the reaction tube, and replace the cap with the reaction tube plug/snapper, a round, white plug with a small hole in the center.

Step 2 - Color Development

1. Push a filtering column onto the tip of the vacuum-sealed ampule containing anhydrous $AlCl_3$ until the column fits snugly.

2. Insert the column and ampule assembly through the hole in the reaction tube plug/snapper up to the blue line on the ampule. While holding the reaction tube, gently pull the ampule to one side in order to snap the ampule tip. The ampule will then slowly draw liquid from the reaction tube.

3. Withdraw the column and ampule assembly from the reaction tube, and invert the assembly. Remove the column.

4. Firmly place a small, silicone ampule cap on the tip of the ampule, and invert the ampule every 2 minutes for 10 minutes. Then let the ampule stand undisturbed for 10 minutes. Depending on the concentration and type of hydrocarbon present, the solvent in the ampule will turn a yellow to orange-brown color. If the ampule appears to be cloudy after 10 minutes of standing, wait an additional 5 to 10 minutes for the cloudiness to settle out. Do not let the ampule stand longer than 1 hour before placing it in the spectrophotometer, as this may result in a small positive bias.

Step 3 - Color Measurement

1. Immediately after allowing the ampule to stand undisturbed for 10 minutes, or after waiting for any cloudiness to settle out, insert the reagent blank ampule into the spectrophotometer to zero the instrument.

2. Remove the reagent blank, insert the test ampule into the spectrophotometer, and record the absorbance.

3. If the absorbance is less than 0.700, use Equation 2-6 to convert the absorbance value to mg/kg TPH in the soil sample on a wet weight basis.

$$\text{TPH concentration in soil sample (mg/kg)} = \frac{[(A \times S) - I] \times V}{W} \tag{2-6}$$

where

A = Absorbance
S = Slope for a specific hydrocarbon mixture ([mg/L/A]; see the test procedure manual)
I = Intercept for a specific hydrocarbon mixture (mg/L; see the test procedure manual)
V = Volume of extract: 20 mL
W = Weight of soil sample: 5 grams

Note: CHEMetrics and AZUR developed S and I values by plotting absorbance on the x-axis and concentration on the y-axis, which is the opposite of conventional practice. Hence, S and I are expressed in concentration units of mg/L.

4. If the absorbance is equal to or greater than 0.700, perform a five-times dilution by first placing 5 mL of the extract supernatant from item 5 of the extraction step in a reaction tube. Add the contents of a double-tipped ampule (20 mL of CH_2Cl_2) to the reaction tube, cap the tube, and shake it briefly. Then follow the color development and color measurement steps described above. Use Equation 2-7 to convert the absorbance value to mg/kg TPH in the soil sample on a wet weight basis.

$$\text{TPH concentration in soil sample (mg/kg)} = \frac{[(A \times S) - I] \times V \times 5}{W} \qquad (2\text{-}7)$$

2.2.1.3 Advantages and Limitations

An advantage of the RemediAid™ starter kit is that it is easy to operate, requiring one operator with minimal skills in basic wet chemistry techniques. The kit provides premeasured amounts of chemicals in specially designed, single-use ampules. The ampules are vacuum-sealed, and therefore the chemicals are not susceptible to degradation. The single-use ampules eliminate the need for measuring and pipetting skills, minimize user contact with reagents, and minimize the possibility of operator error. In addition, the spectrophotometer operates on a 9-volt battery, so an alternating current (AC) power source is not required in the field.

The kit uses a drying agent (anhydrous sodium sulfate) to remove moisture from soil samples. As a result, no correction associated with solvent dilution is required, which is necessary for wet soil samples. The kit also uses Florisil to eliminate interferences from natural organic matter in soil. However, this practice results in removal of polar compounds from the sample extract, including PHC degradation products (California Environmental Protection Agency 1999).

Another advantage of the kit is that it can quantitatively measure all fuel types that contain an aromatic hydrocarbon component. According to the developers, the kit is designed to measure aromatic hydrocarbons regardless of their carbon range. If a site-specific calibration is performed, the kit can measure aliphatic hydrocarbons in the presence of aromatic compounds, but a limitation of the kit is that it does not measure aliphatic hydrocarbons when aromatic compounds are not present in the sample.

2.2.2 Infracal® TOG/TPH Analyzer, Models CVH and HATR-T

The Infracal® TOG/TPH Analyzer, a quantitative device developed by Wilks Enterprise, Inc. (Wilks), is based on infrared analysis as discussed in Section 2.1.2. The Infracal® TOG/TPH Analyzer is identified according to the sample stage used in the device. The device can be operated as either Model CVH or Model HATR-T simply by switching the sample stages. Model CVH uses the CVH sample stage, which contains a quartz cuvette, while Model HATR-T uses the cubic zirconia horizontal attenuated total reflection sample stage. Models CVH and HATR-T have been commercially available since 1996 and 1997, respectively. Model CVH is used when a sample contains either GRO or GRO and EDRO; Model HATR-T cannot measure GRO. This section describes both models of the device, presents their operating procedure, and discusses their advantages and limitations.

2.2.2.1 Device Description

Models CVH and HATR-T include a single-beam, fixed-wavelength, NDIR filter-based spectrophotometer with a dual detector system. As stated above, the only difference between the two models involves the sample stage used.

In Model CVH, infrared radiation from a tungsten lamp is captured using an elliptical source mirror and transmitted through a quartz cuvette that contains a sample extract. The radiation that has passed through the extract enters a dual detector system containing filters that isolate a reference wavelength of 2,500 nm and an analytical wavelength of 3,400 nm. The reference wavelength stabilizes device response and automatically corrects absorbance values for fluctuations in ambient temperature and relative humidity.

Model CVH is suitable for analyzing sample extracts that have been extracted from soil using Freon 113 or other solvents invisible in the measurement range. Model HATR-T, unlike Model CVH, is based on an evaporation technique and measures residual hydrocarbons after volatile organics evaporate from the sample extract. Therefore, analyses using Model HATR-T result in the loss of some volatiles in the GRO range. Vertrel® MCA, a hydrochlorofluorocarbon extraction solvent manufactured by DuPont, is used with Model HATR-T. Although hexane can be used as the extraction solvent with Model HATR-T, Vertrel® MCA is preferred because (1) it achieves measurement stability more quickly than hexane (in 1.5 to 2 minutes instead of 3 to 5 minutes); (2) it has a lower boiling point than hexane, which results in fewer light-end volatile organic compounds being lost in the evaporation process; and (3) it is less flammable than hexane, resulting in fewer disposal concerns. In addition, Model HATR-T does not require a cuvette to contain the sample extract. The extract is transferred directly to the sample stage.

The Infracal® TOG/TPH Analyzer presents results in units selected by the user during calibration, such as mg/kg, mg/L, or absorbance values. According to Wilks, both Models CVH and HATR-T can measure aromatic and aliphatic hydrocarbons. However, Model CVH can measure both GRO and EDRO, but Model HATR-T can primarily measure EDRO. Model CVH has (1) an MDL of 3 mg/kg and is linear up to 5,000 mg/kg in soil; (2) a measurement accuracy of ± 1 percent; and (3) a measurement precision of ± 1 percent. Model HATR-T has an MDL of 20 mg/kg and is linear up to 5,000 mg/kg in soil. The Infracal® TOG/TPH Analyzer uses a point-to-point calibration to correct for non-linearity. The Infracal® TOG/TPH Analyzer can operate in a temperature range of 4 to 43 °C and a relative humidity range of 10 to 60 percent. When the device is not in operation, it can be stored in a temperature range of -18 to 52 °C.

Table 2-4 lists the components of the Infracal® TOG/TPH Analyzer. The device weighs 4.5 pounds and is 6.5 inches long, 6.5 inches wide, and 5 inches deep. Wilks offers users a field sampling kit for TPH in soil, KIT-10410-S, the components of which are also presented in Table 2-4. Additional supplies required for TPH analysis using the Infracal® TOG/TPH analyzer are also listed in the table.

The Infracal® TOG/TPH Analyzer has a standard, nine-pin, female DB9 connector (RS232-C) for serial data communication. Wilks offers an optional software package, InfraWin, that allows the user to connect a personal computer to the device and automatically download, label, and save measurement results; remotely control measurement parameters; generate and store multiple calibration tables; and report measurement results in various numerical and graphical formats. The built-in microprocessor in the device can store up to 10 measurement results for use with its averaging function or for local recall and display. Measurement results may be transferred via the serial communication interface to a serial printer or to an external personal computer. The device may be connected to an external battery pack or an automobile cigarette lighter. The device draws only about 8 watts (0.67 ampere) of power.

Model CVH can be purchased for $4,675. Rental is available for 15 percent of the purchase price per month. The HATR-T sample stage can be purchased for an additional $1,150. The serial printer can be purchased from Wilks for $695. KIT-10410-S can be purchased for $865 and includes enough supplies to perform at least 75 soil analyses. Additional required supplies that can be purchased from Wilks include a set of four 10-mm, quartz cuvettes with Teflon™ stoppers ($575) and the InfraWin software package ($475). Additional required supplies that cannot be purchased from Wilks are listed in Table 2-4.

Table 2-4. Infracal® TOG/TPH Analyzer, Models CVH and HATR-T Components

Spectrophotometer and Accessories
- Infrared spectrophotometer
- CVH or HATR-T sample stage
- Dust cover
- Power supply cable
- Instruction manual

KIT-10410-S Components
- Timer (batteries included)
- Battery-powered balance (batteries included)
- Silica gel (60-200 mesh) (500 grams)
- Teflon™ wash bottle (125 milliliters)
- Glass funnel
- 10-milliliter, graduated cylinder
- 100-milliliter, graduated cylinder with stopper
- Air syringe
- 20-milliliter, glass beaker
- Spatula
- 50-microliter pipette with pipette tips (pack of 50)
- 250-milliliter syringe
- 40-milliliter vials (box of 50)
- Extraction reservoirs (box of 50)
- Reservoir sealer
- Extraction procedure instructions

Additional Required Supplies that Can Be Purchased from Wilks
- 10-millimeter, quartz cuvettes
- 50-millimeter, quartz cuvette and holder

Additional Required Supplies that Cannot be Purchased from Wilks
- Extraction solvents (Freon 113 or Vertrel® MCA)
- Seven standards (104; 208; 260; 389; 519; 778; and 1,038 milligrams per liter) of 3-IN-ONE oil in Freon 113 in sealed cuvettes for Model CVH (standards for Model HATR-T will be prepared in Vertrel® MCA solvent on site)
- 100-microliter, glass syringe
- One 3-ounce bottle of 3-IN-ONE oil
- 4-ounce, high-density polyethylene, disposable bottles
- Disposable eye droppers

According to Wilks, the average sample extraction and analysis time for Models CVH and HATR-T is 10 to 15 minutes per sample. Both models are easy to use. Normal training for using each model involves reading the instruction manual. Wilks also provides technical support over the telephone at no additional cost.

According to Wilks, Models CVH and HATR-T are innovative TPH field measurement devices because their spectrophotometer uses a pulsed, infrared light source instead of a “chopper,” which mechanically “chops” the light beam to turn the radiation signal on and off. The chopper, which is a primary component of most conventional spectrophotometers, requires more maintenance to prevent drift than does the pulsed, infrared light source. In addition, Model HATR-T does not use Freon 113, which is expensive and is being phased out of use.

2.2.2.2 Operating Procedure

Measuring TPH in soil using Models CVH and HATR-T involves the following three steps: (1) extraction, (2) measurement of TPH in the extract, and (3) calculation of the TPH concentration in soil. These steps are described below for both models. In these steps, operating procedures specific to Model CVH are identified as "(a)" and to Model HATR-T are identified as "(b)." Calibration procedures and QC checks required for the Infracal® TOG/TPH Analyzer are presented in Chapter 8.

Step 1 - Extraction

1. Measure 20 grams of soil sample, and place the measured amount in a 40-mL vial.
2. Add 2 to 5 grams of 60-200 mesh silica gel to the vial, depending on the soil's moisture content. Cap and shake the vial, and ensure that its contents are free-flowing.
3. Add 20 mL of (a) Freon 113 or (b) Vertrel® MCA to the vial.
4. Cap the vial, and shake it vigorously for 2 minutes. Then let the vial stand for up to 2 minutes in order to allow the soil and sample extract to separate.
5. Decant the sample extract into an extraction reservoir with a filter frit (to capture large particles) and a silica gel cartridge (to capture small particles and remove natural hydrocarbons). Leave as much of the soil in the vial as possible.
6. Seal the extraction reservoir with a sealer, and insert the tip of the air syringe into the sealer.
7. Place the tip of the extraction reservoir over the vial, and push down on the air syringe plunger so that the sample extract drips slowly into the vial.
8. Discard the first 1 mL (4 or 5 drops) of sample extract in the vial.

Step 2 - Measurement of TPH in Extract

1. (a) Place the tip of the extraction reservoir over a 10-mm, quartz cuvette, and allow the rest of the sample extract to drip into the cuvette until the cuvette is full.

   (b) Place the tip of the extraction reservoir over a beaker, and allow the rest of the sample extract to drip into the beaker. Immediately collect 50 microliters (μL) of the sample extract from the beaker using a pipette.
2. (a) Insert the quartz cuvette into the CVH sample stage, and record the sample extract concentration in mg/L.

(b) Transfer the sample extract from the pipette onto the center of the HATR-T sample stage, allow the sample extract to evaporate, and record the residual sample extract concentration in mg/L.

Step 3 - Calculation of TPH Concentration in Soil

Because the infrared spectrophotometer can measure the soil TPH concentration in mg/kg on a wet weight basis, no additional calculations are required. However, any variations from the soil sample amount or reagent amount specified in Step 1 or any sample dilutions should be factored into a calculation of the TPH concentration.

2.2.2.3 Advantages and Limitations

An advantage of the Infracal® TOG/TPH Analyzer is that it is easy to operate, requiring one person with basic wet chemistry skills. It has no moving parts that require optical alignment or adjustment, and it uses a pulsed, infrared light source instead of a chopper to prevent measurement fluctuations resulting from mechanical wear. The chopper, which is a primary component of most conventional spectrophotometers, requires more maintenance than does the pulsed, infrared light source. In addition, the devices can be operated using either a direct current (DC) power source such as an automobile cigarette lighter or an external battery pack; an AC power source is not required in the field.

Model CVH can measure all TPH fuel types and is designed to measure both aromatic and aliphatic hydrocarbons independent of their carbon range. Model HATR-T can also measure both aromatic and aliphatic hydrocarbons primarily in the EDRO range.

Model CVH can use Freon 113, a chlorofluorocarbon (CFC), as the extraction solvent. CFCs that are discharged to the atmosphere are primary contributors to depletion of the earth's stratospheric ozone layer. The United States, as a party to the Montreal Protocol on Substances that Deplete the Ozone Layer and as required by law under the Clean Air Act of 1990, is committed to controlling and eventually phasing out use of CFCs. As a result, Freon 113 will become increasingly scarce and expensive. However, Model HATR-T allows use of Vertrel® MCA or hexane as the extraction solvent instead of Freon 113.

2.2.3 OCMA-350

The OCMA-350, a quantitative device developed by Horiba Instruments, Incorporated (Horiba), is based on infrared analysis as discussed in Section 2.1.2. The OCMA-350 has been commercially available since 1995. This section describes the device, presents its operating procedure, and discusses its advantages and limitations.

2.2.3.1 Device Description

The OCMA-350 includes a single-beam, fixed-wavelength, NDIR filter-based spectrophotometer. Infrared radiation from a tungsten lamp is transmitted through a cylindrical, quartz cuvette containing a sample extract. The radiation that has passed through the extract enters a detector containing a filter that isolates analytical wavelengths in the 3,400- to 3,500-nm range. The infrared spectrophotometer may be used to analyze soil samples that have been extracted using Horiba's proprietary S-316 extraction solvent, Freon 113, tetrachloroethylene, or carbon tetrachloride. Horiba recommends its proprietary S-316 extraction solvent because it has a higher boiling point (134 °C) and a lower freezing point (-143 °C) than other extraction solvents. In addition, the S-316 extraction solvent is nonflammable, nontoxic, and relatively nonvolatile because of its low vapor pressure.

According to Horiba, the infrared spectrophotometer can measure both aromatic and aliphatic hydrocarbons independent of their carbon range. The spectrophotometer's response to aliphatic hydrocarbons is more sensitive than its response to aromatic hydrocarbons. However, the calibration standards provided by Horiba include aromatic hydrocarbons to compensate for the lower aromatic hydrocarbon response. The spectrophotometer presents results in units selected by the user during calibration, such as mg/kg in soil or absorbance values.

The infrared spectrophotometer has an MDL of 1 mg/kg for TPH and is linear up to 1,000 mg/kg in soil. It can achieve repeatability of ± 2 mg/kg from 0 to 99.9 mg/kg, ± 4 mg/kg from 100 to 200 mg/kg, and ± 10 mg/kg from 201 to 1,000 mg/kg. No information on the spectrophotometer's accuracy is available from Horiba. The OCMA-350 has an operating temperature range of 0 to 40 °C and an operating humidity range of 0 to 90 percent.

Components of the OCMA-350 are listed in Table 2-5. Also listed in the table are additional components available from Horiba, including a Model SR-300 solvent reclaimer to reclaim S-316 extraction solvent and a Model GE-50 ultrasonic mixer to disperse the soil sample in the solvent. Additional components required for measuring TPH in soil that are not available from Horiba are also listed in Table 2-5.

The infrared spectrophotometer weighs 11 pounds and is 7 inches long, 9.8 inches wide, and 11 inches deep. The spectrophotometer can operate using a 110- or 220-volt AC power source. With the addition of a DC to AC converter, the device can be powered by an automobile battery or cigarette lighter. The spectrophotometer is also equipped with a parallel printer port and an RS232-C data port to allow transfer of data to a computer or other data logger. The spectrophotometer keeps a record of the time and date of measurement along with each data set that it records. Spectrophotometer printouts contain the time and date of each measurement along with the concentration or absorbance value, which facilitates recordkeeping.

Table 2-5. OCMA-350 Components

Spectrophotometer and Accessories
- Infrared spectrophotometer
- Proprietary, 10-millimeter, quartz cuvette with cap
- 25-microliter microsyringe
- 10-milliliter syringe
- 10-milliliters of B-heavy oil for preparing span adjustment solution
- 3.15-ampere fuse
- Power supply cable
- Instruction manual and simplified operating instruction sheet

Additional Components Available from Horiba
- S-316 extraction solvent
- Model SR-300 solvent reclaimer
- Model VC-50 ultrasonic mixer

Additional Components Not Available from Horiba
- 40-milliliter vials
- 100 milliliters of isooctane
- 100 milliliters of hexadecane
- 100 milliliters of chlorobenzene
- Anhydrous sodium sulfate
- Stainless-steel spatula
- 11-centimeter-diameter, No. 40 Whatman filter paper
- Glass funnel
- Glass beaker
- Balance

Currently, Horiba does not rent its measurement devices or components. The purchase cost for the infrared spectrophotometer and accessories listed in Table 2-5 is $6,500. Horiba offers the S-316 extraction solvent for $240 for a 1.5-kg bottle and $1,040 for a 7-kg bottle. The Model SR-300 solvent reclaimer is available from Horiba for $1,650. Horiba offers the Model VC-50 ultrasonic mixer for $2,080. During the demonstration, Horiba will use the Model GE-50 ultrasonic mixer, which is no longer available from the developer. According to Horiba, the Model GE-50 and VC-50 ultrasonic mixers are functionally the same. To analyze samples using the OCMA-350, the user must provide the additional components listed in Table 2-5 that are not available from Horiba.

According to Horiba, one person can use the OCMA-350 to perform up to 20 analyses in 1 hour. The device is relatively easy to use. Normal training for using the device is limited to reading the instruction manual and simplified operating instruction sheet. Horiba offers a 1-day training course to discuss operation, maintenance, and service of the OCMA-350, but according to the developer, purchasers of the device rarely choose this option. Horiba also provides technical support over the telephone at no additional charge.

Horiba considers the OCMA-350 to be an innovative TPH field measurement device for soil because previous devices in the OCMA series have been used since the 1970s for measurement of oil in water, but the OCMA-350 allows the user to determine hydrocarbon concentration in soil. The Model SR-300 solvent reclaimer is also considered to be innovative because it recycles the extraction solvent, which is difficult to dispose of, and thus can reduce costs by up to 90 percent.

2.2.3.2 Operating Procedure

Measuring TPH in soil using the OCMA-350 involves the following three steps: (1) extraction, (2) measurement of TPH in the extract, and (3) calculation of the TPH concentration in soil. These steps are described below. Calibration procedures and QC checks required for the OCMA-350 are presented in Chapter 8.

Step 1 - Extraction

1. Measure 5 grams of the soil sample, and place the measured amount in a 40-mL vial.
2. Add 1 gram of anhydrous sodium sulfate to the vial in order to dry the soil. This is done to prevent water damage to the quartz cuvette and clogging of the filter used below. Mix the soil and anhydrous sodium sulfate with a stainless-steel spatula.
3. Add a carefully measured amount of S-316 extraction solvent (nominally 20 mL) to the vial.
4. Cap the vial, and mix the sample for 1 minute. During the demonstration, Horiba will use the Model GE-50 ultrasonic mixer to disperse the soil in the solvent.
5. Place the vial in its upright position, and wait at least 1 minute to allow soil particles to settle.
6. Place an 11-centimeter-diameter, No. 40 Whatman filter paper in a glass funnel.
7. Pour the extract through the filter and funnel into a clean beaker.

Step 2 - Measurement of TPH in Extract

1. Pre-rinse the quartz sample cuvette using 1 to 4 mL of the filtered sample extract.
2. Place about 6 mL of the filtered sample extract in an OCMA-350 quartz cuvette.
3. Insert the cuvette into the infrared spectrophotometer, and press Measure.

Step 3 - Calculation of TPH Concentration in Soil

Because the infrared spectrophotometer can measure the soil TPH concentration in mg/kg on a wet weight basis, no additional calculations are required. However, any variations from the soil sample amount or reagent amount specified in Step 1 or any sample dilution should be factored into a calculation of the TPH concentration.

2.2.3.3 Advantages and Limitations

An advantage of the OCMA-350 is that it is easy to operate, requiring one person with basic wet chemistry skills. In addition, the infrared spectrophotometer can be operated in the field using a 110- or 220-volt AC power source, a gasoline-powered electric generator, or a DC to AC power converter to provide power from a battery.

The OCMA-350 can quantitatively measure all TPH fuel types. According to Horiba, the OCMA-350 can measure both aromatic and aliphatic hydrocarbons independent of their carbon range.

Another advantage of the OCMA-350 is that the proprietary S-316 extraction solvent has desirable characteristics relative to other, conventional solvents. According to Horiba, because of the S-316 extraction solvent's high boiling point (134 °C) and low freezing point (-129 °C), measurements can be made in a wider temperature range than other extraction solvents allow. The S-316 extraction solvent is nonflammable, nontoxic, and relatively nonvolatile.

The OCMA-350 uses a drying agent—anhydrous sodium sulfate as specified in "Methods for Chemical Analysis of Water and Wastes" (MCAWW) Methods 413.2 and 418.1 (EPA 1983)—to remove moisture from soil samples. As a result, no correction associated with solvent dilution is required, which is necessary for wet soil samples. In addition, not all the supplies necessary to analyze samples using the device are available from Horiba; therefore, an OCMA-350 user must obtain some supplies from another source.

2.2.4 PetroFLAG™ Test Kit

The PetroFLAG™ test kit, a quantitative device manufactured by Dexsil® Corporation (Dexsil®), is based on emulsion turbidimetry as discussed in Section 2.1.3. The device has been commercially available since January 1995. This section describes the device, presents its operating procedure, and discusses its advantages and limitations.

2.2.4.1 Device Description

The PetroFLAG™ test kit uses a proprietary, nonpolar organic solvent mixture for extraction that is composed of alcohols, primarily methanol. The device also uses a proprietary developer solution that is polar in nature and that acts as the emulsifying agent. The developer solution also contains water and surfactants that stabilize the emulsion.

According to Dexsil®, the PetroFLAG™ test kit can measure the petroleum products listed in Table 2-6. The device does not distinguish between aromatic and aliphatic hydrocarbons, and it responds to compounds in the C_8 through C_{36} carbon range. MDLs for the device are also listed in Table 2-6 and range from 10 mg/kg for hydraulic fluid to 1,000 mg/kg for weathered gasoline. Based on precision and accuracy tests conducted for diesel and motor oil, the precision and accuracy of the device are estimated to be ± 10 and ± 20 percent, respectively. The device's response factors are based on mineral oil. These response factors are listed in Table 2-6 and range from 2 for weathered gasoline to 10 for transformer oil, indicating that the device is more sensitive to transformer oil than weathered gasoline. If no information is available regarding the type of contamination in a sample, Dexsil® recommends using an average response factor of 5. For accurate measurement of TPH in soil, Dexsil® recommends using its HYDROSCOUT® meter to measure the moisture content of samples so that an appropriate solvent dilution correction may be applied to the TPH concentration.

Table 2-6. PetroFLAG™ Test Kit Method Detection Limits and Response Factors for Petroleum Products Measured

Petroleum Product	Method Detection Limit (milligram per kilogram)	Response Factor
Mineral oil	15	10
Transformer oil	15	10
Grease	15	9
Hydraulic fluid	10	8
Transmission fluid	19	8
Motor oil	19	7
No. 2 fuel oil	25	7
No. 6 fuel oil	18	6
Diesel	13	5
Gear oil	22	5
Low-aromatic diesel	27	4
Pennsylvania crude oil	20	4
Kerosene	28	4
Jet A fuel	27	4
Weathered gasoline	1,000	2

For optimum performance, the PetroFLAG™ test kit should be used in environments with a temperature range of about 4 to 45 °C. The turbidimeter is equipped with a built-in temperature sensor. This sensor measures the ambient temperature while TPH measurements are being made. The turbidimeter then uses the sensor's temperature readings to correct for measurement fluctuations caused by temperature variations. However, the temperature corrections are valid only for ambient temperatures within 10 °C of the calibration temperature. Therefore, if the ambient temperature deviates from the calibration temperature by more than 10 °C, an error condition results, and the turbidimeter has to be recalibrated.

Table 2-7 lists the components of the PetroFLAG™ test kit. The device's components are packaged in the carrying case, which is about 19 inches wide, 14.25 inches long, and 5.5 inches deep. All device reagents are premeasured and sealed in glass ampules; additional reagents can be ordered in multiples of the supply requirements for 10 analyses. The turbidimeter weighs 0.6 pound and is 5.75 inches long, 3.5 inches wide, and 2 inches deep. The complete device weighs less than 10 pounds. The turbidimeter operates on one 9-volt battery, which can last for about 18,000 readings.

Table 2-7. PetroFLAG™ Test Kit Components

- Battery-powered, hand-held, digital turbidimeter (9-volt battery included)
- Battery-powered balance (batteries included)
- Battery-powered timer (batteries included)
- 10 plastic screw-capped, polypropylene tubes
- 10 filter-syringe assemblies
- 2 calibration standards
- 10 breaktop vials of extraction solvent
- 10 glass vials containing developer solution
- Test procedure manual
- Material Safety Data Sheets for all chemicals in kit
- Carrying case

The PetroFLAG™ test kit can be purchased for \$695 and includes enough supplies to perform 10 soil analyses. Additional reagents can be purchased for \$10 to \$15 per analysis, depending on the total quantity purchased. The HYDROSCOUT® meter may be purchased for an additional \$395. Neither the device components nor the HYDROSCOUT® meter is available for rental from Dexsil®.

The PetroFLAG™ test kit enables the user to perform up to 16 analyses in 1 hour. Only one person is required to perform analyses using the device. The device is designed to be used by those with basic wet chemistry skills. A free training videotape for the device is available from Dexsil®, which also provides technical support over the telephone at no additional cost.

According to the developer, the PetroFLAG™ test kit is innovative because it responds to a broad range of PHCs regardless of the source or state of weathering. Also, the proprietary solvent mixture used in the device was selected to provide consistent extraction efficiencies for a range of soil types and conditions, including moisture content and ionic strength.

2.2.4.2 Operating Procedure

Measuring TPH in soil using the PetroFLAG™ test kit involves the following three steps: (1) extraction, (2) filtration and emulsion development, and (3) turbidity measurement and calculation of the TPH concentration. Calibration procedures and QC checks required for the device are presented in Chapter 8.

Step 1 - Extraction

1. Measure 10 grams (± 0.1 gram) of the soil sample, and place the measured amount in a polypropylene tube. To extend the quantification range for samples containing EDRO, either a 1-gram sample can be analyzed or a 10-gram sample can be analyzed using the High Range procedure.
2. Pour one breaktop vial of extraction solvent into the tube, and cap the tube.
3. Set the timer to 5 minutes, and shake the tube for 15 seconds or until the sample is fully wet.
4. Continue to shake the tube intermittently for a minimum of 4 minutes, then let it stand for 1 minute. Samples containing EDRO contaminants can be extracted for up to 1 hour without significant loss.

Step 2 - Filtration and Emulsion Development

1. Remove the plunger from the filter-syringe assembly, and verify that the 0.2-µm filter disk is firmly attached to the syringe barrel.
2. Remove the cap from a 6-mL, glass vial containing developer solution.
3. Remove the cap from the polypropylene tube containing the soil and extraction solvent, and pour the extract supernatant into the syringe barrel. Do not allow soil to enter the syringe, as too much soil might plug the filter.
4. Discard the first few drops of the extract from the filter into a waste container, and then filter the extract into the vial containing developer solution. Add the extract into the vial one drop at a time until the meniscus just enters the vial neck.
5. Cap the vial, and shake it for 10 seconds. Then set the timer to 10 minutes, and let the vial stand. During the 10-minute period, an emulsion associated with the hydrocarbons in the extract is developed. Do not let the vial stand longer than 20 minutes before placing it in the turbidimeter, as this might result in a low bias.

Step 3 - Turbidity Measurement and Calculation of TPH Concentration

1. After the 10-minute development period, place the vial in the turbidimeter, which should have been calibrated using a blank and a single calibration standard.

2. If the primary contaminant in a sample is known or suspected, set the appropriate response factor shown in Table 2-6 on the turbidimeter. If not, use a response factor of 5 as a default value.

3. When the vial is placed in the turbidimeter, a beam of light at a wavelength of 585 nm passes through the vial, and the intensity of the light scattered at an angle of 90 degrees to the initial beam of light is measured. The TPH reading of the sample extract is displayed on the turbidimeter as mg/kg TPH in soil on a wet weight basis.

4. In certain circumstances, the water content of the soil and analyte carbon number range may require a correction factor to account for soil-water content. To correct the TPH concentration in mg/kg on a wet weight basis for solvent dilution associated with the moisture content of a given soil sample, use Equation 2-8.

$$\begin{matrix}\text{mg/kg TPH after correcting} \\ \text{for solvent dilution}\end{matrix} = \begin{matrix}\text{mg/kg TPH before correcting} \\ \text{for solvent dilution}\end{matrix} \times \frac{100 + \text{Percent Moisture}}{100} \qquad (2\text{-}8)$$

2.2.4.3 Advantages and Limitations

An advantage of the PetroFLAG™ test kit is that it is easy to operate, requiring one person with basic wet chemistry skills. In addition, the turbidimeter operates using a 9-volt battery; therefore, an AC power source is not required in the field.

According to Dexsil®, another advantage of the PetroFLAG™ test kit is that a soil moisture content of up to 25 percent does not interfere with soil analysis. Therefore, no chemicals need to be added to a soil sample in order to reduce its moisture content. In addition, the turbidimeter is able to correct for measurement fluctuations caused by temperature variations.

A limitation of the PetroFLAG™ test kit is that it cannot quantitatively measure TPH in the C_6 and C_7, and the C_{37} through C_{40} carbon ranges. According to Dexsil®, the device can measure both aromatic and aliphatic hydrocarbons in the C_8 through C_{36} carbon range. In addition, the device is less sensitive to weathered gasoline than to other petroleum products.

2.2.5 Luminoscope

The Luminoscope was developed by the Oak Ridge National Laboratory under the sponsorship of the U.S. Department of Energy and the EPA in collaboration with Environmental Systems Corporation (ESC). The device produces quantitative results and is based on ultraviolet fluorescence spectroscopy as discussed in Section 2.1.4. The Luminoscope has been commercially available since 1997. This section describes the device, presents its operating procedure, and discusses its advantages and limitations.

2.2.5.1 Device Description

The Luminoscope is based on ultraviolet fluorescence spectroscopy and uses excitation and emission monochromators. The components of the Luminoscope are structured to maintain a constant wavelength interval (*delta lambda*) between the excitation and emission monochromators. This modification of classical fluorescence technology is called synchronous fluorescence and takes advantage of the overlap between the excitation and emission spectra for a sample to produce more sharply defined spectral peaks. According to ESC, this modification maximizes the Luminoscope's capability to differentiate among various aromatic hydrocarbons that may be present in a sample extract. For TPH analyses of soil samples, a *delta lambda* of 18 nm is typically used.

The Luminoscope uses a high-pressure xenon lamp as its light source. The xenon lamp emits light of wavelengths ranging from 200 to 650 nm. The Luminoscope has a spherical, concave mirror that collects back-emitted light and directs it toward the excitation monochromator. A laptop computer with Grams/32 software developed by Galactic Industries is used to control the Luminoscope and to manage data collected by the device. The Luminoscope allows the user to generate emission spectra ranging from 200 to 650 nm.

Several solvents can be used to complete extraction of soil samples for Luminoscope analysis, including methanol, CH_2Cl_2, and cyclohexane. According to ESC, the choice of solvent depends on (1) the carbon range of the contaminant of concern and (2) the solvent that would typically be used to analyze for the contaminant under conventional, laboratory methods. For the demonstration, methanol will be used to extract all soil samples.

The Luminoscope can be used to measure concentrations of PHCs in soil. Because aromatic hydrocarbons fluoresce when they are excited by ultraviolet light, the Luminoscope responds to their concentrations in sample extracts. Although aliphatic hydrocarbons do not fluoresce, off-site laboratory results for TPH analysis of a subset of samples can be used to develop a site-specific calibration curve of luminescence intensity versus TPH concentration. Once the Luminoscope has been used to measure the luminescence intensities of the remaining sample extracts, the calibration curve can be used to calculate the concentrations of TPH present. The Luminoscope can achieve an MDL of 50 micrograms (µg) per kg for TPH. No information is currently available from ESC regarding the accuracy and precision of the device. Interpretation of the spectra generated by the Luminoscope allows the user to report data as GRO and EDRO concentrations based on the carbon range selected. Additional extraction or analysis of the sample extract is not required to report data as GRO and EDRO concentrations.

ESC does not specify an operating temperature range for the Luminoscope; however, the device has been successfully operated at ambient temperatures ranging from -7 to 38 °C. ESC also does not specify a particular storage temperature for the Luminoscope. In addition, according to ESC, humidity levels do not appear to affect the operation of the Luminoscope.

The Luminoscope is 12 inches long, 16 inches wide, and 16 inches deep; weighs 34 pounds; and comes with a carrying case. The Luminoscope can be operated using a standard automobile battery; an appropriate battery converter may be purchased from ESC. To analyze samples using the Luminoscope, a user may also purchase quartz cuvettes and a sampling kit from ESC. The sampling kit contains enough vials, pipettes, test tubes, and filters to analyze 25 samples along with 1 L of extraction solvent. Grams/32, the computer software used to control the Luminoscope and manage its data, is purchased separately. Additional equipment required to operate the Luminoscope that is not provided by ESC includes a balance, centrifuge, test tube shaker, and laptop computer.

According to ESC, about 40 samples can be analyzed by one on-site technician using the Luminoscope over an 8-hour period. Although it is not necessary for operation of the device, ESC recommends 3 days of training in fluorescence theory, device operation, sample preparation, and data display. The cost of this training is included in the purchase cost of the Luminoscope. ESC also provides technical support over the telephone at no additional cost.

The Luminoscope's purchase cost is $26,500. Quartz cuvettes cost an additional $110 each. Depending on a user's requirements, ESC can prepare a sampling kit that contains enough support equipment to analyze 25 samples; such a kit costs about $115. To analyze samples using the Luminoscope, the user must also provide a balance, centrifuge, test tube shaker, and laptop computer that are not available for purchase from ESC. The software used to operate the Luminoscope, Grams/32, costs an additional $2,400. The Luminoscope and Grams/32 software may also be rented from ESC for $600 per day. This cost covers a technician to operate the device and enough support equipment to analyze about 40 samples. The Luminoscope purchase and rental costs do not include travel and per diem costs for the training instructor and technician, respectively.

According to ESC, the Luminoscope is innovative when compared with conventional ultraviolet fluorescence spectroscopes because the device uses synchronous fluorescence to take advantage of the overlap between the absorption and emission spectra for a sample extract to generate more sharply defined spectral peaks. This feature enhances the Luminoscope's capability to differentiate among various aromatic hydrocarbons that may be present in a sample extract. The Luminoscope is also able to separately report TPH concentrations for GRO and EDRO without additional extraction or analysis.

2.2.5.2 Operating Procedure

Measuring TPH in soil using the Luminoscope involves the following two steps: (1) extraction and (2) concentration measurement. These steps are described below. Calibration procedures and QC checks required for the Luminoscope are presented in Chapter 8.

Step 1 - Extraction

1. Measure 2 grams of soil sample, and place the measured amount in a test tube.
2. Add 10 mL of the appropriate solvent to the test tube.
3. Manually shake the test tube until, based on visual observation, more than 90 percent of the sample is suspended in the solvent. If multiple test tubes need to be shaken, use a test tube shaker.
4. Spin the test tube in a centrifuge to separate the soil.
5. Pour the extract into a second test tube.
6. If any soil particles are visible, use a syringe with a detachable filter to transfer the extract to a cuvette. If soil particles are not visible, transfer the extract directly to a cuvette.

Step 2 - Concentration Measurement

1. Analyze the sample over a predetermined wavelength range. For TPH analyses, a wavelength range of 250 to 400 nm is typically used.
2. Use the Grams/32 software to integrate the area under the peaks of the sample spectrum. To report results as a TPH concentration, integrate the area under the curve from about 275 to 340 nm. To report results as GRO and EDRO concentrations, integrate the areas under the curve from about 275 to 300 nm and 300 to 340 nm, respectively.
3. Use a calibration curve to convert area counts reported by the Luminoscope into a TPH, GRO, or EDRO concentration.

2.2.5.3 Advantages and Limitations

An advantage of the Luminoscope is that it is easy to operate, requiring one person with basic analytical chemistry skills. In addition, the device is operated using a DC power source such as an automobile cigarette lighter; therefore, an AC power source is not required in the field.

Another advantage of the Luminoscope is that it can quantitatively measure all fuel types in the µg/kg range that contain aromatic hydrocarbons. In addition, interpretation of the spectra generated by the Luminoscope allows the user to report data as GRO and EDRO concentrations without additional sample extraction or analysis.

According to ESC, an advantage of the Luminoscope over conventional ultraviolet fluorescence spectroscopes is that the device uses synchronous fluorescence to take advantage of the overlap between absorption and emission spectra for a sample extract. This feature maximizes the Luminoscope's ability to differentiate among various aromatic hydrocarbons that may be present in a sample extract.

A limitation of the Luminoscope is that it requires calibration and calculation of TPH concentrations using site-specific soil sample concentrations measured by an off-site laboratory during a presampling or postsampling effort. The presampling effort allows the Luminoscope user to determine on-site TPH concentrations while in the field but increases the mobilization costs for a project. The postsampling effort does not increase mobilization costs, but the Luminoscope user cannot determine TPH concentrations in the field; only luminescence intensity readings can be taken. In addition, a laptop computer must be used to analyze samples and to report data generated using the Luminoscope.

2.2.6 UVF-3100A

The UVF-3100A, a quantitative device developed by siteLAB® Corporation (siteLAB®), is based on ultraviolet fluorescence spectroscopy as discussed in Section 2.1.4. The UVF-3100A is manufactured for siteLAB® by Turner Designs and has been modified and distributed for environmental use by siteLAB®. The UVF-3100A has been commercially available since October 1998. This section describes the device, presents its operating procedure, and discusses its advantages and limitations.

2.2.6.1 Device Description

The siteLAB® portable fluorometer is fitted with excitation and emission filters that are appropriate for TPH analysis of soil samples. In addition, siteLAB® has developed and provides software that can be used to manage and present data generated by the UVF-3100A.

The UVF-3100A uses a mercury vapor lamp with a predominant emission of 254-nm wavelength as its light source. Light from the lamp is directed through an excitation filter of 254 nm before it irradiates a sample extract held in a quartz cuvette. Depending on the analysis being conducted, the fluorometer is fitted with an appropriate emission filter that corresponds to the wavelength at which the sample extract is expected to fluoresce. For GRO, an emission filter with a bandwidth of 280 nm is used, and for EDRO, an emission filter with a bandwidth between 300 and 400 nm is used. These filters are used because GRO and EDRO aromatic hydrocarbons fluoresce within these wavelength ranges. Both the excitation and emission filters are fitted into sleeves that fit into ports in the fluorometer. To analyze soil samples using the UVF-3100A, methanol is used as the extraction solvent.

The UVF-3100A can be used to measure petroleum fuel products. Because aromatic hydrocarbons fluoresce when they are excited by ultraviolet light, the fluorometer can measure their concentrations in sample extracts. Aliphatic hydrocarbons do not fluoresce; therefore, the fluorometer cannot quantify aliphatic hydrocarbon concentrations. However, siteLAB® software can estimate aliphatic hydrocarbon fractions and individual PAH or BTEX concentrations by generating response factors based on aromatic and aliphatic hydrocarbon ratios for two to five site-specific samples that are sent to an off-site laboratory for GC analysis. siteLAB® has determined MDLs for the UVF-3100A by analyzing sand blanks. The resulting MDLs for petroleum fuel products in soil range from 0.08 to 6.9 mg/kg and are listed in Table 2-8.

Table 2-8. UVF-3100A Method Detection Limits

Petroleum Fuel Product	Method Detection Limit for Soil (milligram per kilogram)
No. 2 fuel oil	0.50
No. 4 fuel oil	0.20
No. 6 fuel oil	0.08
Diesel	0.60
50 percent weathered diesel	0.34
Gasoline	6.9
50 percent weathered gasoline	3.9
Motor oil	1.0
Extended diesel range organics-polynuclear aromatic hydrocarbons	0.04
Gasoline range organics-benzene, toluene, ethylbenzene, and xylenes	0.10

The operating temperature range for the UVF-3100A is 0 to 38 °C. The lowest operating temperature is based on the possibility of the fluorometer's quartz crystal display freezing. According to siteLAB®, the UVF-3100A does not have a storage temperature or operating humidity restriction.

The siteLAB® UVF-3100A Extraction System includes the fluorometer and support equipment listed in Table 2-9. siteLAB® separately provides a 20 Sample Extraction Kit that contains the equipment listed in Table 2-9. Calibration kits for a variety of TPH standards are also available from siteLAB®. Each calibration kit includes five linear calibration standards and one reference standard.

Table 2-9. UVF-3100A Components

UVF-3100A Extraction System

- Fluorometer
- Alternating current power adapter
- Direct current power converter
- RS-232 cable
- Quartz cuvettes (2)
- Timer (batteries included)
- Certified clean sand (500 grams)
- High-performance liquid chromatography-grade methanol (1 liter)
- Solvent dispenser bottle
- 5-millimeter volumetric flask
- 10-millimeter volumetric flask
- Tissue wipes
- 2 stainless-steel spatulas
- Adjustable pipette
- Test tube rack
- Battery-powered balance (9-volt battery included)
- Markers
- Shaker/mixer can
- siteLAB® software
- Portable field case
- Instruction manual and simplified instruction sheet

20 Sample Extraction Kit

- 20 extraction jars
- 20 weighing boats
- 20 pipette tips
- 20 syringes with detachable filters
- 40 10-milliliter test tubes
- 40 stainless-steel mixing balls

The UVF-3100A Extraction System and 20 Sample Extraction Kit fit in a portable field case that is 12 inches long, 36 inches wide, and 24 inches deep and weighs 55 pounds. The UVF-3100A may be operated using a using a DC power source such as an automobile cigarette lighter; therefore, an AC power source is not required in the field.

Connecting the UVF-3100A to a computer allows downloading and manipulation of calibration and sample data using siteLAB® software, although a computer connection is not needed to collect or read data. To connect the device to a computer, an RS-232 cable connection is used. At a minimum, the computer must support the Microsoft

Windows 95 operating system and have Microsoft Excel software installed. If a computer that does not meet these requirements is used, a special computer program and technical support can be provided by siteLAB® to assist the user in manipulating data.

Using the UVF-3100A, 40 to 50 samples can be analyzed by one on-site technician in an 8-hour period. Each sample takes about 5 to 10 minutes to process; analysis time is 5 to 10 seconds. Although it is not required for operation of the UVF-3100A, siteLAB® recommends 0.5 to 1 day of training by a siteLAB® instructor in device operation and data management. The cost of this training is included in the purchase cost of the UVF-3100A. siteLAB® also provides technical support over the telephone at no additional cost.

The UVF-3100A Extraction System has a purchase price of $11,999. siteLAB® also rents the UVF-3100A at a rate of $1,250 per day. The rental cost covers a technician and all the support equipment required to operate the device. siteLAB® does not rent the UVF-3100A without a technician. The purchase and rental costs do not include travel and per diem costs for an instructor or technician. In the New England region, siteLAB® rents the device at a rate of $150 per hour for the UVF-3100A and a technician; a 4-hour rental minimum is required to obtain this rate, and travel costs for the technician may also be applied. The 20 Sample Extraction Kit costs $299. In addition, calibration kits for a variety of TPH standards cost $199 each. siteLAB® provides software upgrades at no cost.

siteLAB® considers the UVF-3100A to be innovative because the device adapts a laboratory technology for field use. The device is able to separately report aromatic hydrocarbon concentrations for GRO and EDRO analyses.

2.2.6.2 Operating Procedure

Measuring TPH in soil using the UVF-3100A involves the following two steps: (1) extraction and (2) concentration measurement. The UVF-3100A can measure both GRO and EDRO components of sample extracts. Both analyses may be performed on one sample extract; however, the emission filter must be replaced and the device must be recalibrated between the GRO and EDRO analyses. The two-step operating procedure for the device is described below. Calibration procedures and QC checks required for the UVF-3100A are presented in Chapter 8.

Step 1 - Extraction

1. Measure 10 grams of soil sample, and place the measured amount in a high-density polypropylene sample extraction jar.
2. Add two steel mixing balls to the jar. For clay soil, add three mixing balls to the jar.

3. Add 10 mL of methanol to the jar, and cap the jar.

4. Manually shake the jar for 2 minutes. If multiple jars must be shaken, use the shaker/mixer can, which can hold up to five jars.

5. If soil particles are visible in the jar, allow the soil to settle for 1 to 2 minutes.

6. Use a syringe with a detachable filter to transfer 3 mL of filtered extract into a test tube.

7. Cap the test tube, and let it stand until concentration measurement is performed.

Step 2 - Concentration Measurement

1. Decant the extract from the test tube into a quartz cuvette. Place the cuvette in the sample chamber of the UVF-3100A.

2. The device displays concentrations in ng/L, µg/L, mg/L, parts per trillion, parts per billion (ppb), ppm, or fluorescence units. Device readings may be downloaded to a computer and compiled with other data as part of a spreadsheet or may be manually recorded from the UVF-3100A display.

3. The concentration measured represents only the aromatic hydrocarbons present in the sample extract. The aliphatic hydrocarbon concentration may be estimated using the UVF-3100A software.

2.2.6.3 Advantages and Limitations

An advantage of the UVF-3100A is that it is easy to operate, requiring one person with basic analytical chemistry skills. In addition, the device is operated using a DC power source such as an automobile cigarette lighter; therefore, an AC power source is not required in the field. After sample extraction, analysis takes less than 10 seconds.

Another advantage of the UVF-3100A is that it can quantitatively measure all TPH fuel types that contain aromatic hydrocarbons. The device does not measure aliphatic hydrocarbons. However, the device's software can estimate aliphatic hydrocarbon fractions based on response factors developed using off-site laboratory analytical results for site-specific samples. The UVF-3100A can also measure both GRO and EDRO compounds of all fuel types regardless of weathering. However, between GRO and EDRO analyses, the emission filter must be changed and the device must be recalibrated; then the sample extract must be reanalyzed. The UVF-3100A uses reusable, certified standards for calibration.

A limitation of the UVF-3100A is that response factors must be developed to measure aliphatic hydrocarbon concentrations. These response factors are developed using site-specific soil sample concentrations measured by an off-site laboratory during a presampling or postsampling effort. A presampling effort allows the UVF-3100A user to determine TPH concentrations while in the field but increases the mobilization costs of a project. A postsampling effort does not increase the mobilization costs, but the UVF-3100A user cannot determine aliphatic hydrocarbon concentrations in the field; only aromatic concentration readings can be taken.

2.2.7 EnSys Petro Test System

The EnSys Petro Test System, a semiquantitative device manufactured by Strategic Diagnostics, Inc. (SDI), is based on a combination of immunoassay, specifically ELISA, and colorimetry as discussed in Section 2.1.5. The device has been commercially available since 1992. The device conforms to SW-846 Method 4030 for screening PHCs using immunoassay detection (EPA 1996). This section describes the device, presents its operating procedure, and discusses its advantages and limitations.

2.2.7.1 Device Description

The EnSys Petro Test System includes the SDI Sample Extraction Kit, the EnSys Petro 12T Soil Test Kit, and the EnSys/EnviroGard® Common Accessory Kit. The EnSys Petro Test System includes antibody-coated test tubes containing monoclonal antibodies, which are produced using m-xylene as the antigen. The enzyme conjugate used to produce color is composed of m-xylene as the target compound and horseradish peroxidase as the enzyme. The washing step is performed with a dilute detergent solution. Color development is achieved using hydrogen peroxide as the substrate and tetramethylbenzidine (TMB) as the chromogen. The stop solution added to terminate color development is 0.5 percent sulfuric acid. A differential spectrophotometer that emits light in the visible range of the electromagnetic spectrum at 450-nm wavelength is used to measure the absorbance of the sample extract and of a reference standard containing 3 mg/L m-xylene during color measurement. The concentration of PHCs in the sample extract is then determined by comparing the absorbance readings associated with the sample extract and reference standard.

According to SDI, the EnSys Petro Test System can be used to measure the following petroleum products in soil: gasoline, diesel, Jet A fuel, JP-4, kerosene, No. 2 fuel oil , No. 6 fuel oil, and mineral spirits. The device is not designed to respond to every petroleum product component that may be present in a sample extract. The monoclonal antibodies used in the device are specific to a subset of petroleum product components. According to SDI, the subset includes a large portion of the aromatic hydrocarbons and a few aliphatic hydrocarbons in the C_6 through C_{22} carbon range.

The MDLs of the EnSys Petro Test System for a variety of aromatic and aliphatic hydrocarbons are presented in Table 2-10. Except for C_6H_6, which has an MDL of 400 mg/kg, the aromatic hydrocarbons listed in Table 2-10 have MDLs that are less than 40 mg/kg, indicating a high degree of selectivity. A few aliphatic hydrocarbons, such as 2-methylpentane and isooctane, also have low MDLs. Table 2-10 also presents the MDLs for various petroleum fuel products. These MDLs generally range from 10 mg/kg (gasoline) to 40 mg/kg (mineral spirits). The MDLs for machine oil, brake fluid, motor oil, grease, and mineral oil are all greater than 1,000 mg/kg, indicating that the EnSys Petro Test System is not as sensitive to these formulated petroleum products. Additional information on EnSys Petro Test System performance is available in SW-846 Method 4030 (EPA 1996).

The operating temperature range for the EnSys Petro 12T Soil Test Kit is 16 to 38 °C. According to SDI, the test kit does not have an operating humidity restriction. The test kit should be stored at less than 27 °C when not in use. The shelf life of the test kit is typically 1 year after its date of manufacture; lot-specific expiration date information is provided on the test kit packaging. The chromogen and substrate solutions should not be exposed to direct sunlight during test kit operation or storage.

The components of the EnSys Petro Test System are listed in Table 2-11. The SDI Sample Extraction Kit contains enough components to perform 12 soil sample extractions. The EnSys Petro 12T Soil Test Kit contains enough components to process up to 12 samples (for example, 10 soil sample extracts and duplicate calibration standards). The components of the EnSys/EnviroGard® Common Accessory Kit are multi-use items and do not require frequent replacement. All components of the accessory kit are housed in a hard-plastic carrying case to prevent component damage during kit transport. The components of the extraction kit and soil test kit are shipped in cardboard boxes.

The spectrophotometer (Artel DP™ Differential Photometer) included in the EnSys/EnviroGard® Common Accessory Kit is designed to provide an immediate, direct comparison of the absorbance of two liquid samples (for example, a soil sample extract and a reference standard) by means of a digital display; the display indicates the difference in absorbance between the two samples. The spectrophotometer emits light at a factory-preset wavelength of 450 nm. This spectrophotometer is 3.4 inches long, 5.3 inches wide, and 2.6 inches tall and weighs 0.8 pound. The power supply for the spectrophotometer consists of four rechargeable nickel-cadmium batteries; the spectrophotometer cannot be operated using an AC power source. The batteries require 8 to 10 hours to achieve a full recharge after discharge and last for about 500 readings between recharges. The operating temperature range for the spectrophotometer is 10 to 40 °C. According to SDI, the spectrophotometer does not have an operating humidity restriction. The spectrophotometer should be stored at a temperature between -20 and 65 °F when not in use.

Table 2-10. EnSys Petro Test System Method Detection Limits

Compound or Substance	Method Detection Limit (milligram per kilogram)[a]
Petroleum fuel product	
Gasoline	10
Diesel	15
Jet A fuel	15
JP-4	15
Kerosene	15
No. 2 fuel oil	15
No. 6 fuel oil	25
Formulated petroleum product	
Mineral spirits	40
Machine oil	>1,000[b]
Brake fluid	>1,000[b]
Unused motor oil	>1,000[b]
Grease	>1,000[b]
Mineral oil	>1,000[b]
Aromatic hydrocarbon	
Benzene	400
Toluene	40
Ethylbenzene	7
o-Xylene	8.5
m-Xylene	8
p-Xylene	4.5
Styrene	7
1,2-Dichlorobenzene	2.5
Hexachlorobenzene	10
Naphthalene	0.8
Acenaphthalene	0.5
Biphenyl	10
Creosote	1.5
Aliphatic hydrocarbon	
2-Methylpentane	35
Hexanes (mixed)	65
Heptane	130
Isooctane	8.5
Undecane	>1,000[b]
Trichloroethylene	>1,000[b]
Methyl-tert-butyl-ether	>1,000[b]

Notes:

> = Greater than

[a] Minimum soil concentration necessary to obtain a positive result more than 95 percent of the time

[b] Highest concentration tested; positive result not obtained at this concentration

Source: SDI 1999

The purchase cost for the SDI Sample Extraction Kit designed for the EnSys Petro Test System is $120. The purchase cost for the EnSys Petro 12T Soil Test Kit is $366. The EnSys/EnviroGard® Common Accessory Kit can be purchased for $1,999 or rented for $175 per day, $450 per week, or $800 per month. Individual kit components may also be purchased separately from SDI.

According to SDI, 12 samples (including soil sample extracts and reference standards) can be analyzed as one batch by one person using the EnSys Petro Test System in approximately 45 minutes. The device is easy to operate and

Table 2-11. EnSys Petro Test System Components

SDI Sample Extraction Kit

- 12 extraction jars with screw caps (each jar contains 3 stainless-steel mixing balls)
- 12 filter units (tops and bottoms)
- 12 ampule crackers
- 12 dilution ampules (for EnSys Petro Test System only)
- 12 wooden spatulas
- 12 weigh canoes
- 12 disposable transfer pipettes
- 12 ampules containing 100 percent methanol solvent
- User guide

EnSys Petro 12T Soil Test Kit

- 12 monoclonal antibody-coated tubes
- 16 conjugate tubes
- 1 80-milliliter bottle of phosphate buffer solution
- 1 15-milliliter bottle of chromogen (tetramethylbenzidine)
- 1 15-milliliter bottle of substrate (hydrogen peroxide)
- 1 15-milliliter bottle of stop solution (0.5 percent sulfuric acid)
- 3 15-milliliter vials of Petro standard (3 milligrams per liter m-xylene)
- 24 Microman® positive displacement pipettor tips
- 3 5-milliliter Combitips® for the repeater pipettor
- 1 12.5-milliliter Combitip® for the repeater pipettor
- 12 ampule crackers
- 3 amber vials (for storage of remnant solution from cracked ampules)
- 3 disposable transfer pipettes
- 1 480-milliliter bottle of dilute detergent solution
- 2 wash bottles
- User guide

EnSys/EnviroGard® Common Accessory Kit

- 1 battery-powered Artel DP™ Differential Photometer
- 1 battery-powered ACCULAB® digital balance
- 1 battery-powered digital timer
- 1 Gilson M-25 Microman® positive displacement pipettor
- 1 Eppendorf™ repeater pipettor
- 1 5-milliliter Combitip® for the repeater pipettor
- 1 12.5-milliliter Combitip® for the repeater pipettor
- 1 50-milliliter Combitip® for the repeater pipettor
- 1 wash bottle
- 1 foam workstation
- User guide
- Carrying case

is designed to be used by those with basic wet chemistry skills. In addition to the user guide that accompanies each kit of the device, SDI provides technical support over the telephone at no additional cost. SDI also offers a 1-day, on-site training program for $999, which covers instructor travel, instructor per diem, and one EnSys Petro Test System for training purposes.

According to SDI, the EnSys Petro Test System is innovative because the device uses biologically engineered antibodies to measure PHCs in soil. The device measures most aromatic hydrocarbons and some aliphatic hydrocarbons in the C_6 through C_{22} carbon range.

2.2.7.2 Operating Procedure

Measuring TPH in soil using the EnSys Petro Test System involves the following five steps: (1) extraction, (2) sample and standard preparation, (3) washing, (4) color development, and (5) color measurement and estimation of TPH concentration. These steps are described below. Calibration procedures and QC checks required for the device are presented in Chapter 8.

Step 1 - Extraction

1. Measure 10 ± 0.1 grams of soil sample, and placed the measured amount in an extraction jar.
2. Pour the contents of one solvent ampule (containing 20 mL of 100 percent methanol) into the jar, and cap the jar.
3. Shake the jar vigorously for 1 minute.
4. Allow the soil to settle for 1 minute or until a liquid solvent layer (supernatant) is observed above the soil. If the supernatant is not observed within 15 minutes, decreasing the soil to solvent ratio may be required. Also, when a clay soil sample is being extracted, the soil might absorb all the methanol, leaving little or no excess liquid to filter. Under these circumstances, add another 10 mL of methanol to the jar, and shake the jar vigorously for an additional 1 to 2 minutes. Factor this dilution into the calculations for interpreting the photometric results during Step 5.
5. Transfer at least half a bulb pipette (about 1.5 mL) of supernatant into the bottom portion of the filter unit; do not transfer more than one full bulb of supernatant.
6. Press the top portion of the filter unit (the piece with the cap and filter) into the bottom portion (containing the sample extract) until the unit snaps together or until most of the liquid has passed upward through the filter.

Step 2 - Sample and Standard Preparation

1. Add 1.25 mL of phosphate buffer solution to each conjugate tube. The phosphate buffer solution (1) hydrates the enzyme conjugate (which is freeze-dried) and (2) helps maintain the pH in the antibody-coated tube (thus preventing the antibodies from denaturing) when the enzyme conjugate is added to the antibody-coated tube.
2. Transfer 60 µL of sample extract from the filter unit to each of the conjugate tubes designated for sample extract.
3. Add 60 µL of standard (3-mg/L m-xylene) to each of the two conjugate tubes designated for the reference standard.
4. Fit an antibody-coated tube on top of each conjugate tube.

5. Set the timer for 10 minutes.

6. Invert all the connected tube pairs so that the liquid is poured into the antibody-coated tubes.

7. Invert all the tube pairs several more times during the 10-minute period to mix the solutions. Make sure that each tube pair is positioned with the antibody-coated tube on the bottom after each inversion.

8. Disconnect the conjugate tubes, and discard them.

Step 3 - Washing

1. Vigorously shake out each antibody-coated tube's contents into a sink or suitable container.

2. Fill each antibody-coated tube to overflowing with a vigorous stream of dilute detergent solution. Then vigorously shake out the tube contents into a sink or suitable container. Repeat this washing activity three more times. After the final wash, remove as much water as possible by tapping the inverted tube on absorbent paper.

Step 4 - Color Development

1. Add 200 µL of chromogen (TMB) to each antibody-coated tube.

2. Set the timer for 2.5 minutes.

3. Add 200 µL of substrate (hydrogen peroxide) to each antibody-coated tube. Addition of the substrate turns the reaction mixture blue.

4. At the end of 2.5 minutes, add 200 µL of stop solution (0.5 percent sulfuric acid) to each antibody-coated tube. Addition of the stop solution turns the reaction mixture yellow.

Step 5 - Color Measurement and Estimation of TPH Concentration

1. Place the two tubes that contain the reference standard in the differential photometer.

2. Switch the reference standard tubes until the photometer reading is negative or zero. If the reading is less than -0.30, the results are outside QC limits. In this case, re-extract the soil samples, and repeat Steps 2 through 4.

3. Remove and discard the reference standard tube in the right well of the photometer; the reference standard tube in the left well contains the lower standard concentration, which will result in a conservative estimate of the sample extract concentration.

4. Place the sample extract tube in the right well of the photometer, and record the absorbance reading. If the photometer reading is negative or zero, the TPH concentration in the sample extract is equal to or greater than that in the reference standard (3-mg/L m-xylene). If the photometer reading is positive, the TPH concentration in the sample extract is less than that in the reference standard (3-mg/L m-xylene).

5. Convert the concentration of the reference standard to petroleum fuel product equivalents. For example, the 3-mg/L m-xylene standard is equivalent to 8 mg/kg m-xylene in soil on a wet weight basis. According to Table 2-10, the gasoline fuel product equivalent to 8 mg/kg m-xylene in soil on a wet weight basis is 10 mg/kg gasoline in soil on a wet weight basis. Similarly, for diesel, the equivalent concentration is 15 mg/kg in soil on a wet weight basis. The petroleum fuel product equivalent is selected based on the petroleum fuel product known to be present where the sample was collected. Therefore, if the absorbance reading is negative and gasoline is believed to be present in the soil, the TPH concentration in the sample would be estimated to be greater than 10 mg/kg gasoline; if the absorbance reading is positive or zero, the TPH concentration in the sample would be estimated to be less than or equal to 10 mg/kg gasoline.

6. If the absorbance reading is negative, the analysis may be performed at multiple detection levels by diluting the sample extract with methanol and assaying the sample extract again. Interpret the resulting photometer reading as described in item 5; however, multiply the petroleum fuel product equivalent by the dilution factor to determine the approximate soil sample concentration. Methanol dilutions must be conducted at least four times apart to adequately discriminate between measurements.

2.2.7.3 Advantages and Limitations

An advantage of the EnSys Petro Test System is that it is easy to operate, requiring one person with basic wet chemistry skills. In addition, the spectrophotometer is battery-operated; therefore, an AC power source is not required in the field.

A limitation of the device is that it does not measure all TPH fuel types. According to SDI, the device is designed to measure a large portion of the aromatic hydrocarbons but only a few of the aliphatic hydrocarbons in the C_6 through C_{22} carbon range. In addition, spectrophotometer results are not reported in terms of TPH. Instead, absorbance results are converted to petroleum fuel product equivalents for only one type of fuel—for example, gasoline or diesel. The device is not designed to account for more than one fuel type that may be present in a sample extract, and the individual fuel types that can be measured are limited to those for which SDI has developed petroleum fuel product equivalents (see Table 2-10).

Another limitation of the EnSys Petro Test System is that it does not generate quantitative results. The semiquantitative results produced by the device can only indicate whether the PHC concentration in a sample extract is above or below a particular level. The concentration range of the sample extract can be narrowed by increasing the number of dilutions in a sample batch. However, increasing the number of dilutions increases the analytical time and cost per sample.

Chapter 3
Demonstration Site Descriptions

This chapter describes the three sites selected for conducting the demonstration. The first site is referred to as the Navy BVC site; it is located in Port Hueneme, California, and contains three sampling areas. The second site is referred to as the Kelly AFB site; it is located in San Antonio, Texas, and contains one sampling area. The third site is referred to as the PC site; it is located in north-central Indiana and contains one sampling area. After review of the information available on these and other candidate sites, the Navy BVC, Kelly AFB, and PC sites were selected based on the following criteria:

- **Site Diversity**—Collectively, the three sites contain sampling areas with the different soil types and the different levels and types of PHC contamination needed to evaluate the field measurement devices described in Chapter 2.
- **Access and Cooperation**—The site representatives are interested in supporting the demonstration by providing site access for collection of soil samples required for the demonstration. In addition, because the field measurement devices will be demonstrated at the Navy BVC site using soil samples from all three sites, the Navy BVC site representatives will provide the site support facilities required for the demonstration and will support a visitors' day during the demonstration. As a testing location for the Department of Defense National Environmental Technology Test Site Program, the Navy BVC site is used to demonstrate technologies and systems for characterizing or remediating soil, sediment, and groundwater contaminated with fuel hydrocarbons or waste oil.

The site descriptions in Sections 3.1 through 3.3 are based on data collected in January 2000 during the predemonstration investigation sampling activities as well as on the information provided by the site representatives. The predemonstration investigation samples were analyzed for GRO and EDRO using SW-846 Method 8015B (modified) by Tetra Tech's SITE team laboratory, Severn Trent Laboratories in Tampa, Florida (STL Tampa East). Physical characterization of these samples was done in the field by a geologist. Some of the predemonstration investigation samples were also analyzed by the measurement device developers at their facilities. Table 3-1 summarizes key site characteristics, including the contamination type, predemonstration investigation sampling depth intervals, associated TPH concentrations, and soil type in each sampling area.

Table 3-1. Summary of Site Characteristics

Site	Sampling Area	Type of Contamination	Approximate Sampling Depth Interval (foot bgs)	TPH Concentration Range (mg/kg)	Type of Soil
Navy BVC	FFA	EDRO (weathered diesel with carbon range from C_{10} through C_{40})	Upper layer[a]	38 to 470	Silty sand
			Lower layer[a]	7,700 to 11,000	
	NEX Service Station Area	GRO and EDRO (fairly weathered gasoline with carbon range from C_7 through C_{14})	5 to 6	21 to 22	Silty sand
			6 to 7	15 to 18	
			7 to 8	25 to 60	
			8 to 9	23 to 65	
			9 to 10	40 to 1,600	
			10 to 11	24 to 300	
	PRA	EDRO (heavy lubricating oil with carbon range from C_{14} through C_{40+})	1.5 to 2.5	1,500 to 2,700	Silty sand with some clay
Kelly AFB	B-38 Area	GRO and EDRO (heavy lubricating oil with carbon range from C_{24} through C_{30})	13 to 17	9	Clayey silt in upper depth interval and sandy clay with significant gravel in deeper depth interval
			29 to 30	9 to 18	
PC	SFT Area	GRO and EDRO (combination of slightly weathered gasoline, kerosene, JP-5, and diesel with carbon range from C_6 through C_{32})	2 to 4	27 to 1,300	Sandy silt and clay with increasing clay content in deeper depth intervals
			4 to 6	200 to 1,300	
			6 to 8	46 to 600	
			8 to 10	49 to 260	

Notes:

AFB = Air Force Base
bgs = Below ground surface
BVC = Base Ventura County
C = Carbon
EDRO = Extended diesel range organic
FFA = Fuel farm area
GRO = Gasoline range organic
mg/kg = Milligram per kilogram
NEX = Naval Exchange
PC = Petroleum company
PRA = Phytoremediation area
SFT = Slop Fill Tank
TPH = Total petroleum hydrocarbons

[a] Because of soil conditions encountered in the FFA, the sampling depth intervals could not be accurately determined. Sample collection was initiated approximately 10 feet bgs, and attempts were made to collect 4-foot-long soil cores. This approach resulted in varying degrees of core tube penetration up to 17 feet bgs. At each location in the area, the sample cores were divided into two samples based on visual observations. The upper layer of the soil core, which consisted of yellowish-brown, silty sand, made up one sample, and the lower layer of the soil core, which consisted of grayish-black, silty sand and smelled of hydrocarbons, made up the second sample.

The primary purpose of the predemonstration investigation was to assess existing conditions and confirm available information on physical and chemical characteristics of soil in each demonstration area. The demonstration approach presented in Chapter 4 is based on the predemonstration investigation results as well as available historical data.

3.1 Navy Base Ventura County Site

The Navy BVC site in Port Hueneme, California, covers about 1,600 acres along the south California coast. Three areas at the Navy BVC site were selected as sampling areas for the demonstration: (1) the Fuel Farm Area (FFA), (2) the Naval Exchange (NEX) Service Station Area, and (3) the Phytoremediation Area (PRA). These areas are briefly described below and are shown in Figure 3-1.

3.1.1 Fuel Farm Area

The FFA is a tank farm in the southwest corner of the Navy BVC site. The area contains five tanks and was constructed to refuel ships and to supply heating fuel for the Navy BVC site. Tank No. 5114 along the south edge of the FFA was used to store marine diesel. After Tank No. 5114 was deactivated in 1991, corroded pipelines leading into and out of the tank leaked and contaminated the surrounding soil with diesel.

Predemonstration investigation samples were collected at three locations in the FFA using a Geoprobe® (see Figure 3-2). The horizontal area of contamination is estimated to be about 20 feet wide and 90 feet long. Samples were collected at the three locations from east to west and about 5 feet apart. During the predemonstration investigation, soil in the area was found to generally consist of silty sand, and the soil cores contained two distinct layers. The upper layer consisted of yellowish-brown, silty sand with TPH concentrations ranging from 38 to 470 mg/kg. The lower layer consisted of grayish-black, silty sand with TPH concentrations ranging from 7,700 to 11,000 mg/kg. Gas chromatograms showed that FFA soil samples contained (1) weathered diesel, (2) hydrocarbons in the C_{10} through C_{28} carbon range with the hydrocarbon hump maximizing at C_{17}, and (3) hydrocarbons in the C_{12} through C_{40} carbon range with the hydrocarbon hump maximizing at C_{20}.

3.1.2 Naval Exchange Service Station Area

The NEX Service Station Area lies in the northeast portion of the Navy BVC site. About 11,000 gallons of regular and unleaded gasoline was released from UST lines in this area between September 1984 and March 1985. Although the primary soil contaminant in this area is gasoline, EDRO is also of concern because (1) another spill north of the area may have resulted in a commingled plume of gasoline and diesel and (2) a significant portion of weathered gasoline is associated with EDRO.

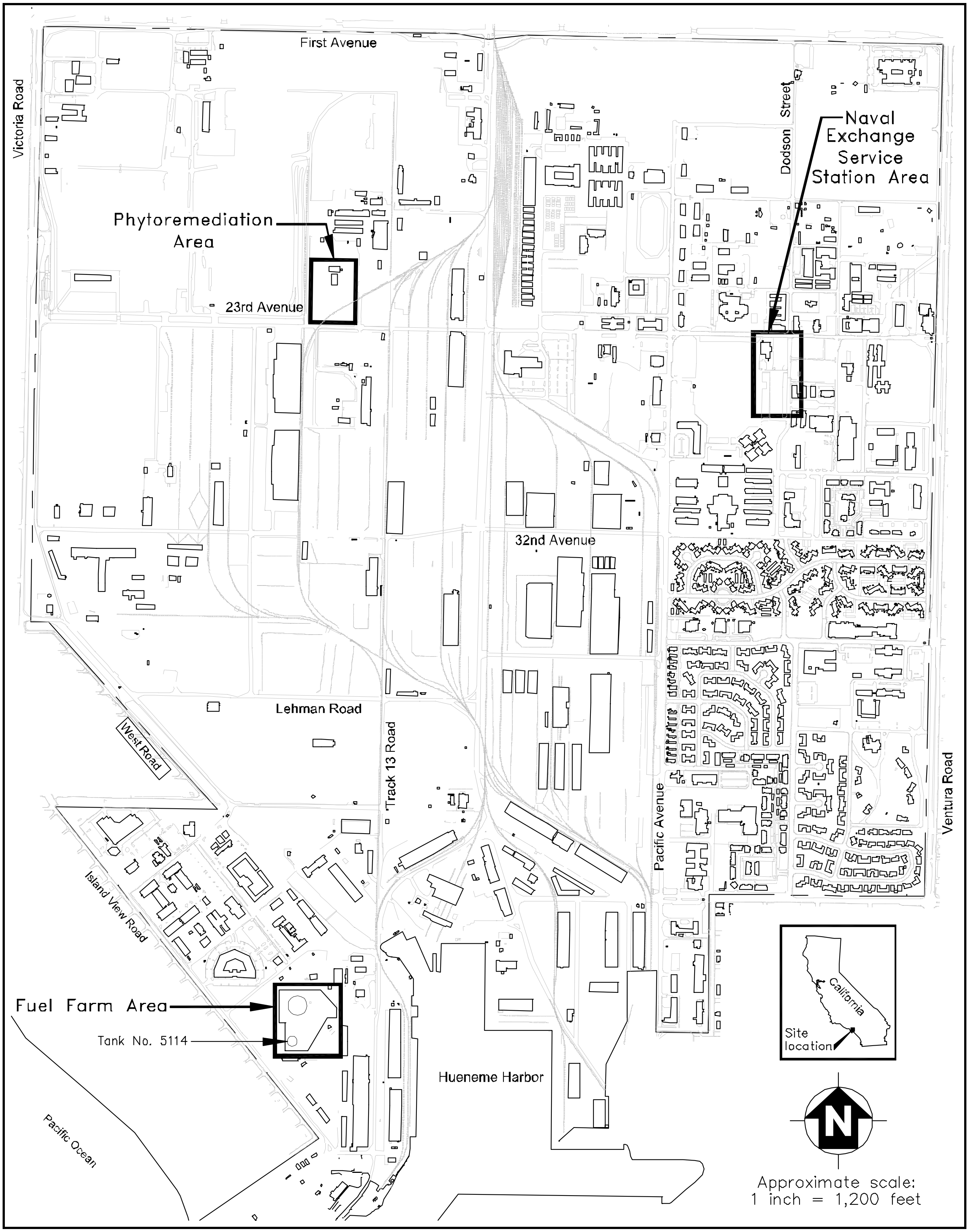


Figure 3-1. Navy Base Ventura County site map.

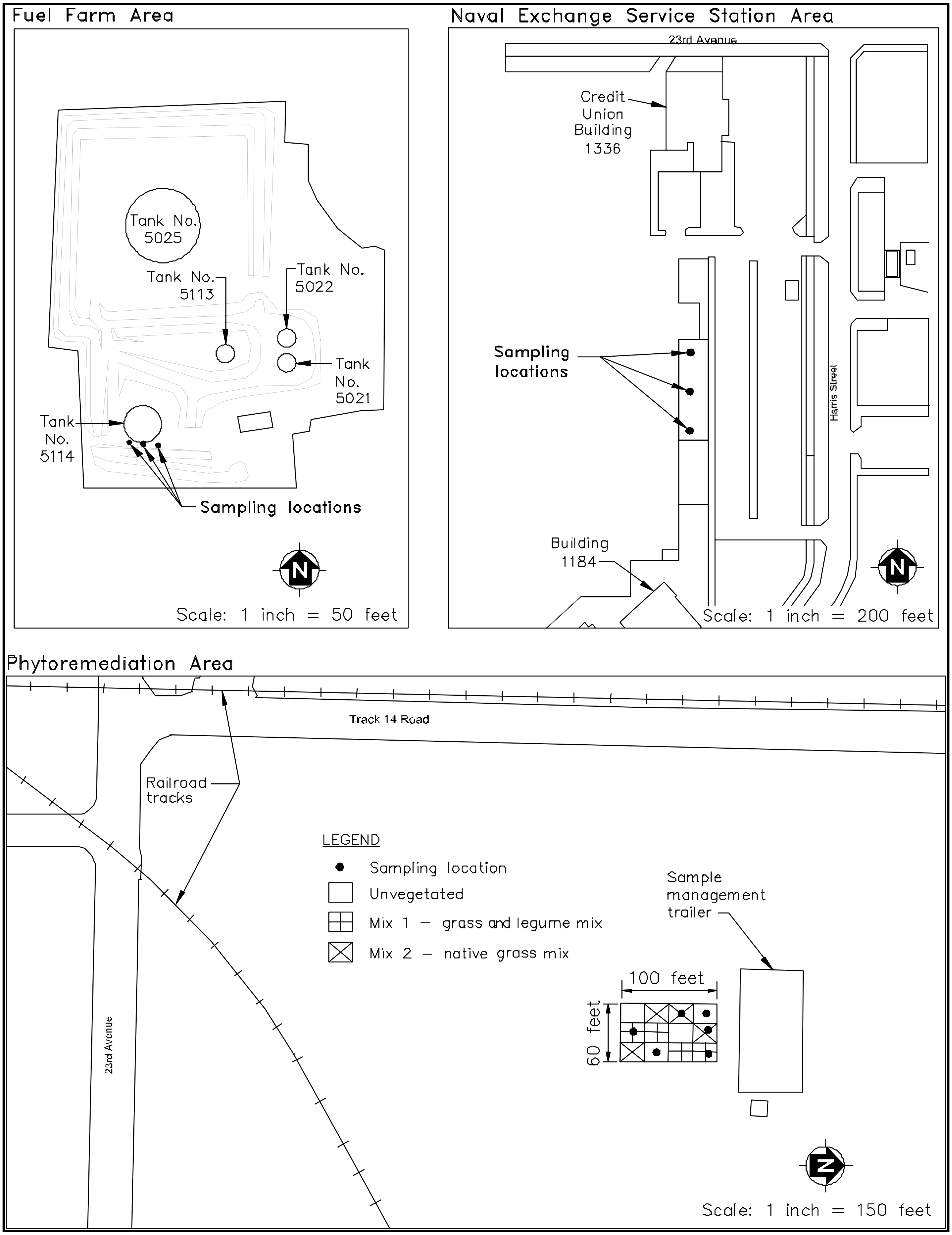


Figure 3-2. Navy Base Ventura County site sampling locations.

Predemonstration investigation samples were collected at three locations in the NEX Service Station Area using a Geoprobe® (see Figure 3-2). Samples were collected from 5 to 11 feet below ground surface (bgs) at 1-foot depth intervals. The horizontal area of contamination is estimated to be about 450 feet wide and 750 feet long. Samples were collected at the three locations from south to north and about 60 feet apart. During the predemonstration investigation, soil in the area was found to generally consist of (1) brown, silty sand in the depth intervals above the water table (which was about 9 feet bgs) and (2) black, silty sand in the depth intervals at and below the water table.

The highest TPH concentrations in the NEX Service Station Area were detected in the depth interval that contained the water table (up to 1,600 mg/kg in the 9- to 10-foot bgs depth interval) at the middle and south sampling locations; at the north sampling location, however, a relatively low concentration of TPH was detected in this depth interval (40 mg/kg). The TPH concentrations in the (1) top four depth intervals ranged from 15 to 65 mg/kg and (2) bottom depth interval (10 to 11 feet bgs) ranged from 24 to 300 mg/kg. Gas chromatograms showed that NEX Service Station Area soil samples contained (1) fairly weathered gasoline with a high aromatic hydrocarbon content and (2) hydrocarbons in the C_7 through C_{14} carbon range. Benzene, toluene, ethylbenzene, and xylene (BTEX) analytical results for the 9- to 10-foot bgs depth interval at the middle sampling location revealed a concentration of 347 mg/kg; BTEX made up 39 percent of the total GRO and 27 percent of the TPH at this location.

3.1.3 Phytoremediation Area

The PRA lies north of the FFA and west of the NEX Service Station Area at the Navy BVC site. The PRA consists of soil from a fuel tank removal project conducted at the Naval Weapons Station in Seal Beach, California. The area is contained within concrete railings and is 60 feet wide, 100 feet long, and about 3 feet deep. It consists of 12 cells of equal size (20 by 25 feet) that have three different types of cover: (1) unvegetated cover, (2) a grass and legume mix (Mix 1), and (3) a native grass mix (Mix 2). There are four replicate cells of each cover type.

Predemonstration investigation samples were collected from the 1.5- to 2.5-foot bgs depth interval at six locations in the PRA using a split-core sampler (see Figure 3-2). During the predemonstration investigation, soil in the area was found to generally consist of silty sand with some clay. The TPH concentrations in the samples ranged from 1,500 to 2,700 mg/kg. Gas chromatograms showed that PRA soil samples contained (1) heavy lubricating oil and (2) hydrocarbons in the C_{14} through C_{40} carbon range with the hydrocarbon hump maximizing at C_{32}.

3.2 Kelly Air Force Base Site

The Kelly AFB site covers approximately 4,660 acres and is about 7 miles from the center of San Antonio, Texas (see Figure 3-3). One area at Kelly AFB, the B-38 Area, was selected as a sampling area for the demonstration. The B-38 Area lies along the east boundary of Kelly AFB and is part of an active UST farm that serves the government vehicle refueling station at the base. During UST removal and upgrading activities in December 1992, soil contamination from leaking diesel and gasoline USTs was observed in the UST and associated piping excavations.

Predemonstration investigation samples were collected in the 13- to 17- and 29- to 30-foot bgs depth intervals at four locations in the B-38 Area using a Geoprobe® (see Figure 3-4). The B-38 Area is estimated to be about 150 square feet in size. The water table in the area fluctuates between 16 and 24 feet bgs. During the predemonstration investigation, soil in the area was found to generally consist of (1) clayey silt in the upper depth interval above the water table interval with a TPH concentration of about 9 mg/kg and (2) sandy clay with significant gravel in the deeper depth interval below the water table interval with TPH concentrations ranging from 9 to 18 mg/kg. Gas chromatograms showed that B-38 Area soil samples contained (1) heavy lubricating oil and (2) hydrocarbons in the C_{24} through C_{30} carbon range.

3.3 Petroleum Company Site

One area at the PC site in north-central Indiana, the Slop Fill Tank (SFT) Area, was selected as a sampling area for the demonstration (see Figure 3-5). The SFT Area lies in the west-central portion of the PC site and is part of an active fuel tank farm. Although the primary soil contaminant in this area is gasoline, EDRO is also of concern because of a heating oil release that occurred north of the area.

Predemonstration investigation samples were collected at five locations in the SFT Area using a Geoprobe® (see Figure 3-5). Samples were collected from 2 to 10 feet bgs at 2-foot depth intervals. The SFT Area is estimated to be 20 feet long and 20 feet wide. Four of the sampling locations were spaced about 15 feet apart to form the corners of a square, and the fifth sampling location was at the center of the square. During the predemonstration investigation, soil in the area was found to generally consist of sandy silt and clay with increasing clay content in deeper depth intervals. Soil in the top three depth intervals consisted of brown, sandy silt with clay that smelled increasingly of hydrocarbons with depth and had TPH concentrations ranging from 27 to 1,300 mg/kg. Soil in the deepest depth interval consisted of sandy clay with increasing clay content with depth, exhibited a moderate to strong hydrocarbon odor, and had TPH concentrations ranging from 49 to 260 mg/kg.

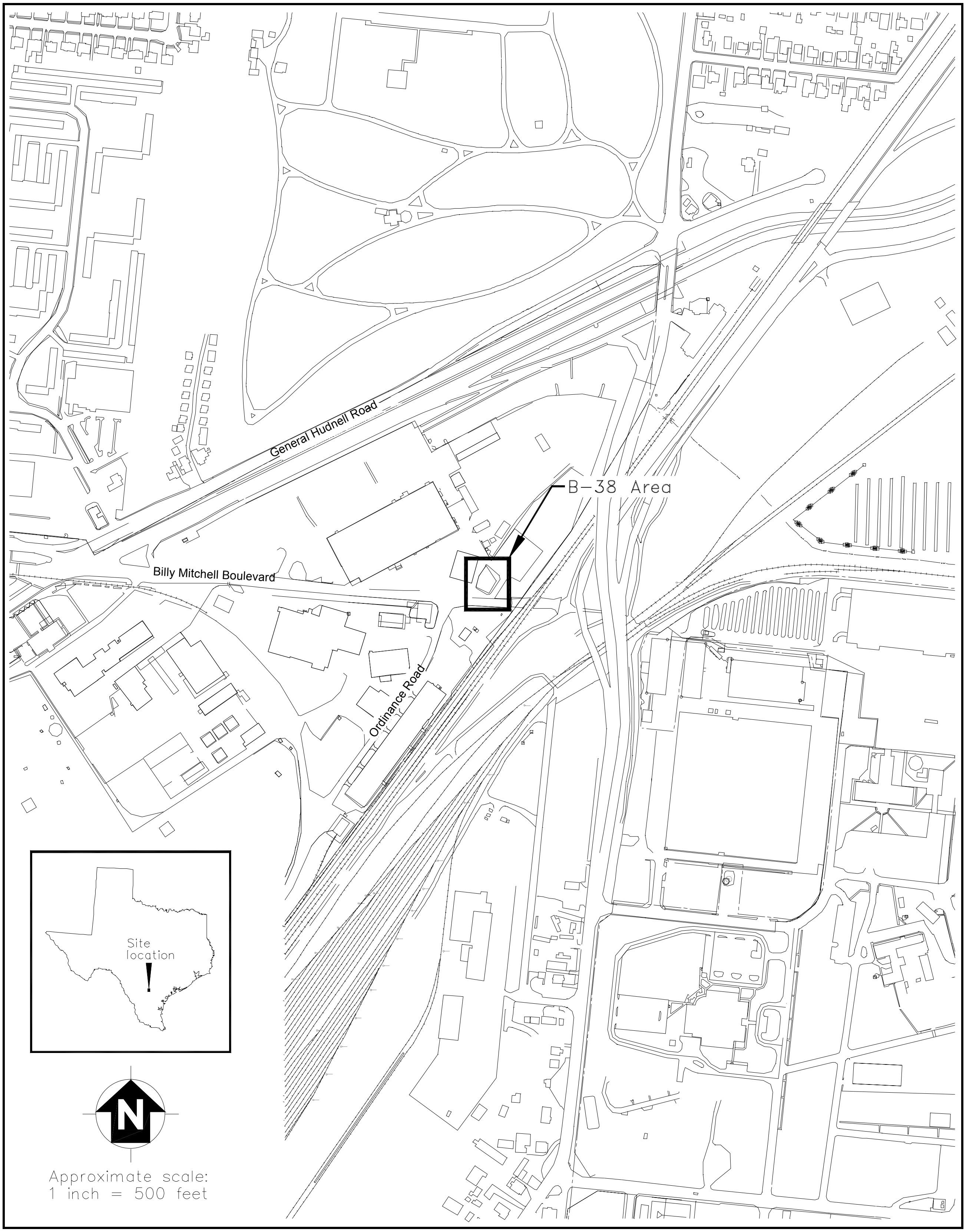


Figure 3-3. Kelly Air Force Base site map.

B-38 Area

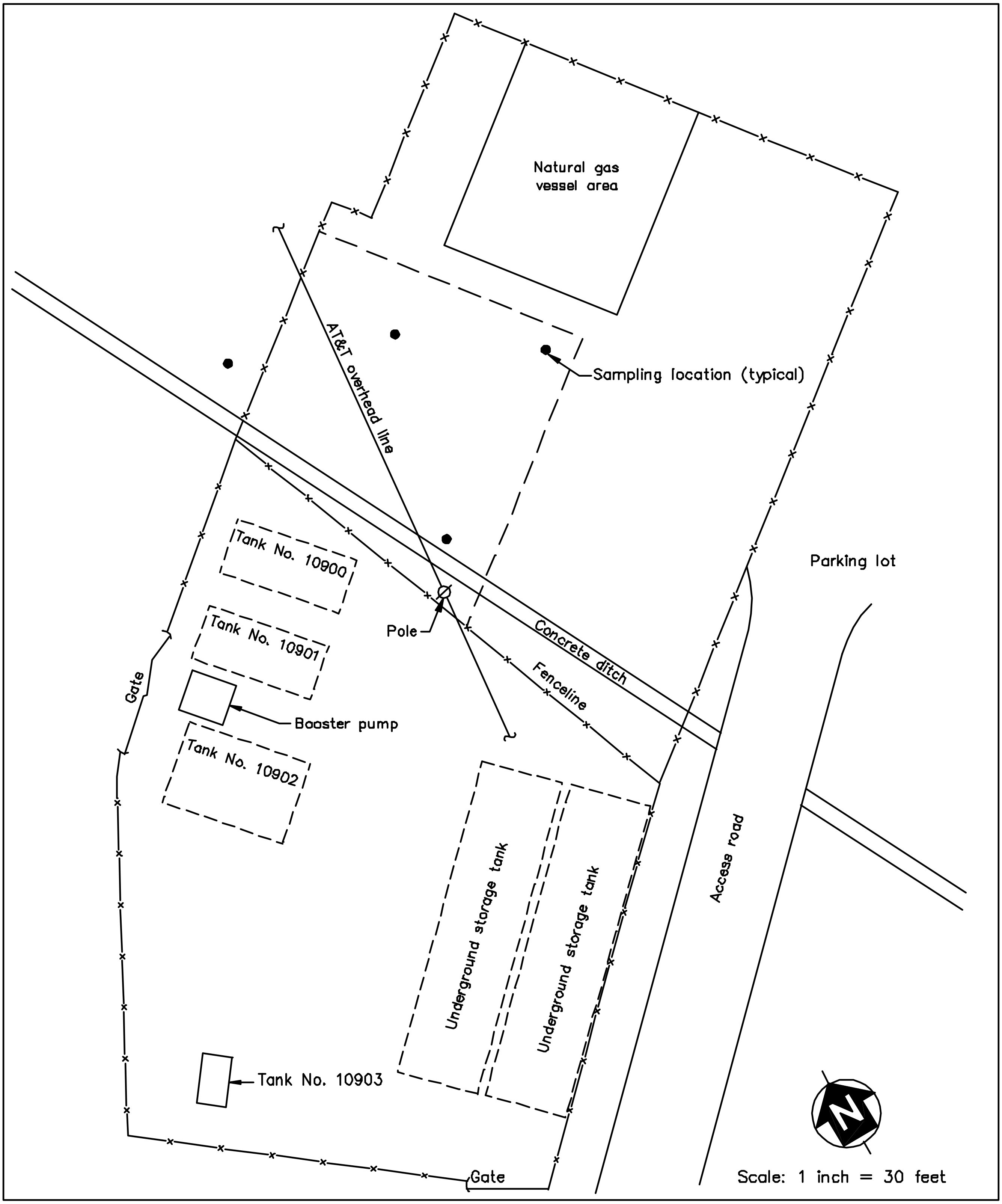


Figure 3-4. Kelly Air Force Base site sampling locations.

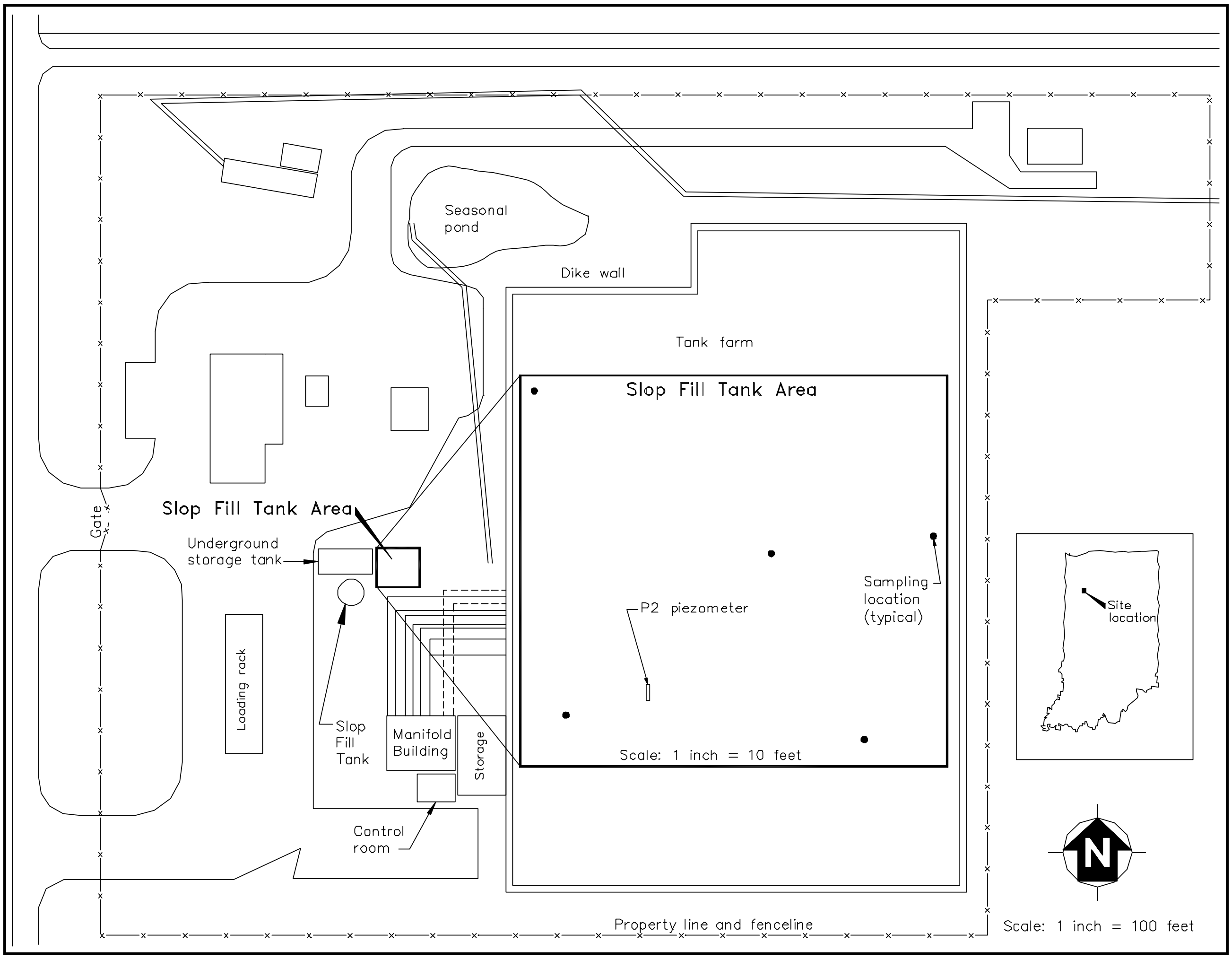


Figure 3-5. Petroleum company site sampling locations.

Gas chromatograms showed that SFT Area soil samples contained (1) slightly weathered gasoline, kerosene, JP-5, and diesel and (2) hydrocarbons in the C_6 through C_{20} carbon range. There was also evidence of an unidentified petroleum product containing hydrocarbons in the C_{24} through C_{32} carbon range. BTEX analytical results for the deepest depth interval revealed concentrations of 26, 197, and 67 mg/kg at the northwest, center, and southwest sampling locations, respectively. At the northwest location, BTEX made up 13 percent of the total GRO and 5 percent of the TPH. At the center location, BTEX made up 16 percent of the total GRO and 7 percent of the TPH. At the southwest location, BTEX made up 23 percent of the total GRO and 18 percent of the TPH.

Chapter 4
Demonstration Approach

This chapter presents the objectives, design, data analysis procedures, and schedule for the innovative TPH field measurement device demonstration.

4.1 Demonstration Objectives

The primary goal of the SITE MMT Program is to develop reliable performance and cost data on innovative, field-ready technologies. A SITE demonstration must provide detailed and reliable performance and cost data so that potential technology users have adequate information to make sound judgments regarding an innovative technology's applicability to a specific site and to compare the technology to conventional technologies.

The demonstration has both primary and secondary objectives. Primary objectives are critical to the technology evaluation and require the use of quantitative results to draw conclusions regarding a technology's performance. Secondary objectives pertain to information that is useful but will not necessarily require the use of quantitative results to draw conclusions regarding a technology's performance.

The primary objectives for the demonstration of the individual field measurement devices are as follows:

P1. Determine the MDL

P2. Evaluate the accuracy and precision of TPH measurement for a variety of contaminated soil samples

P3. Evaluate the effect of interferents on TPH measurement

P4. Evaluate the effect of soil moisture content on TPH measurement

P5. Measure the time required for TPH measurement

P6. Estimate costs associated with TPH measurement

The secondary objectives for the demonstration of the individual field measurement devices are as follows:

S1. Document the skills and training required to properly operate the device

S2. Document health and safety concerns associated with operating the device

S3. Document the portability of the device

S4. Evaluate the device's durability based on its materials of construction and engineering design

S5. Document the availability of the device and associated spare parts

The objectives for the demonstration were developed based on input from MMT Program stakeholders, general user expectations of field measurement devices, characteristics of the demonstration areas, the time available to complete the demonstration, and device capabilities that the developers participating in the demonstration intend to highlight.

4.2 Demonstration Design

In January 2000, Tetra Tech conducted a predemonstration sampling and analysis investigation to assess existing conditions and confirm available information on physical and chemical characteristics of soil in each demonstration area. Based on information from the predemonstration investigation as well as available historical data, a demonstration design was developed to address the demonstration objectives. Input regarding the demonstration design was obtained from developers participating in the demonstration. Appendix A presents several findings of the predemonstration investigation, developer review comments on the predemonstration investigation results, and Tetra Tech responses to the developers' comments. Table 4-1 summarizes the demonstration area characteristics and demonstration design.

Tetra Tech will collect core samples of soil from the demonstration areas at the Navy BVC, Kelly AFB, and PC sites described in Chapter 3. The soil core samples collected at the Kelly AFB and PC sites will be shipped to the Navy BVC site 6 days prior to the start of field analysis activities. Tetra Tech will homogenize the soil core sample collected from a given depth interval at a given sampling location in a given area and will aliquot soil samples for each of the developers and the reference laboratory. In addition, Tetra Tech will obtain performance evaluation (PE) samples from Environmental Resource Associates (ERA) in Arvada, Colorado, for distribution to the developers and the reference laboratory. Field analysis of all environmental and PE samples will be conducted at the Navy BVC site. The environmental samples collected at the three demonstration sites will be used to address the demonstration objectives. However, if environmental samples will not allow a given objective to be effectively addressed, PE

Table 4-1. Demonstration Approach

Site	Area	Primary Objective to be Addressed	Expected Contamination Type[a]	Approximate Sampling Depth Interval (foot bgs)	Expected Concentration	Soil Characteristics	Measurement Devices[b]
Navy BVC	FFA	P2	EDRO (weathered diesel with carbon range from C_{10} through C_{40})	Upper layer[c]	L to M	Silty sand	Group I and Group II with Model HATR-T
				Lower layer[c]	H		
	NEX Service Station Area	P2	GRO and EDRO (fairly weathered gasoline with carbon range from C_7 through C_{14})	7 to 8	L	Silty sand	Group I and Group II with Model CVH
				8 to 9	L		
				9 to 10	L to H		
				10 to 11	L to M		
	PRA	P2	EDRO (heavy lubricating oil with carbon range from C_{14} through C_{40})	1.5 to 2.5	H	Silty sand with some clay	Group I not including the PetroFLAG™ test kit and EnSys Petro Test System[d] and Group II with Model HATR-T
Kelly AFB	B-38 Area	P2	GRO and EDRO (additional information unavailable)	18 to 20	L	Clayey silt in upper depth interval and sandy clay with significant gravel in deeper depth interval[e]	Group I and Group II with Model CVH
				20 to 22	L		
PC	SFT Area	P2	GRO and EDRO (combination of slightly weathered gasoline, kerosene, JP-5, and diesel with carbon range from C_6 through C_{32})	2 to 4	L to H	Sandy silt and clay with increasing clay content in deeper depth intervals	Group I and Group II with Model CVH
				4 to 6	M to H		
				6 to 8	L to M		
				8 to 10	L to M		
Reference soil 1 (PE sample)		P1, P2	GRO (methanol as carrier)	Not applicable	L	Sand	Group I not including the PetroFLAG™ test kit
			GRO (Freon 113 as carrier)	Not applicable	L		Group II with Model CVH
			EDRO (methanol as carrier)	Not applicable	L		Group I
			EDRO (Freon 113 as carrier)	Not applicable	L		Group II with Model HATR-T
Reference soil 2 (PE sample)		P2	GRO (methanol as carrier)	Not applicable	M	Silty sand	Group I not including the PetroFLAG™ test kit
			GRO (Freon 113 as carrier)	Not applicable	M		Group II with Model CVH
			EDRO (methanol as carrier)	Not applicable	M		Group I
			EDRO (Freon 113 as carrier)	Not applicable	M		Group II with Model HATR-T
		P3	GRO (trace levels) EDRO (low levels)	Not applicable	L	Silty sand	Group I and Group II with both Models CVH and HATR-T
		P2, P3, P4	GRO	Not applicable	H	Silty sand	Group I and Group II with Model CVH
			EDRO	Not applicable	H		Group I and Group II with Model HATR-T
Reference soil 2 (PE sample) (continued)		P3	I(1 through 4)L1[f] + GRO	Not applicable	H	Silty sand	Group I and Group II with Model CVH
			I(1 through 4)L2[f] + GRO	Not applicable	H		
			I(3 through 6)L1[f] + EDRO	Not applicable	H		Group I and Group II with Model HATR-T
			I(3 through 6)L2[f] + EDRO	Not applicable	H		

Table 4-1. Demonstration Approach (Continued)

Site	Area	Primary Objective to be Addressed	Expected Contamination Type[a]	Approximate Sampling Depth Interval (foot bgs)	Expected Concentration	Soil Characteristics	Measurement Devices[b]
Liquid (PE sample)		P3	GRO	Not applicable	H	Not applicable; these are liquid samples	Group I and Group II with Model CVH
			EDRO	Not applicable	H		Group I and Group II with Model HATR-T
			I(1 and 2)L1[f]	Not applicable	M to H		Group I and Group II with Model CVH
			I(3 and 4)L1[f]	Not applicable	M to H		Group I and Group II with both Models CVH and HATR-T
			I(5)L1[f]	Not applicable	M to H		Group I and Group II with Model HATR-T
			I(1 and 2)L2[f]	Not applicable	M to H		Group I and Group II with Model CVH
			I(3 and 4)L2[f]	Not applicable	M to H		Group I and Group II with both Models CVH and HATR-T
			I(5)L2[f]	Not applicable	M to H		Group I and Group II with Model HATR-T
Reference soil 2 (PE sample)		P3	I(6)L1[f]	Not applicable	M to H	Silty sand	Group I and Group II with Model HATR-T
			I(6)L2[f]	Not applicable	M to H		
		P4	GRO at 18 percent moisture	Not applicable	H	Silty sand	Group I and Group II with Model CVH
			EDRO at negligible moisture (none added)	Not applicable	H		Group I and Group II with Model HATR-T

Notes:

\> = Greater than
< = Less than
AFB = Air Force Base
bgs = Below ground surface
BVC = Base Ventura County
C = Carbon
EDRO = Extended diesel range organics
FFA = Fuel Farm Area
GRO = Gasoline range organics
H = High (>1,000 mg/kg)
I(1)L1 = Interferent 1 at level 1
L = Low (<100 mg/kg)
M = Medium (100 to 1,000 mg/kg)
mg/kg = Milligram per kilogram
NEX = Naval Exchange
PC = Petroleum company
PE = Performance evaluation
PRA = Phytoremediation Area
SFT = Slop Fill Tank
TPH = Total petroleum hydrocarbons

Unless otherwise specified, all soil PE samples will be prepared at 9 percent moisture.

Reference soil 1 is Ottawa sand, and reference soil 2 is processed garden soil (sandy silt). Reference soil 2 may contain trace levels of GRO and low levels of EDRO.

The boiling points and vapor pressures of (1) interferents I(1) and I(2) have characteristics similar to those of GRO, (2) interferents I(3) and I(4) have characteristics similar to those of GRO and EDRO, and (3) interferents I(5) and I(6) have characteristics similar to those of EDRO. PE samples containing interferents I(1) and I(2) will not be prepared with EDRO because these interferents are not expected to impact EDRO analysis. Similarly, PE samples containing interferents I(5) and I(6) will not be prepared with GRO because these interferents are not expected to impact GRO analysis. In addition, practical difficulties (solubility constraints) associated with preparing the I(1) or I(2) + EDRO and I(5) or I(6) + GRO PE samples will not allow use of these samples in the demonstration. Expected concentrations for samples containing interferents are based on expected analytical results for the samples and not on the concentrations of interferents that will be added to the samples.

[a] Expected contamination types for environmental samples are based on predemonstration investigation results. Expected contamination types for PE samples are based on planned preparation methods for PE samples.

Table 4-1. Demonstration Approach (Continued)

[b] Group I includes the PetroFLAG™ test kit, Luminoscope, UVF-3100A, and EnSys Petro Test System. Group II includes the RemediAid™ starter kit, OCMA-350, and Model CVH or Model HATR-T or both.

[c] Because of soil conditions encountered in the FFA during the predemonstration investigation, the sampling depth intervals could not be accurately determined. Sample collection was initiated approximately 10 feet bgs, and attempts were made to collect 4-foot-long soil cores. This approach resulted in varying degrees of core tube penetration up to 17 feet bgs. During the demonstration, for each sampling location in the area, the sample cores will be divided into two samples based on visual observations. The upper layer of the soil core, which will likely consist of yellowish-brown, silty sand, will make up one sample, and the lower layer of the soil core, which will likely consist of grayish-black, silty sand and smell of hydrocarbons, will make up the second sample.

[d] Dexsil® chose not to demonstrate the PetroFLAG™ test kit using PRA samples because the device's performance is affected by the presence of the natural organic material present in PRA soil. Also, SDI chose not to demonstrate the EnSys Petro Test System using PRA samples because, based on predemonstration investigation results, the primary contaminant in the PRA is a heavy lubricating oil in the C_{32} range, and this device can only be used to measure hydrocarbons up to C_{22}.

[e] The depth intervals sampled during the demonstration will differ from those sampled during the predemonstration investigation; therefore, the soil characteristics may vary.

[f] Interferents will be added to the PE samples at two levels ranging from 50 to 500 percent of the TPH concentrations.

samples will be used to supplement environmental samples as necessary. Each field measurement device will be evaluated based primarily on how it compares with the reference method selected for the demonstration. PE samples will be used to verify that the reference method's performance is acceptable. However, during the comparison with the field measurement device results, the reference method results will not be adjusted based on the recoveries observed during analysis of the PE samples. Any problems encountered when the reference method or a given field measurement device is used to analyze the PE samples (for example, consistent high or low bias) will be appropriately mentioned in the ITVRs.

To facilitate effective use of available information on both the environmental and PE samples, the developers and the reference laboratory will be informed of (1) whether each sample is an environmental or PE sample, (2) the area that each environmental sample was collected in, and (3) the expected contamination type and concentration range of each sample. This information will be included in each sample identification number. Each sample will be identified as having an expected low (less than 100 mg/kg), medium (100 to 1,000 mg/kg), or high (greater than 1,000 mg/kg) TPH concentration. The expected concentration ranges will be based on predemonstration investigation results or the amount of TPH added during PE sample preparation. For each PE sample containing interferents, the expected TPH concentration will be based on the expected analytical result for the sample and not on the concentrations of the interferents added to the sample.

Some PE samples will also contain interferents specifically added to evaluate the effective of interferents on TPH measurement; the interferent nature and concentration range will be identified; however, the specific compounds used as interferents will not be identified. The expected concentration ranges are meant to be used only as a guide by the developers and reference laboratory. Additional information regarding sample collection, preparation, and labeling is included in Chapter 7. During the demonstration, each developer will operate its own field measurement device or devices. Tetra Tech will make observations to evaluate each device as described in the demonstration design.

The performance and cost of each field measurement device will be compared only to those of the reference method; the field measurement devices will not be compared to one another. Developers may choose not to analyze samples collected in a particular area or a particular class of samples, depending on the intended use of their devices. However, the developers must notify Tetra Tech if they choose not to analyze any of the demonstration samples.

The general approach for addressing each primary objective is discussed below. Specific procedures for addressing each primary objective are presented in Section 4.3.

- To determine the MDL for each field measurement device (primary objective P1), low-level GRO and EDRO PE samples will be analyzed. The low-level PE samples will be prepared using methanol or Freon 113 as a carrier. Details on PE sample preparation are provided in Chapter 7. The target concentrations of the PE samples will be set to meet the following criteria: (1) at the minimum acceptable recoveries, the samples will contain measurable TPH concentrations, and (2) the sample TPH concentrations will generally be ten times the MDLs claimed by the developers and the reference laboratory. Each developer and the reference laboratory will analyze seven GRO and seven EDRO PE samples to statistically determine the MDLs for GRO and EDRO soil samples. Table 4-2 presents the expected MDLs for each measurement device based on data provided by the developers. The reference method's MDLs for GRO and EDRO soil samples are 0.43 and 6.3 mg/kg, respectively.

Table 4-2. Expected Method Detection Limits for Each Field Measurement Device

	Method Detection Limit (milligram per kilogram)	
Device	Gasoline Range Organics	Extended Diesel Range Organics
RemediAid™ starter kit	8[a]	40[a]
Model CVH	3	3[b]
Model HATR-T	Not recommended	20
OCMA-350	1	1
PetroFLAG™ test kit	1,000	20
Luminoscope	0.050	0.050
UVF-3100A	3.9	0.080
EnSys Petro Test System	10	15

Notes:

[a] According to CHEMetrics, the method detection limits for liquid samples for gasoline range organics and extended diesel range organics are 2 and 10 milligrams per liter, respectively; Tetra Tech calculated the method detection limits for soil samples assuming 5 grams of soil are extracted using 20 milliliters of solvent.

[b] During the demonstration, if a sample does not contain gasoline range organics, Wilks will use Model HATR-T; otherwise, Wilks will use Model CVH. Model CVH can be converted into Model HATR-T simply by changing the sample stage, and the reverse is also true. Therefore, the demonstration design does not include separate evaluation of Models CVH and HATR-T.

Because SDI's EnSys Petro Test System reports results relative to an action level or as a concentration range, MDLs cannot be statistically determined for this device. SDI will be asked to report whether the GRO and EDRO PE samples contain TPH concentrations above 10 and 15 mg/kg, respectively. These concentrations are close to SDI's reported MDLs for the EnSys Petro Test System. In addition, Dexsil® has chosen not to analyze the low-level GRO PE samples because the PetroFLAG™ test kit has an MDL of 1,000 mg/kg for GRO.

- To estimate the accuracy and precision of each field measurement device (primary objective P2), both environmental and PE samples will be analyzed. To evaluate analytical precision, one set of blind field triplicate environmental samples will be collected from each depth interval at one location in each demonstration area. Blind triplicate low-, medium-, and high-level PE samples will also be used to address P2 because TPH concentrations in environmental samples collected during the demonstration may differ from the analytical results for predemonstration investigation samples. The low- and medium-level PE samples will be prepared using methanol or Freon 113 as a carrier. Details on PE sample preparation are provided in Chapter 7.

Information regarding analytical precision will be collected by having the developers and the reference laboratory analyze extract duplicates. For environmental samples, one sample from each depth interval will be designated as an extract duplicate. Each sample selected as an extract duplicate will be collected from a location where field triplicates are collected. For PE samples, one low-, one medium-, and one high-level GRO sample and one low-, one medium-, and one high-level EDRO sample will be designated as extract duplicates.

The evaluation of analytical accuracy will be based on the assumption that a field measurement device may be used to (1) determine whether the TPH concentration in a given area exceeds an action level (a state or other action level) or (2) perform a preliminary characterization of soil in a given area. To evaluate whether the TPH concentration in a soil sample exceeds an action level, each developer and the reference laboratory will be asked to determine whether TPH concentrations in a given area or PE sample type exceed the action levels listed in Table 4-3. The action levels chosen for environmental samples are based on the predemonstration investigation analytical results and state action levels. The action levels chosen for the PE samples are based in part on ERA's acceptance limits for PE samples; therefore, each PE sample is expected to have at least the TPH concentration indicated in Table 4-3.

Table 4-3. Action Levels to be Used to Evaluate Analytical Accuracy

Site	Area	Expected Concentration[a]	Primary Objective P2 Action Level (mg/kg)
Navy BVC	FFA	L to H	100
	NEX Service Station Area	L to H	50
	PRA	H	1,500
Kelly AFB	B-38 Area	L	100
PC	SFT Area	L to H	500
PE samples (GRO)		L	10
		M	200
		H	2,000
PE samples (EDRO)		L	15
		M	200
		H	2,000

Notes:

< = Less than
> = Greater than
AFB = Air Force Base
BVC = Base Ventura County
EDRO = Extended diesel range organics
FFA = Fuel Farm Area

GRO = Gasoline range organics
H = High (>1,000 mg/kg)
kg = Kilogram
L = Low (<100 mg/kg)
M = Medium (100 to 1,000 mg/kg)
mg = Milligram

NEX = Naval Exchange
PC = Petroleum company
PE = Performance evaluation
PRA = Phytoremediation Area
SFT = Slop Fill Tank

[a] The expected concentrations shown cover all the depth intervals in each area. Table 4-1 shows the depth intervals to be sampled in each area and the expected concentration for each depth interval. The action level for each area will be used as the basis for evaluating sample analytical results regardless of the expected concentrations for the various depth intervals.

- To evaluate the effect of interferents on each field measurement device's ability to accurately measure TPH (primary objective P3), high-level soil PE samples that contain GRO or EDRO with or without an interferent will be analyzed. Six different interferents, I(1) through I(6), will be evaluated. The boiling points and vapor pressures of (1) interferents I(1) and I(2) are similar to those of GRO, (2) interferents I(3) and I(4) are similar to those of GRO and EDRO, and (3) interferents I(5) and I(6) are similar to those of EDRO. Separate PE samples will be prepared for GRO and EDRO analyses with the following exceptions: PE samples containing I(1) and I(2) will not be prepared with EDRO and PE samples containing I(5) and I(6) will not be prepared with GRO because these interferents are not expected to impact the analyses and because practical difficulties such as solubility constraints are associated with preparation of such samples. Both GRO and EDRO PE samples will be spiked with a high concentration of TPH (greater than 1,000 mg/kg). Interferents will be added to the samples at two concentrations ranging from 50 to 500 percent of the TPH concentrations.

Appropriate control samples will be prepared and analyzed to address P3. These samples include reference soil, reference soil plus GRO or EDRO, reference soil plus interferent, and reference soil plus interferent plus GRO or EDRO samples. When a soil sample containing only a given interferent cannot be prepared because of solubility constraints, liquid samples containing the interferent will be prepared and used as quasi-control samples to evaluate the effect of the interferent alone on the result obtained by each measurement device and the reference method. Each PE sample will be prepared in triplicate. Average and variability information for the triplicates will be used to interpret the effect of the interferent.

- To evaluate the effect of soil moisture content on each field measurement device's ability to accurately measure TPH (primary objective P4), high-level soil PE samples that contain GRO or EDRO will be analyzed. For GRO, PE samples will be prepared at two moisture levels: (1) about 9 percent moisture because this is the minimum level required to containerize PE samples in EnCores (these samples are already accounted for under primary objective P2) and (2) about 18 percent because this is the saturation level of the soil. For EDRO, PE samples will be prepared at two moisture levels: (1) negligible moisture (none added) and (2) about 9 percent (these samples are already accounted for under P2). Each PE sample will be prepared in triplicate. Average and variability information for the triplicates will be used to interpret the effect of moisture.

- The time required for TPH measurement (primary objective P5) or the sample throughput (the number of TPH measurements made per unit of time) will be determined by measuring the time required for each activity associated with TPH measurement, including field measurement device setup, sample extraction, sample analysis, and data package preparation. Tetra Tech will provide each developer with investigative samples stored in coolers. The developers will unpack the coolers and check the chain-of-custody forms to verify that they have received the correct samples. Time measurement will begin when a developer begins to set up the device. The total time required to complete analysis of all investigative samples will be recorded. Analysis will be considered complete and the time measurement will stop when the developer provides Tetra Tech with a summary table of results, a run log, and any supplementary information that the developer chooses. The summary table must list all samples analyzed and their respective TPH concentrations (and GRO and EDRO concentrations, if applicable) in mg/kg on a wet weight basis.

  For the purposes of the demonstration, investigative samples will include environmental, field triplicate, and PE samples provided to the developers by Tetra Tech as well as the extract duplicates specified by Tetra Tech. The total number of samples recorded as being analyzed in a given period by a developer will be based on the number of investigative samples analyzed. If a developer conducts multiple extractions, dilutions, reanalyses, or QC checks, these will be noted by Tetra Tech but will not be included in the number of investigative samples analyzed, and the total time expended will not be adjusted because any additional work performed by the developer is considered to be required in order to provide data of adequate quality.

  In addition to the total time expended to analyze the investigative samples, Tetra Tech will measure the time required to analyze two "batches" of samples. A "batch" will be a group of samples that the developer prepares or analyzes in one sequence. Tetra Tech will measure only the time expended for the first and last batches of samples. Tetra Tech will note the elements that comprise each batch, such as environmental samples, developer QC samples, multiple dilutions, re-extractions, and others. In addition to measuring the time required to analyze the first and last batches of samples, Tetra Tech will confirm that each analytical step is completed by the developer as described in Chapter 2. For example, if a developer claims that part of the device operating procedure is to shake a sample extract for 3 minutes, Tetra Tech will verify that the sample was actually shaken for 3 minutes.

Each developer will be made aware of when the timing of each activity begins and ends. If the developer's entire field team chooses to take a break, this time will not be counted as part of the analytical time. If a device is not working or requires repair, this fact will be noted, but the resulting downtime will not be counted as part of the analytical time.

For the reference laboratory, the total analytical time will begin to be measured when the laboratory receives all investigative samples, and the time measurement will continue until Tetra Tech receives a complete data package from the laboratory.

- To estimate the costs associated with TPH measurement (primary objective P6), the following five cost categories will be considered: capital, labor, supplies, investigation-derived waste (IDW), and support equipment. A summary of the costs that will be estimated for each measurement device is provided below.

    1. The capital cost will be estimated based on price lists for purchasing, renting, or leasing each field measurement device. If the device has to be purchased, the capital cost will include no salvage value for the device after work is completed.

    2. The labor cost will be estimated based on the number of people required to analyze samples during the demonstration for each field measurement device. The labor rate will be based on a standard hourly rate for a technician. During the demonstration, the skill level required will be confirmed based on input from the developer regarding operation of the device and Tetra Tech's observation of the skills required to operate the device or to interpret data in order to calculate TPH concentrations. In addition, the labor cost will be based on (1) the actual number of hours required to complete analysis of all investigative samples as measured under primary objective P5 and (2) the assumption that a technician who has worked for a portion of a day would be paid for an entire 8-hour day.

    3. The cost of supplies will be estimated based on any supplies required to analyze all investigative samples during the demonstration using each field measurement device but not included in the capital cost category, such as a balance, extraction solvent, glassware, pipettes, spatulas, agitators, and so on. Tetra Tech will note the type and quantity of all supplies brought to the field as well as the quantity of supplies used by each developer to analyze all the investigative samples during the demonstration.

       If a developer typically provides all supplies to a user, the developer's costs will be used to estimate the cost of supplies. If the supplies required to analyze a set number of samples are covered by the purchase cost of a measurement device, this cost will not be broken out separately as part of the cost of supplies. However, the cost of any additional supplies required to analyze all the investigative samples will be included in the cost of supplies. If a developer provides supplies as part of a refill kit, the cost for the number of kits required to analyze all the investigative samples will be included in the cost of supplies. If a developer creates refill kits specific to a user's needs, the associated cost of supplies will be based on the cost of the refill kits that the developer uses during the demonstration. Unless a developer allows a user to return unused portions of a refill kit, the cost of supplies will be estimated under the assumption that no salvage value is associated with unused refill kit supplies. If unused supplies can be returned to a developer, the quantities of unused supplies will be noted during the demonstration, and the appropriate credit will be applied to the cost of supplies minus any restocking charge.

If a developer typically does not provide all required supplies to a user, Tetra Tech will estimate the cost of supplies using independent vendor quotes. Tetra Tech will note the identification numbers and manufacturers of supplies used by the developer during the demonstration and will attempt to obtain pricing information for these supplies. If the costs of these supplies are not available, Tetra Tech will use the prices of comparable supplies to estimate the cost of supplies. If unused supplies can be returned to a vendor or manufacturer, the quantities of unused supplies will be noted during the demonstration and the appropriate credit will be applied to the cost of supplies minus any restocking charge.

4. The IDW disposal cost will be estimated for each device. Each developer will be provided with two 20-gallon laboratory packs: one for flammable wastes and one for corrosive wastes, as necessary. IDW generated may include decontamination fluids, decontamination equipment, spent solvents, unused chemicals that a user cannot return to the developer or an independent vendor, used EnCores, TPH-contaminated soil samples, and soil extracts. The cost for disposal of personal protective equipment (PPE), glassware, and plastic utensils used during the demonstration will not be included in the disposal cost because these wastes can be disposed of in a dumpster along with municipal garbage. The cost for disposal of laboratory packs will be included in the overall analysis cost for each developer. Each developer will provide containers to containerize individual wastes, and the cost of these containers will not be included in the total analysis cost.

5. The support equipment cost will be estimated for each field measurement device based on information provided by the developer and any support equipment such as tables, chairs, and a tent observed to be required for the device during the demonstration. Tetra Tech will note the type and quantity of all support equipment used by each developer during the demonstration.

The cost per analysis will not be estimated for the field measurement devices because the cost per analysis would decrease as more samples were analyzed and because the initial capital cost would be distributed across a greater number of samples. This decrease in cost per analysis cannot be fairly compared to the reference laboratory's fixed cost per analysis.

Secondary objectives will be addressed based on observations made during the demonstration. Because of the number of developers involved, several Tetra Tech personnel will be required to make simultaneous observations during the demonstration. Two Tetra Tech personnel will be assigned to observe a pair of developers for the entire demonstration. These personnel will make observations and take notes regarding TPH measurement activities. To ensure observational consistency, Tetra Tech personnel will discuss and compare observations made and notes taken regarding each device at the end of each day of the demonstration. Developers will also be given the opportunity to review and comment on the notes taken by Tetra Tech regarding their devices at the end of each day of the demonstration. Any issues that may arise will be resolved with the direction of the EPA, if necessary. The approach for addressing each secondary objective is discussed below.

- The skills and training required for proper device operation (secondary objective S1) will be addressed by observing and noting the skills required to operate each device during the demonstration, assessing how easy the device is to operate, and discussing any necessary user training with the developer.

- Health and safety concerns associated with device operation (secondary objective S2) will be addressed by observing and noting possible health and safety concerns during the demonstration, such as the types of hazardous substances that must be handled by a user of a device during analysis, the number of times that hazardous substances must be transferred from one container to another during the analytical procedure, and any direct user exposure to hazardous substances.

- The portability of a given device (secondary objective S3) will be addressed by observing and noting the weight and size of the device and its required support equipment as well as how easily the device is set up for use during the demonstration.

- The durability of a given device (secondary objective S4) will be addressed by noting the materials of construction of the device and its required support equipment. Also, Tetra Tech will note any device failures or any repairs that may be necessary during extended use of the device. Any downtime required to make device repairs during the demonstration will be noted.

- The availability of a given device and associated spare parts (secondary objective S5) will be addressed by discussing the availability of the replacement device with the developer and determining whether spare parts are available in retail stores or only from the developer. In addition, if the replacement device or spare parts are required during the demonstration, their availability will be noted.

Demonstration field and laboratory data will be collected to address the demonstration objectives. The procedures that will be used to analyze these data are presented in Section 4.3. Critical and noncritical measurement are identified in Chapter 7. Specific procedures for collecting samples and making measurements to address the objectives as well as the numbers of samples to be collected and the types of analyses to be completed are also presented in Chapter 7.

4.3 Data Analysis Procedures

As discussed in Section 4.2, demonstration field and laboratory data will be used to meet the specific project objectives discussed in Section 4.1. Section 4.3 presents and describes data analysis procedures that will be used to address each primary objective as well as cost categories that will be assessed as part of the field measurement device cost analysis. Depending on the primary objective involved, some data will be analyzed using statistical approaches such as hypothesis testing, some data will be analyzed using simple descriptive parameters such as means and standard deviations, and other data will be used without use of statistical procedures. Data analysis will be completed separately for field measurement devices and the reference method unless otherwise noted below. A given field measurement device's TPH results will be compared only to the reference method TPH results; one field measurement device's TPH results will not be compared to another's. This section describes the data analysis procedures that will be used to address the primary objectives only. Secondary objectives are not discussed in this section because these objectives do not involve quantitative data analysis.

The statistical hypothesis testing procedures described in this section are based on the assumption that the data will be normally distributed. This assumption will be verified using the Wilk-Shapiro test. If the normality assumption is not satisfied, a nonparametric test such as the Wilcoxon signed rank test will be used for data analysis. Statistical software such as *Statistix* will be used to perform all statistical calculations for data analysis.

4.3.1 Primary Objective P1: Method Detection Limit

To determine the MDLs for GRO and EDRO for each field measurement device under primary objective P1, Tetra Tech will use each device's TPH results for seven low-level GRO and seven low-level EDRO soil PE samples to calculate the variance and standard deviation for the seven replicate samples using Equations 4-1 and 4-2.

$$S^2 = \frac{1}{n-1}\left[\sum_{i=1}^{n} x_1^2 - \frac{\left(\sum_{i=1}^{n} x_i\right)^2}{n}\right] \tag{4-1}$$

$$S = \sqrt{S^2} \tag{4-2}$$

where

S^2 = Variance of replicate TPH results

n = Number of sample replicates

x_i = TPH result in mg/kg on a wet weight basis for the ith sample

S = Standard deviation of replicate TPH results

The Student's t value and standard deviation will be used to calculate the MDL as shown in Equation 4-3.

$$\mathrm{MDL} = t_{(n-1,1-\infty=0.99)}(S) \tag{4-3}$$

where

$t_{(n-1,\ 1-\infty=0.99)}$ = Student's t value appropriate for a 99 percent confidence level and a standard deviation estimate with n-1 degrees of freedom (3.143 for n = 7)

Tetra Tech will follow the same procedure to determine the MDLs achieved by the reference laboratory for the reference method. However, because all GRO samples will be analyzed for both GRO (C_6 through C_{10}) and EDRO (C_{10} through C_{40}) and the reference laboratory will report EDRO analytical results for the diesel range (C_{10} through C_{28}) and the extended diesel range (C_{28} through C_{40}) separately, the TPH result for a given sample will be calculated by summing the three values reported by the reference laboratory. After calculating the seven TPH results, Tetra Tech will use Equations 4-1 through 4-3 to determine the MDL. For each sample that contains only EDRO, the TPH result will be calculated using the two values reported by the reference laboratory (for C_{10} through C_{28} and C_{28} through C_{40}) instead of three values. To avoid potential confusion regarding which GRO value should be added to which EDRO value, each replicate will be uniquely identified by the Tetra Tech field team.

The procedure described above is based on 40 *Code of Federal Regulations* (CFR) Part 136, Appendix B, Revision 1.11. This procedure appears to be based on an assumption that the replicates used are homogeneous enough to allow proper measurement of the analytical precision, which will be true for the demonstration because of the PE sample preparation procedures that will be followed. If the replicates used are not subsamples of a homogenous matrix, the resulting high sample variability may not allow proper measurement of TPH analytical precision and subsequent determination of MDLs.

For field measurement devices that cannot provide quantitative measurements, such as SDI's EnSys Petro Test System, MDLs cannot be determined using the statistical procedure described above. Therefore, SDI will be provided with seven low-level GRO samples and asked to report how many samples contain TPH concentrations above 10 mg/kg, a value close to the MDL claimed by the developer. Similarly, SDI will be provided with seven low-level EDRO samples and asked to report how many samples contain TPH concentrations above 15 mg/kg, a value close to the MDL claimed by the developer.

4.3.2 Primary Objective P2: Accuracy and Precision

To determine whether a field measurement device can accurately and precisely measure TPH under primary objective P2, Tetra Tech will compare device TPH measurement results with reference method TPH results.

The evaluation of a field measurement device's ability to accurately measure TPH will be based on the assumption that a field measurement device may be used to (1) determine whether the TPH concentration in a given area exceeds an action level (a state or other action level) or (2) perform a preliminary characterization of soil in a given area. These scenarios are addressed below.

Sample TPH results obtained using the reference method and a given field measurement device will be compared to the action levels presented in Table 4-3 in order to determine whether a particular sample's TPH concentration is above the action level. The findings obtained using the reference method and the device will be compared to determine how many times the device's findings agreed with those of the reference method for a particular area or for all areas taken together.

To complete a preliminary characterization of soil in a given area using a field measurement device, the device user may have to demonstrate to a regulatory agency that (1) no statistically significant difference exists between the results of the laboratory method selected for the project (the reference method) and the device results, indicating that the device may be used as a substitute for the laboratory method, or (2) a consistent correlation exists between device results and laboratory method results, indicating that the device results can be adjusted using the established correlation.

Tetra Tech will evaluate whether a statistically significant difference exists between a given field measurement device's results and the reference method results by performing a two-tailed, paired, Student's t-test. The null hypothesis will be that the mean difference between the device results and the reference method results equals zero. Assuming that the data are normally distributed, Tetra Tech will perform the t-test with a 0.05 significance level using Equation 4-4.

$$t = \frac{\bar{d}}{S_d / \sqrt{n}} \tag{4-4}$$

where

t = Critical t value

$\bar{d}$ = Mean difference between TPH results of a given field measurement device and the reference method

S_d = Standard deviation of d

n = Number of samples (for a particular area or for all areas taken together)

Equations 4-5 and 4-6 will be used to calculate $\bar{d}$ and S_d, respectively.

$$\bar{d} = \frac{\sum_{i=1}^{n}\left(x_{2i} - x_{1i}\right)}{n} \tag{4-5}$$

$$S_d = \sqrt{\frac{\sum\left(d - \bar{d}\right)^2}{n - 1}} \tag{4-6}$$

where

n = Number of samples (for a particular area or for all areas taken together)

x_{2i} = TPH concentration measured in the ith sample using the reference method

x_{1i} = TPH concentration measured in the ith sample using a given field measurement device

d = Difference between TPH results of a given field measurement device and the reference method

In addition, the ratio of the TPH results of a given field measurement device to the TPH results of the reference method will be calculated. The ratio will be used to develop a frequency distribution in order to determine how many of the results of the device and the reference method are within 30 percent, within 50 percent, and outside the 50 percent window. A ratio of 0.7 to 1.3 would imply that a device result is within 30 percent of the reference method result. Similarly, a ratio of 0.5 to 1.5 would imply that a device result is within 50 percent of the reference method result. A ratio less than 0.5 or greater than 1.5 would imply that the device result is outside the 50 percent window.

To determine whether a consistent correlation exists between the TPH results of a given field measurement device and the reference method, a linear regression will be performed to estimate the square of the correlation coefficient (R^2), the slope, and the intercept of each regression equation. Separate regression equations will be developed for each demonstration area and for PE samples. The reliability of these regression equations will be tested using the F-test; the regression equation probability from the F-test will be used to evaluate whether the correlation between the TPH results of the device and the reference method is merely by chance. A low probability (5 percent or less) would suggest that the correlation is unlikely to have occurred by chance and that the device is an acceptable substitute for the reference method.

To evaluate a given field measurement device's ability to precisely measure TPH, Tetra Tech will calculate the relative standard deviation (RSD) of the reference method and device TPH results for the triplicate samples. The RSD will be calculated using Equation 4-7.

$$\text{RSD} = \frac{\text{Standard deviation}}{\text{Mean}} \times 100 \tag{4-7}$$

A low RSD indicates high precision. For a given set of replicate samples, the RSD of a given field measurement device's TPH results will be compared with that of the reference method's TPH results to determine whether the reference method is more precise than the device or vice versa. In addition, Tetra Tech will compare the action level findings of the reference method and a given field measurement device to evaluate whether the findings are consistent for a given set of replicates.

Using the TPH results for extract duplicates, Tetra Tech will calculate the analytical precision for a given field measurement device and the reference method. The relative percent difference (RPD) of the TPH results will be calculated for the device and the reference method using Equation 4-8.

$$\text{RPD} = \frac{\text{Difference between TPH results for extract duplicates}}{\text{Average TPH result for extract duplicates}} \times 100 \tag{4-8}$$

A low RPD indicates high precision. The RPD values for a given pair of extract duplicates will be compared to determine whether the reference method has higher analytical precision than a given field measurement device or vice versa.

For the field measurement device to be demonstrated by SDI, precision and accuracy will be evaluated only for the action level scenarios. SDI will be asked to report its results using the tightest concentration range possible relative to a given action level. To evaluate how broad the range is, the ratio of the upper limit to the lower limit of the range will be calculated.

4.3.3 Primary Objective P3: Effect of Interferents

To evaluate the effect of interferents on each field measurement device's ability to accurately measure TPH under primary objective P3, Tetra Tech will calculate the means and standard deviations of the TPH results for the following triplicate PE samples: reference soil, reference soil plus TPH (GRO or EDRO), reference soil plus TPH (GRO or EDRO) plus interferent (level 1), and reference soil plus TPH (GRO or EDRO) plus interferent (level 2). Tetra Tech will review the means for each group of samples to qualitatively evaluate whether the data show any trend—that is, to evaluate whether an increase in the interferent concentration resulted in an increase or decrease in the measured TPH concentration. Tetra Tech will also perform a one-way analysis of variance to determine whether the group means are the same or different. If they are different, the analysis will also identify which means are different from one another. This analysis will be performed at a significance level of 0.05.

Tetra Tech may be able to qualitatively evaluate SDI's field measurement device in order to address P3, but this evaluation can occur only if SDI reports different TPH concentration ranges for the samples collected.

4.3.4 Primary Objective P4: Effect of Soil Moisture Content

To evaluate the effect of soil moisture content on each field measurement device's ability to accurately measure TPH under primary objective P4, Tetra Tech will calculate the means and standard deviations of the TPH results for triplicate PE samples containing GRO or EDRO at two moisture levels. Tetra Tech will compare the means to determine whether the device and reference method results were impacted by moisture—that is, to determine whether an increase in moisture resulted in an increase or decrease in the measured TPH concentration. During the comparison, Tetra Tech will consider the variability associated with each mean. For example, if the mean concentrations are within one standard deviation for a given field measurement device but are within two standard deviations for the reference method, Tetra Tech may conclude that the effect of moisture is more significant for the reference method.

Tetra Tech may be able to qualitatively evaluate SDI's field measurement device in order to address P4, but this evaluation can occur only if SDI reports different TPH concentration ranges for the samples collected.

4.3.5 Primary Objective P5: Time Required for TPH Measurement

Tetra Tech will measure the time required for each activity associated with field analysis to address primary objective P5. Activities that will be timed include field measurement device setup, sample extraction, sample analysis, and data package preparation. Sample homogenization and sample containerization will be conducted by Tetra Tech; therefore, the time required for these activities will not be included in the sample analysis time required by the developers. For the reference method, the time required for sample analysis will be the number of days that elapse from the time that the reference laboratory receives the samples to the time that Tetra Tech receives the laboratory's full data package.

4.3.6 Primary Objective P6: Costs Associated with TPH Measurement

Tetra Tech will estimate the costs associated with field analysis to address primary objective P6. The costs associated with analyzing all the investigative samples will be estimated and documented separately for each field measurement device except Wilks' Models CVH and HATR-T, for which costs will be estimated and documented jointly. The elements to be included in the cost analysis are capital, labor, supply, IDW disposal, and support equipment costs; these elements are discussed in detail in Section 4.2. The total cost to analyze all the investigative samples will be estimated for each device. For the reference method, the cost associated with TPH measurement will be equal to the analytical cost per sample multiplied by the number of samples analyzed by a given device.

4.4 Demonstration Schedule

This section discusses the overall project schedule and provides a detailed field activity schedule for the demonstration. Table 4-4 presents the overall project schedule from work plan preparation to completion of the ITVRs for the seven TPH field measurement devices and the DER. The predemonstration investigation field activities were completed in January 2000. Demonstration field activities are scheduled to occur between June 5 and 18, 2000, as discussed below. Draft ITVRs will be available for peer and developer review on March 19, 2001, and final ITVRs will be submitted to the EPA on October 1, 2001.

Table 4-5 presents the field activity schedule for the demonstration. Field mobilization activities are scheduled to be conducted on June 5, 6, and 7, 2000. Demonstration soil sampling activities are scheduled to be conducted at the demonstration sites on June 7 and 8, 2000. Soil sample homogenization, preparation, and distribution will be conducted from June 9 through 11, 2000. The developers will analyze practice samples and will meet individually

Table 4-4. Schedule for Innovative Total Petroleum Hydrocarbon Field Measurement Device Demonstration Project

Task	Start Date	Finish Date
Task 1. Project Management and Work Plan Preparation	**07/06/99**	**07/28/01**
Project management	07/06/99	07/28/01
Work plan preparation	07/06/99	08/06/99
Task 2. Technology Developer and Demonstration Site Selection	**07/19/99**	**09/30/99**
Task 3. Predemonstration Sampling and Analysis Investigation	**10/01/99**	**02/02/00**
Task 4. Demonstration Plan Preparation	**05/01/00**	**06/05/00**
EPA and developer review of draft demonstration plan	05/01/00	05/15/00
Response to comments	05/16/00	05/30/00
Final demonstration plan	05/16/00	06/05/00
Task 5. Field Demonstration	**06/05/00**	**06/18/00**
Navy BVC site preparation and mobilization	06/05/00	06/07/00
Soil sample collection at all sites	06/07/00	06/08/00
Field analysis activities	06/13/00	06/16/00
Visitors' day	06/15/00	06/15/00
Demobilization	06/17/00	06/18/00
Task 6. Laboratory Analysis	**06/13/00**	**07/31/00**
Pre-audit	01/25/00	01/26/00
Sample analysis	06/13/00	07/31/00
In-process audit	07/18/00	07/19/00
Task 7. ITVR Preparation	**06/26/00**	**10/01/01**
ITVR 1	06/26/00	10/01/01
ITVR 1, draft 1	06/26/00	10/31/00
EPA project manager review of draft 1	11/01/00	11/08/00
Draft 2	11/09/00	11/23/00
Peer and developer review of draft 2	03/19/01	04/16/01
Draft 3	04/17/01	07/25/01
EPA project manager review of draft 3	07/26/01	08/08/01
Final ITVR 1	08/09/01	10/01/01
ITVRs 2 through 4	06/26/00	10/01/01
ITVRs 2 through 4, draft 1	06/26/00	12/22/00
EPA project manager review of draft 1	12/26/00	01/04/01
Draft 2	01/05/01	01/19/01
Peer and developer review of draft 2	03/19/01	04/16/01
Draft 3	04/17/01	07/25/01
EPA project manager review of draft 3	07/26/01	08/08/01
Final ITVRs 2 through 4	08/09/01	10/01/01
ITVRs 5 through 7	06/26/00	10/01/01
ITVRs 5 through 7, draft 1	06/26/00	02/13/01
EPA project manager review of draft 1	02/14/01	02/21/01
Draft 2	02/22/01	03/16/01
Peer and developer review of draft 2	03/19/01	04/16/01
Draft 3	04/17/01	07/25/01
EPA project manager review of draft 3	07/26/01	08/08/01
Final ITVRs 5 through 7	08/09/01	10/01/01

Table 4-4. Schedule for Innovative Total Petroleum Hydrocarbon Field Measurement Device Demonstration Project (Continued)

Task	Start Date	Finish Date
Task 8. DER Preparation	**06/26/00**	**10/01/01**
DER, draft 1	06/26/00	01/15/01
EPA project manager review of draft 1	01/16/01	01/23/01
Draft 2	01/24/01	02/07/01
Peer and developer review of draft 2	03/19/01	04/16/01
Draft 3	04/17/01	07/25/01
EPA project manager review of draft 3	07/26/01	08/08/01
Final DER	08/09/01	10/01/01

Notes:

BVC = Base Ventura County
DER = Data evaluation report
EPA = U.S. Environmental Protection Agency
ITVR = Innovative technology verification report

Table 4-5. Field Activity Schedule for Demonstration

Date	Activity		Site/Area
	Morning	Afternoon	
June 5 and 6, 2000 (Monday and Tuesday)	Tetra Tech conducts field mobilization activities.		Navy BVC/PRA
June 7, 2000 (Wednesday)	Tetra Tech conducts field mobilization activities.	Tetra Tech collects soil cores in the PRA.	
	Tetra Tech collects soil cores in the B-38 and SFT Areas and ships the cores to the Navy BVC site in Port Hueneme, California.		Kelly AFB/B-38 Area PC/SFT Area
June 8, 2000 (Thursday)	Tetra Tech collects soil cores in the FFA.	Tetra Tech collects soil cores in the NEX Service Station Area.	Navy BVC/FFA and NEX Service Station Area
June 9 through 11, 2000 (Friday through Sunday)	Tetra Tech homogenizes samples collected from each area and prepares samples for distribution to developers and STL Tampa East-Tampa.		Navy BVC/PRA
June 12, 2000[a] (Monday)	Each developer analyzes practice samples. Tetra Tech and the EPA meet with each developer for about 1 hour. During this meeting, Tetra Tech observes the developer analyzing samples and answers any questions that arise. In addition, Tetra Tech ships samples (except soil PE samples for GRO analysis) to STL Tampa East for next-day delivery.		
June 13, 2000[a] (Tuesday)	All samples except soil PE samples for GRO analysis are distributed to each developer at 7:30 a.m., and field analysis activities begin.		
June 14, 2000[a] (Wednesday)	Field analysis activities continue. At a minimum, all developers complete analyses of all environmental samples that contain GRO. Tetra Tech ships the soil PE samples for GRO analysis to STL Tampa East for next-day delivery.		
June 15, 2000[a] (Thursday)	Soil PE samples for GRO analysis are distributed to each developer at 7:30 a.m. Visitors' day activities are conducted, and field analysis activities continue. (Developer participation in the visitors' day activities is voluntary.)		
June 16, 2000[a] (Friday)	Field analysis activities continue.		
June 17 and 18, 2000[a] (Saturday and Sunday)	Field analysis activities are conducted only if, because of bad weather or other unforeseen circumstances, sample analysis has not been completed on June 16, 2000. Tetra Tech conducts demobilization activities.		

Notes:

AFB = Air Force Base
BVC = Base Ventura County
EPA = U.S. Environmental Protection Agency
FFA = Fuel Farm Area
GRO = Gasoline range organics
NEX = Naval Exchange
PC = Petroleum company
PE = Performance evaluation
PRA = Phytoremediation Area
SFT = Slop Fill Tank
STL Tampa East = Severn Trent Laboratories in Tampa, Florida
Tetra Tech = Tetra Tech EM Inc.

[a] The workday will begin at 7:30 a.m. and end at 5:30 p.m. to comply with Navy BVC site work guidelines.

with Tetra Tech and the EPA on June 12, 2000. Field analysis activities will be conducted from June 13 through 16, 2000. Visitors' day activities will be conducted in the PRA on June 15, 2000. Demobilization activities will be completed over a 2-day period immediately following completion of field analysis activities at the Navy BVC site.

Chapter 5
Confirmatory Process

The performance results for each innovative TPH field measurement device will be compared to the performance results for an off-site laboratory measurement method—that is, a reference method. This chapter describes the rationale for the selection of the reference method (Section 5.1) and the reference laboratory (Section 5.2).

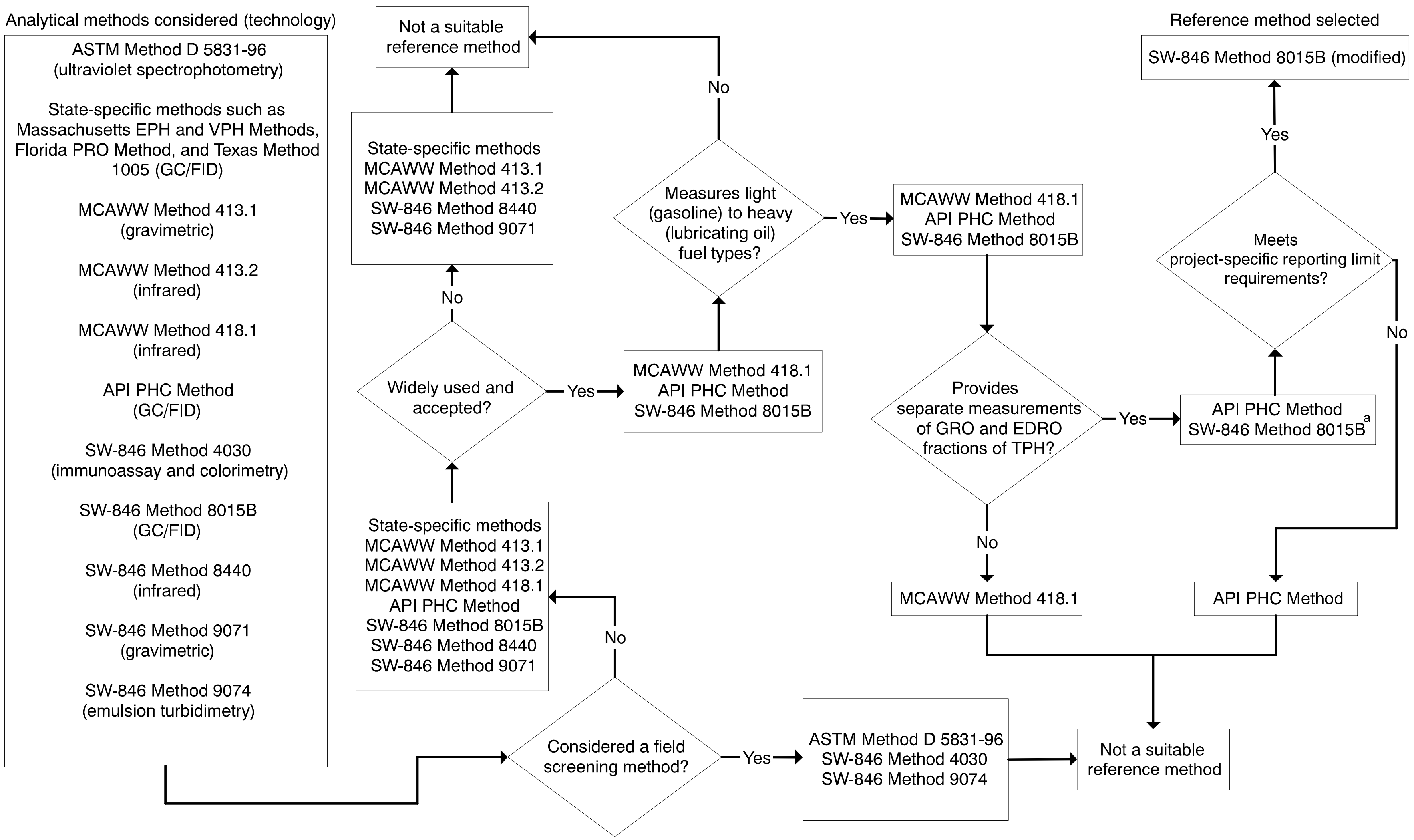
5.1 Reference Method Selection

During the demonstration, environmental and PE samples will be analyzed for TPH by the reference laboratory using SW-846 Method 8015B (modified). This section describes the analytical methods considered for the demonstration and provides a rationale for the reference method selected. Project-specific procedures for sample preparation and analysis are summarized in Chapter 9.

The reference method to be used was selected based on the following criteria:

- It is not a field screening method.
- It is widely used and accepted.
- It measures light (gasoline) to heavy (lubricating oil) fuel types.
- It can provide separate measurements of GRO (C_6 through C_{10}) and EDRO (greater than C_{10} through C_{40}) fractions of TPH.
- It meets project-specific reporting limit requirements.

The analytical methods considered for the demonstration and the reference method selected based on the above-listed criteria are illustrated in a flow diagram in Figure 5-1. The reference method selection process is briefly discussed below.



Notes:

API = American Petroleum Institute, ASTM = American Society for Testing and Materials, C = carbon, DRO = diesel range organics (greater than C_{10} through C_{28}), EDRO = extended diesel range organics (greater than C_{10} through C_{40}), EPH = extractable petroleum hydrocarbon, GC/FID = gas chromatograph/flame ionization detector, GRO = gasoline range organics (C_6 through C_{10}), MCAWW = "Methods for Chemical Analysis of Water and Wastes," PHC = petroleum hydrocarbon, PRO = petroleum range organics, SW-846 = "Test Methods for Evaluating Solid Waste," TPH = total petroleum hydrocarbons, VPH = volatile petroleum hydrocarbon

[a] SW-846 Method 8015B provides separate GRO and DRO measurements and, when modified, can also provide EDRO measurements.

Figure 5-1. Reference method selection process.

The analytical methods considered for the demonstration were identified based on a review of SW-846, MCAWW, American Society for Testing and Materials (ASTM), API, and state-specific methods and collectively represent six different measurement technologies. Of the methods considered, those identified as field screening methods, such as SW-846 Method 4030, were eliminated from further consideration in the reference method selection process.

A literature review was conducted to determine whether the remaining methods are widely used and accepted in the United States (Association for Environmental Health and Sciences [AEHS] 1999). As a result of this review, state-specific methods such as the Massachusetts Extractable Petroleum Hydrocarbon (EPH) and Volatile Petroleum Hydrocarbon (VPH) Methods (Massachusetts Department of Environmental Protection 2000), the Florida Petroleum Range Organic (PRO) Method (Florida Department of Environmental Protection 1996), and Texas Method 1005 (Texas Natural Resource Conservation Commission 2000) were eliminated from the selection process. Also eliminated were the gravimetric and infrared methods except for MCAWW Method 418.1 (EPA 1983). The use and acceptability of MCAWW Method 418.1 will likely decline because the extraction solvent used in this method is a CFC (Freon 113), and use of CFCs will eventually be phased out under the Montreal Protocol. However, because several states still accept the use of MCAWW Method 418.1 for measuring TPH, the method was retained for further consideration in the selection process (AEHS 1999).

Of the remaining methods, MCAWW Method 418.1, the API PHC Method, and SW-846 Method 8015B can measure light (gasoline) to heavy (lubricating oil) fuel types. However, GRO and EDRO fractions cannot be measured separately using MCAWW Method 418.1. As a result, this method was eliminated from the selection process.

Both the API PHC Method and SW-846 Method 8015B can be used to separately measure the GRO (C_6 through C_{10}) and DRO (greater than C_{10} through C_{28}) fractions of TPH. These methods can also be modified to extend the DRO range to EDRO (greater than C_{10} through C_{40}) by using a calibration standard that includes even-numbered alkanes in the EDRO range.

Based on a review of state-specific action levels for TPH, a TPH reporting limit of 10 mg/kg is considered to be appropriate for the demonstration. Because the TPH reporting limit for the API PHC Method (50 to 100 mg/kg) is greater than 10 mg/kg, this method was eliminated from the selection process (API 1994). SW-846 Method 8015B (modified) can meet the reporting limit requirements for the demonstration. For GRO, SW-846 Method 8015B (modified) has a reporting limit of 5 mg/kg, and for EDRO, this method has a reporting limit of 10 mg/kg. Therefore, SW-846 Method 8015B (modified) satisfies all the criteria established for selecting the reference method. In addition, this method provides a fingerprint (chromatograph) of TPH components.

Several states, including Massachusetts, Alaska, Louisiana, and North Carolina, have implemented or are planning to implement a TPH contamination fractionation approach based on the aliphatic and aromatic hydrocarbon fractions of TPH. The approach used in these states involves performing a sample extract fractionation procedure and two analyses to determine the aliphatic and aromatic hydrocarbon concentrations in a sample. However, the ability of an analytical method to determine aliphatic and aromatic hydrocarbon concentrations was not considered in the reference method selection process because

- The approach is used in only a few states.
- Variations exist among the sample extract fractionation and analysis procedures used in different states.
- The repeatability and versatility of sample extract fractionation and analysis procedures are not well documented.
- In some of the above-mentioned states, TPH-based action levels are still used.

5.2 Reference Laboratory Selection

This section provides the rationale for the selection of the reference laboratory. STL Tampa East was selected as the reference laboratory because it (1) has been performing TPH analyses for many years, (2) has passed many external audits by successfully implementing a variety of TPH analytical methods, and (3) agreed to implement project-specific analytical requirements. In January 2000, a project-specific audit of the laboratory was conducted and determined that STL Tampa East satisfactorily implemented the reference method during the predemonstration investigation. In addition, STL Tampa East successfully analyzed double-blind PE samples and blind field triplicates for GRO and EDRO during the predemonstration investigation. Furthermore, in 1998 STL Tampa East was one of four recipients and in 1999 was one of six recipients of the Seal of Excellence Award issued by the American Council of Independent Laboratories. In each instance, this award was issued based on the results of PE sample analyses and client satisfaction surveys. Thus, the selection of the reference laboratory was based primarily on performance and not cost.

Chapter 6
Demonstration Organization and Responsibilities

This chapter identifies key project personnel and summarizes their demonstration responsibilities. Figure 6-1 is an organizational chart that shows key project personnel and the lines of communication among them. Table 6-1 at the end of this chapter presents the key demonstration participants.

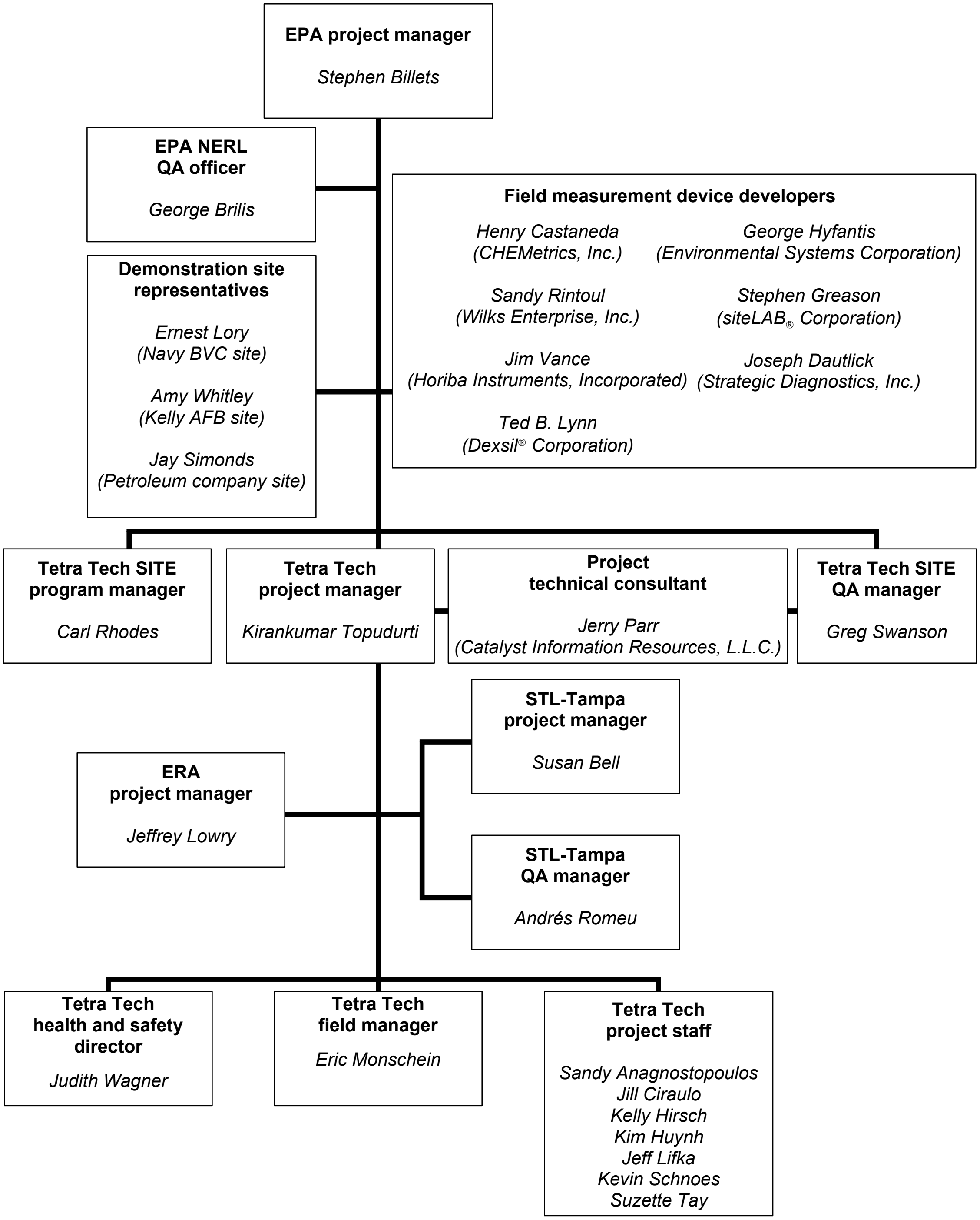
During the demonstration, the organizations identified below may choose to follow the Tetra Tech health and safety procedures identified in Chapter 13. However, each organization is directly and fully responsible for the health and safety of its own employees; Tetra Tech assumes no such responsibility for non-Tetra Tech personnel.

6.1 EPA Project Personnel

The EPA project manager, Stephen Billets, has overall responsibility for the project. Dr. Billets will review and concur with the project deliverables, including the demonstration plan, ITVRs, and DER. The EPA QA officer at the EPA NERL, George Brilis, is also responsible for reviewing and concurring with the demonstration plan.

6.2 Tetra Tech Project Personnel

The Tetra Tech project manager, Kirankumar Topudurti, is responsible for conducting day-to-day management of Tetra Tech project personnel, maintaining direct communication with the EPA and the developers, and ensuring that all Tetra Tech personnel involved in the demonstration understand and comply with the demonstration plan. Dr. Topudurti is also responsible for distributing the draft and final demonstration plans to all key project personnel and for reviewing measurement and analytical data obtained during the demonstration. Tetra Tech project personnel will assist Dr. Topudurti in preparing project deliverables and in performing day-to-day project activities. In consultation with the EPA, Tetra Tech project personnel are responsible for the following elements of the demonstration:



Notes:

AFB = Air Force Base
BVC = Base Ventura County
EPA = U.S. Environmental Protection Agency
ERA = Environmental Resource Associates
NERL = National Exposure Research Laboratory

QA = Quality assurance
SITE = Superfund Innovative Technology Evaluation
STL Tampa East = Severn Trent Laboratories in Tampa, Florida
Tetra Tech = Tetra Tech EM Inc.

Figure 6-1. Organizational chart.

- Developing and implementing all elements of this demonstration plan
- Scheduling and coordinating the activities of all demonstration participants
- Performing on-site sample collection in the five demonstration areas
- Coordinating the PE sample preparation by ERA
- Performing sample preparation at the Navy BVC site for the developers and STL Tampa East
- Overseeing operation of the innovative TPH field measurement devices and documenting the operation of each device during the demonstration
- Coordinating meetings among the EPA, the developers, STL Tampa East, and the site representatives
- Summarizing, evaluating, interpreting, and documenting demonstration data for inclusion in the ITVRs and DER
- Evaluating and reporting on the performance and cost of each device
- Preparing draft, final, and camera-ready versions of seven ITVRs (one for each device)
- Preparing draft and final versions of the DER

The project technical consultant, Jerry Parr of Catalyst Information Resources, L.L.C. (Catalyst), will support the Tetra Tech project manager and will provide technical assistance and guidance throughout all phases of the project. Tetra Tech subcontracted Mr. Parr specifically for this project because of his extensive experience in the area of PHC measurement technologies. Mr. Parr's specific responsibilities include the following:

- Providing technical assistance and guidance during development of the demonstration plan
- Auditing field and laboratory operations to determine whether the operations are properly performed
- Providing technical reviews of the demonstration plan, ITVRs, DER, and other project documents
- Providing technical assistance and guidance throughout all phases of the project

The Tetra Tech field manager, Eric Monschein, is responsible for day-to-day field operations and for reporting to the Tetra Tech project manager. Mr. Monschein will monitor sample preparation and analysis activities to ensure that procedures set forth in the demonstration plan are followed. Mr. Monschein will also ensure that chain-of-custody procedures and applicable U.S. Department of Transportation (DOT) shipping regulations are followed for sample shipment to STL Tampa East. Specific responsibilities of the Tetra Tech field manager include the following:

- Managing field staffing and mobilization activities

- Overseeing and performing sample collection and preparation activities
- Overseeing sample analysis activities
- Overseeing the activities of project personnel in the field
- Overseeing operation of the innovative TPH field measurement devices and documenting the operation of each device during the demonstration
- Providing required planning, scheduling, cost control, documentation, and data management for field activities
- Managing demobilization activities, including IDW disposal, as required by the demonstration site representatives
- Immediately communicating any deviation from the demonstration plan during field activities to the Tetra Tech project manager and discussing appropriate resolutions of the deviation

Tetra Tech project personnel will assist Mr. Monschein in conducting day-to-day field activities during the demonstration, including sample collection, sample preparation, measurement of time associated with TPH field measurements, and oversight of TPH field measurements.

At the Navy BVC site, Mr. Monschein will oversee sample collection activities in the FFA and NEX Service Station Area. In these areas, sample collection using a Geoprobe® will be conducted by Tetra Tech's subcontractor. In the PRA at the Navy BVC site, Mr. Monschein or other Tetra Tech project personnel will perform split core sample collection activities. The samples collected in the three areas at the Navy BVC site will be transferred by Mr. Monschein to the sample management trailer in the PRA. At the Kelly AFB site, Jill Ciraulo of Tetra Tech will oversee sample collection activities and perform sample shipment to the Navy BVC site. At the Kelly AFB site, sample collection using a Geoprobe® will be conducted by Tetra Tech's subcontractor. At the PC site, Suzette Tay of Tetra Tech will oversee sample collection activities and perform sample shipment to the Navy BVC site. At the PC site, sample collection using a Geoprobe® will be conducted by a subcontractor to the demonstration site representative.

Kevin Schnoes of Tetra Tech will cut open the soil sample core tube liners and perform soil classification activities at the sample management trailer in the PRA. Tetra Tech's health and safety representative (HSR), Judith Wagner, will review the site-specific health and safety procedures, and she or her designee will audit field procedures during the demonstration to ensure compliance with the health and safety procedures presented in Chapter 13 of the demonstration plan.

Tetra Tech's SITE program manager, Carl Rhodes, is responsible for project review and for allocating Tetra Tech resources. The Tetra Tech SITE QA manager, Greg Swanson, is responsible for overall project QA. Dr. Swanson will be available to resolve any project-specific QA issues.

6.3 Developer Personnel

The developers of the seven field measurement devices are responsible for providing, mobilizing, operating, and demobilizing their respective devices. The developer responsibilities include the following:

- Providing Tetra Tech with information on the devices
- Reviewing and concurring with the demonstration plan pertaining to the devices
- Notifying Tetra Tech in writing of device-specific requirements, such as the type of power supply and the amount of work space needed, so that proper arrangements can be made for field demonstration of the devices
- Providing the personnel and all supplies needed for demonstration of the devices unless otherwise arranged in advance with Tetra Tech
- Analyzing the samples specified in the demonstration plan
- Analyzing appropriate QC samples (for example, blanks or standards) in accordance with the developer specifications
- Providing device-specific demonstration results to Tetra Tech at the end of the demonstration
- Reviewing and commenting on the device-specific ITVRs

6.4 Demonstration Site Representatives

The representatives for the three demonstration sites are Ernest Lory for the Navy BVC site, Amy Whitley for the Kelly AFB site, and Jay Simonds for the PC site. All work performed at each demonstration site will be scheduled, coordinated, and conducted with the permission of the individual demonstration site representative, who will be the primary contact for the Tetra Tech project manager. The demonstration site representatives are responsible for obtaining the site access and permission necessary for performing sampling activities at the sites. The demonstration site representatives are also responsible for reviewing and concurring with the demonstration plan.

At the Navy BVC site, Mr. Lory's specific responsibilities include the following:

- Providing Tetra Tech with background information on the three demonstration areas
- Providing logistical support for conducting visitors' day activities, which will include providing a meeting room and transporting visitors between the meeting room and PRA, if necessary
- Preparing the sample management trailer area and providing utilities, a power source when necessary, work space with tables and chairs, distilled water for field blanks, and logistical support needed for the demonstration
- Providing access to the sample management trailer in the PRA

At the Kelly AFB site, Ms. Whitley's specific responsibilities include the following:

- Providing Tetra Tech with background information the B-38 Area
- Providing Tetra Tech with access to the B-38 Area

At the PC site, Mr. Simonds' specific responsibilities include the following:

- Providing Tetra Tech with background information on the SFT Area
- Procuring a Geoprobe® subcontractor to collect soil core samples
- Providing Tetra Tech with the soil core samples
- Providing Tetra Tech with access to the SFT Area

6.5 Laboratory Project Personnel

The reference laboratory for the project, STL Tampa East, will perform laboratory analyses of environmental and PE samples provided by Tetra Tech during the demonstration. The STL Tampa East project manager, Susan Bell, is responsible for overall planning, scheduling, budgeting, and reporting of laboratory activities. All STL Tampa East work will be conducted under the direct supervision of Ms. Bell, who will be the primary contact for the Tetra Tech project manager. Ms. Bell is also responsible for reviewing and concurring with the demonstration plan, and she will immediately discuss appropriate resolutions of any deviation from the STL Tampa East activities specified in the plan with the Tetra Tech project manager. STL Tampa East's QA manager, Andrés Romeu, will assist Ms. Bell in ensuring adherence to all QA/QC elements specified in the demonstration plan that pertain to the analyses performed at their laboratory.

ERA will prepare all soil and liquid PE samples requested by Tetra Tech and will ship them to the Navy BVC site. The ERA project manager, Jeffrey Lowry, is responsible for all planning, scheduling, and budgeting of laboratory activities and for preparation of PE samples. All PE sample preparation work will be conducted under the direct supervision of Mr. Lowry, who will be the primary contact for the Tetra Tech project manager.

Table 6-1. Demonstration Participants

Organization	Participant	Contact Information
U.S. Environmental Protection Agency	Dr. Stephen Billets Mr. George Brilis	National Exposure Research Laboratory 944 East Harmon Avenue Las Vegas, NV 89119 Telephone: (702) 798-2232 Fax: (702) 798-2261 E-mail: billets.stephen@epamail.epa.gov
Tetra Tech EM Inc.	Dr. Kirankumar Topudurti Mr. Eric Monschein Ms. Sandy Anagnostopoulos Ms. Jill Ciraulo Ms. Kelly Hirsch Ms. Kim Huynh Mr. Jeff Lifka Mr. Kevin Schnoes Ms. Suzette Tay	200 East Randolph Drive, Suite 4700 Chicago, IL 60601 Telephone: (312) 856-8700 Fax: (312) 938-0118 E-mail: topuduk@ttemi.com
	Ms. Judith Wagner	3550 Salt Creek Lane, Suite 105 Arlington Heights, IL 60005 Telephone: (847) 255-4166 Fax: (847) 255-8528 E-mail: wagnerj@ttemi.com
	Mr. Carl Rhodes	250 West Court Street, Suite 200 W Cincinnati, OH 45202 Telephone: (513) 241-0149 Fax: (513) 241-0354 E-mail: rhodesc@ttemi.com
	Dr. Greg Swanson	591 Camino de la Reina, Suite 640 San Diego, CA 92108 Telephone: (619) 718-9676 Fax: (619) 718-9698 E-mail: swansog@ttemi.com
Catalyst Information Resources, L.L.C.	Mr. Jerry Parr	1153 Bergen Parkway, # 238 Evergreen, CO 80439 Telephone: (303) 670-7823 Fax: (303) 670-2964 E-mail: catalyst@eazy.net
Navy Base Ventura County	Mr. Ernest Lory	NFESC 1100 23rd Avenue Port Hueneme, CA 93043 Telephone: (805) 982-1299 Fax: (805) 982-4304 e-mail: loryee@nfesc.navy.mil
Kelly Air Force Base	Ms. Amy Whitley	SA-ALC/EMRR 307 Tinker Drive (Bldg. 306) Kelly AFB, TX 78241 Telephone: (210) 925-3100 (Ext. 214) Fax: (210) 925-1814 e-mail: amy.whitley@kelly.af.mil
Handex of Indiana	Mr. Jay Simonds	8579 Zionsville Road Indianapolis, IN 46268 Telephone: (317) 228-6240 Fax: (317) 228-6243 e-mail: jsimonds@handexmail.com
CHEMetrics	Mr. Henry Castaneda	CHEMetrics, Inc. Route 28 Calverton, VA 20138 Telephone: (800) 356-3072 Fax: (540) 788-4856 E-mail: henryc@chemetrics.com

Table 6-1. Demonstration Participants (Continued)

Organization	Participant	Contact Information
Wilks	Ms. Sandy Rintoul	Wilks Enterprise, Inc. 345 Riverview Drive Boulder Creek, CA 95006 Telephone: (831) 338-7459 Fax: (831) 338-3393 E-mail: srintoul@dellnet.com
Horiba	Mr. Jim Vance	Horiba Instruments, Incorporated 17671 Armstrong Avenue Irvine, CA 92614 Telephone: (800) 4HORIBA, ext. 170 Fax: (949) 250-0924 E-mail: jim.vance@horiba.com
Dexsil®	Dr. Ted B. Lynn	Dexsil® Corporation One Hamden Park Drive Hamden, CT 06517 Telephone: (203) 288-3509 Fax: (203) 248-6523 E-mail: tblynn@dexsil.com
ESC	Dr. George Hyfantis	Environmental Systems Corporation 200 Tech Center Drive Knoxville, TN 37912 Telephone: (865) 688-7900 Fax: (865) 687-8977 E-mail: ghyfantis@envirosys.com
siteLAB®	Mr. Stephen Greason	siteLAB® Corporation 94 Highland Street Portsmouth, NH 03801 Telephone: (877) SITELAB Fax: (603) 436-9008 E-mail: sgreason@site-lab.com
SDI	Mr. Joseph Dautlick	Strategic Diagnostics, Inc. 111 Pencader Drive Newark, DE 19702 Telephone: (800) 544-8881, ext. 222 Fax: (302) 456-6770 E-mail: jdautlick@sdix.com
Severn Trent Laboratories	Ms. Susan Bell Mr. Andrés Romeu	5910 Breckenridge Parkway, Suite H Tampa, FL 33610 Telephone: (813) 621-0784 Fax: (813) 623-6021 E-mail: bellsu@quanterra.com
Environmental Resource Associates	Mr. Jeffrey Lowry	5540 Marshall Street Arvada, CO 80002 Telephone: (800) 372-0122 Fax: (303) 421-0159 E-mail: jlowry@eraqc.com

Chapter 7
Field Sampling Procedures

This chapter discusses the field sampling procedures to be used during the demonstration. Specifically, this chapter addresses the sampling procedures (Section 7.1), sample handling and shipping procedures (Section 7.2), sampling equipment decontamination and IDW disposal procedures (Section 7.3), and field documentation procedures (Section 7.4) to be used during the demonstration.

The critical and noncritical measurements for the demonstration are summarized in Table 7-1. Critical measurements will be used to address primary objectives. Although critical measurements generally involve a greater level of QC than noncritical measurements, several critical measurements for the demonstration do not have significant QC procedures. For example, noting the number of technicians required to operate an innovative TPH field measurement device is considered to be a critical measurement because the number of technicians required is associated with estimating the time and cost requirements for each field measurement activity (primary objectives P5 and P6). However, the number of technicians will be noted only once for each device during the demonstration because the number is not likely to change or to be subject to measurement error. Noncritical measurements will provide additional information regarding the devices but are not critical to addressing the primary objectives. A number of demonstration measurements, such as those for sampling locations and depth intervals, are classified as noncritical because these measurements were made during the predemonstration investigation and because it is adequate if the demonstration sampling locations and depth intervals are within 1 to 2 feet of the predemonstration investigation sampling locations and depth intervals. The demonstration will take advantage of as much predemonstration investigation data as possible. Because of the qualitative nature of the demonstration's secondary objectives, measurement and sampling activities will not be conducted primarily to address secondary objectives.

Table 7-1. Critical and Noncritical Measurements

Measurement	Critical or Noncritical Measurement	Rationale (Primary or Secondary Objective)
Measurements Made Specifically During Sample Collection		
Sampling locations and depth intervals	Noncritical	Collect samples at locations and depth intervals close to those of predemonstration investigation
Water level measurements at Kelly AFB, B-38 Area	Noncritical	Locate soil sampling depth intervals 2 feet above and 2 feet below the water table
Photoionization detector measurements at Kelly AFB, B-38 Area	Noncritical	Characterize soil sample concentrations as nondetect, low, medium, or high
Measurements Made During Demonstration as a Whole		
STL Tampa East and developer TPH measurements	Critical	Evaluate method detection limits (P1), precision and accuracy (P2), effect of interferents (P3), and effect of moisture (P4)
Percent moisture for soil samples	Critical	Document moisture content of samples (P4)
Number of technicians required	Critical	Estimate time and cost associated with each field measurement activity (P5 and P6)
Time required for sample analysis activities	Critical	Estimate time and cost associated with each field measurement activity (P5 and P6)
Volume of investigation-derived waste generated	Critical	Estimate cost associated with each field measurement activity (P6)
Extra items required to perform analysis	Critical	Estimate cost associated with each field measurement activity (P6)
Personal protective equipment required	Noncritical	Document health and safety concerns associated with operating each device (S2)
Support facilities required	Noncritical	Document power sources and amount of work space required (S5)
Time required for homogenization of environmental samples	Noncritical	Document sample preparation procedures
Physical soil classification using USCS	Noncritical	Characterize environmental samples for physical parameters (for example, sand, silt, and clay content)

Notes:

AFB = Air Force Base
STL Tampa East = Severn Trent Laboratories in Tampa, Florida
TPH = Total petroleum hydrocarbons
USCS = Unified Soil Classification System

7.1 Sampling Procedures

The demonstration will involve both environmental and PE samples. Environmental samples will be collected by Tetra Tech or its subcontractor in core tube liners, sectioned as appropriate to represent approximate depth intervals at each sampling location, homogenized, containerized, and labeled. Homogenous PE samples will be prepared, containerized, and shipped to Tetra Tech at the Navy BVC site by ERA in Arvada, Colorado. Tetra Tech will not open any of the PE sample containers but will assign them sample identification numbers and distribute them to STL Tampa East and the developers.

For the demonstration, environmental samples will be collected in the areas that were used for the January 2000 predemonstration investigation: (1) the FFA, NEX Service Station Area, and PRA at the Navy BVC site; (2) the B-38 Area at the Kelly AFB site; and (3) the SFT Area at the PC site. These areas are shown in Figures 3-2, 3-4, and 3-5 of Chapter 3. Samples will be collected in all areas except the PRA using a subcontractor-operated Geoprobe®; in the PRA, samples will be collected by Tetra Tech personnel using a Split Core Sampler. Where a subcontractor-operated Geoprobe® is used, Tetra Tech personnel will be present to supervise sample collection and to determine whether enough sample material is collected in a given depth interval. Except in the PRA, after sampling is completed at a location, the boring will be grouted with bentonite to the ground surface. In the PRA, planter soil will be used to fill the borings.

All samples will be managed at the sample management trailer in the PRA at the Navy BVC site. Samples collected at the Kelly AFB and PC sites will be shipped in core tube liners to the sample management trailer, where Tetra Tech will section the core tube liners as needed, record soil characteristics using the Unified Soil Classification System (USCS), homogenize the sample material collected from a given depth interval at a particular sampling location, and prepare samples for shipment to STL Tampa East and for transfer to the developers on site. Sample collection, handling, and shipping procedures to be used at each of the three demonstration sites are detailed in Sections 7.1.1 and 7.1.2. Sample preparation procedures are detailed in Section 7.1.3.

Tetra Tech is aware that some of the sample collection and homogenization procedures to be used may result in significant loss of GRO from the samples. Specifically, the core tube liners used to transport samples to the Navy BVC site and the homogenization techniques described in Section 7.1.3 may result in GRO losses of up to one order of magnitude. Despite these losses, based on observations during the predemonstration investigation, the demonstration samples are expected to contain adequate concentrations of GRO after homogenization. Moreover, the homogenization procedures will ensure that STL Tampa East and the developers receive the same sample material for analysis, which is necessary to perform a meaningful comparison of the reference method and device analytical results.

7.1.1 Sample Collection

This section describes environmental sample collection activities to be performed at the three demonstration sites. Homogenous PE samples will be prepared, containerized, and shipped to Tetra Tech at the Navy BVC site by ERA in Arvada, Colorado. Tetra Tech will not open any of the PE sample containers but will assign them sample identification numbers and distribute them to STL Tampa East and the developers.

During the demonstration, Tetra Tech will collect samples from locations and depth intervals immediately adjacent to the predemonstration investigation sampling locations and depth intervals with a few exceptions; based on predemonstration investigation results, some of the sampling locations and depth intervals have been modified for the demonstration. To allow later identification of the predemonstration investigation sampling locations at each site, Tetra Tech marked them with either metal or plastic stakes. Also, Tetra Tech's field logbooks contain sketches of the sampling locations and measurements of each location with respect to a permanent reference point; these measurements were made using a measuring tape and compass.

During the predemonstration investigation, Tetra Tech found that in some sampling areas the TPH concentrations were highest at or near the water table because of contamination floating on the groundwater surface. For example, based on discussions between Tetra Tech and the demonstration site representative for Kelly AFB, the contamination is expected to be highest at or near the water table. Four groundwater monitoring wells exist in the B-38 Area. Therefore, Tetra Tech will use a water-level indicator to measure the depth to groundwater in each of the monitoring wells before collecting soil samples. In addition, Tetra Tech will identify any monitoring wells or piezometers in the other sampling areas and, where possible, will measure the depth to groundwater in the areas before collecting soil samples. The measurements will be used to modify sampling depth intervals during sample collection, as necessary.

During the predemonstration investigation, the diameter of the core tube liners used and the soil depth intervals selected generally allowed collection of enough sample volume for analysis. In some cases, however, additional sample volume was needed for collection of field triplicates. The additional sample volume was obtained by advancing an additional Geoprobe® boring within 1 foot of the initial sampling location. Because all samples will be distributed to both STL Tampa East and the developers during the demonstration, and because triplicates will be collected at a frequency of one per depth interval, additional sample volume may be required. Therefore, during the demonstration, additional Geoprobe® borings may be advanced immediately adjacent to the initial sampling locations in order to obtain adequate sample volume.

When core tube liners containing samples arrived at the sample management trailer at the Navy BVC site during the predemonstration investigation, the core tube liners were visually examined, and depth intervals were identified for the liners. The depth intervals were identified based on Tetra Tech field logbook notes taken during sample collection that documented (1) the length of the Geoprobe® rods in the ground when filling of the core tube liners started and (2) visual observations of staining or differing soil types in various depth intervals. For a given sampling location, the topmost portion of the entire length of the sample corresponded to the depth at which the core tube liner was introduced for sample collection, and the soil recovered in each core tube liner was measured using that top depth as a reference point. For example, when Tetra Tech attempted to collect a sample from 2 to 4 feet bgs and only a 1-foot length of soil was recovered in the core tube liner, the soil sample's depth interval was identified as 2 to 3 feet bgs. Tetra Tech used this approach because soil recoveries were sometimes less than the length of the core tube liners. This approach did not account for soil compression or loss of soil during sample collection. Tetra Tech will use a consistent method of identifying depth intervals for samples in each area during the demonstration.

Table 7-2 summarizes the proposed demonstration sampling depth intervals, the expected contamination types and concentrations for each interval, and the rationale for the selection of intervals based on the objectives to be addressed and the analyses to be performed by STL Tampa East. Table 7-3 summarizes the proposed demonstration sampling depth intervals, expected contamination types and concentrations, objectives to be addressed, numbers of environmental and QA/QC samples to be collected, and numbers of analyses to be performed for environmental samples. The sampling depth intervals for the demonstration have been slightly modified from those used during the predemonstration investigation in the FFA, NEX Service Station Area, and B-38 Area. An explanation of each modification is provided in the following site-specific sections. Table 7-4 summarizes the expected contamination types and concentrations, objectives to be addressed, numbers of samples and analyses, and numbers of sample containers for the PE samples that will be prepared by ERA.

Contamination types and concentration ranges shown in Tables 7-2, 7-3, and 7-4 are based on predemonstration investigation results in the case of environmental samples and discussions with ERA in the case of PE samples. In these tables, a low (L) concentration corresponds to a TPH value of less than 100 mg/kg; a medium (M) concentration corresponds to a TPH range of 100 to 1,000 mg/kg; and a high (H) concentration corresponds to a TPH value of more than 1,000 mg/kg. The concentration information should be used only as a guide to determine appropriate dilutions for sample analysis. Certain information, such as the specific compounds to be used as interferents in preparing the PE samples required to address primary objective P3, is purposely not included in this demonstration plan.

7.1.1.1 Navy Base Ventura County Site

This section describes the sampling locations, depth intervals, and types of sampling equipment to be used in the three demonstration areas at the Navy BVC site.

Table 7-2. Sampling Depth Intervals, Sampling Parameters, and Associated Rationale

Site	Area	Depth Interval (foot bgs)	Expected Contamination Type[a]	Expected Concentration	Rationale	
					Primary Objective to be Addressed	Analyses by STL Tampa East
Navy BVC	FFA	Upper layer[b]	EDRO	L to M	P2 (precision and accuracy) at low to medium levels	Only EDRO because these samples will not contain GRO
		Lower layer[b]		H	P2 (precision and accuracy) at high levels	
	NEX Service Station Area	7 to 8	GRO and EDRO	L	P2 (precision and accuracy) at low levels	GRO and EDRO because these samples will likely contain both
		8 to 9		L		
		9 to 10		L to H	P2 (precision and accuracy) at low to high levels	
		10 to 11		L to M	P2 (precision and accuracy) at low to medium levels	
	PRA	1.5 to 2.5	EDRO	H	P2 (precision and accuracy) at high levels	Only EDRO because these samples will not contain GRO
Kelly AFB	B-38 Area	18 to 20	GRO and EDRO	L	P2 (precision and accuracy) at low levels	GRO and EDRO because these samples will likely contain both
		20 to 22		L		
PC	SFT Area	2 to 4	GRO and EDRO	L to H	P2 (precision and accuracy) at low to high levels	GRO and EDRO because these samples will likely contain both
		4 to 6		M to H	P2 (precision and accuracy) at medium to high levels	
		6 to 8		L to M	P2 (precision and accuracy) at low to medium levels	
		8 to 10		L to M	P2 (precision and accuracy) at low to medium levels	
Reference soil 1 (PE sample)			GRO	L	P1 (MDL) and P2 (precision and accuracy) at low levels	GRO and EDRO because the TPH source used for GRO spiking will likely contain some EDRO
Reference soil 2 (PE sample)			GRO	M	P2 (precision and accuracy) at medium levels	
			GRO	H	P2 (precision and accuracy) at high levels Control for P3 (interferents) and low levels for P4 (moisture)	
Reference soil 1 (PE sample)			EDRO	L	P1 (MDL) and P2 (precision and accuracy) at low levels	Only EDRO because these samples will not contain GRO
Reference soil 2 (PE sample)			EDRO	M	P2 (precision and accuracy) at medium levels	
			EDRO	H	P2 (precision and accuracy) at high levels Control for P3 (interferents) and high levels for P4 (moisture)	
			GRO (trace levels) EDRO (low levels)	L	Control sample for P3 (interferents)	GRO and EDRO because, based on predemonstration investigation results, reference soil 2 will contain trace levels of GRO and low levels of EDRO
			I(6)	M to H	Control samples for P3 (interferents)	Only EDRO because these samples will not contain GRO
			I(1) + GRO	H	P3 (interferents) at two interferent levels for GRO	GRO and EDRO because the TPH source used for GRO spiking will likely contain some EDRO
			I(2) + GRO	H		
Reference soil 2 (PE sample) (continued)			I(3) + GRO	H		
			I(4) + GRO	H		

Table 7-2. Sampling Depth Intervals, Sampling Parameters, and Associated Rationale (Continued)

Site	Area	Depth Interval (foot bgs)	Expected Contamination Type^a	Expected Concentration	Rationale	
					Primary Objective to be Addressed	Analyses by STL Tampa East
			I(3) + EDRO	H	P3 (interferents) at two interferent levels for EDRO	GRO and EDRO because these interferents will likely contain both
			I(4) + EDRO	H		
			I(5) + EDRO	H	P3 (interferents) at two interferent levels for EDRO	Only EDRO because these samples will not contain GRO The contribution of trace levels of GRO (< 15 mg/kg) found in reference soil 2 during the predemonstration investigation is considered to be insignificant in medium- and high-level EDRO samples.
			I(6) + EDRO	H		
			GRO at 18 percent moisture	H	P4 (moisture) at high levels	GRO and EDRO because the TPH source used for GRO spiking will likely contain some EDRO
			EDRO at negligible moisture (none added)	H	P4 (moisture) at negligible moisture	Only EDRO because these samples will not contain GRO
Liquid PE samples			GRO	H	Control samples for P3 (interferents)	GRO and EDRO because the GRO source used will likely contain some EDRO
			EDRO	H		Only EDRO because this sample will not contain GRO
			I(1)	M to H	Control samples for P3 (interferents)	Only GRO only because these samples will not contain EDRO
			I(2)	M to H		
			I(3)	M to H	Control samples for P3 (interferents)	GRO and EDRO because these interferents will likely contain both
			I(4)	M to H		
			I(5)	M to H	Control samples for P3 (interferents)	Only EDRO because this sample will not contain GRO

Notes:

\> = Greater than
< = Less than
AFB = Air Force Base
bgs = Below ground surface
BVC = Base Ventura County
EDRO = Extended diesel range organics
FFA = Fuel Farm Area
GRO = Gasoline range organics
H = High (>1,000 mg/kg)
L = Low (<100 mg/kg)
M = Medium (100 to 1,000 mg/kg)
MDL = Method detection limit
mg/kg = Milligram per kilogram
NEX = Naval Exchange
PC = Petroleum company
PE = Performance evaluation
PRA = Phytoremediation Area
SFT = Slop Fill Tank
STL Tampa East = Severn Trent Laboratories in Tampa, Florida
TPH = Total petroleum hydrocarbons

Unless otherwise specified, soil PE samples will be prepared at 9 percent moisture.

Reference soil 1 is Ottawa sand, and reference soil 2 is processed garden soil (sandy silt). Reference soil 2 may contain native trace levels of GRO and low levels of EDRO.

Table 7-2. Sampling Depth Intervals, Sampling Parameters, and Associated Rationale (Continued)

Two different carriers will be used to meet the needs of individual developers. Methanol will be used as the carrier for the following devices: the PetroFLAG™ test kit, Luminoscope, UVF-3100A, and EnSys Petro Test System. Freon will be used as the carrier for the following devices: the RemediAid™ starter kit, OCMA-350, Model CVH, and Model HATR-T.

[a] The boiling points and vapor pressures of (1) interferents I(1) and I(2) have characteristics similar to those of GRO, (2) interferents I(3) and I(4) have characteristics similar to those of GRO and EDRO, and (3) interferents I(5) and I(6) have characteristics similar to those of EDRO. PE samples containing interferents I(1) and I(2) will not be prepared with EDRO because these interferents are not expected to impact EDRO analysis. Similarly, PE samples containing interferents I(5) and I(6) will not be prepared with GRO because these interferents are not expected to impact GRO analysis. In addition, practical difficulties (solubility constraints) associated with preparing the I(1) or I(2) + EDRO and I(5) or I(6) + GRO PE samples will not allow use of these samples in the demonstration. Expected concentrations for PE samples containing interferents are based on expected analytical results for the samples and not on the concentrations of interferents to be added to the samples.

[b] Because of soil conditions encountered in the FFA during the predemonstration investigation, the sampling depth intervals in this area could not be accurately determined. Sample collection was initiated approximately 10 feet bgs, and attempts were made to collect 4-foot-long soil cores. This approach resulted in varying degrees of core tube penetration up to 17 feet bgs. During the demonstration, for each sampling location in the area, each sample core will be divided into two samples based on visual observations. The upper layer of the soil core, which will likely consist of yellowish-brown, silty sand, will make up one sample, and the lower layer of the soil core, which will likely consist of grayish-black, silty sand and smell of hydrocarbons, will make up the second sample.

Table 7-3. Environmental Samples

Site	Area	Depth Interval (foot bgs)	Expected Contamination Type	Expected Concentration	Primary Objective to be Addressed	Number of Sampling Locations	Total Number of Samples, Including Field Triplicates, to Each Developer and STL Tampa East[a]	Number of MS/MSD[b] Pairs	Number of Analyses by STL Tampa East[c]		Number of Sample Containers to STL Tampa East		Total Number of 25-Gram EnCores to All Developers
									GRO	EDRO	5-Gram EnCores[d]	4-Ounce Jars[e]	
Navy BVC	FFA	Upper layer[f]	EDRO	L to M	P2	3	5	1	0	7	0	10	35
		Lower layer[f]		H	P2	3	5	1	0	7	0	10	35
	NEX Service Station Area	7 to 8	GRO and EDRO	L	P2	3	5	1	7	7	14	10	35
		8 to 9		L	P2	3	5	1	7	7	14	10	35
		9 to 10		L to H	P2	3	5	1	7	7	14	10	35
		10 to 11		L to M	P2	3	5	1	7	7	14	10	35
	PRA	1.5 to 2.5	EDRO	H	P2	6 (4 vegetated and 2 unvegetated)	8	1	0	10	0	16	40[g]
Kelly AFB	B-38 Area	18 to 20	GRO and EDRO	L	P2	4	6	1	8	8	16	12	42
		20 to 22		L	P2	4	6	1	8	8	16	12	42
PC	SFT Area	2 to 4	GRO and EDRO	L to H	P2	5	7	1	9	9	18	14	49
		4 to 6		M to H	P2	5	7	1	9	9	18	14	49
		6 to 8		L to M	P2	5	7	1	9	9	18	14	49
		8 to 10		L to M	P2	5	7	1	9	9	18	14	49
Total							**78**	**13**	**80**	**104**	**160**	**156**	**530**

Notes:

\> = Greater than
< = Less than
AFB = Air Force Base
bgs = Below ground surface
BVC = Base Ventura County
EDRO = Extended diesel range organics
FFA = Fuel Farm Area
GRO = Gasoline range organics
H = High (>1,000 mg/kg)
L = Low (<100 mg/kg)
M = Medium (100 to 1,000 mg/kg)
mg/kg = Milligram per kilogram
MS/MSD = Matrix spike/matrix spike duplicate
NEX = Naval Exchange
PC = Petroleum company
PRA = Phytoremediation area
SFT = Slop Fill Tank
STL Tampa East = Severn Trent Laboratories in Tampa, Florida

[a] Field triplicates will be collected at a frequency of one per depth interval in each sampling area. Three samples will be prepared for each field triplicate.

[b] MS/MSD samples will be collected at a frequency of one per depth interval in each sampling area for analysis by STL Tampa East. MS/MSD samples will not be analyzed by the developers. For MS/MSD analysis for GRO, four additional 5-gram EnCores will be provided to STL Tampa East. No additional sample containers will be required for MS/MSD analysis for EDRO.

[c] All environmental samples will also be analyzed for moisture by STL Tampa East; however, no additional sample volume will be required for these analyses.

Table 7-3. Environmental Samples (Continued)

[d] Number of 5-gram EnCores to STL Tampa East = number of GRO analyses x 2

[e] Number of 4-ounce jars to STL Tampa East = total number of samples, including field triplicates, x 2

[f] Because of soil conditions encountered in the FFA during the predemonstration investigation, the sampling depth intervals in this area could not be accurately determined. Sample collection was initiated approximately 10 feet bgs, and attempts were made to collect 4-foot-long soil cores. This approach resulted in varying degrees of core tube penetration up to 17 feet bgs. During the demonstration, for each sampling location in the area, each sample core will be divided into two samples based on visual observations. The upper layer of the soil core, which will likely consist of yellowish-brown, silty sand, will make up one sample, and the lower layer of the soil core, which will likely consist of grayish-black, silty sand and smell of hydrocarbons, will make up the second sample.

[g] It is assumed that only five devices will be used to analyze PRA samples because SDI and Dexsil® will not analyze PRA samples.

Table 7-4. Performance Evaluation Samples

Sample Type[a,b,c]	Expected Concentration	Primary Objective to be Addressed	Total Number of Samples to Each Developer and STL Tampa East	Number of Analyses by STL Tampa East[d]		Number of Sample Containers to STL Tampa East			Number of 25-Gram EnCores to Developers	Number of Ampules to Developers
				GRO	EDRO	5-Gram EnCores[e]	4-Ounce Jars[f]	Ampules		
Soil Samples										
Reference soil 1 + GRO (methanol as carrier)	L	P1, P2	7	7	7	14	7	0	21[g]	0
Reference soil 1 + GRO (Freon as carrier)	L	P1, P2	7	7	7	14	7	0	21	0
Reference soil 2 + GRO (methanol as carrier)	M	P2	3	3	3	6	3	0	9[g]	0
Reference soil 2 + GRO (Freon as carrier)	M	P2	3	3	3	6	3	0	9	0
Reference soil 2 + GRO	H	P2, P3, P4	3	3	3	6	3	0	21	0
Reference soil 1 + EDRO (methanol as carrier)	L	P1, P2	7	0	7	0	7	0	28	0
Reference soil 1 + EDRO (Freon as carrier)	L	P1, P2	7	0	7	0	7	0	21	0
Reference soil 2 + EDRO (methanol as carrier)	M	P2	3	0	3	0	3	0	12	0
Reference soil 2 + EDRO (Freon as carrier)	M	P2	3	0	3	0	3	0	9	0
Reference soil 2 + EDRO	H	P2, P3, P4	3	0	3	0	3	0	21	0
Reference soil 2	L	P3	3	3	3	6	3	0	24[h]	0
Reference soil 2 + I(1)L1 + GRO	H	P3	3	3	3	6	3	0	21	0
Reference soil 2 + I(1)L2 + GRO	H	P3	3	3	3	6	3	0	21	0
Reference soil 2 + I(2)L1 + GRO	H	P3	3	3	3	6	3	0	21	0
Reference soil 2 + I(2)L2 + GRO	H	P3	3	3	3	6	3	0	21	0
Reference soil 2 + I(3)L1 + GRO	H	P3	3	3	3	6	3	0	21	0
Reference soil 2 + I(3)L2 + GRO	H	P3	3	3	3	6	3	0	21	0
Reference soil 2 + I(4)L1 + GRO	H	P3	3	3	3	6	3	0	21	0
Reference soil 2 + I(4)L2 + GRO	H	P3	3	3	3	6	3	0	21	0
Reference soil 2 + I(3)L1 + EDRO	H	P3	3	3	3	6	3	0	21	0
Reference soil 2 + I(3)L2 + EDRO	H	P3	3	3	3	6	3	0	21	0
Reference soil 2 + I(4)L1 + EDRO	H	P3	3	3	3	6	3	0	21	0
Reference soil 2 + I(4)L2 + EDRO	H	P3	3	3	3	6	3	0	21	0
Reference soil 2 + I(5)L1 + EDRO	H	P3	3	0	3	0	3	0	21	0
Reference soil 2 + I(5)L2 + EDRO	H	P3	3	0	3	0	3	0	21	0
Reference soil 2 + I(6)L1 + EDRO	H	P3	3	0	3	0	3	0	21	0
Reference soil 2 + I(6)L2 + EDRO	H	P3	3	0	3	0	3	0	21	0
Reference soil 2 + I(6)L1	M to H	P3	3	0	3	0	3	0	21	0
Reference soil 2 + I(6)L2	M to H	P3	3	0	3	0	3	0	21	0
Reference soil 2 + GRO at 18 percent moisture	H	P4	3	3	3	6	3	0	21	0

Table 7-4. Performance Evaluation Samples (Continued)

Sample Type[a,b,c]	Expected Concentration	Primary Objective to be Addressed	Total Number of Samples to Each Developer and STL Tampa East	Number of Analyses by STL Tampa East[d]		Number of Sample Containers to STL Tampa East			Number of 25-Gram EnCores to Developers	Number of Ampules to Developers
				GRO	EDRO	5-Gram EnCores[e]	4-Ounce Jars[f]	Ampules		
Reference soil 2 + EDRO at negligible moisture (none added)	H	P4	3	0	3	0	3	0	21[i]	0
Liquid Samples										
GRO	H	P3	3	3	3	0	0	6	0	21
EDRO	H	P3	3	0	3	0	0	3	0	21
I(1)L1	M to H	P3	3	3	0	0	0	3	0	21
I(1)L2	M to H	P3	3	3	0	0	0	3	0	21
I(2)L1	M to H	P3	3	3	0	0	0	3	0	21
I(2)L2	M to H	P3	3	3	0	0	0	3	0	21
I(3)L1	M to H	P3	3	3	3	0	0	6	0	24[h]
I(3)L2	M to H	P3	3	3	3	0	0	6	0	24[h]
I(4)L1	M to H	P3	3	3	3	0	0	6	0	24[h]
I(4)L2	M to H	P3	3	3	3	0	0	6	0	24[h]
I(5)L1	M to H	P3	3	0	3	0	0	3	0	21
I(5)L2	M to H	P3	3	0	3	0	0	3	0	21
Total			**145**	**92**	**133**	**130**	**109**	**51**	**616**[j]	**264**

Notes:

\> = Greater than
< = Less than
EDRO = Extended diesel range organics
GRO = Gasoline range organics
H = High (>1,000 mg/kg)
L = Low (<100 mg/kg)
M = Medium (100 to 1,000 mg/kg)
mg/kg = Milligram per kilogram
STL Tampa East = Severn Trent Laboratories in Tampa, Florida
VOA = Volatile organic analysis

Unless otherwise specified, soil performance evaluation samples will be prepared at 9 percent moisture, the minimum moisture required for containerizing samples using EnCores.

Two different carriers will be used to meet the needs of individual developers. Methanol will be used as the carrier for the following devices: the PetroFLAG™ test kit, Luminoscope, UVF-3100A, and EnSys Petro Test System. Freon will be used as the carrier for the following devices: the RemediAid™ starter kit, OCMA-350, Model CVH, and Model HATR-T.

Samples that will contain interferent alone have an expected device response of M to H because it is not certain how each interferent will quantitatively respond to TPH analysis.

[a] Reference soil 1 is Ottawa sand, and reference soil 2 is processed garden soil (sandy silt). Reference soil 2 may contain trace levels of GRO and low levels of EDRO.

[b] The boiling points and vapor pressures of (1) interferents I(1) and I(2) have characteristics similar to those of GRO, (2) interferents I(3) and I(4) have characteristics similar to those of GRO and EDRO, and (3) interferents I(5) and I(6) have characteristics similar to those of EDRO. PE samples containing interferents I(1) and I(2) will not be prepared with EDRO because these

Table 7-4. Performance Evaluation Samples (Continued)

interferents are not expected to impact EDRO analysis. Similarly, PE samples containing interferents I(5) and I(6) will not be prepared with GRO because these interferents are not expected to impact GRO analysis. In addition, practical difficulties (solubility constraints) associated with preparing the I(1) or I(2) + EDRO and I(5) or I(6) + GRO PE samples will not allow use of these samples in the demonstration. Expected concentrations for PE samples containing interferents are based on expected analytical results for the samples and not on the concentrations of interferents to be added to the samples.

[c] Performance evaluation samples containing interferents will be prepared at two different interferent levels, L1 and L2.

[d] All performance evaluation samples will also be analyzed for moisture by STL Tampa East; however, no additional sample volume will be required for these analyses.

[e] Number of 5-gram EnCores to STL Tampa East = number of GRO analyses x 2

[f] Number of 4-ounce jars to STL Tampa East = number of EDRO analyses

[g] It is assumed that only three devices will be used to analyze low- and medium-level GRO samples with the methanol carrier because Dexsil® will not demonstrate the PetroFLAG™ test kit using low- and medium-level GRO samples.

[h] This number includes enough samples for Wilks to use both Models CVH and HATR-T. Both models need to be used in order to provide enough control samples to address primary objective P3 (effects of interferents).

[i] These samples will be containerized in VOA vials because soil with less than 9 percent moisture cannot be containerized in EnCores.

[j] The total number of EnCores is actually 595 because VOA vials will be used for 21 samples as explained in footnote i.

7.1.1.1.1 Fuel Farm Area

Soil samples will be collected by Tetra Tech in the FFA at three locations south of Tank No. 5114 (see Figure 3-2). Specifically, the sampling locations will consist of the three locations sampled during the predemonstration investigation and three of the four locations sampled by site representatives in September 1999. The three demonstration sampling locations lie about 30 feet apart. Based on historical data regarding the extent of area contamination and Tetra Tech's knowledge of the area gained during the predemonstration investigation, soil samples will be collected in two depth intervals at the three sampling locations. During the predemonstration investigation, Tetra Tech could not identify exact sampling depth intervals because of the soil types encountered and the recoveries obtained. The two depth intervals were classified as the upper layer and lower layer, with the upper layer consisting of yellowish-brown, silty sand and the lower layer consisting of grayish-black, silty sand with a hydrocarbon odor. During the demonstration, based on visual observations, Tetra Tech will attempt to sample the same depth intervals as were sampled during the predemonstration investigation. As stated in Chapter 3, hydrocarbon contamination in the FFA is not homogeneous. To the extent possible, Tetra Tech will not mix soil types in a given soil sample. Based on soil profiles, a field decision will be made regarding whether the sampling depth intervals should be modified to achieve as much homogeneity as possible in each soil sample.

Tetra Tech's subcontractor will conduct sample collection activities using a Geoprobe® with push rods containing approximately 1.7-inch-diameter, plastic liners that can be capped at both ends. Based on the history of contamination in the FFA, soil samples collected in this area will be analyzed for EDRO by STL Tampa East.

7.1.1.1.2 Naval Exchange Service Station Area

Soil samples will be collected by Tetra Tech in the NEX Service Station Area at three locations between Building 1184 and Credit Union Building 1336 (see Figure 3-2). Specifically, the sampling locations will consist of the three locations sampled during the predemonstration investigation and sampled by site representatives in September 1999. The sampling locations are about 30 feet apart. Based on historical data regarding the extent of area contamination and Tetra Tech's knowledge of the area gained during the predemonstration investigation, soil samples will be collected in 1-foot depth intervals from 7 to 11 feet bgs at the three sampling locations. These intervals were selected based on soil contamination characteristics observed during predemonstration investigation sampling in the area. The 5- to 6- and 6- to 7-foot bgs depth intervals sampled during the predemonstration investigation will not be sampled during the demonstration because TPH concentrations in these intervals were negligible. To the extent possible, Tetra Tech will not mix soil types in a given soil sample. Based on soil profiles, a field decision will be made regarding whether the sampling depth intervals should be modified to achieve as much homogeneity as possible in each soil sample.

Sampling will be conducted using a Geoprobe® with push rods containing approximately 1.7-inch-diameter, plastic liners that can be capped at both ends. Based on the history of contamination in the NEX Service Station Area and Tetra Tech's knowledge of the area gained during the predemonstration investigation, soil samples collected in this area will be analyzed for both GRO and EDRO by STL Tampa East.

7.1.1.1.3 Phytoremediation Area

Soil samples will be collected by Tetra Tech in the PRA at six locations using a Split Core Sampler containing a 2-inch-diameter, plastic liner. Specifically, one soil sample will be collected from each of six cells; each cover type (that is, unvegetated, native grass mix, and grass and legume mix) will be represented twice in the sampling (see Figure 3-2). Samples will be collected from 1.5 to 2.5 feet bgs. Based on the results of previous investigations in the PRA, the samples collected in this area will be analyzed for EDRO by STL Tampa East.

7.1.1.2 Kelly Air Force Base Site

At the Kelly AFB site, soil samples will be collected in the B-38 Area at the four locations sampled during the predemonstration investigation (see Figure 3-4). The depth intervals sampled during the demonstration will be different from those sampled during the predemonstration investigation because the latter contained low or nondetect TPH concentrations. Based on discussions with the demonstration site representative after the predemonstration investigation, it was determined that most of the contamination in the B-38 Area can be found at or near the water table. Therefore, the depth intervals to be sampled during the demonstration will be located 2 feet above and 2 feet below the water table. For the purposes of this demonstration plan, Tetra Tech assumes that the surface of the water table will be about 20 feet bgs during the demonstration. This assumption is based on previous groundwater-level data provided by the site representative. The exact depth to groundwater will be measured by Tetra Tech using a water-level indicator in four nearby monitoring wells at the time of the demonstration. Tetra Tech will then calculate an average depth to groundwater and will collect the soil samples 2 feet above and 2 feet below the average water table depth. Therefore, soil samples will likely be collected in two depth intervals—18 to 20 and 20 to 22 feet bgs—at the four sampling locations.

Sampling will be conducted using a Geoprobe® with push rods containing approximately 1.7-inch-diameter, plastic liners that can be capped at both ends. Based on the history of contamination in the B-38 Area and Tetra Tech's knowledge of the area gained during the predemonstration investigation, soil samples collected in this area will be analyzed for both GRO and EDRO by STL Tampa East.

7.1.1.3 Petroleum Company Site

At the PC site, soil samples will be collected in the SFT Area at five sampling locations. Specifically, samples will be collected from the four corners and center of the square sampling area shown in Figure 3-5, which are the five locations sampled during the predemonstration investigation. Based on the known extent of area contamination, soil samples will be collected in 2-foot depth intervals from 2 to 10 feet bgs at the five sampling locations.

Sampling will be conducted using a Geoprobe® with push rods containing approximately 1.7-inch-diameter, plastic liners that can be capped at both ends. Based on the history of contamination in the SFT Area and Tetra Tech's knowledge of the area gained during the predemonstration investigation, soil samples collected in this area will be analyzed for both GRO and EDRO by STL Tampa East.

7.1.2 Sample Handling and Shipping

Following its recovery, each soil sample collected using a lined Geoprobe® or Split Core Sampler will be removed intact in the liner from the sampling equipment. If necessary, the liner will be cut to a length that will fit in a cooler. After the liner is removed and cut, it will be immediately closed with plastic caps at both ends to minimize volatilization of contaminants. The liner caps will be of two colors to designate the top and bottom of the soil sample. To ensure that the caps will not be detached from the liner, the caps will be taped to the liner. The liner will then be labeled. The label will contain information on the site, sampling area, sampling location, and depth interval from which the sample was collected.

The samples collected at the Kelly AFB and PC sites will be placed in coolers containing ice and will be shipped by overnight courier to the sample management trailer at the Navy BVC site. At this location, the soil samples collected at all three demonstration sites will be profiled, homogenized, and prepared for shipment to STL Tampa East and for transfer to the developers on site. PE samples will be shipped to Tetra Tech at the Navy BVC site by ERA in Arvada, Colorado. Tetra Tech will not open any of the PE sample containers but will assign them sample identification numbers and distribute them to STL Tampa East and the developers.

7.1.3 Sample Preparation

This section describes environmental, PE, and QC sample preparation activities that will be conducted during the demonstration.

7.1.3.1 Environmental Samples

After the liners containing environmental samples are transported to the sample management trailer at the Navy BVC site, Tetra Tech will cut the liners longitudinally. Tetra Tech will then profile the samples to determine where the soil cores have to be sectioned if their depth intervals are found to differ from the target depth intervals based on soil characteristics. The soil samples will be characterized by a Tetra Tech geologist using the USCS.

Because the TPH concentrations encountered in the B-38 Area at Kelly AFB during the predemonstration investigation were low or nondetects, photoionization detector (PID) readings will also be used to select the depth intervals to be sampled for this area. The PID readings will be used qualitatively to determine whether contamination is present at low, medium, or high levels or not at all. If the PID does not detect any measurable contamination, Tetra Tech will make a field decision regarding whether to analyze samples from the B-38 Area.

Each core sample will be transferred to a stainless-steel bowl. The presence of any unrepresentative material such as sticks, roots, and stones will be noted in a field logbook, and such material will be removed to the extent possible using gloved hands. Any lump of clay in the sample that is greater than about 1/8 inch in diameter will be crushed between gloved fingers before homogenization.

Tetra Tech will homogenize each soil sample by stirring the soil for at least 2 minutes using a stainless-steel spoon or gloved hands until the sample is visibly homogeneous. During or immediately following homogenization, Tetra Tech will pour off any free water from the stainless-steel bowl containing the soil sample into a container designated for IDW. During the predemonstration investigation, nitrile gloves were used by the field sampling team for most sample preparation; however, when the supply of nitrile gloves was depleted, locally available plastic gloves were used. Use of plastic gloves resulted in phthalate contamination in the EDRO range in the predemonstration investigation sample analytical results. Therefore, during the demonstration, only nitrile gloves will be used by the field sampling team.

Tetra Tech will then place the samples in (1) EnCores for GRO and TPH analyses and (2) STL Tampa East-provided, 4-ounce, glass sample jars for EDRO analysis. The 4-ounce, glass sample jars will be filled after all the EnCores have been filled for a given sample. A sample that will be submitted for GRO analysis will be aliquoted from the mixing bowl into an EnCore of approximately 5-gram capacity. Similarly, a sample that will be submitted for TPH analysis will be aliquoted from the mixing bowl into an EnCore of approximately 25-gram capacity. A sample to be submitted for EDRO analysis will be placed in 4-ounce, glass jars. Using a quartering technique, Tetra Tech will fill each sample container by alternately spooning soil from one quadrant of the mixing bowl and then from the opposite quadrant until the container is full. After a sample container is filled, it will be immediately closed to minimize volatilization of contaminants. The top of each sample container will be carefully wiped with a clean, disposable laboratory wipe to remove any soil particles that might be present. To minimize the time required for sample homogenization and filling of sample containers, these activities will be conducted by two or three Tetra Tech personnel simultaneously.

Some time will elapse between the filling of the first EnCore and the filling of the last EnCore for the developers. Therefore, to distribute the EnCores to the developers in such a way that the order in which they were filled is not critical, the following procedure will be used. The seven EnCores per sampling location for the developers will be labeled 1 through 7 by Tetra Tech prior to the demonstration. When filling these EnCores, Tetra Tech will always start with number 1 and fill them sequentially through number 7. When field triplicates are collected, 21 EnCores will be filled for the developers. These samplers will be labeled as three batches of 1 through 7 and will be filled one batch at a time in numerical sequence. The field triplicates can thus be used to check Tetra Tech's soil homogenization procedures. The numbered EnCores that the developers receive will be varied so that all the developers receive approximately the same distribution of EnCores of a given number. When a sample is prepared for submittal to STL Tampa East for GRO analysis, the 5-gram EnCores for STL Tampa East will always be filled after one-half of the 25-gram EnCores designated for a given depth interval and location have been filled.

Table 7-5 summarizes the sample container, preservation, and holding time requirements for (1) GRO, EDRO, and percent moisture analyses to be performed by STL Tampa East and (2) TPH analyses to be performed by the developers. STL Tampa East will receive two EnCores, each containing approximately 5 grams of soil, for GRO analysis and two 4-ounce, glass jars filled to two-thirds of their capacity with soil to be analyzed for EDRO and percent moisture. The 4-ounce jars will be filled to two-thirds of their capacity so that STL Tampa East can mix each sample before separating aliquots for EDRO and percent moisture analyses. Field triplicates will be collected at a rate of one per depth interval per area. For GRO analysis, when matrix spike/matrix spike duplicate (MS/MSD) or field triplicate samples are required, four additional 5-gram EnCores will be filled with soil and sent to STL Tampa East. For EDRO analysis, only field triplicates require additional sample volume. For each environmental field triplicate, four additional 4-ounce, glass jars will be filled with soil and sent to STL Tampa East. Each developer will receive one EnCore sample containing about 25 grams of soil for TPH analysis. MS/MSDs and field triplicates

Table 7-5. Sample Container, Preservation, and Holding Time Requirements

Parameter[a]	Medium	Container	Preservation	Holding Time (days) Extraction	Holding Time (days) Analysis
GRO	Soil	Two 5-gram EnCores	4 ± 2 °C	2[b]	14
EDRO	Soil	Two 4-ounce, glass jars with Teflon™-lined lids	4 ± 2 °C	14[b]	40
Percent moisture	Soil	Two 4-ounce, glass jars with Teflon™-lined lids	4 ± 2 °C	Not applicable	7
Total petroleum hydrocarbons	Soil	One 25-gram EnCore	4 ± 2 °C	Performed on site[c]	
GRO and EDRO	Liquid	One 2-milliliter ampule	Not applicable	See note d	

Notes:

± = Plus or minus
EDRO = Extended diesel range organics
GRO = Gasoline range organics

[a] Severn Trent Laboratories in Tampa, Florida, will measure percent moisture using part of the soil sample from a container designated for extended diesel range organic analysis.

[b] The extraction holding time will start on the day that samples are shipped.

[c] If gasoline range organics are likely to be present, extraction will take place within 2 days. Otherwise, all extractions and analyses will be completed on site within 7 days.

[d] Severn Trent Laboratories in Tampa, Florida, will crack open each ampule and will immediately add a specified aliquot of the sample to methanol for GRO analysis and to methylene chloride for EDRO analysis in such a way that the final volumes of the extracts for GRO and EDRO analyses are 5.0 and 1.0 milliliters, respectively. Once the extracts are prepared, the GRO and EDRO analyses will be performed within 14 and 40 days, respectively.

will be designated by Tetra Tech for a given area based on the expected contaminant concentration range and visual observations of soil characteristics. These samples will be selected in such a way that they reflect a variety of concentration ranges and all the soil types in a given area. Unused soil will be collected as IDW and will be managed in the manner described in Section 7.3.

7.1.3.2 PE Samples

All PE samples will be prepared by ERA and shipped to Tetra Tech at the sample management trailer at the Navy BVC site. PE samples will consist of soil samples and liquid samples. ERA will prepare soil PE samples using two reference soils. Reference soil 1 will consist of Ottawa sand and will be used to prepare low-level PE samples. Reference soil 2 will consist of processed garden soil (sandy silt) and will be used to prepare the rest of the soil PE samples. Reference soil 2 may contain native trace levels of GRO and low levels of EDRO. However, for medium- and high-level PE samples, reference soil 2 will be used because the sandy silt will allow a more realistic evaluation of contaminant recovery than Ottawa sand and because the trace levels of background contamination present in the sandy silt will not impact the integrity of the samples. Each soil PE sample will be spiked with a known type of contaminant at a known concentration as summarized in Table 7-4.

To prepare the soil PE samples, ERA will spike the required volume of soil based on the number of PE samples and the quantity of soil per PE sample requested by Tetra Tech. ERA will then homogenize the soil by manually mixing it. ERA will use a GRO-range spiking material and an EDRO-range spiking material, and spiking will be done at three levels: low, medium, and high. A low-level sample will correspond to a TPH value of less than 100 mg/kg; a medium-level sample will correspond to a TPH range of 100 to 1,000 mg/kg; and a high-level sample will correspond to a TPH value of more than 1,000 mg/kg. Some soil samples will also be spiked with interferents I(1) through I(6) at two different levels, L1 and L2. The interferent levels will range from 50 to 500 percent of the expected TPH concentration in a given sample. Whenever possible, the interferents will be added at levels that best represent real-world conditions.

To spike each low- and medium-level soil sample, ERA must use a "carrier" to distribute the contaminant evenly throughout the sample. During the predemonstration investigation, methanol and Freon 113 were used as carriers for low-level soil PE samples. After reviewing STL Tampa East data for these samples, Tetra Tech determined that the samples prepared using methanol contained lower levels of TPH than those prepared using Freon 113 (up to 40 percent lower). According to the manufacturer of EnCores, using methanol as the carrier in an EnCore may have caused the sampler to swell, opening the pores on the sampler's viton ring; as a result, some of the volatiles in the sample may have escaped. To address this potential loss of volatiles, ERA will spike the low- and medium-level PE samples prepared using methanol with slightly higher levels than those actually requested by Tetra Tech. The low- and medium-level PE samples will be used to address primary objectives P1 and P2, but most of the PE samples will be used to address primary objective P3. This objective will be addressed using these high-level PE samples because these samples do not require a carrier. Tetra Tech is attempting to minimize the use of PE samples that require a carrier because when carriers are used, two sets of PE samples must be sent to STL Tampa East, one prepared with Freon 113 and one prepared with methanol. As a result, sample preparation and analysis costs increase.

The liquid PE samples will be prepared to address primary objective P3 and will each consist of one of five interferents, I(1) through I(5), at two different levels. Neat materials will be used as liquid PE samples; no carrier will be used. Each liquid PE sample will consist of approximately 2 mL of liquid in a flame-sealed, glass ampule. ERA will prepare the samples in such a way that a minimum amount of liquid corresponds to 1 gram of soil. The amount of liquid that corresponds to 1 gram of soil will range from 2.5 to 12.5 µL depending on the interferent used. During the demonstration, both STL Tampa East and the developers will be given a table informing them of the amount of liquid sample to be used per gram of soil extracted. STL Tampa East or a given developer will analyze the liquid PE samples using an amount of liquid that corresponds to the amount of soil that would normally be used for analysis. For example, if a particular device requires 5 grams of soil for analysis, and if 5 µL per gram is the amount of liquid sample corresponding to 1 gram of soil, 25 µL of liquid sample would be used for the analysis. Thus, the 1 mL of liquid in each PE sample will be adequate for conducting multiple analyses, if necessary.

ERA will provide Tetra Tech with a certified value and acceptance limits for each PE sample prepared. The certified values will be derived differently for EDRO PE samples and GRO PE samples. For an EDRO PE sample, the certified value will be equal to the concentration of spiking material used. For a GRO PE sample, the certified value will be derived by analyzing a portion of the spiked, homogenized sample using a GC/FID method. After homogenization of the soil spiked with GRO, ERA will collect and analyze three soil samples: one before filling any EnCores, one after all the 5-gram EnCores are filled, and one after all the 25-gram EnCores are filled. ERA will then report the average value as the certified value for that batch of PE samples. This method will be used to determine the GRO PE sample certified values because a significant portion of the GRO spiking material could be lost during homogenization due to the volatility of the spiking material.

The acceptance limits will be derived by ERA based on historical mean recovery values for similar types of samples. The acceptance limits will be evenly distributed about the historical mean recovery values. However, these acceptance limits will not be evenly distributed about the certified values because the certified values will not have been corrected for the historical mean recovery values. Therefore, the acceptance limits may seem to be biased low or high, depending on whether the certified values are higher or lower than the historical mean recovery values.

ERA will group like PE samples together in a resealable bag and will place all samples in a cooler containing ice for overnight shipment to Tetra Tech at the Navy BVC site. When Tetra Tech receives the PE samples, Tetra Tech will label them with the appropriate sample identification numbers and will place them in an appropriate cooler for shipment to STL Tampa East or for transfer to the developers at the site. Detailed sample handling and shipping procedures are provided in Section 7.2. PE samples spiked with GRO only will be sent to STL Tampa East for analysis for both GRO and EDRO because the spiking material used by ERA for GRO PE samples may contain some EDRO and because reference soil 2 may contain native low levels of EDRO. Therefore, for GRO PE samples, STL Tampa East will receive two EnCores, each containing approximately 5 grams of soil sample, and one 4-ounce, glass jar containing at least 100 grams of soil for EDRO analysis. Only one jar will be provided for EDRO PE samples rather than two because of the significant costs associated with sample preparation by ERA. All PE samples will be prepared in triplicate; therefore, for each PE triplicate, four additional 5-gram EnCores and two additional 4-ounce, glass jars will be sent to STL Tampa East. In the event that a PE sample cannot be used by STL Tampa East because its container is broken during shipment, an aliquot from one of the other two remaining triplicates will be used for extraction and analysis. However, the triplicates will be submitted to STL Tampa East as blind samples; therefore, Tetra Tech will inform STL Tampa East of which triplicate should be used as a backup for an unusable sample.

7.1.3.3 QC Samples

The following field QC samples will be collected during the demonstration: (1) MS/MSD samples, (2) field triplicate samples, (3) extraction duplicates, (4) field blanks, and (5) temperature blanks. Table 7-3 identifies the planned numbers of MS/MSD and field triplicate samples. MS/MSD samples will be collected for environmental samples at a frequency of one per depth interval in each sampling area for analysis by STL Tampa East. This frequency exceeds the typical frequency of one per analytical batch. MS/MSDs for PE samples will be analyzed by STL Tampa East at a frequency of one per analytical batch. MS/MSD samples will not be analyzed by the developers.

Blind field triplicate samples will be prepared by Tetra Tech for environmental samples at a frequency of one per depth interval in each sampling area. All PE samples will be prepared by ERA in triplicate as blind samples.

Tetra Tech will designate certain environmental samples as extraction duplicates, meaning that duplicate analyses will be performed on the sample extracts by the developers and STL Tampa East. Extraction duplicate samples will be used to address the analytical precision component of primary objective P2. These samples will be designated at a rate of one per depth interval in each sampling area; each extraction duplicate will be one of the field triplicate samples. Therefore, the developers and STL Tampa East will each receive 13 environmental samples designated as extraction duplicates. Tetra Tech will designate an extraction duplicate by adding "ED" to the end of the sample designation (see Section 7.2). If a device's operating procedure requires that all the sample extract be used for one analysis, extraction duplicates will not be analyzed by that device.

Tetra Tech will designate certain soil PE samples as extraction duplicates, meaning that duplicate analyses will be performed on the sample extracts by the developers and STL Tampa East. Extraction duplicate samples will be used to address the analytical precision component of primary objective P2. Only PE samples that contain soil with GRO or soil with EDRO at low, medium, or high levels and that do not contain interferents will be designated as extraction duplicates. Therefore, each developer will receive 6 PE samples designated as extraction duplicates, and STL Tampa East will receive 10 PE samples designated as extraction duplicates (because STL Tampa East will analyze low- and medium-level PE samples containing methanol and Freon). If a device's operating procedure requires that all the sample extract be used for one analysis, extraction duplicates will not be analyzed by that device. Tetra Tech will designate an extraction duplicate by adding "ED" to the end of the sample designation (see Section 7.2).

Field blanks consisting of distilled water in beakers exposed to the atmosphere throughout the day will be available to the developers at the Navy BVC site during the demonstration. If they choose to, the developers may analyze the field blank samples to assess whether atmospheric conditions at the site may have impacted device measurements.

Temperature blanks consisting of potable water in sealed containers will be prepared by Tetra Tech at a frequency of one per cooler for both STL Tampa East and the developers.

The QA objectives for the demonstration are presented in Chapter 10.

7.2 Sample Handling and Shipping Procedures

Each environmental sample will have a unique sample designation and will be identified by sampling area, expected type of contamination, expected concentration range, sampling location, sample number, and QC identification, as appropriate. Each PE sample will also have a unique sample designation that identifies it as a PE sample. Each PE sample designation will also identify the expected contamination type and range, whether the sample is soil or liquid, and the sample number. PE sample numbers will be assigned for Tetra Tech's reference only. Each sample container will be labeled with the unique sample designation, date, time, preservative, initials of Tetra Tech personnel filling the container, and analysis to be performed. Examples of sample designations are listed below.

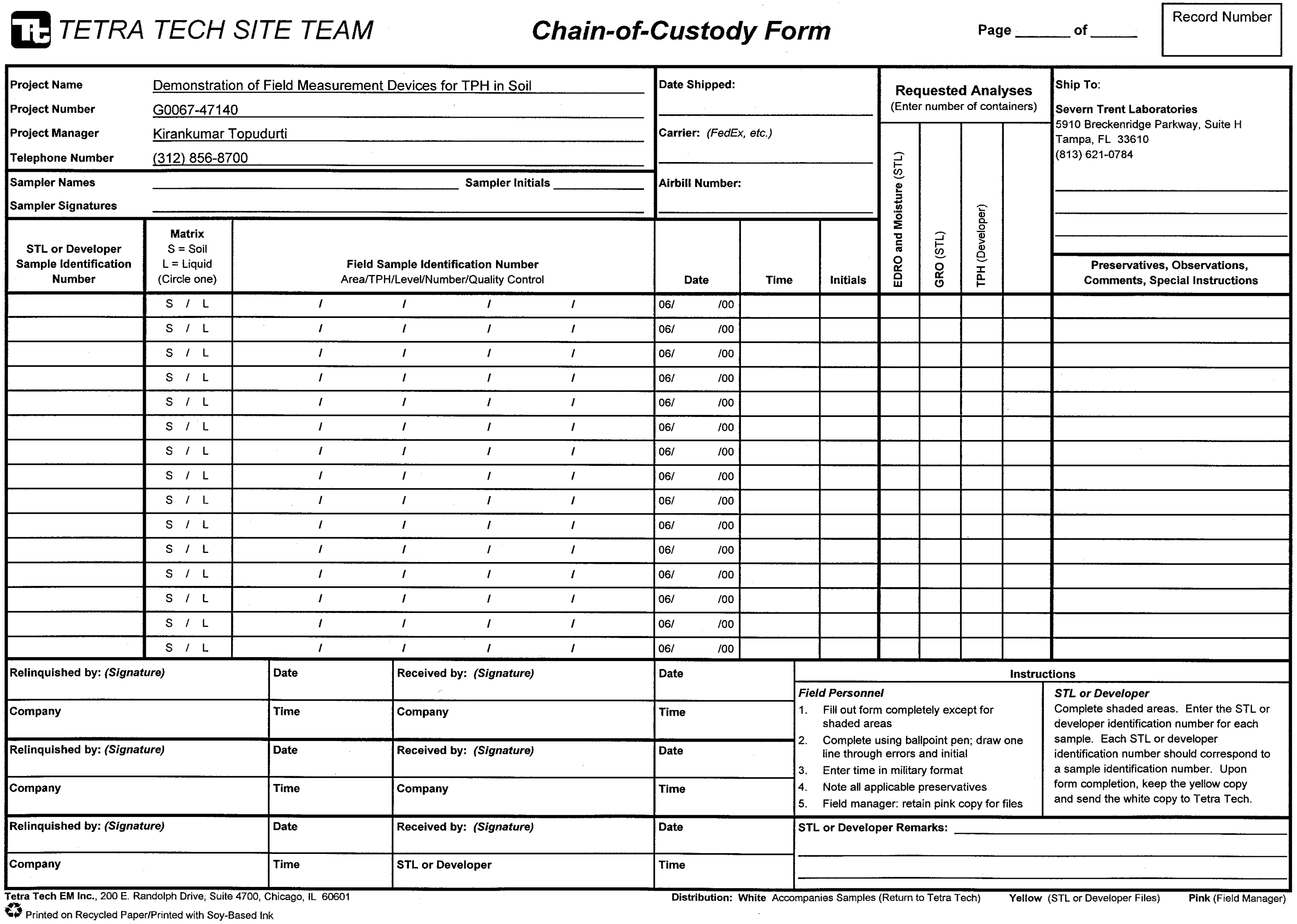
•	FFA/E/LM/A01	FFA, EDRO analysis by STL Tampa East, low- to medium-concentration range, location A, sample number 01
•	NEX/GE/L/B02/ED	NEX Service Station Area, GRO and EDRO analyses by STL Tampa East, low-concentration range, location B, sample number 02, extraction duplicate
•	PRA/E/H/C02	PRA, EDRO analysis by STL Tampa East, high-concentration range, location C, sample number 02
•	B38/GE/L/B01	B-38 Area, GRO and EDRO analyses by STL Tampa East, low-concentration range, location B, sample number 01
•	SFT/GE/MH/D04/MSMSD	SFT Area, GRO and EDRO analyses by STL Tampa East, medium- to high-concentration range, location D, sample number 04, MS/MSD analyses by STL Tampa East
•	PE/E/M/S01	PE sample, EDRO analysis by STL Tampa East, medium-concentration range, soil sample number 01
•	PE/GE/H/L02	PE sample, GRO and EDRO analyses by STL Tampa East, high-concentration range, liquid sample number 02

Tetra Tech will maintain master data sheets that list each sample designation used during the demonstration and the corresponding sampling area, location, and depth interval.

Tetra Tech's sample custody will begin when samples are placed in iced coolers in the possession of the designated field sample custodian. Samples for STL Tampa East analysis will be placed in coolers containing ice and will be shipped by overnight courier to STL Tampa East. Chain-of-custody forms will be completed and will accompany each sample shipment to STL Tampa East. Samples for the developers will be placed in coolers containing ice and chain-of-custody forms and will be given directly to each developer at the Navy BVC site. Chain-of-custody forms will be filled out and initialed by Tetra Tech personnel. As indicated in Figure 7-1, the following information will be provided on each chain-of-custody form:

Project Name	Demonstration of Field Measurement Devices for TPH in Soil
Project Number	G0067-47140
Project Manager	Kirankumar Topudurti
Telephone Number	Tetra Tech project manager's telephone number: (312) 856-8700
Sampler Names, Initials, and Signatures	The sampling technicians' printed names, initials, and signatures
STL Tampa East or Developer Sample Identification Number	Unique sample identification number assigned by STL Tampa East or the developer after receipt of samples
Matrix	Sample matrix (S for soil and L for liquid)
Field Sample Identification Number	Tetra Tech-assigned field sample identification number
Date	Date of sample collection
Time	Time of filling sample container
Initials	Sampling technician's initials
Requested Analyses	The number of sample containers for each requested analysis

When all appropriate line items are completed, Tetra Tech's field manager or his designee will confirm the completeness of all descriptive information on the form and will sign and date the form. Each individual who subsequently assumes responsibility for the samples will sign the chain-of-custody form. For samples shipped by courier service, the courier service will not sign the chain-of-custody form; the airbill invoice will serve as part of the chain-of-custody documentation. Use of the chain-of-custody form will end when STL Tampa East or the developer receives the samples and enters the STL Tampa East or developer sample identification numbers on the form. The Tetra Tech field manager will retain the pink copy of the chain-of-custody form for the project files.



TETRA TECH SITE TEAM **Chain-of-Custody Form** Page ______ of ______

Record Number

Project Name Demonstration of Field Measurement Devices for TPH in Soil
Project Number G0067-47140
Project Manager Kirankumar Topudurti
Telephone Number (312) 856-8700
Sampler Names ______ Sampler Initials ______
Sampler Signatures ______

Date Shipped: ______
Carrier: *(FedEx, etc.)* ______
Airbill Number: ______

Requested Analyses
(Enter number of containers)

Ship To:
Severn Trent Laboratories
5910 Breckenridge Parkway, Suite H
Tampa, FL 33610
(813) 621-0784

STL or Developer Sample Identification Number	Matrix S = Soil L = Liquid (Circle one)	Field Sample Identification Number Area/TPH/Level/Number/Quality Control	Date	Time	Initials	EDRO and Moisture (STL)	GRO (STL)	TPH (Developer)		Preservatives, Observations, Comments, Special Instructions
	S / L	/ / / /	06/ /00							
	S / L	/ / / /	06/ /00							
	S / L	/ / / /	06/ /00							
	S / L	/ / / /	06/ /00							
	S / L	/ / / /	06/ /00							
	S / L	/ / / /	06/ /00							
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	S / L	/ / / /	06/ /00							
	S / L	/ / / /	06/ /00							
	S / L	/ / / /	06/ /00							

Relinquished by: *(Signature)*	Date	Received by: *(Signature)*	Date
Company	Time	Company	Time
Relinquished by: *(Signature)*	Date	Received by: *(Signature)*	Date
Company	Time	Company	Time
Relinquished by: *(Signature)*	Date	Received by: *(Signature)*	Date
Company	Time	STL or Developer	Time

Instructions

Field Personnel

1. Fill out form completely except for shaded areas
2. Complete using ballpoint pen; draw one line through errors and initial
3. Enter time in military format
4. Note all applicable preservatives
5. Field manager: retain pink copy for files

STL or Developer

Complete shaded areas. Enter the STL or developer identification number for each sample. Each STL or developer identification number should correspond to a sample identification number. Upon form completion, keep the yellow copy and send the white copy to Tetra Tech.

STL or Developer Remarks: ______

Tetra Tech EM Inc., 200 E. Randolph Drive, Suite 4700, Chicago, IL 60601
♻ Printed on Recycled Paper/Printed with Soy-Based Ink

Distribution: White Accompanies Samples (Return to Tetra Tech) **Yellow** (STL or Developer Files) **Pink** (Field Manager)

Figure 7-1. Chain-of-custody form.

All samples for STL Tampa East analysis will be packaged and labeled for shipment in compliance with current DOT and International Air Transport Association (IATA) regulations for dangerous materials. Only plastic coolers will be used for shipping samples. Each cooler shipped to STL Tampa East will be lined with two 6-mil, plastic bags. Styrofoam or bubble wrap will be used in the coolers to absorb shocks. After the sample containers are packaged, the inner 6-mil, plastic bag around the containers will be sealed to prevent leaks by twisting the top and securely taping the bag closed. To meet preservation requirements, ice will be placed in the coolers along with the sample containers. The white and yellow copies of the chain-of-custody forms will accompany each cooler. These forms will be enclosed in a waterproof, plastic bag that will be taped to the underside of the cooler lid.

All samples for the developers will be packaged in the same manner as the samples for STL Tampa East. Coolers containing samples for developers will be given directly to the developers by Tetra Tech personnel at the Navy BVC site.

Each cooler prepared for shipment to STL Tampa East will be securely taped shut. Sample custody seals will be placed on the front and back of each cooler to reveal any unauthorized tampering with samples before analysis. The Tetra Tech field manager or his designee will sign and date the custody seals and will affix the seals at the time of sample packaging. Reinforced or other suitable tape (such as duct tape) will be wrapped at least twice around the cooler near each end where the hinges are located.

Each cooler to be shipped to STL Tampa East will be marked in accordance with DOT regulations for shipping hazardous materials (49 CFR Part 172 and IATA Dangerous Goods Regulations, 31st Edition, January 1, 1987). In addition to complete mailing addresses, each cooler will clearly display “This End Up” arrows on all four sides and a label on the exterior lid indicating the originator’s and recipient’s addresses.

When selecting means of sample shipment to STL Tampa East, Tetra Tech field personnel will ensure that allowable sample holding times will not be exceeded. When commercial common carriers are used to ship samples, all samples will be shipped “Priority One/Overnight.” If necessary, samples may be shipped using a reliable commercial carrier such as Federal Express. If commercial carriers are used, airbills will be completed and attached to the exterior lids of the coolers. Multiple-shipment labels will be used when more than one cooler is being shipped.

The STL Tampa East sample coordinator or a designee will receive samples and assume custody of them until they have been properly logged in the laboratory and stored in secured areas. The laboratory sample management procedures to be followed are presented in Section 8.2.

Tetra Tech's field logbooks will contain the information entered by Tetra Tech on the chain-of-custody forms and will be maintained by the field manager or his designee on site. The developers will have an opportunity to cross-check all samples with the chain-of-custody forms before the demonstration officially begins. The demonstration will officially end for a particular developer when all of the developer's sample analytical results are provided to Tetra Tech on site.

7.3 Sampling Equipment Decontamination and Investigation-Derived Waste Disposal Procedures

The Tetra Tech field team will take steps to minimize the volume of IDW generated. Nondisposable sampling equipment will be decontaminated by scrubbing it with an Alconox® solution, washing it with potable water, and then rinsing it with distilled water. Decontamination of nondisposable sampling equipment will be conducted prior to sampling at each site as well as between sampling areas. All IDW generated, including unused sample material and decontamination water that has come into contact with grossly contaminated material, will be managed and disposed of in accordance with site-specific IDW management practices.

Solid wastes expected to be generated during the demonstration include disposable glassware, EnCores, unused or extra soil samples, and PPE. Used, disposable, empty glassware and used, empty EnCores will be disposed of as general refuse in a dumpster or garbage can provided by Tetra Tech at the Navy BVC site. Unused or extra soil samples and used PPE such as gloves will be placed in an on-site drum designated for nonhazardous solid waste; Tetra Tech will provide this drum.

Liquid wastes expected to be generated during the demonstration include decontamination water and spent or excess chemicals from the devices. Decontamination water will be placed in an on-site drum designated for nonhazardous liquid waste; Tetra Tech will provide this drum. Spent or excess chemicals from the field devices will be disposed of in 20-gallon laboratory packs in accordance with 40 CFR 261.5 regulations pertaining to conditionally exempt small-quantity generators (those generating less than 100 kilograms or 220 pounds of hazardous waste during a calendar month). Spent or excess chemicals will be separated as either corrosive or flammable wastes and will be placed in separate laboratory packs. Tetra Tech will provide each developer with one laboratory pack for corrosive wastes and one for flammable wastes, as needed. The developers will be responsible for providing any secondary containment necessary within the laboratory packs. If the developers choose to, they may retain their spent or excess chemicals rather than dispose of them at the Navy BVC site. However, if they choose to retain such chemicals, and if this option would not normally be available to users of their devices, then the amount of the chemicals will be measured by Tetra Tech so that a disposal cost can be estimated. After the demonstration, the laboratory packs will be transported to either a waste storage area at the Navy BVC site or an off-site, state-approved hazardous waste facility.

7.4 Field Documentation Procedures

Field documentation will include use of field logbooks, chain-of-custody forms, and photographs. Tetra Tech will be responsible for all field documentation. Field logbooks will be labeled with the project name and number. Each page of each field logbook will be sequentially numbered. Completed logbook pages will be signed and dated by the individual responsible for the entries. An error in a field logbook or chain-of-custody form will have one line drawn through it, and this line will be initialed and dated by the person making the correction. All photographs will be logged in the field logbooks, and each such entry will include the date, time, orientation, and subject of the photograph. Specific information regarding samples (such as profiling information, visual observations, or deviations from this demonstration plan) will also be documented in the field logbooks. At the end of each day of the demonstration, each developer will have the opportunity to review the field logbooks pertaining to the developer's activities and to verify the accuracy and completeness of the observations recorded in the logbook.

Chapter 8
Calibration Requirements and Sample Management Procedures for Innovative Field Measurement Devices

This chapter identifies calibration requirements and describes sample management procedures for the seven innovative TPH field measurement devices. IDW disposal and field documentation procedures to be used for the demonstration are described in Chapter 7. The operating procedures for the devices are presented in Section 2.2.

8.1 Calibration Requirements

This section describes the calibration requirements for each TPH field measurement device. Table 8-1 summarizes the calibration requirements for each device, including the frequency of continuing calibration verification, the calibration acceptance criterion, and corrective action.

8.1.1 RemediAid™ Starter Kit

The RemediAid™ Starter Kit is calibrated using slope and intercept values (response factors) designated by the developer for calibration curves for common hydrocarbons. These response factors are presented in the device user's manual and in Table 8-2. When the hydrocarbon or hydrocarbons present in a sample are unknown, the slope and intercept values for "unknown" hydrocarbons in Table 8-2 are used to determine TPH concentration in soil. These values represent the averages of the slope and intercept values for the other hydrocarbons listed in the table. Alternatively, the users may generate their own calibration curves using site-specific data following guidelines presented in the user's manual. For the demonstration, predemonstration results will be used to determine the type of hydrocarbons present in soil samples at a given site; therefore, CHEMetrics will use the response factors presented in Table 8-2.

Table 8-1. Summary of Total Petroleum Hydrocarbon Field Measurement Device Calibration Requirements

Measurement Device	Calibration	Frequency	Acceptance Criterion	Corrective Action
RemediAid™ Starter Kit	Select slope and intercept values provided by developer for common hydrocarbons	NA	NA	NA
Infracal® TOG/TPH Analyzer, Models CVH and HATR-T	Calibration verification using all standards	At the beginning of each day	2 percent deviation from expected values	1. Repeat calibration 2. If problem persists, contact developer for technical support
	Zero calibration	Before beginning sample analyses; after every 10 samples; and at end of analyses	± 2 absorbance units	1. Repeat zero calibration 2. If problem persists, contact developer for technical support
OCMA-350	Two-point calibration using blank and standard provided by developer	Zero calibration after every 10 samples; zero and standard calibrations under conditions specified in Section 8.1.3	± 5 mg/L	1. Repeat initial calibration if discrepancy during zero calibration is $ 5 mg/L 2. If problem persists, contact developer for technical support
PetroFLAG™ Test Kit	Two-point calibration using blank and standard provided by developer	Zero and standard calibrations after every 10 samples or during drastic (± 10 °C) temperature fluctuations	If device is not calibrated properly, error message will flash	1. Repeat zero and standard calibrations until no error message flashes at end of calibration, indicating that valid calibration is complete 2. If problem persists, contact developer for technical support
Luminoscope	Three-point calibration using off-site laboratory analytical results for site-specific samples	Off site once before main analysis event	Square of correlation coefficient of calibration curve > 0.90	1. Repeat calibration 2. If problem persists, contact developer for technical support
	Calibration check using anthracene and naphthalene	At beginning and end of each day	5 percent deviation from expected values	1. Conduct instrument maintenance 2. If problem persists, contact developer for technical support
UVF-3100A	Separate six-point calibrations for GRO and EDRO using standards provided by developer	At beginning of main analysis event; after every 20 samples, rerun methanol blanks and single- or multi-point (hi and low) calibration standards	20 percent deviation from expected values	1. Prepare new standards using reference standard provided by developer, and repeat calibration 2. If problem persists, contact developer for technical support
EnSys Petro Test System	Initial (three-point) calibration	Every analysis batch	NA	NA
	Zero calibration	At beginning of each day	First, absolute absorbance value of instrument reading does not exceed 0.02	1. Prepare new calibration blanks, and repeat zero calibration 2. If problem persists, contact developer for technical support
			Second, when position of calibration blanks is switched, absorbance value changes sign	1. Repeat zero calibration 2. If problem persists, contact developer for technical support

Table 8-1. Summary of Total Petroleum Hydrocarbon Field Measurement Device Calibration Requirements (Continued)

Notes:

\> = Greater than
$ = Greater than or equal to
± = Plus or minus
EDRO = Extended diesel range organics
GRO = Gasoline range organics
L = Liter
mg = Milligram
NA = Not applicable

Table 8-2. Response Factors for Common Hydrocarbons

Hydrocarbon	Slope Value (milligram per liter)	Intercept Value (milligram per liter)
Unleaded gasoline	338.6	17.3
Weathered gasoline	108.0	2.4
Diesel	254.6	19.7
Brent crude	223.5	4.3
Lube oil	703.3	25.1
Benzene, toluene, ethylbenzene, and xylenes	87.5	8.1
Leaded gasoline	197.7	8.4
Polynuclear aromatic hydrocarbons	0.975	0.009
Unknown[a]	195.0	5.5

Note:

[a] When the hydrocarbon or hydrocarbons of interest are unknown, the slope and intercept values for "unknown" hydrocarbons are used for calibration; these values represent the averages of the slope and intercept values for the other hydrocarbons listed.

Once calibration is complete and a calibration curve is generated, the device is ready for sample analysis. The operating procedure for the RemediAid™ Starter Kit is detailed in Section 2.2.1.2.

8.1.2 Infracal® TOG/TPH Analyzer, Models CVH and HATR-T

Models CVH and HATR-T of the Infracal® TOG/TPH Analyzer can be calibrated (1) at the factory using a standard hydrocarbon mixture established by the EPA or another regulatory agency before they are distributed to users, (2) by the user using site-specific samples, or (3) by the user using calibration standards prepared on site. For the demonstration, Models CVH and HATR-T will be calibrated off site using seven- and five-point calibrations, respectively. Calibration standards for Models CVH and HATR-T will be prepared by dissolving 3-IN-ONE oil in Freon 113 and Vertrel® MCA, respectively. Model CVH will be calibrated with seven standards provided by a supplier contracted by the developer. Model HATR-T will be calibrated with five standards prepared by the user. Calibration curves will be generated for each model that will consist of absorbance values on the x-axis and corresponding concentrations on the y-axis for the calibration standards; the TPH concentration in each sample will be determined based on the sample's absorbance as read by the device.

Calibration of the Infracal® TOG/TPH Analyzer involves two steps: zero calibration and standard calibration. According to the developer, the device must be zeroed before standard calibration or sample analysis. The typical calibration procedures for Models CVH and HATR-T are presented below. For the demonstration, the developer will perform standard calibrations according to these procedures for both models off site before the main analysis event.

Step 1 - Zero Calibration

Model CVH

1. Rinse a quartz cuvette with Freon 113 until the cuvette is clean.

2. Fill the cuvette with clean Freon 113.

3. Insert the cuvette in the TPH analyzer sample stage such that the frosted side of the cuvette is facing you.

4. Press and hold the ZERO button until bAL appears on the display. Then a reading will appear.

5. Press the RUN button. The display should read 00 ± 2. If it does not, repeat the five calibration steps.

Model HATR-T

1. Hold the HATR crystal plate vertically over a waste container, and squirt a small stream of Vertrel® MCA from the wash bottle onto the HATR sample stage.

2. Shake the crystal plate to dry it, or use an antistatic wipe (for example, a Kimwipe) to wipe the crystal plate.

3. Place the crystal plate on the HATR sample stage.

4. Press and hold the ZERO button until the timer value is displayed. The timer will begin counting down to zero. Press and release the RUN button to override the timer. bAL will appear on the display, and then a reading will appear.

5. Press the RUN button. The timer will begin counting down to zero. Press the RUN button again to override the timer. The display should read 00 ± 2. If it does not, repeat the five calibration steps.

Step 2 - Standard Calibration

Model CVH

1. Insert the sealed cuvette containing the lowest concentration standard in the TPH analyzer sample stage such that the frosted side of the cuvette is facing you.

2. Record the absorbance reading of the standard.

3. Repeat Steps 1 and 2 for the remaining six standards.

4. After the absorbance readings for all calibration standards are recorded, press the CAL button once and then press the RECALL button repeatedly until “Edit” appears on the display. Press the CAL button again.

5. When prompted for the number of calibration standards used, press the UP and DOWN Arrow buttons until the display reads “7.”

6. Press the CAL button. When prompted for the absorbance and concentration of each standard, use the UP and DOWN Arrow buttons to enter the appropriate pairs of values (absorbance and concentration), beginning with the lowest concentration standard. Press the CAL button between each entry. Once all values have been entered, the display will read "idLE."

Model HATR-T

1. Put 100 mL of Vertrel® MCA in a 100-mL graduated cylinder.

2. Pour 3-IN-ONE oil into a 40-mL vial and label the vial "Stock Oil."

3. Put the appropriate amount of stock oil (see Table 8-3), beginning with the highest concentration, in the 100-mL graduated cylinder using a 100-µL glass syringe.

Table 8-3. Calibration Standards for Model HATR-T

Standard Concentration (milligram per liter)	Volume of Solvent (milliliter)	Volume of Stock Oil (microgram per liter)
4,000	100	400
2,000	100	200
1,000	100	100
500	100	50
250	100	25

4. Place a stopper on the graduated cylinder and shake for 1 minute.

5. Transfer the calibration standard into a 40-mL vial and label the vial with the date and concentration.

6. Repeat Steps 1 through 3 under Zero Calibration procedures for Model HATR-T.

7. Pour 6 to 8 mL of a calibration standard, beginning with the lowest concentration, into an extraction reservoir.

8. Seal the extraction reservoir with a sealer, and insert the tip of the air syringe into the sealer.

9. Place the tip of the extraction reservoir over a waste container and push down on the air syringe plunger so that about 1 mL of the standard drips into the container.

10. Place the tip of the extraction reservoir over a beaker and push down on the air syringe plunger so that about 1 or 2 mL of the standard drips into the beaker. Immediately collect 50 µL of the standard from the beaker using a pipette.

11. Transfer the standard from the pipette onto the center of the Model HATR-T sample stage, and record the absorbance.

12. Repeat Steps 7 through 11 for each standard in order of increasing concentration.

13. Perform Steps 4 through 6 under Standard Calibration procedure for Model CVH. (Note: In Step 5, press the UP and DOWN Arrow buttons until the display reads "5" instead of "7.")

Once calibration is complete, the device is ready for sample analysis. The operating procedure for the Infracal® TOG/TPH Analyzer is detailed in Section 2.2.2.2.

8.1.3 OCMA-350

The OCMA-350 requires two-point calibration involving a solvent blank and a calibration standard. The OCMA-350 can be calibrated using (1) the device default calibration settings for standard measurement conditions or (2) user-specified calibration settings. The default calibration settings are listed below.

- Zero calibration value: 0 mg/L
- Standard calibration value: 200 mg/L
- Solvent volume: 1 mL
- Sample mass: 1 gram

The user can change these settings by using the panel buttons on the OCMA-350 to specify new values, as necessary.

Calibration of the OCMA-350 involves two steps: zero calibration and standard calibration. According to the developer, the user must conduct zero calibration before sample analysis under one or more of the following conditions:

1. The OCMA-350 quartz cuvette is replaced.
2. A different lot of solvent is being used.
3. The device is turned off for more than 1 week.
4. A new calibration standard is prepared.
5. The device is being used continuously. (Zero calibration is required every 4 hours.)
6. The device is moved.
7. The ambient temperature and humidity have significantly changed (more than 5 °C and more than 10 percent, respectively).

The user must also conduct standard calibration before sample analysis under conditions 4, 5, 6, and 7. Horiba recommends use of B-heavy oil, which is provided with the OCMA-350, as the default calibration standard when the hydrocarbon content of the soil is unknown. Based on the results of the predemonstration investigation sampling, Horiba will use diesel as the calibration standard for the demonstration. According to the developer, the device user should use one lot of S-316 extraction solvent both for zero and standard calibration and for measurement. The zero and standard calibration steps are presented below.

Step 1 - Zero Calibration

1. Partially fill an OCMA-350 quartz cuvette with 1 to 4 mL of solvent.
2. Cap and shake the cuvette to pre-rinse it, and then pour out the solvent.
3. Put about 6 mL of solvent in the cuvette.
4. Insert the cuvette into the OCMA-350.
5. Press MEAS. and then ZERO CAL. on the OCMA-350. The device should read 0 mg/L.

Step 2 - Standard Calibration

1. Put 100 mL of solvent in a 250-mL volumetric flask.
2. Put 55.6 µL of diesel calibration standard in the flask.
3. Swirl the contents of the flask just enough to dissolve the diesel in the solvent.
4. Add solvent to fill the flask to the 250-mL mark.
5. Close the flask with a stopper, and swirl the flask contents until they are well mixed.
6. Partially fill an OCMA-350 quartz cuvette with 1 to 4 mL of diesel.
7. Cap and shake the cuvette, and then pour out its contents.
8. Put about 6 mL of diesel in the cuvette.
9. Insert the cuvette into the OCMA-350.
10. Press MEAS. and then SPAN CAL. on the OCMA-350. The device should read 200 mg/L.

Once calibration is complete, the device is ready for sample analysis. The operating procedure for the OCMA-350 is detailed in Section 2.2.3.2.

8.1.4 PetroFLAG™ Test Kit

The PetroFLAG™ Test Kit is factory-calibrated using default calibration settings for standard measurement conditions before it is issued to the user. However, to maximize field measurement accuracy, the device can also be calibrated in the field, where calibration standards provided by the developer are used to generate a two-point calibration curve.

Calibration of the PetroFLAG™ Test Kit in the field involves two steps: zero calibration and standard calibration. As part of the device, Dexsil® provides (1) an extra extraction solvent vial to be used as a blank for zero calibration and (2) a calibration standard for standard calibration. The blank and calibration standard are intended for one-time use. The zero calibration and standard calibration steps are presented below.

Step 1 - Zero Calibration

1. Add the contents of a break-top vial of extraction solvent provided by the developer to a soil extraction tube labeled “blank.”
2. Process the “blank” in accordance with the operating procedure for analyzing soil samples presented in Section 2.2.4.2.
3. After 5 seconds, the device display should read “0.”

Step 2 - Standard Calibration

1. Add the contents of the break-top vial containing the calibration standard (1,000 mg/L of mineral oil dielectric fluid) provided by the developer to the soil extraction tube labeled “standard.”
2. Process the “standard” in accordance with the operating procedure for analyzing soil samples presented in Section 2.2.4.2.
3. After 5 seconds, the device display should read “1,000.”

If the device is not calibrated properly or if the concentration is not correct, an error message will flash until NEXT is pushed. The user will be prompted to recalibrate the device until a valid calibration is completed. Once calibration is complete, the device is ready for sample analysis. The operating procedure for the PetroFLAG™ Test Kit is detailed in Section 2.2.4.2.

8.1.5 Luminoscope

The Luminoscope will be calibrated before the demonstration using off-site laboratory analytical results and device readings for each demonstration area. The calibration curve will consist of absorbance values on the x-axis and corresponding off-site laboratory sample analytical results on the y-axis; the TPH concentration in each sample will be determined based on the sample's luminescence as read by the device. Calibration curves will be generated for each demonstration area using the off-site laboratory sample analytical results and the Luminoscope results from the predemonstration investigation. This section briefly discusses additional calibration that will be conducted in the field.

A calibration check of the Luminoscope involves taking measurements in the field using a Starna quartz cuvette containing anthracene and naphthalene standards (0.2 mg/L and 0.8 mg/L, respectively) at the beginning and end of each day. According to the developer, calibration using a known contaminant should be performed to check the device's ability to consistently analyze for a known contaminant. Once the calibration check is complete, the device is ready for sample analysis. The operating procedure for the Luminoscope is detailed in Section 2.2.5.2.

8.1.6 UVF-3100A

The UVF-3100A requires six-point calibration involving five calibration standards and a calibration blank. Separate calibration curves have been generated for GRO and EDRO. Specifically, the curves have been generated at concentrations of 0.1, 0.5, 1.0, 5.0, and 10.0 mg/L for GRO and at concentrations of 0.05, 0.1, 0.5, 1.0, and 1.5 mg/L for EDRO. GRO and EDRO will be measured separately by using a filter to change the device's wavelengths. The components of each set of calibration standards provided by the developer are presented in Table 8-4.

Table 8-4. Components of Calibration Standards

Gasoline Range Organics	Extended Diesel Range Organics	
Benzene	Acenaphthene	Fluoranthene
Chlorobenzene	Acenaphthylene	Fluorene
1,2-Dichlorobenzene	Anthracene	Indeno(1,2,3-cd)pyrene
1,3-Dichlorobenzene	Benz(a)anthracene	2-Methylnaphthalene
1,4-Dichlorobenzene	Benzo(a)pyrene	Naphthalene
Ethylbenzene	Benzo(b)fluoranthene	Phenanthrene
Toluene	Benzo(g,h,i)perylene	Pyrene
m-Xylene	Benzo(k)fluoranthene	
o-Xylene	Chrysene	
p-Xylene	Dibenz(a,h)anthracene	

Calibration of the UVF-3100A involves two steps: standard calibration and zero calibration. The calibration steps are presented below.

Step 1 - Standard Calibration

1. Select the multioptional direct concentration mode for the UVF-3100A by pressing ENT at the HOME screen, 1 for setup, and then 1 again for mode. Press ESC to return to the previous screen, and then press 2 to choose the calibration procedure. Use the — key to choose "Direct Conc" for the direct concentration calibration procedure. Press ESC to return to the previous screen. To choose the unit of measure, press 3, and then use the — key to choose the required unit. Press ESC twice to return to the Setup/Cal screen.

2. To access the calibration sequence, press 2 at the Setup/Cal screen. The direct concentration calibration sequence will appear.

3. When the device prompts you for the "maximum range," press 9 to enter the range of expected concentration values.

4. Enter 5 as the number of calibration standards that will be used, and press ENT.

5. When the device prompts you for the "Hi Std Conc," press 1 to accept the default value or press 9 to enter a new value. Enter the actual concentration of the highest standard being used, and press ENT. The developer recommends that the Hi Std be about 80 percent of the maximum range entered. Additionally, the standards used should not be too close in concentration; the difference in concentration between any two standards should not be less than 10 percent of the maximum range entered.

6. Fill a clean cuvette with the Hi Std, and insert the cuvette into the sample adapter in the sample chamber. Press *. The device will adjust its sensitivity, which is displayed as the "SENS FACTOR," to the level appropriate for the standard and will then analyze the standard.

7. Remove the Hi Std cuvette. The device will prompt you to enter the actual concentration of the second standard. Repeat the process described in items 5 and 6 until all five standards have been analyzed. Press * and then ENT when each standard has been analyzed.

Step 2 - Zero Calibration

1. When the five calibration standards have been analyzed, the UVF-3100A will prompt you to insert a calibration blank. Insert the cuvette containing blank solvent into the sample adapter in the sample chamber, and press ENT.

2. Wait for the blank reading to stabilize, and then press 0. The device will read the blank as having zero concentration, and the display will automatically return to the HOME screen.

Once calibration is complete, the device is ready for sample analysis. The operating procedure for the UVF-3100A is detailed in Section 2.2.6.2.

8.1.7 EnSys Petro Test System

This section discusses calibration procedures for the EnSys Petro Test System. This device does not require generation of a calibration curve because it provides semiquantitative results.

Calibration procedures for the EnSys Petro Test System involve conducting zero calibration of the spectrophotometer. The procedures for zero calibration are presented below.

Zero Calibration

1. Prepare two calibration blanks by filling two conjugate tubes with potable water.

2. Insert the two tubes into the spectrophotometer. The reading on the display should be 0.00 ± 0.02. If the reading is outside this range, reverse the positions of the tubes.

3. If the reading changes from positive to negative or from negative to positive when the tubes' positions are reversed, there is a lack of equality between the two calibration blanks. In this case, prepare a new pair of calibration blanks and conduct zero calibration.

   If the reading remains about the same when the tubes' positions are reversed, the spectrophotometer zero needs to be adjusted. In this case, with the two tubes still in the spectrophotometer, use a small screwdriver to turn trimmer R-17 at the rear of the spectrophotometer until the display reads zero.

Once zero calibration is complete, the device is ready for sample analysis. The operating procedure for the EnSys Petro Test System is detailed in Section 2.2.7.2.

8.2 Sample Management Procedures

Demonstration samples collected for the developers for TPH analysis will be presented to the developers by Tetra Tech at the Navy BVC site. These samples will be in coolers and will be accompanied by chain-of-custody forms. Each developer will be responsible for the samples it receives. When a developer receives a sample cooler, the developer will open the cooler and carefully check its contents for evidence of breakage or leakage. The developer will verify that all information on the sample container labels is correct and consistent with the chain-of-custody forms.

Any discrepancy between sample container labels and chain-of-custody forms, any broken or leaking sample containers, or any other abnormal situation will be reported to the Tetra Tech field manager, who will implement corrective action.

Tetra Tech will provide sample data sheets to the developers in order to ensure that TPH field measurement device results are recorded in a consistent manner. The results generated by the devices will be entered on the appropriate sample data sheets.

Chapter 9
Laboratory Sample Preparation and Analytical Methods, Calibration Requirements, and Sample Management Procedures

Soil samples collected during the demonstration will be analyzed for GRO, EDRO, and percent moisture by STL Tampa East. STL Tampa East will also analyze liquid PE samples for GRO and EDRO.

This section describes the laboratory sample preparation and analytical methods, calibration requirements, and sample management procedures proposed for the demonstration.

9.1 Laboratory Sample Preparation and Analytical Methods

The laboratory sample preparation and analytical methods proposed for the demonstration are summarized in Table 9-1. Chapter 5 describes the rationale for the selection of the GRO and EDRO reference method. The SW-846 methods listed in Table 9-1 for GRO and EDRO analyses were tailored to meet the definition of TPH for the project (see Chapter 1). Project-specific procedures for soil sample preparation and analysis for GRO and EDRO are summarized in Tables 9-2 and 9-3, respectively. To prepare the liquid PE samples, STL Tampa East will add an aliquot of PE sample to the extraction solvent used for soil samples. A specified aliquot of the liquid PE sample will be diluted in methanol for GRO analysis and in methylene chloride for EDRO analysis such that the final volume of the solution for GRO and EDRO analyses is 5.0 and 1.0 mL, respectively. The solution will then be analyzed for GRO and EDRO using the same procedures as are used for soil samples. For this reason, liquid sample analyses are not separately discussed in this chapter.

9.2 Calibration Requirements

This section describes the calibration procedures, acceptance criteria, and corrective action procedures for GRO, EDRO, and percent moisture laboratory analyses. For these analyses, calibration data will be recorded on

Table 9-1. Laboratory Sample Preparation and Analytical Methods

Parameter	Method Reference (Step)	Method Title
Gasoline range organics	Based on SW-846 Method 5035 (extraction)	Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples
	Based on SW-846 Method 5030B (purge and trap)	Purge-and-Trap for Aqueous Samples
	Based on SW-846 Method 8015B (analysis)	Nonhalogenated Volatile Organics by Gas Chromatography
Extended diesel range organics	Based on SW-846 Method 3540C (extraction)	Soxhlet Extraction
	Based on SW-846 Method 8015B (analysis)	Nonhalogenated Volatile Organics by Gas Chromatography
Percent moisture	Based on MCAWW Method 160.3[a]	Residue, Total (Gravimetric, Dried at 103-105 °C)

Notes:

MCAWW = “Methods for Chemical Analysis of Water and Wastes”
SW-846 = “Test Methods for Evaluating Solid Waste”

[a] MCAWW Method 160.3 will be modified to include calculation and reporting of percent moisture in solid samples.

printouts from instrument data systems and calibration summary forms similar to the EPA Contract Laboratory Program (CLP) forms. These records will be made a permanent part of the project files. The calibration records will include tracking numbers for standards so that the source and method of preparation of each standard solution used may be identified. The frequencies, acceptance criteria, and corrective actions required for calibration of laboratory analytical measurement equipment are outlined in Table 9-4. All calibration standards for GRO and EDRO analyses will be prepared using commercially available (Supelco or equivalent) standards or using standards verified against independently prepared, separate source standards.

9.3 Sample Management Procedures

Demonstration samples collected for GRO, EDRO, and percent moisture analyses will be shipped to STL Tampa East. The laboratory sample coordinator, Carol McNulty, or her designated alternate will receive the samples and assume custody of them until they have been properly logged in the laboratory and stored in secured areas.

When a sample shipment is received at the laboratory, the cooler containing the samples will be inspected for warning labels and security breaches before it is opened. The laboratory sample coordinator will open the cooler and carefully check its contents for evidence of breakage or leakage. The interior of the cooler will also be inspected for the chain-of-custody form and other information or instructions. The temperature of the temperature blank in the cooler will be included in the sample receipt log along with the date and the signature of the person making the entry.

Table 9-2. Summary of Project-Specific Procedures for Gasoline Range Organic Analysis

SW-846 Method Reference (Step)	Project-Specific Procedures
5035 (Extraction)	
Low-level (0.5 to 200 µg/kg) or high-level (>200 µg/kg) samples may be prepared.	Because the project-specific reporting limit for GRO is 5 milligrams per kilogram, all samples to be analyzed for GRO will be prepared using procedures for high-level samples.
Samples may be collected with or without use of a preservative solution.	Samples will be collected without use of a preservative.
A variety of sample containers, including EnCores, may be used when high-level samples are collected without use of a preservative.	Samples will be containerized in EnCores.
Samples collected in EnCores should be transferred to vials containing the extraction solvent as soon as possible or analyzed within 48 hours.	Samples will be weighed and extracted within 2 calendar days of their shipment. The holding time for analysis will be 14 days after extraction. A full set of quality control samples (method blanks, MS/MSDs, and LCS/LCSDs) will be prepared at this time.
For samples not preserved in the field, a solubility test should be performed using methanol, PEG, and hexadecane to determine an appropriate extraction solvent.	Because the reference laboratory obtained acceptable results for PE samples extracted with methanol during the predemonstration investigation, samples will be extracted with methanol.
Removal of unrepresentative material from the sample is not discussed.	During sample homogenization, field sampling technicians will attempt to remove unrepresentative material such as sticks, roots, and stones if present in the sample; the reference laboratory will not remove any remaining unrepresentative material.
Procedures for adding surrogates to the sample are inconsistently presented. Section 2.2.1 indicates that surrogates should be added to an aliquot of the extract solution. Section 7.3.3 indicates that soil should be added to a vial containing both the extraction solvent (methanol) and surrogate spiking solution.	The soil sample will be ejected into a volatile organic analysis vial, an appropriate amount of surrogate solution will be added to the sample, and then methanol will be quickly added.
Nine mL of methanol should be added to a 5-gram (wet weight) soil sample.	Five mL of methanol will be added to the entire soil sample contained in a 5-gram EnCore.
When practical, dispersing the sample to allow contact with the methanol is recommended by shaking or using other mechanical means for 2 minutes without opening the sample container. When shaking is not practical, the sample should be dispersed with a narrow, metal spatula, and the sample container should be immediately resealed.	The sample will be dispersed using a stainless-steel spatula to allow contact with the methanol. The volatile organic analysis vial will then be capped and shaken vigorously until the soil is dispersed in methanol, and the soil will be allowed to settle.
5030B (Purge and Trap)	
Screening of samples before the purge and trap procedure is recommended using one of the two following techniques: Use of an automated headspace sampler (see SW-846 Method 5021) connected to a GC equipped with a photoionization detector in series with an electrolytic conductivity detector Extraction of the samples with hexadecane (see SW-846 Method 3820) and analysis of the extracts using a GC equipped with a flame ionization detector or electron capture detector	Samples will be screened with an automated headspace sampler (see SW-846 Method 5021) connected to a GC equipped with a flame ionization detector.
SW-846 Method 5030B indicates that contamination by carryover can occur whenever high-level and low-level samples are analyzed in sequence. Where practical, analysis of samples with unusually high concentrations of analytes should be followed by an analysis of organic-free reagent water to check for cross-contamination. Because the trap and other parts of the system are subject to contamination, frequent bake-out and purging of the entire system may be required.	According to the reference laboratory, a sample extract concentration equivalent to 10,000 ng on-column is the minimum concentration of GRO that could result in carryover. Therefore, if a sample extract has a concentration that exceeds the minimum concentration for carryover, the next sample in the sequence will be evaluated as follows: (1) if the sample is clean (has no chromatographic peaks), no carryover has occurred; (2) if the sample has detectable analyte concentrations (chromatographic peaks), it will be reanalyzed under conditions in which carryover would not occur.
The sample purge device used must demonstrate adequate performance.	A Tekmar 2016 autosampler and a Tekmar LSC 2000 concentrator will be used. Based on quality control sample results, the reference laboratory has demonstrated adequate performance using these devices.

Table 9-2. Summary of Project-Specific Procedures for Gasoline Range Organic Analysis (Continued)

SW-846 Method Reference (Step)	Project-Specific Procedures
5030B (Purge and Trap) (Continued)	
Purge and trap conditions for high-level samples are not clearly specified. According to SW-846, manufacturer recommendations for the purge and trap devices should be considered when this method is implemented. The following purge and trap general conditions are recommended for samples that are water-miscible (methanol extract): Purge gas: nitrogen or helium Purge gas flow rate: 20 mL/min Purge time: 15 ± 0.1 min Purge temperature: 85 ± 2 °C Desorb time: 1.5 min Desorb temperature: 180 °C Backflush inert gas flow rate: 20 to 60 mL/min Bake time: not specified Bake temperature: not specified Multiport valve and transfer line temperatures: not specified	The purge and trap conditions that will be used are listed below. These conditions are based on manufacturer recommendations for the purge device specified above and the VOCARB 3000 trap. Purge gas: helium Purge gas flow rate: 35 mL/min Purge time: 8 min with 2-min dry purge Purge temperature: ambient temperature Desorb time: 1 min Desorb temperature: 250 °C Backflush inert gas flow rate: 35 mL/min Bake time: 7 min Bake temperature: 270 °C Multiport valve and transfer line temperatures: 115 and 120 °C
8015B (Analysis)	
GC Conditions	
The following GC conditions are recommended: Column: 30-m x 0.53-mm-ID, fused-silica capillary column chemically bonded with 5 percent methyl silicone, 1.5-µm field thickness Carrier gas: helium Carrier gas flow rate: 5 to 7 mL/min Makeup gas: helium Makeup gas flow rate: 30 mL/min Injector temperature: 200 °C Detector temperature: 340 °C Temperature program: Initial temperature: 45 °C Hold time: 1 min Program rate: 45 to 100 °C at 5 °C/min Program rate: 100 to 275 °C at 8 °C/min Hold time: 5 min Overall time: 38.9 min	The HP 5890 Series II will be used as the GC. The following GC conditions will be used based on manufacturer recommendations: Column: 30-m x 0.53-mm-ID, fused-silica capillary column chemically bonded with 5 percent methyl silicone, 1.5-µm field thickness Carrier gas: helium Carrier gas flow rate: 15 mL/min Makeup gas: helium Makeup gas flow rate: 15 mL/min Injector temperature: 200 °C Detector temperature: 200 °C Temperature program: Initial temperature: 25 °C Hold time: 3 min Program rate: 25 to 120 °C at 25 °C/min Hold time: 4 min Program rate: 120 to 245 °C at 25 °C/min Hold time: 5 min Overall time: 20.4 min
Calibration	
The chromatographic system may be calibrated using either internal or external standards.	The chromatographic system will be calibrated using external standards.
Calibration should be performed using samples of the specific fuel type contaminating the site. When such samples are not available, recently purchased, commercially available fuel should be used.	Calibration will be performed using a commercially available, 10-component GRO standard that contains 35 percent aliphatic hydrocarbons and 65 percent aromatic hydrocarbons.
ICV is not required.	ICV will be performed using a second-source standard.
CCV should be performed at the beginning of every 12-hour work shift and at the end of an analytical sequence. CCV throughout the 12-hour shift is also recommended; however, the frequency is not specified.	CCV will be performed at the beginning of each analytical batch, after every tenth analysis, and at the end of the analytical batch.
CCV should be performed using a fuel standard.	CCV will be performed using a commercially available, 10-component GRO standard that contains 35 percent aliphatic hydrocarbons and 65 percent aromatic hydrocarbons.
According to SW-846 Method 8000, CCV should be performed at the same concentration as the midpoint concentration of the initial calibration curve; however, the concentration of each calibration point is not specified.	CCV will be performed at a concentration equivalent to 2,000 ng on-column.

Table 9-2. Summary of Project-Specific Procedures for Gasoline Range Organic Analysis (Continued)

SW-846 Method Reference (Step)	Project-Specific Procedures
8015B (Analysis) (Continued)	
Calibration (Continued)	
Method sensitivity check is not required.	The method sensitivity check will be performed daily using a calibration standard at a concentration equivalent to 100 ng on-column. See Table 9-4 for details.
Retention Time Windows	
The retention time range (window) should be established using 2-methylpentane (C_6) and 1,2,4-trimethylbenzene (C_{10}) during initial calibration. Three measurements should be made over a 72-hour period; the results should be used to determine the average retention time. As a minimum requirement, the retention time should be verified using a midlevel calibration standard at the beginning of each 12-hour shift. Additional analysis of the standard throughout the 12-hour shift is strongly recommended.	The retention time range will be established using the opening or first CCV specific to each analytical batch. The first eluter, C_6 (2-methylpentane), and last eluter, C_{10} (1,2,4-trimethylbenzene), of the GRO standard will be used to establish each day's retention time range.
Quantitation	
Quantitation is performed by summing the areas of all chromatographic peaks eluting within the retention time range established using 2-methylpentane (C_6) and 1,2,4-trimethylbenzene (C_{10}). Subtraction of the baseline rise for the method blank resulting from column bleed is generally not required.	Quantitation will be performed by summing the areas of all chromatographic peaks from C_6 (2-methylpentane) through C_{10} (1,2,4-trimethylbenzene). This range includes C_{10} (n-decane). Baseline rise subtraction will not be performed.
Quality Control	
Spiking compounds for MS/MSDs and LCSs are not specified.	Spiking compounds for MS/MSDs and LCSs are discussed in Sections 10.2.1.3 and 10.2.1.5, respectively.
According to SW-846 Method 8000, spiking levels for MS/MSDs are determined differently for compliance and noncompliance monitoring applications. For noncompliance applications, the laboratory may spike the sample (1) at the same concentration as the reference sample (LCS), (2) at 20 times the estimated quantitation limit for the matrix of interest, or (3) at a concentration near the middle of the calibration range.	MS/MSD spiking levels will be targeted to be between 50 and 150 percent of the unspiked sample concentration. The reference laboratory will use historical information to adjust the spike amounts or to adjust sample amounts to a preset spike amount. The spiked samples and unspiked samples will be prepared such that the sample mass and extract volume used for analysis will be the same.
According to SW-846 Method 8000, in-house laboratory acceptance criteria for MS/MSDs and LCSs should be established. As a general rule, the recoveries of most compounds spiked into a sample should fall within the range of 70 to 130 percent, and this range should be used as a guide in evaluating in-house performance.	Reference laboratory acceptance criteria for MS/MSDs and LCSs are specified in Table 10-2. The acceptance criteria are based on laboratory historical data.
The LCS should consist of an aliquot of a clean (control) matrix that is similar to the sample matrix.	The LCS/LCSD matrix will be Ottawa sand.
No LCSD is required.	LCSD spiking compounds, concentrations, and acceptance criteria are discussed in Section 10.2.1.5.
The surrogate compound and spiking concentration are not specified. According to SW-846 Method 8000, establishing in-house laboratory acceptance criteria for surrogate recoveries is recommended.	The surrogate compound will be 4-bromofluorobenzene. See Section 10.2.1.2 for details.
The method blank matrix is not specified.	The method blank matrix will be Ottawa sand.
The extract duplicate is not specified.	The extract duplicate will be performed. See Section 10.2.1.4 for details.

Notes:

± = Plus or minus
> = Greater than
μg/kg = Microgram per kilogram
μm = Micrometer
C = Carbon
CCV = Continuing calibration verification
GC = Gas chromatograph
GRO = Gasoline range organics
ICV = Initial calibration verification
ID = Inside diameter
LCS = Laboratory control sample
LCSD = Laboratory control sample duplicate
m = Meter
min = Minute
mL = Milliliter
mm = Millimeter
MS = Matrix spike
MSD = Matrix spike duplicate
ng = Nanogram
PE = Performance evaluation
PEG = Polyethylene glycol
SW-846 = "Test Methods for Evaluating Solid Waste"

Table 9-3. Summary of Project-Specific Procedures for Extended Diesel Range Organic Analysis

SW-846 Method Reference (Step)	Project-Specific Procedures
3540C (Extraction)	
Any free water present in the sample should be decanted and discarded. The sample should then be thoroughly mixed, and any unrepresentative material such as sticks, roots, and stones should be discarded.	During sample homogenization, field sampling technicians will attempt to remove unrepresentative material such as sticks, roots, and stones. In addition, the field sampling technicians will decant any free water present in the sample. The reference laboratory will not decant water or remove any unrepresentative material from the sample. The reference laboratory will mix the sample with a wooden tongue depressor.
Ten grams of soil sample should be blended with 10 grams of anhydrous sodium sulfate.	Thirty grams of sample will be blended with at least 30 grams of anhydrous sodium sulfate. For medium- and high-level samples, 6 and 2 grams of soil will be used for extraction, respectively, and proportionate amounts of anhydrous sodium sulfate will be added. The amount of anhydrous sodium sulfate used will not be measured gravimetrically but will be sufficient to ensure that free moisture is effectively removed from the sample.
Extraction should be performed using 300 mL of extraction solvent.	Extraction will be performed using 200 mL of extraction solvent.
Acetone and hexane (1:1 v/v) or methylene chloride and acetone (1:1 v/v) may be used as the extraction solvent. Note: Methylene chloride and acetone do not constitute a constant-boiling solvent and thus do not provide a suitable solvent for SW-846 Method 3540C. Methylene chloride was used as an extraction solvent for method validation of SW-846 Method 3540C.	Methylene chloride will be used as the extraction solvent.
The micro Snyder column technique or nitrogen blowdown technique may be used to adjust (concentrate) the soil extract to the required final volume.	Kuderna Danish and nitrogen evaporation will be used as the concentration techniques.
Procedures for addressing contamination carryover are not specified.	According to the reference laboratory, a sample extract concentration of 100,000 µg/mL is the minimum concentration of EDRO that could result in carryover. Therefore, if a sample extract has a concentration that exceeds the minimum concentration for carryover, the next sample in the sequence will be evaluated as follows: (1) if the sample is clean (has no chromatographic peaks), no carryover has occurred; (2) if the sample has detectable analyte concentrations (chromatographic peaks), it will be reanalyzed under conditions in which carryover would not occur.
8015B (Analysis)	
GC Conditions	
The following GC conditions are recommended: Column: 30-m x 0.53-mm-ID, fused-silica capillary column chemically bonded with 5 percent methyl silicone, 1.5-µm field thickness Carrier gas: helium Carrier gas flow rate: 5 to 7 mL/min Makeup gas: helium Makeup gas flow rate: 30 mL/min Injector temperature: 200 °C Detector temperature: 340 °C Temperature program: Initial temperature: 45 °C Hold time: 3 min Program rate: 45 to 275 °C at 12 °C/min Hold time: 12 min Overall time: 34.2 min	An HP 6890 GC will be used with the following conditions: Column: 30-m x 0.53-mm-ID, fused-silica capillary column chemically bonded with 5 percent methyl silicone, 1.5-µm field thickness Carrier gas: hydrogen Carrier gas flow rate: 1.9 mL/min Makeup gas: hydrogen Makeup gas flow rate: 23 mL/min Injector temperature: 250 °C Detector temperature: 345 °C Temperature program: Initial temperature: 40 °C Hold time: 2 min Program rate: 40 to 345 °C at 30 °C/min Hold time: 5 min Overall time: 17.2 min
Calibration	
The chromatographic system may be calibrated using either internal or external standards.	The chromatographic system will be calibrated using external standards.

Table 9-3. Summary of Project-Specific Procedures for Extended Diesel Range Organic Analysis (Continued)

SW-846 Method Reference (Step)	Project-Specific Procedures
8015B (Analysis) (Continued)	
Calibration (Continued)	
Calibration should be performed using samples of the specific fuel type contaminating the site. When such samples are not available, recently purchased, commercially available fuel should be used.	Calibration will be performed using a commercially available standard that contains even-numbered alkanes from C_{10} through C_{40}.
ICV is not required.	ICV will be performed using a second-source standard that contains even-numbered alkanes from C_{10} through C_{40}.
CCV should be performed at the beginning of every 12-hour work shift and at the end of an analytical sequence. CCV throughout the 12-hour shift is also recommended; however, the frequency is not specified.	CCV will be performed at the beginning of each analytical batch, after every tenth analysis, and at the end of the analytical batch.
CCV should be performed using a fuel standard.	CCV will be performed using a standard that contains only even-numbered alkanes from C_{10} through $C_{40.}$
According to SW-846 Method 8000, CCV should be performed at the same concentration as the midpoint concentration of the initial calibration curve; however, the concentration of each calibration point is not specified.	CCV will be performed at a concentration equivalent to 3,750 ng on-column.
Method sensitivity check is not required.	The method sensitivity check will be performed daily using a calibration standard at a concentration equivalent to 75 ng on-column.
Retention Time Windows	
The retention time range (window) should be established using C_{10} and C_{28} alkanes during initial calibration. Three measurements should be made over a 72-hour period; the results should be used to determine the average retention time. As a minimum requirement, the retention time should be verified using a midlevel calibration standard at the beginning of each 12-hour shift. Additional analysis of the standard throughout the 12-hour shift is strongly recommended.	Two retention time ranges will be established using the opening CCV for each analytical batch. The first range, labeled diesel range organics, will be marked by the end of the C_{10} (n-decane) peak through C_{28} (n-octacosane). The second range, labeled oil range organics, will be marked by the end of the C_{28} (n-octacosane) peak through C_{40} (tetracontane)
Quantitation	
Quantitation is performed by summing the areas of all chromatographic peaks eluting between C_{10} (n-decane) and C_{28} (n-octacosane).	Quantitation will be performed by summing the areas of all chromatographic peaks from greater than C_{10} (n-decane) through C_{28} (n-octacosane). A separate quantitation will also be performed to sum the areas of all chromatographic peaks from greater than C_{28} (n-octacosane) through C_{40} (tetracontane). Separate average response factors for each carbon range will be used for quantitation. The quantitation results will then be summed to determine the total EDRO concentration. All calibrations, CCVs, ICVs, and associated batch quality control will be controlled for the entire EDRO range (greater than C_{10} [n-decane] through C_{40} [tetracontane]) using a single quantitation performed over the entire EDRO range.
Subtraction of the baseline rise for the method blank resulting from column bleed is appropriate.	The reference laboratory will identify any occurrences of baseline rise in the data package. The need to subtract baseline rise will be evaluated during data validation.
Because phthalate esters contaminate many types of products commonly found in the laboratory, consistent quality control should be practiced.	Phthalate peaks, if present, will not be included in quantitation.
Quality Control	
Spiking compounds for MS/MSDs and LCSs are not specified.	Spiking compounds for MS/MSDs and LCSs are discussed in Sections 10.2.1.3 and 10.2.1.5, respectively.

Table 9-3. Summary of Project-Specific Procedures for Extended Diesel Range Organic Analysis (Continued)

SW-846 Method Reference (Step)	Project-Specific Procedures
8015B (Analysis) (Continued)	
Quality Control (Continued)	
According to SW-846 Method 8000, spiking levels for MS/MSDs are determined differently for compliance and noncompliance monitoring applications. For noncompliance applications, the laboratory may spike the sample (1) at the same concentration as the reference sample (LCS), (2) at 20 times the estimated quantitation limit for the matrix of interest, or (3) at a concentration near the middle of the calibration range.	MS/MSD spiking levels will be targeted to be between 50 and 150 percent of the unspiked sample concentration. The reference laboratory will use historical information to adjust the spike amounts or to adjust sample amounts to a preset spike amount. The spiked samples and unspiked samples will be prepared such that the sample mass and extract volume used for analysis will be the same.
According to SW-846 Method 8000, in-house laboratory acceptance criteria for MS/MSDs and LCSs should be established. As a general rule, the recoveries of most compounds spiked into a sample should fall within the range of 70 to 130 percent, and this range should be used as a guide in evaluating in-house performance.	Reference laboratory acceptance criteria for MS/MSDs and LCSs are specified in Table 10-2.
The LCS should consist of an aliquot of a clean (control) matrix that is similar to the sample matrix.	The LCS/LCSD matrix will be Ottawa sand.
No LCSD is required.	LCSD spiking compounds, concentrations, and acceptance criteria are discussed in Section 10.2.1.5.
The surrogate compound and spiking concentration are not specified. According to SW-846 Method 8000, establishing in-house laboratory acceptance criteria for surrogate recoveries is recommended.	The surrogate compound will be o-terphenyl. See Section 10.2.1.2 for details.
The method blank matrix is not specified.	The method blank matrix will be Ottawa sand.
The extract duplicate is not specified.	The extract duplicate will be performed. See Section 10.2.1.4 for details.

Notes:

µg = Microgram
µm = Micrometer
C = Carbon
CCV = Continuing calibration verification
EDRO = Extended diesel range organics
GC = Gas chromatograph
ICV = Initial calibration verification
ID = Inside diameter
LCS = Laboratory control sample
LCSD = Laboratory control sample duplicate
m = Meter
min = Minute
mL = Milliliter
mm = Millimeter
MS = Matrix spike
MSD = Matrix spike duplicate
SW-846 = “Test Methods for Evaluating Solid Waste”
v/v = Volume per volume

Table 9-4. Summary of Laboratory Calibration Requirements

Parameter	Method Reference	Calibration	Frequency	Acceptance Criterion	Corrective Action
Gasoline range organics and extended diesel range organics	SW-846 Method 8015B	Initial calibration[a,b]	Before sample analysis	RSD # 20 percent if average response factors used; correlation coefficient $ 0.990 if least-squares linear regression used	1. Check calculation 2. Check instrument operating conditions and adjust if necessary 3. Repeat initial calibration
		ICV (single-point)[c]	Beginning of analytical batch immediately after initial calibration	Instrument response within 25 percent of response obtained during initial calibration	1. Check calculation 2. Check instrument operating conditions and adjust if necessary 3. Reanalyze CCV standard 4. Repeat initial calibration 5. Inform reference laboratory quality assurance manager and Tetra Tech project manager
		CCV (single-point)[d]	Beginning of each analytical batch (opening), after every tenth analysis, and at end of analytical batch (closing)	Instrument response within 25 percent (closing CCV) and 15 percent (all other CCVs) of response obtained during initial calibration	1. Check calculation 2. Check instrument operating conditions and adjust if necessary 3. Reanalyze CCV standard 4. Repeat initial calibration 5. Reanalyze all samples analyzed since last acceptable calibration verification 6. Inform reference laboratory quality assurance manager and Tetra Tech project manager
		Method sensitivity check (single-point)[e]	Daily after opening CCV	Detection of standard	Inform reference laboratory quality assurance manager and Tetra Tech project manager
Percent moisture	MCAAW Method 160.3	Calibration check for oven temperature using NIST-traceable oven thermometer	Daily before sample analysis	Thermometer reading between 103 and 105 °C	Adjust thermostat to bring oven temperature between 103 and 105 °C
		Verification of oven thermometer reading	Annually	No deviation from reference thermometer	Establish and apply temperature correction factor

Notes:

$ = Greater than or equal to
# = Less than or equal to
C = Carbon
CCV = Continuing calibration verification
ICV = Initial calibration verification
MCAWW = “Methods for Chemical Analysis of Water and Wastes”
mg/kg = Milligram per kilogram
ng = Nanogram
RSD = Relative standard deviation
SW-846 = “Test Methods for Evaluating Solid Waste”
Tetra Tech = Tetra Tech EM Inc.

[a] For gasoline range organic analysis, initial calibration will be performed using a commercially available, 10-component gasoline range organic standard that contains 35 percent aliphatic hydrocarbons and 65 percent aromatic hydrocarbons. The standard will contain the following analytes on a percent weight basis: 2-methylpentane (15 percent); 2,2,4-trimethylpentane (15 percent); heptane (5 percent); benzene (5 percent); toluene (15 percent); ethylbenzene (5 percent); m-xylene (10 percent); o-xylene (10 percent); p-xylene (10 percent); and 1,2,4-trimethylbenzene (10 percent). The initial calibration curve will be prepared at the following levels: 100 ng; 500 ng; 1,000 ng; 2,000 ng; 4,000 ng; and 10,000 ng on-column.

Table 9-4. Summary of Laboratory Calibration Requirements (Continued)

For extended diesel range organic analysis, initial calibration will be performed using a commercially available extended diesel range organic standard that contains even-numbered alkanes from C_{10} through C_{40}. The initial calibration curve will be prepared at the following levels: 75 ng; 375 ng; 750 ng; 2,250 ng; 3,750 ng; 5,250 ng; and 7,500 ng.

[b] During initial calibration, only the high point on the noncompliant curve may be removed; subsequently, the linear range will be adjusted. If a calibration standard other than the high point appears to be biased, the point may not be removed until bias is demonstrated. Bias will be demonstrated by reanalyzing the standard. If the reanalyzed standard is not consistent with the others (relative to the calibration curve trend line), the suspect standard can be removed from the curve only with the approval of the reference laboratory quality assurance manager.

[c] ICV will be performed using a second-source standard. For GRO analysis, the ICV will be performed at a concentration equivalent to 2,000 ng on-column. For EDRO analysis, the ICV will be performed at a concentration equivalent to 3,750 ng on-column.

[d] CCV will be performed using the commercially available, 10-component gasoline range organic standard that contains 35 percent aliphatic hydrocarbons and 65 percent aromatic hydrocarbons and the commercially available extended diesel range organic standard that contains even-numbered alkanes from C_{10} through C_{40}. For GRO analysis, the CCV will be performed at a concentration equivalent to 2,000 ng on-column. For EDRO analysis, the CCV will be performed at a concentration equivalent to 3,750 ng on-column.

[e] The method sensitivity check for GRO analysis will be performed using a calibration standard at a concentration equivalent to 100 ng on-column. The method sensitivity check for EDRO analysis will be performed using a calibration standard at a concentration equivalent to 75 ng on-column.

The sample coordinator will verify that all information on the sample container labels is correct and consistent with the information on the chain-of-custody form and will sign the receipt log. The yellow copy of the chain-of-custody form will be retained in the laboratory's project files, and the white copy will be returned to Tetra Tech in order to verify sample receipt.

Any discrepancy between the sample container labels and chain-of-custody form, any broken or leaking sample containers, or any other abnormal situation will be reported to the laboratory project manager. The laboratory project manager will inform the Tetra Tech project manager of any such problem, and corrective actions will be discussed and implemented. The problem and its resolution will be noted in a nonconformance memorandum, which will be initialed and dated (electronically) by the laboratory project manager.

Each shipment of samples received at STL Tampa East will be assigned a unique lot number. Each lot will be divided into groups of 20 samples or less. In addition, each sample in the shipment will be assigned a sequential sample number, and each sample container will be assigned a unique work order number. A laboratory sample label specifying the lot number, sample number, and work order number will be attached to each sample container. A worksheet will be prepared that specifies the samples to be analyzed, the analyses to be performed, the level of QC for the project, and any other necessary information. The worksheet, accompanied by a copy of the chain-of-custody form, will be given to the laboratory project manager for review and approval. Copies of the approved worksheet will be given to the laboratory group leaders, who will schedule the extractions and analyses in accordance with applicable sample holding times and project schedules. Bench sheets initiated at the first point of sample preparation will accompany the samples throughout the analytical sequence.

Samples for GRO and EDRO analyses will be stored in designated refrigerators. Unless otherwise specified by Tetra Tech, an aliquot from the samples for EDRO analysis will be used for percent moisture determination. A logbook will be maintained for each refrigerator, and the refrigerator's temperature will be recorded each working day. All samples will be stored at 4 ± 2 °C. A sample storage logbook or form will be used to document each instance when a sample is removed from or replaced in a storage area.

All unused samples and associated extracts will be stored for 6 months after their receipt or generation at the laboratory. If a longer storage period is needed based on Tetra Tech's review of the analytical data, the Tetra Tech project manager will so notify the laboratory in writing prior to the end of the 6-month storage period. All unused samples will be stored in their original containers, and all associated extracts will be stored in bottles or vials with Teflon™-lined caps or septa. Unused samples and extracts will be stored at 4 ± 2 °C. The laboratory will contact the Tetra Tech project manager before disposing of any unused samples or extracts.

The laboratory will be responsible for properly disposing of all samples and extracts as well as all wastes associated with sample analysis. However, if a sample, extract, or waste is expected to be incompatible with a laboratory waste stream, the laboratory will notify the Tetra Tech project manager of this problem in writing, and the problem will be resolved before the demonstration begins. However, based on the results of the predemonstration investigation, no such problem is expected to occur.

Chapter 10
QA/QC Procedures

The purpose of QA/QC is to ensure generation of high-quality, scientifically valid, and legally defensible data that meet the demonstration objectives. QA objectives, internal QC checks, calculation of data quality indicators, QA reports, and special QC requirements for the demonstration of the innovative TPH field measurement devices are discussed below.

10.1 QA Objectives

The overall QA objective for the demonstration is to produce well-documented data of known quality. Data quality will be measured in terms of the data's reporting limits, precision and accuracy, completeness, representativeness, and comparability.

Depending on the measurement parameter involved, individual QA objectives or acceptance criteria were set based on either demonstration objectives or STL Tampa East's and the technology developers' experience in analyzing the predemonstration investigation samples and similar environmental samples. If analytical or measurement data fail to meet the QA objectives described in this section, corrective actions will be taken. Corrective actions associated with the reference method, field measurement device, and sample collection internal QC checks are discussed in detail in Section 10.2. In the DER, Tetra Tech will explain why any QA objectives were not met (for example, because of matrix interferences) and will describe the usefulness and limitations of all data.

10.1.1 Reporting Limits

The reporting limits for reference method, field measurement device, and sample collection parameters are presented in Table 10-1. The project-required reporting limits for GRO and EDRO presented in Table 10-1 were identified based on (1) a review of state action levels for TPH, (2) detection levels achieved by the field measurement devices

Table 10-1. Reporting Limits for Reference Method, Field Measurement Device, and Sample Collection Parameters

Parameter	Matrix	Method Reference[a]	Reporting Unit	Reporting Limit
Reference Method				
TPH measurement				
GRO	Soil	SW-846 Method 8015B (modified)	mg/kg (wet weight basis)	5[b]
EDRO	Soil	SW-846 Method 8015B (modified)	mg/kg (wet weight basis)	10[b]
GRO	Liquid	SW-846 Method 8015B (modified)	mg/L in the extract	0.02[b]
EDRO	Liquid	SW-846 Method 8015B (modified)	mg/L in the extract	0.075[b]
Percent moisture	Soil	MCAWW Method 160.3	Percent	1
Analytical cost	Not applicable	Not applicable	Dollar	10
Time required for sample analysis	Not applicable	Not applicable	Day	1
Field Measurement Devices				
TPH measurement	Soil and liquid	See Note[c]	mg/kg (wet weight basis)	See Note[d]
Number of developer technicians	Not applicable	See Section 4.2 (P6)	Person	1
Number of samples analyzed	Not applicable	See Section 4.1 (P5)	Sample	1
Time required for sample analysis activities	Not applicable	See Section 4.2 (P5)	Minute	1
Number of measurement kits required to perform analyses	Not applicable	See Section 4.2 (P6)	Kit	1
Items not included in measurement kit that are required to perform analyses	Not applicable	See Section 4.2 (P6)	Variable	1
Cost of measurement kits	Not applicable	See Section 4.2 (P6)	Dollar	10
Personal protective equipment items used during sample analyses	Not applicable	See Section 4.2 (S2)	Variable[e]	1
Power source used during sample analyses	Not applicable	None	Variable[e]	1
Work space used during sample analyses	Not applicable	None	Variable[e]	1
Volume of IDW generated	Soil and liquid	See Section 4.2 (P6)	Laboratory pack	1
Sample Collection				
Sampling location	Not applicable	See Section 7.1.1	Foot	0.5
Groundwater level	Groundwater	See Section 7.1.1.2	Inch	1
Depth interval sampled	Soil	See Section 7.1.1	Inch	1
Photoionization detector measurement	Air	Manufacturer instructions	Part per million	5
Physical description of sample	Soil	USCS	See Note[f]	Not applicable
Time required for sample homogenization	Not applicable	See Section 7.1.3.1	Minute	1
Cooler temperature	Water	See Section 7.1.3.3	°C	1

Notes:

EDRO = Extended diesel range organics
EPA = U.S. Environmental Protection Agency
GRO = Gasoline range organics
IDW = Investigation-derived waste
MCAWW = "Methods for Chemical Analysis of Water and Wastes"
mg/kg = Milligram per kilogram
mg/L = Milligram per liter
SW-846 = "Test Methods for Evaluating Solid Waste"
TPH = Total petroleum hydrocarbons
USCS = Unified Soil Classification System

Table 10-1. Reporting Limits for Reference Method, Field Measurement Device, and Sample Collection Parameters (Continued)

[a] SW-846 method reference: EPA 1996b; MCAWW method reference: EPA 1983; manufacturer instruction reference: HNU Systems, Inc. 1985

[b] Reporting limits will be adjusted for dilutions made during sample preparation and analysis.

[c] See Chapter 2 for a description of the operating procedure for each field measurement device.

[d] See Table 4-2 for developer-claimed method detection limits for each field measurement device.

[e] The personal protective equipment, power source, and work space used will vary depending on the field measurement device being demonstrated.

[f] Tetra Tech EM Inc. will record qualitative observations regarding soil sample color, composition, and water content, as appropriate.

(see Table 4-2), and (3) reporting limits achieved by STL Tampa East. These reporting limits are the concentrations at or above which the objectives for precision and accuracy can be met. The general criterion applied by STL Tampa East for evaluation of MDL data and their use to support reporting limit data is that the method detection limit for a target parameter should be lower than the reporting limit by a factor of 2 to 5. STL Tampa East performs MDL studies annually. STL Tampa East's last MDL study for (1) GRO in soil was done in September 1999 and (2) EDRO in soil was done in March 2000. MDLs for the reference method and field measurement devices will be determined under primary objective P1 as discussed in Section 4.3.

10.1.2 Precision and Accuracy

QA objectives for precision and accuracy depend on the types of samples to be collected, the analyses to be performed, and the ultimate use of the analytical data. Table 10-2 summarizes the precision (RPD) and accuracy (percent recovery [%R]) acceptance criteria for the reference method, field measurement device, and sample collection parameters. Specific QC samples that will be used to estimate precision and accuracy, such as MS/MSDs and laboratory control sample/laboratory control sample duplicates, are discussed in Section 10.2. The equations that will be used to estimate precision and accuracy are presented in Section 10.3.

10.1.3 Completeness

Completeness is determined by assessing the amount of valid data obtained from a measurement system compared to the amount of data planned to be obtained. The percent completeness (%C) is calculated by dividing the number of samples yielding valid data by the total number of samples planned for collection and multiplying by 100. The project QA objective for completeness is 100 percent for each parameter. If completeness is less than 100 percent, Tetra Tech will prepare documentation explaining why this objective was not met and what impact, if any, the lower percentage will have on meeting the demonstration objectives. The equation that will be used to calculate %C is presented in Section 10.3.

10.1.4 Representativeness

The QA objective for representativeness is to obtain samples and measurements that represent demonstration area conditions. For example, predemonstration investigation results have been used to select demonstration sampling locations and depth intervals that possess appropriate physical and chemical characteristics for assessing the

Table 10-2. Internal Quality Control Checks, Frequencies, Acceptance Criteria, and Corrective Actions for Reference Method, Field Measurement Device, and Sample Collection Parameters

Parameter	Analytical Method	QC Check	Frequency[a]	Acceptance Criterion	Corrective Actions
Reference Method					
GRO and EDRO	SW-846 Method 8015B (modified)	Method blank (instrument blank for EDRO liquid PE samples)	One per analytical batch	#Reporting limit	1. Check calculations 2. Assess and eliminate sources of contamination 3. Reanalyze blank 4. Inform Tetra Tech project manager 5. Flag impacted data
		Surrogate (not performed for EDRO liquid PE samples)	Every sample, including every QC sample	GRO[b]: 39 to 163 %R EDRO[b]: 45 to 143 %R	1. Check calculations 2. Check instrument operating conditions and adjust if necessary 3. Reanalyze impacted samples 4. Inform Tetra Tech project manager 5. Flag impacted data
		MS/MSD (not performed for EDRO liquid PE samples)	One per analytical batch	GRO: RPD # 25 33 to 115 %R EDRO: RPD # 45 46 to 124 %R	1. Check calculations 2. Check LCS/LCSD and extract duplicate results to determine whether they meet acceptance criteria 3. Inform Tetra Tech project manager 4. Flag impacted data
		Extract duplicate (for soil samples only)	One per field triplicate	GRO: RPD # 25 EDRO: RPD # 45	1. Check calculations 2. Check MS/MSD and LCS/LCSD results to determine whether they meet acceptance criteria 3. Inform Tetra Tech project manager 4. Flag impacted data
		LCS/LCSD (not performed for EDRO liquid PE samples)	One per analytical batch	GRO: RPD # 25 33 to 115 %R EDRO: RPD # 45 46 to 124 %R	1. Check calculations 2. Check instrument operating conditions and adjust if necessary 3. Check MS/MSD and extract duplicate results to determine whether they meet acceptance criteria 4. Inform Tetra Tech project manager 5. Reanalyze entire batch, including QC samples 6. Flag impacted data
Percent moisture	MCAWW Method 160.3	Laboratory duplicate	One per analytical batch	RPD # 20	1. Check calculations 2. Reanalyze impacted samples 3. Inform Tetra Tech project manager 4. Flag impacted data
Field Measurement Devices					
TPH	All field measurement devices	Simultaneous measurements of time required for sample analysis activities	One per day prior to sample analysis activities	RPD # 10	1. Repeat measurements using primary stopwatches 2. Conduct measurements using primary stopwatches and secondary stopwatch; conduct measurements with primary stopwatch that meets acceptance criterion when compared to secondary stopwatch 3. Replace primary stopwatch identified as unacceptable
	RemediAid™ starter kit	None specified	Not applicable	Not applicable	Not applicable

Table 10-2. Internal Quality Control Checks, Frequencies, Acceptance Criteria, and Corrective Actions for Reference Method, Field Measurement Device, and Sample Collection Parameters (Continued)

Parameter	Analytical Method	QC Check	Frequency^a	Acceptance Criterion	Corrective Actions
TPH (Continued)	Models CVH and HATR-T	None specified	Not applicable	Not applicable	Not applicable
	OCMA-350	Sample duplicate	One per analytical batch	None specified	None specified
		Blank spike (clean sand spiked with diesel)	One per 40 samples	None specified	None specified
	PetroFLAG™ test kit	None specified	Not applicable	Not applicable	Not applicable
	Luminoscope	Reagent blank	Two per day; one prior to and one after sample analysis activities	Peak shift of less than 5 nm and change in intensity of less than 15 counts	1. Clean cuvette and restart instrument 2. Reanalyze reagent blank
		Solvent blank	One per analytical batch	Increase in peak height of less than 5 counts and no additional peaks on chromatogram	Replace solvent and reanalyze blank
		Cuvette decontamination solution blanks	One per analytical batch	No visible peaks on chromatogram	Clean cuvette and reanalyze new decontamination solution
	UVF-3100A	Two dilutions of soil sample extract	One per analytical batch	RPD $\leq$ 20	1. Check calculations 2. Reanalyze analytical batch
		Blank spike (clean sand spiked with calibration standards)	One per analytical batch	80 to 120 %R	1. Check calculations 2. Analyze solvent 3. Analyze sand blank 4. Analyze spiking material 5. Reanalyze blank spike
		MS	One per analytical batch	80 to 120 %R	1. Check calculations 2. Analyze solvent 3. Analyze spiking material 4. Reanalyze MS
	EnSys Petro Test System	m-Xylene reference standard duplicates	One per analytical batch	Difference in absorbance value between standards is $\geq$ -0.30	Reanalyze soil sample extracts in batch since last successful QC check

Table 10-2. Internal Quality Control Checks, Frequencies, Acceptance Criteria, and Corrective Actions for Reference Method, Field Measurement Device, and Sample Collection Parameters (Continued)

Parameter	Analytical Method	QC Check	Frequency[a]	Acceptance Criterion	Corrective Actions
Sample Collection					
GRO, EDRO, and TPH	SW-846 Method 8015B (modified) and all field measurement devices	Field triplicates[c]	See Table 7-3	RSD # 50	Not applicable
Time required for sample homogenization	None	Simultaneous measurements	Once per day prior to sample collection activities	RPD # 10	1. Repeat measurements using primary stopwatches 2. Conduct measurements using primary stopwatches and secondary stopwatch; conduct measurements with primary stopwatch that meets acceptance criterion when compared to secondary stopwatch 3. Replace primary stopwatch identified as unacceptable
Cooler temperature	None	Temperature blank	One per cooler containing soil samples for GRO, EDRO, or TPH analysis	4 ± 2 °C	1. Replace thermometer 2. Repeat measurement 3. Inform Tetra Tech project manager

Notes:

$ = Greater than or equal to
# = Less than or equal to
± = Plus or minus
EDRO = Extended diesel range organics
GRO = Gasoline range organics
LCS = Laboratory control sample
LCSD = Laboratory control sample duplicate
MCAWW = “Methods for Chemical Analysis of Water and Wastes”
MS = Matrix spike
MSD = Matrix spike duplicate
nm = Nanometer
QC = Quality control
%R = Percent recovery
RPD = Relative percent difference
SW-846 = “Test Methods for Evaluating Solid Waste”
Tetra Tech = Tetra Tech EM Inc.
TPH = Total petroleum hydrocarbons

[a] For the demonstration, an analytical batch of soil samples will be defined as 20 or fewer samples, including QC samples.

[b] The acceptance criterion is applicable when the dilution factor is less than five.

[c] Tetra Tech will submit field triplicate samples as blind samples; no laboratory corrective action is applicable.

performance of the field measurement devices. In addition, to ensure that each sample analyzed by the developers and STL Tampa East is representative of the material collected during the demonstration, (1) all soil collected in a specified depth interval at a given sampling location will be homogenized in the field, and (2) the PE samples prepared by ERA will be homogenized by ERA. Also, sampling procedures will be implemented in the field to minimize bias associated with TPH volatilization during filling of sample containers. These procedures are discussed in Section 7.1.3.1.

10.1.5 Comparability

Tetra Tech will ensure data comparability by (1) using standard methods such as SW-846 (GRO and EDRO) and MCAWW (percent moisture) methods, (2) consistently reporting results in the standard units shown in Table 10-1, and (3) presenting data in tabular and graphic formats that allow effective comparisons.

10.2 Internal QC Checks

Internal QC will consist of checks used to ensure that QA objectives are met. These checks are also intended to identify any need for corrective action. Internal QC checks apply to the reference method, field measurement devices, and sample collection. A discussion of the internal QC checks to be used for the demonstration is provided below.

10.2.1 Reference Method QC Checks

With regard to reference method QC checks, QC checks will be used to (1) demonstrate the absence of interferents and contamination from laboratory glassware and reagents, (2) verify that the measurement system is in control, (3) evaluate the precision and accuracy of laboratory analyses, and (4) ensure the comparability of data. Reference method QC checks for soil samples will consist of method blanks, surrogates, MS/MSDs, extract duplicates, and LCS/LCSDs for GRO and EDRO analyses, and laboratory duplicates will be used as QC checks for percent moisture analysis.

Reference method QC checks for liquid PE samples will consist of method blanks, surrogates, MS/MSDs, and LCS/LCSDs for GRO analysis. Because EDRO analysis does not contain a preparation step, surrogates, MS/MSDs, and LCS/LCSDs will not be performed for EDRO analysis. However, an instrument blank will be performed for EDRO analyses as a method blank equivalent.

The reference method QC checks for both environmental and PE samples are discussed below; the frequencies, acceptance criteria, and corrective actions for the QC checks are presented in Table 10-2.

10.2.1.1 Method and Instrument Blanks

Method or instrument blanks will be used to verify that steps in the analytical procedures do not introduce contaminants that affect analytical results, as applicable. Each method blank for soil samples will be prepared by adding all reagents and surrogates, as appropriate, to Ottawa sand. Liquid PE sample method blanks for GRO analysis will be prepared by adding 100 µL of methanol and surrogates, as appropriate, to 5 mL of deionized water. These blanks will then undergo all the procedures required for sample preparation. The method blanks will be analyzed along with environmental and PE samples prepared under identical conditions. For liquid PE samples analyzed for EDRO, instrument blanks that consist of the solvent (methylene chloride) without the surrogate will be analyzed.

10.2.1.2 Surrogates

Surrogates will be spiked into each soil sample for GRO and EDRO analyses before extraction to determine whether significant matrix effects exist within the samples and to measure the efficiency of analyte recovery during sample preparation and analysis. Each surrogate spike will be prepared by adding a known amount of the surrogate to a sample; the compound will be similar to the compounds that the sample will be analyzed for. The calculated %R of the spike will be used as a measure of the accuracy of the total analytical method. STL Tampa East will use 4-bromofluorobenzene and o-terphenyl as the surrogate spiking compounds for GRO and EDRO analyses, respectively. For GRO analysis, 0.25 mL of 40-mg/L 4-bromofluorobenzene will be added to each soil sample. For EDRO analysis, 1.0 mL of 100-mg/L o-terphenyl will be added to each soil sample.

A surrogate will also be spiked into each liquid PE sample for GRO analysis. STL Tampa East will spike 5 µL of 40-µg/mL 4-bromofluorobenzene into 5 mL of deionized water.

10.2.1.3 MS/MSDs

MS/MSD results will be evaluated to determine the accuracy and precision of the analytical results with respect to the effects of the sample matrix. The matrix spiking solutions for soil samples will be (1) the 10-component GRO calibration standard for GRO analysis and (2) the EDRO standard that contains even-numbered alkanes from C_{10} through C_{40} for EDRO analysis. For GRO analysis, 0.25 mL of 400-mg/L spiking solution will be added to each soil sample designated as an MS/MSD. For EDRO analysis, 1.0 mL of 1,500-mg/L spiking solution will be added to each soil sample designated as an MS/MSD. If the MS/MSD recovery limits specified in Table 10-2 are not met, the failure to meet the recovery limits will not be attributed to the initial, inappropriate spiking levels. In such cases, target spiking levels for MS samples will be targeted to be between 50 and 150 percent of the unspiked sample concentration. The reference laboratory will use historical information to adjust the spike amounts or will adjust sample amounts to a preset spike amount. The spiked and unspiked samples will be prepared such that the sample mass and extract volume used for analysis will be the same. The MS/MSD acceptance criteria presented in Table 10-2 are based on STL Tampa East historical data and STL Tampa East's experience in analyzing the predemonstration investigation samples.

MS/MSDs for liquid PE samples for GRO analysis will also be prepared. STL Tampa East will spike 5 µL of 40-µg/mL GRO spike solution and surrogate into 5 mL of deionized water and an appropriate volume of diluted sample.

10.2.1.4 Extract Duplicates

For GRO and EDRO analyses, extract duplicates will be evaluated to determine the precision associated with laboratory analytical procedures following soil sample extraction. STL Tampa East will sample duplicate aliquots of the GRO and EDRO extracts for analysis. Comparison of MS/MSD RPD results with extract duplicate RPD results should indicate whether any out-of-control situation associated with MS/MSD precision occurred during the extraction or analytical procedures.

10.2.1.5 LCS/LCSDs

For GRO and EDRO analyses, LCS/LCSD results will be evaluated to determine whether observed deviations for MS/MSD samples and extract duplicates were caused by a matrix effect. Ottawa sand will be spiked with matrix spiking compounds to generate soil LCS/LCSDs. The spiking compounds will be the same as those for MS/MSDs. The spiking levels for GRO and EDRO soil LCS/LCSDs will be 20 and 50 mg/kg, respectively. Five mL of deionized water and 100 µL of methanol will be spiked with 5 µL of 40-µg/mL GRO spike solution to generate liquid PE sample LCS/LCSDs for GRO analysis. The LCS/LCSD acceptance criteria presented in Table 10-2 are based on STL Tampa East historical data.

10.2.1.6 Laboratory Duplicate

For percent moisture analysis, laboratory duplicates will be evaluated to determine the precision associated with laboratory analytical procedures for the entire method. STL Tampa East will sample duplicate aliquots of soil samples for GRO and EDRO analyses.

10.2.2 Field Measurement Device QC Checks

QC checks for field measurement devices will be used to evaluate the quality of field TPH measurements. The frequencies, acceptance criteria, and corrective actions for QC checks proposed by the developers for the demonstration are presented in Table 10-2. In many cases, the devices do not have established QC checks. Tetra Tech discussed such situations with the developers, explained the significance of QC checks, and gave the developers an opportunity to propose QC checks and associated frequencies, acceptance criteria, and corrective actions. As shown in Table 10-2, QC check information for the devices is incomplete or unavailable in a few cases. During the demonstration, if the technology developers choose to modify the proposed QC checks listed in Table 10-2, Tetra Tech will record the modifications in field logbooks.

10.2.3 Sample Collection QC Checks

QC checks for sample collection will be used to evaluate the quality of sample collection activities. In general, the QC checks will be used to (1) assess the representativeness of the samples and (2) ensure that the degree to which the analytical data are representative of actual demonstration area conditions is known and documented. QC checks for sample collection will consist of field triplicates, the time required for sample homogenization, and temperature blanks. Acceptance criteria and associated corrective actions for sample collection QC checks are presented in Table 10-2. No internal QC checks are recommended for the PID. According to the PID manufacturer, the quality of sample measurements is controlled by performing a daily calibration check using a disposable cylinder containing isobutylene. The PID is readjusted if its reading deviates from the known concentration.

Field triplicate samples for GRO, EDRO, and TPH analyses will be collected to evaluate whether a sample is adequately homogenized in the field prior to filling of sample containers. Such an evaluation can be made by taking into account the analytical precision results achieved for MS/MSDs, LCS/LCSDs, and extract duplicates. Field triplicate samples will be submitted to the developers and STL Tampa East as blind samples (that is, the developers and STL Tampa East will not know which samples are replicates). The developers and STL Tampa East will not be responsible for comparing sample analytical results to the acceptance criteria. Tetra Tech will conduct this comparison after data verification and validation are complete.

Other sample collection parameters identified in Table 10-1 (for example, sampling location and groundwater level) do not require QC checks because (1) measurement precision cannot be verified with repeated measurements and (2) measurement accuracy cannot be assessed using another procedure that is considered to be more reliable or accurate.

10.3 Calculation of Data Quality Indicators

This section presents the procedures that will be used to calculate the following data quality indicators: precision, accuracy, completeness, and representativeness. Data analysis procedures that will be used to determine the MDLs of each field measurement device and the reference method are presented under primary objective P1 in Section 4.3.

10.3.1 Precision

Precision will be evaluated using the RPD when two measurements are made and using the RSD when more than two measurements are made. The RPD for GRO, EDRO, and TPH analyses will be estimated for MS/MSD samples, extract duplicates, LCS/LCSD, and laboratory duplicates, as applicable. The RPD between the analyte concentrations measured in the MS and MSD samples will be calculated using Equation 10-1.

$$\mathrm{RPD} = \frac{|\mathrm{MS} - \mathrm{MSD}|}{0.5(\mathrm{MS} + \mathrm{MSD})} \times 100 \qquad (10\text{-}1)$$

where

RPD = Relative percent difference
MS = Analyte concentration in MS sample
MSD = Analyte concentration in MSD sample

In Equation 10-1, MS and MSD will be replaced by LCS and LCSD for LCS/LCSD analyte concentrations and by E and ED for extract and extract duplicate analyte concentrations, respectively. The RPD between percent moisture measurements will also be calculated using Equation 10-1, where MS and MSD will be replaced by S and D for the sample and duplicate sample percent moisture measurements, respectively. In addition, the RPD will be estimated for sample homogenization time and sample analysis time in the field. For these parameters, two simultaneous stopwatch readings (T_1 and T_2) will replace the MS/MSD analyte concentrations (MS and MSD) in Equation 10-1. The QC check procedure for time measurement will end when either T_1 or T_2 equals 5 minutes.

The RSD will be used to estimate precision for soil field triplicate analyses. The RSD among field triplicate analytical results will be calculated using Equation 4-7.

10.3.2 Accuracy

Accuracy will be estimated for sample analyses for GRO and EDRO by calculating %R for MS and LCS samples using Equation 10-2. The developers will use the same equation to calculate accuracy for spiked samples, as applicable.

$$\%R = \frac{\left(C_j - C_o\right)}{C_t} \times 100 \tag{10-2}$$

where

$\%R$ = Percent recovery
C_j = Measured concentration in spiked sample aliquot
C_o = Measured concentration in unspiked sample aliquot
C_t = Actual concentration of spike added

Equation 10-2 will also be used to calculate %R for surrogates, where

C_j = Measured surrogate concentration in spiked sample aliquot
C_o = Zero assumed because surrogate will not be present in sample
C_t = Actual concentration of surrogate added

Estimation of accuracy for percent moisture analyses and sample collection measurements is not applicable to the demonstration. The accuracy of field measurement devices will be addressed under primary objective P2, which is discussed in Chapter 4.

10.3.3 Completeness

Completeness will be reported as the percentage of measurements judged to be valid. Equation 10-3 will be used to calculate %C.

$$\%C = \frac{V}{T} \times 100 \qquad (10\text{-}3)$$

where

%C = Percent completeness
V = Number of measurements judged to be valid
T = Total number of measurements planned

10.3.4 Representativeness

Representativeness will be evaluated in relation to the demonstration design. Field triplicate samples will be collected in each demonstration area, and their analytical results will be compared to determine representativeness. In addition, the results of the MS/MSD and extract duplicate sample analyses (see Section 10.3.1) may also be helpful in evaluating the representativeness of the demonstration data.

10.4 QA Reports

Effective management of demonstration data collection efforts will require timely assessment and review of these efforts. Effective interaction and feedback among project team members will therefore be essential. When appropriate, the Tetra Tech project manager will discuss QA issues with the EPA project manager as they arise. The Tetra Tech project manager will also summarize QA issues and their resolutions in monthly status reports to the EPA project manager. QA issues may pertain to the following matters:

- Deviations from the demonstration plan
- Corrective action activities
- Outstanding issues and proposed resolutions
- Audit results

10.5 Special QC Requirements

For the demonstration, Tetra Tech has identified special QC requirements. First, STL Tampa East will analyze soil samples for GRO and EDRO using a modified version of SW-846 Method 8015B that requires special QC procedures. The project-specific procedures for sample preparation and analysis for GRO and EDRO are summarized in Tables 9-2 and 9-3, respectively. Second, field triplicates, MS/MSDs, and extract duplicates will be collected at a frequency of one per depth interval in each sampling area for analysis by the developers and STL Tampa East, which corresponds to a frequency of about one for every four environmental samples. Typically, field replicates are collected at a frequency of 1 for every 10 environmental samples and MS/MSDs are collected at a frequency of 1 for every 20 environmental samples. Extract duplicates are typically not collected for soil organic analyses.

Chapter 11
Audits and Corrective Actions

Demonstration measurement systems and associated data will be assessed both on a day-to-day basis by Tetra Tech project personnel (routine assessments) and on a periodic basis by independent personnel (audits). Corrective actions will be formulated and implemented in response to any data quality issues that arise during routine assessments or audits. Routine assessments and corrective actions for field measurement device and laboratory calibrations are presented in Chapters 8 and 9, respectively. Chapter 10 presents routine assessments and corrective actions associated with field and laboratory QC procedures.

Although routine assessment is generally the most effective means to identify data quality issues, personnel directly involved in a project may not always recognize a data quality issue. Therefore, audits will be conducted to provide an independent view of demonstration measurement systems and data as well as additional assurance that data quality issues are being identified and appropriate corrective actions are being taken.

QA audits are independent assessments of measurement systems and associated data and are more rigorous than routine assessments. QA audits may be internal or external and most commonly incorporate technical system reviews and analysis of blind or double-blind PE samples. For the demonstration, internal QA audits will be conducted by Tetra Tech's project technical consultant, Jerry Parr of Catalyst, at the direction of the Tetra Tech SITE QA manager. The Tetra Tech project manager will be present during all audits conducted for the demonstration. External QA audits will be conducted by an independent organization such as the EPA.

As part of the predemonstration investigation, Tetra Tech's field sampling activities, including sample collection, homogenization, containerization, packaging, and shipment, were audited by Catalyst. STL Tampa East's sample receipt and storage, chain-of-custody, preparation, analysis, and data reporting procedures for the predemonstration investigation were also audited by Tetra Tech and Catalyst. The findings of these internal technical system audits (TSA) were discussed with the EPA project manager and other demonstration participants.

System audits, performance audits, and associated corrective action procedures are described below.

11.1 System Audits

System audits include thorough evaluations of field and laboratory sampling and measurement systems. For the demonstration, such audits will be conducted at the direction of the EPA project manager, the EPA QA officer, or the Tetra Tech SITE QA manager.

For the demonstration, Catalyst will conduct an internal TSA of field sampling and measurement systems during Tetra Tech's sampling activities and the developers' TPH measurement activities, respectively. In addition, Tetra Tech and Catalyst will conduct an in-process TSA of STL Tampa East's critical measurement activities. Specifically, STL Tampa East will be audited to examine its measurement of GRO, EDRO, and percent moisture in soil samples and of GRO and EDRO in liquid PE samples.

The activities that will be audited during field sampling, field measurement, and laboratory measurement TSAs are summarized in Table 11-1.

Table 11-1. Activities to be Assessed During Field Sampling, Field Measurement, and Laboratory Measurement Technical System Audits

Field Sampling Activity	Field Measurement Activity	Laboratory Measurement Activity
Sample collection at Navy BVC site Sample homogenization Sample containerization Field QA/QC Field documentation Decontamination Sample labeling, packaging, and shipping Project management and QA activities that may impact data quality	Standards preparation (as applicable) Calibration Sample measurement Data reporting QA/QC procedures Project management and QA activities that may impact data quality	Sample receipt and storage Internal chain-of-custody procedures Sample preparation Standards preparation and storage; use of second-source standards Calibration QA/QC procedures Data reduction, validation, and reporting Project management and QA activities that may impact data quality

Notes:

BVC = Base Ventura County
QA = Quality assurance
QC = Quality control

External TSAs of field sampling and field and laboratory measurement activities may also be conducted by the EPA at the discretion of the EPA project manager and QA officer. If the EPA elects to perform a field TSA, Tetra Tech will coordinate an internal TSA with the EPA's TSA and will schedule the audits to occur on consecutive days. The internal TSA will then be identified as a pre-audit and will be used to identify issues for resolution during the EPA's TSA. If the EPA elects not to perform an external field or laboratory TSA, Tetra Tech will include the EPA project manager and QA officer in the debriefing for each internal TSA and will submit all TSA documentation to the EPA for review.

Internal TSAs will be conducted in accordance with (1) Tetra Tech's internal guidance for SITE projects and (2) applicable EPA technical directives and guidance. Based on Tetra Tech's internal guidance, the audit process to be implemented by the assigned auditor for a laboratory audit is summarized below.

- A checklist is developed based on the EPA-approved demonstration plan and analytical methods identified in the demonstration plan.
- Actual laboratory activities are observed and compared to the activities described in the EPA-approved demonstration plan and in the analytical methods using the checklist.
- Nonconformances and corrective actions are discussed on site; any immediate corrective action is observed and documented, when possible.
- A draft TSA report is prepared to document any observed nonconformance as well as any immediate, corrective action that was implemented.
- The draft TSA report is reviewed by Tetra Tech personnel for technical, editorial, and overall quality.
- The draft TSA report is distributed to the laboratory, the EPA project manager and QA officer, the Tetra Tech project manager and SITE QA manager, and the Catalyst project technical consultant.
- Any laboratory response to the draft TSA report is reviewed to assess its impact on the issue or proposed corrective action.
- A final TSA report is prepared, subjected to Tetra Tech's internal review process, and distributed to the laboratory, the EPA project manager and QA officer, the Tetra Tech project manager and SITE QA manager, and the Catalyst project technical consultant.

11.2 Performance Audits

As part of the predemonstration investigation, double-blind PE samples were analyzed by the developers and STL Tampa East. The findings and this performance audit were discussed with the EPA project manager and all other demonstration participants.

As directed by the EPA project manager, a performance audit of field measurement device and STL Tampa East measurement activities will be conducted for GRO and EDRO analyses of soil and liquid samples during the demonstration. Tetra Tech will obtain PE samples for GRO and EDRO analyses from ERA and will have the developers and STL Tampa East analyze them as blind samples. Chapter 4 discusses the PE samples that will be used for the demonstration. The results of the PE sample analyses will be reviewed by Tetra Tech and will be reported to the EPA project manager, the developers, and the STL Tampa East project manager. Performance audit findings, any nonconformances, and their resolutions will be documented in the DER for the demonstration.

11.3 Corrective Action Procedures

If a problem is detected during a system or performance audit, the following procedures will be followed:

- The Tetra Tech project manager will immediately discuss the problem and any corrective action to be taken with the field or laboratory personnel responsible, the Catalyst project technical consultant, and all other appropriate personnel.

- The Tetra Tech project manager, the Catalyst project technical consultant, the developer or the laboratory project and QA managers (as appropriate), and the EPA project manager will develop a plausible course of corrective action.

- The Tetra Tech project manager and the developer or the laboratory project manager (as appropriate) will implement the corrective action and assess its effectiveness.

- The audit report and associated response will serve as the documentation of the problem and corrective action. The Tetra Tech project manager and the developer or laboratory project manager (as appropriate) will be responsible for ensuring that corrective actions identified through the audit process are fully implemented.

Chapter 12
Data Management

To ensure that demonstration data are scientifically valid, defensible, and comparable, appropriate procedures will be used to perform data management. This chapter describes (1) data reduction, (2) data review, (3) data reporting, and (4) data storage procedures for the demonstration.

12.1 Data Reduction

Each analytical method selected for the demonstration and each innovative TPH field measurement device's instruction manual contain detailed instructions and equations for calculating compound concentrations and other parameters. Data will be reduced to the units presented in Table 10-1 using the procedures described in the analytical methods. When appropriate, TPH results will be corrected for solvent dilution to account for moisture present in soil samples. For example, if a soil sample contains 20 percent moisture, the GRO analytical result will be multiplied by 1.2 to calculate the GRO concentration in mg/kg on a wet weight basis and adjusted for methanol dilution. For liquid PE samples, concentrations will be reported in mg/L of diluted extract. These concentrations will be converted to mg/L of sample by Tetra Tech using the appropriate dilution factor.

12.2 Data Review

A review of field and laboratory analytical data will be conducted by each developer and STL Tampa East, respectively. Tetra Tech will also conduct a review of all field and laboratory data. The review processes that will be used for field and laboratory analytical data are described below.

12.2.1 Data Review by Developers

Each developer will review all results generated by its field measurement device. At a minimum, the developer will report measurement results in mg/kg TPH on a wet weight basis. The developer's GRO and EDRO measurements may also be reported, if applicable. The developer's assessment of the investigative sample data and QC results will be summarized for discussion with the Tetra Tech and EPA project managers.

12.2.2 Data Review by STL Tampa East

STL Tampa East will report measurement results for GRO, DRO, oil range organics (greater than C_{28} through C_{40}), and percent moisture. All soil sample GRO, DRO, and oil range organic results will be reported in mg/kg on a wet weight basis. Review of 100 percent of the data will be conducted by STL Tampa East in accordance with a three-level review process. Level 1 and 2 reviews will be conducted by a laboratory analyst and supervisor, respectively, and will serve as a validation of the analytical data by those involved in analyzing the samples. The Level 3 review will be performed by the laboratory project manager and will serve as a final check of the completeness of each data package. Specific actions that will be performed during the three-level review process are presented below.

During the Level 1 review, a laboratory analyst will verify the following:

- Sample preparation information is correct and complete and includes documentation of standards and sample amounts.
- Analysis information is correct and complete and includes proper identification of analysis outputs (charts, chromatograms, and others).
- Analytical results are correct and complete, and include calculations or verifications of instrument calibrations, QC results, and quantitative sample results with appropriate qualifiers.
- The appropriate analytical methods have been followed and are identified in the project records.
- Proper documentation procedures have been followed.
- All nonconformances have been documented.
- Project-specific sample preparation and analytical requirements have been met.
- The data generated have been reported with the number of significant figures specified by the analytical methods or as appropriate.

Following the Level 1 review, the Level 2 review will be performed by the laboratory analyst's supervisor or a designee to ensure that the Level 1 review has been completed correctly. The Level 2 review will include an evaluation of all items required for each raw data package. Also included in this review will be an assessment of data acceptability based on the following elements:

- Adherence of the procedures used to the required analytical methods
- Correct interpretation of chromatograms
- Correctness of numerical input when computer programs are used (checked randomly)
- Correct identification and quantitation of constituents with appropriate qualifiers
- Acceptability of QC results
- Documentation that instruments were operating in accordance with method specifications (including those for calibrations and performance checks)
- Documentation of dilution factors and standard concentrations
- Adherence to sample holding times

The Level 2 review will also serve as a verification that the process followed by the laboratory analyst is correct with regard to the following:

- The analytical procedures follow the methods and specific instructions in the project QA summary memorandum issued by the laboratory project manager.
- Any nonconformances have been addressed through corrective actions that are defined in a laboratory nonconformance memorandum.
- Valid interpretations have been made during examination of the data, and the review comments of the laboratory analyst are correct.
- Each raw data package contains all the necessary documentation for data review and report production, and results are reported in a manner consistent with the method used for preparation of data reports.

Following the Level 2 review, the Level 3 review will be performed by the laboratory project manager. This review will serve to verify the completeness of each data package and to ensure that demonstration requirements have been met for the analyses performed. The laboratory project manager will verify the following:

- Analytical results are presented for every sample in the analytical batch, reporting group, or sample lot.

- Every demonstration parameter or target compound is reported with either a value or reporting limit.
- The correct units and correct numbers of significant figures are used.
- All identifications of nonconformances, including holding time violations, and data evaluation statements that involve data quality are accompanied by clearly expressed comments from the laboratory.
- Each final data report is completely legible, contains all the supporting documentation required for the demonstration, and is in the format required by Tetra Tech.

In addition to the three-level review described above, the STL Tampa East QA manager will perform a full review of the first data package for each analysis to ensure that appropriate data generation procedures are followed. This level of review is not routinely performed by STL Tampa East and has been specifically required by Tetra Tech.

During laboratory reviews of data, problems associated with analytical nonconformances will be identified, and the problems and proposed resolutions will be discussed with the Tetra Tech project manager. Analytical nonconformances are defined as QC data lying outside a specific QA objective range (control limits) for precision or accuracy for a given analytical method. If QC data are outside control limits, the laboratory will determine the probable causes of the problems. If necessary, the samples involved will be re-prepared or reanalyzed, and typically only the reanalysis results will be reported. However, if a problem involves the sample matrix, both initial and reanalysis results will be reported and identified in the data package. If sample reanalysis is not feasible, the initial analysis results will be reported, and these results will be flagged and identified in the data package.

12.2.3 Data Review by Tetra Tech

In addition to the three-level review process that will be used by STL Tampa East, the Tetra Tech project manager or his designee (for example, Mr. Parr of Catalyst) will review all laboratory and field measurement device results, including case or sample lot narratives and QC sample results, based on demonstration objectives. The Tetra Tech project manager or his designee will also conduct a complete data validation for 10 percent of the data as an independent check on reference method or device performance. If this validation reveals no oversights or problems, Tetra Tech will consider all data to be acceptable. If oversights or problems are identified, however, a complete data validation for 100 percent of the data will be conducted by the Tetra Tech project manager or his designee. This data validation will be conducted using EPA CLP "National Functional Guidelines for Organic Data Review" (EPA 1994a), as applicable. Tetra Tech's assessment of the data and QC results will be summarized for discussion with the EPA project manager and incorporation into the DER.

During its data review, Tetra Tech will identify project outlier data and will report these data to the EPA project manager. Project outlier data are defined as sample data outside specified acceptance limits established about the central tendency estimator (the arithmetic mean) of the data set for a given area or for all areas taken together. For data known or assumed to be normally distributed, the specified acceptance limits will be the 95 percent confidence limits defined by the Student's two-tailed t-distribution. Consistent procedures will be used to identify outliers for both laboratory and field data. No data will be rejected simply because they are statistical outliers. However, Tetra Tech will conduct a thorough check to identify the reasons for the outliers and will provide the EPA project manager with an explanation of why some data appear to be outliers.

12.3 Data Reporting

Each developer and STL Tampa East will prepare and submit data packages reporting the results of field and laboratory measurements, respectively. STL Tampa East will also prepare and submit electronic data deliverables (EDD). Tetra Tech will use the data submittals to prepare the ITVR for each field measurement device and the DER for the entire demonstration. Described below are the data reporting requirements for (1) developer data packages, (2) STL Tampa East data packages, (3) STL Tampa East EDDs, (4) ITVRs, and (5) the DER.

12.3.1 Developer Data Packages

The developers will compile field measurement device results on standard forms provided by Tetra Tech. The forms will contain sample identification numbers and spaces for a developer to enter GRO, EDRO, and TPH results, as appropriate.

12.3.2 STL Tampa East Data Packages

STL Tampa East will prepare full EPA CLP-style data packages in accordance with instructions provided in Exhibit B of the most recent EPA CLP statement of work for organic analysis. Each full data package will contain all the information in the summary data package and all associated raw data for the samples in one sample lot. A sample lot is a group of 20 or fewer samples for a given work order that are received over a period of 14 days or less. The summary data package will consist of a case narrative, copies of all associated chain-of-custody forms and sample receipt notices, sample results, and QA/QC summaries. The case narrative will include the following items:

- The laboratory name, project name, project number, sample lot number, and work order number as well as a table that cross-references field and laboratory sample identification numbers

- A general description of the work performed, including sample preparation and analysis procedures and methods used

- A discussion of any deviations from the guidelines and procedures specified in the analytical method, work order, or demonstration plan

- A detailed discussion of all sample shipping, receipt, preparation, analysis, and QC deficiencies

- Copies of all nonconformance and corrective action forms describing the nature of each nonconformance and the reanalysis or other corrective action taken

- A thorough explanation of all cases of manual integration unless each manual integration and its rationale are clearly indicated in the raw data sheets

- A statement identifying samples whose results may have been impacted by baseline rise

- The following statement, signed and dated by the laboratory project manager or a duly authorized designee, with the signer's name and title clearly printed below:

  > "I certify that these data are technically accurate, complete, and in compliance with the terms and conditions of the contract other than the conditions detailed above. Release of the data contained in this hard copy data package and its electronic data deliverable submitted on diskette has been authorized by the laboratory project manager or a designee as verified by the following signature."

All documentation in each full data package, including chromatograms, instrument printouts, calibration records, and QC results, will be clearly labeled with the laboratory standard number or laboratory QC sample number, as applicable. All raw data deliverables will be complete enough and presented in such a way that requantification of the final results using the raw data would be possible and straightforward. Table 12-1 outlines the required full data package format; this format reflects the ordering of a full data package for organic analysis in accordance with EPA CLP statement of work OLM04.1 (EPA 1999).

When required by an analytical method, EPA CLP-style forms will be modified or supplemented with additional information, forms, or documentation. STL Tampa East will provide Tetra Tech with two bound copies of each full data package by July 31, 2000.

Table 12-1. Full Data Package Format for Gasoline Range Organic and Extended Diesel Range Organic Analyses

Section I	**Case Narrative**
1.	Case narrative
2.	Copies of nonconformance and corrective action forms
3.	Chain-of-custody forms
4.	Copies of condition upon sample receipt forms
Section II	**Sample Results** for the following:
1.	Environmental samples, including sample surrogate recoveries, dilutions, and reanalyses.
	All results will include concentration units, reporting limits, and method detection limits.
Section III	**Quality Assurance and Quality Control Summary Results** for the following:
1.	Method and instrument blanks
2.	Surrogate recoveries
3.	Matrix spike and matrix spike duplicate recoveries and relative percent differences
4.	Laboratory control sample and laboratory control sample duplicate recoveries and relative percent differences
5.	Extract duplicate relative percent differences
6.	Other quality control results, as applicable.
	All results will include concentration units and reporting limits.
Section IV	**Sample Raw Data** and all associated raw data for the following:
1.	Environmental samples, including sample screenings, dilutions, and reanalyses.
	All chromatograms will identify start and stop retention times used for integration.
Section V	**Quality Assurance and Quality Control Raw Data** and all associated raw data for the following:
1.	Method and instrument blanks
2.	Matrix spike and matrix spike duplicate samples
3.	Laboratory control samples and laboratory control sample duplicates
4.	Extract duplicates
5.	Other quality control results, as applicable.
	All chromatograms will identify start and stop retention times used for integration.
Section VI	**Standard Raw Data** and all associated raw data for the following:
1.	Initial calibrations and retention time information
2.	Continuing calibration verifications and retention time information
Section VII	**Other Raw Data**
1.	Percent moisture for soil samples for extended diesel range organic analysis
2.	Sample extraction and cleanup logs
3.	Instrument analysis log for each instrument used
4.	Standard preparation logs specifying initial and final concentrations for each standard used
5.	The formula and an example calculation for the initial calibration and continuing calibration
6.	The formula and an example calculation for sample results

The completed data packages will be approved by the STL Tampa East and Tetra Tech project managers before they are used to prepare the ITVRs and DER.

12.3.3 STL Tampa East Electronic Data Deliverables

For each sample lot, STL Tampa East will provide Tetra Tech with an EDD. All results for an sample lot will be complied in one electronic file on a 3.5-inch computer diskette. The diskette will be clearly labeled with the following information:

- Laboratory name
- Project name and number
- Sample lot number
- Work order number
- EDD file name and date prepared

Creation of associated extract files will be an automated process. STL Tampa East will use an automated laboratory information management system to produce the EDD in accordance with project requirements. Manual creation of the deliverable (that is, data entry by hand) is unacceptable, and manual editing will be avoided or kept to a minimum. STL Tampa East will verify all EDDs internally before submitting them to Tetra Tech. Each EDD will exactly correspond to the hard copy data, and no duplicated data will be submitted. STL Tampa East will provide Tetra Tech with one copy of each EDD by July 31, 2000.

12.3.4 Innovative Technology Verification Reports

In accordance with the demonstration plan, Tetra Tech will evaluate the performance and cost data collected for each field measurement device demonstrated and will prepare an ITVR for the device. Each ITVR will be a focused report of about 100 pages and will primarily include the following:

- An introduction
- A description of the field measurement device
- Site descriptions and the demonstration design
- A description of the reference method and its performance
- A description of the device's performance

- An economic analysis
- A summary of demonstration results

Tetra Tech will prepare individual ITVRs in accordance with the format specified in the "Handbook for Preparing Office of Research and Development Reports" (EPA 1995); its March 16, 1998, update; and project-specific guidance from the EPA project manager. The reports will be written in such a way that a reader with a basic science background can understand their contents and make an informed decision regarding the performance of the devices. The ITVRs will undergo a rigorous review process that will include reviews by the EPA project manager, the developers, and external peer reviewers.

12.3.5 Data Evaluation Report

Tetra Tech will prepare a DER containing tabular summaries of investigative and QA/QC data from the demonstration as well as results of technical system and performance audits. The DER will primarily discuss the following:

- Predemonstration investigation activities
- Demonstration activities
- Postdemonstration activities
- Deviations from the demonstration plan
- Investigative sample data
- QA/QC data
- Audit results

12.4 Data Storage

The STL Tampa East analysts responsible for performing measurements will enter raw data into logbooks or on data sheets. In accordance with standard document control procedures, the laboratory will maintain on file the original logbooks or data sheets, which will be signed and dated by the laboratory analysts responsible for them. Similar procedures will be used for all data entered directly into the laboratory information management system. Separate instrument logs will also be maintained by the laboratory to allow reconstruction of the run sequences for individual instruments. STL Tampa East will maintain all raw data, including raw instrument output on tape or diskette, on file for 5 years after the submittal of the data packages to Tetra Tech. Data documents will be kept in secure archive file cabinets accessible only to designated laboratory personnel. The data will be disposed of upon receipt of EPA instructions to do so or after 5 years, whichever is sooner.

A central project file for the demonstration will be established in the document control room of Tetra Tech's Chicago, Illinois, office. This file will be a repository for all relevant field and laboratory project documentation. All project documentation will be placed in the central file within 1 week of its receipt or generation by Tetra Tech. The document control room will be accessible only to authorized personnel and will be secure. Tetra Tech will offer the central file to the EPA at the end of the demonstration project but will maintain the central file until the end of the SITE contract if requested to do so.

Chapter 13
Health and Safety Procedures

This chapter defines health and safety requirements and designates protocols to be followed during demonstration activities at the Navy BVC, Kelly AFB, and PC sites. These activities will include oversight of Geoprobe® operation, soil sampling and sample management, and oversight of innovative TPH field measurement devices operation. This chapter addresses items specified under Occupational Safety and Health Administration (OSHA) Title 29 of the CFR, Section 1910.120 (b), "Final Rule," and will be available to all personnel who may be exposed to hazardous conditions on site, including Tetra Tech, subcontractor, and developer personnel participating in the demonstration and all site visitors, such as regulatory agency representatives. All personnel on site, including Tetra Tech and subcontractor employees and site visitors, must be informed of site emergency response procedures and any potential fire, explosion, health, or safety hazards associated with on-site activities. This chapter summarizes potential hazards and defines protective measures planned for the demonstration activities. Developers, EPA personnel, subcontractors, and site visitors may choose to follow the Tetra Tech health and safety procedures described in this chapter. However, each employer is directly and fully responsible for the health and safety of its own employee; Tetra Tech assumes no responsibility for non-Tetra Tech personnel.

The health and safety procedures described in this chapter have been reviewed and approved by the Tetra Tech HSR or a designee and the Tetra Tech project manager (see the Reviews and Approvals form at the end of this chapter). Protocols established in this chapter are based on site conditions, health and safety hazards known or anticipated to be present on site, and available site data. The health and safety procedures described in this chapter are intended solely for use during the proposed activities described in this demonstration plan. Specifications herein are subject to review and revision based on actual conditions encountered in the field during demonstration activities. Significant revisions to the health and safety procedures must be approved by the Tetra Tech project manager and the Tetra Tech HSR. Tetra Tech employees must also follow safety requirements taught during safety training and described in the Tetra Tech, Inc., "Health and Safety Manual."

This chapter is organized in the following 10 sections:

- Health and Safety Personnel and Procedure Enforcement (Section 13.1)
- Site Background (Section 13.2)
- Site-Specific Hazard Evaluation (Section 13.3)
- Training Requirements (Section 13.4)
- Personal Protection Requirements (Section 13.5)
- Medical Surveillance (Section 13.6)
- Environmental Monitoring and Sampling (Section 13.7)
- Site Control (Section 13.8)
- Decontamination (Section 13.9)
- Emergency Response Planning (Section 13.10)

13.1 Health and Safety Personnel and Procedure Enforcement

This section describes responsibilities of project personnel; summarizes requirements for subcontractors, developers, and visitors who wish to enter the Navy BVC, Kelly AFB, and PC sites; and discusses enforcement of health and safety procedures.

13.1.1 Project Personnel

The following personnel and organizations are associated with planned activities at the demonstration sites. The organizational structure will be reviewed and updated as necessary during the course of the project.

Name	Responsibility	Telephone No.
Client Representative:		
Stephen Billets	EPA project manager	(702) 798-2232
Site Representatives:		
Navy BVC site: Ernest Lory	Site contact	(805) 982-1299
Kelly AFB site: Amy Whitley	Site contact	(210) 925-3100
PC site: Jay Simonds	Site contact	(317) 228-6240

Name	Responsibility	Telephone No.
Tetra Tech Personnel:		
Kirankumar Topudurti	Project manager	(312) 856-8742
Eric Monschein	Field manager	(312) 856-8753
Jill Ciraulo	Site safety coordinator (SSC)	(312) 946-6479
Judith Wagner	HSR	(847) 818-7192
Subcontractors:		
Navy BVC site: Vironex Environmental Field Services	Geoprobe® operator	(800) 847-6639
Kelly AFB site: Venture II Environmental Drilling, Inc.	Geoprobe® operator	(800) 662-5412
PC site: Handex of Indiana	Geoprobe® operator	(317) 228-6240

The Tetra Tech project manager, field manager, SSC, and HSR will be responsible for implementation and enforcement of the health and safety procedures. Their duties and the expectations for Tetra Tech employees are described in the following sections.

13.1.1.1 Project Manager and Field Manager

The Tetra Tech project manager has ultimate responsibility for ensuring implementation of the requirements set forth in this chapter. Some of this responsibility may be fulfilled through delegation of duties to site-dedicated personnel that report directly to the project manager. The project manager will regularly confer with site personnel regarding health and safety compliance.

The Tetra Tech field manager will oversee and direct demonstration activities and will have day-to-day responsibility for ensuring implementation of the health and safety procedures. Subcontractor compliance with the health and safety procedures will be monitored by the field manager. The field manager will report any health and safety-related issues directly to the project manager.

13.1.1.2 Site Safety Coordinator

The Tetra Tech SSC will be appointed by the Tetra Tech project manager and will be responsible for field implementation of tasks and procedures discussed in this chapter, including air monitoring, establishing a decontamination protocol, and ensuring the signing of the Daily Tailgate Safety Meeting form (Form HST-2 in Appendix C) by all personnel working on site. The SSC will have advanced field work experience and will be familiar with health and safety requirements specific to the project. The SSC will also maintain the Daily Site Log (Form SSC-1 in Appendix C).

13.1.1.3 Health and Safety Representative

The Tetra Tech HSR is responsible for administration of the company health and safety program. The HSR will act in an advisory capacity to the Tetra Tech project manager and personnel regarding project-specific health and safety issues. The project manager will establish a liaison between representatives of the EPA; representatives of the Navy BVC, Kelly AFB, and PC sites; and the HSR for matters relating to health and safety.

13.1.1.4 Tetra Tech Employees

Tetra Tech employees are expected to fully participate in implementing the site-specific health and safety procedures by obtaining necessary training, attending site safety meetings, always wearing designated PPE, complying with site safety and health rules, and advising the Tetra Tech SSC of health and safety concerns at the sites.

13.1.2 Subcontractors and Developers

Subcontractor personnel and the developers will be provided with a copy of this chapter. Subcontractor personnel and the developers must comply with all applicable 29 CFR 1910.120 training, fit testing, and medical surveillance requirements, as applicable. Subcontractors and the developers are responsible for providing PPE required for their personnel (see Section 13.5.1, Protective Equipment and Clothing) and are directly responsible for the health and safety of their employees. The developers will have access only to the PRA at the Navy BVC site.

13.1.3 Visitors

All site visitors will be briefed on the site-specific health and safety procedures. Site visitors will be escorted by Tetra Tech personnel during the visitors' day activities.

13.1.4 Health and Safety Procedure Enforcement

The health and safety procedures described in this chapter apply to all demonstration activities and all Tetra Tech personnel working on the Navy BVC, Kelly AFB, and PC sites. Violators of the procedures will be verbally notified upon the first violation, and the violation will be noted by the Tetra Tech SSC in a field logbook. Upon a second violation, the violator will be notified in writing, and the Tetra Tech project manager and the violator's supervisor will be notified. A third violation will result in a written notification and the violator's eviction from the site. The written notification will be sent to Tetra Tech human resources development and the Tetra Tech HSR.

Personnel will be encouraged to report to the Tetra Tech SSC any conditions or practices that they consider to be detrimental to their health or safety or that they believe are in violation of applicable health and safety standards. Such reports may be made orally or in writing. Personnel who believe that an imminent danger threatens human health or the environment will be encouraged to bring the matter to the immediate attention of the SSC for resolution.

At least one copy of this chapter will be available on site for all site personnel. Minor changes to the health and safety procedures discussed in this chapter will be discussed at the beginning of each work day by the SSC at the daily tailgate safety meeting and will be noted in the field logbook. Significant procedure revisions must be discussed with the Tetra Tech HSR and project manager.

13.2 Site Background

The following sections describe the demonstration sites and the activities planned for the demonstration.

13.2.1 Demonstration Site Descriptions

Chapter 3 describes the Navy BVC, Kelly AFB, and PC sites. Figures showing the site locations and layouts are also included in Chapter 3.

13.2.2 Planned Demonstration Activities

Sample collection and handling procedures to be followed at each of the three sites are fully described in Chapter 7. The planned demonstration activities include the following tasks:

- Oversight of soil sample collection using a subcontractor-operated Geoprobe® at the Kelly AFB and PC sites and in two areas of the Navy BVC site (the FFA and NEX Service Station Area)
- Collection of soil samples by Tetra Tech in the PRA of the Navy BVC site using a Split Core Sampler
- Management of all soil samples collected at the Navy BVC, Kelly AFB, and PC sites including sectioning core tube liners, characterizing soil samples, homogenizing soil samples, preparing samples for shipment to STL Tampa East, and transferring samples to the developers at the Navy BVC site
- Oversight of TPH field measurement activities at the Navy BVC site

13.3 Site-Specific Hazard Evaluation

Demonstration activities and physical features of the demonstration sites may expose field personnel to a variety of hazards. This section provides information on potential hazards related to demonstration activities and the nature of hazardous material impacts. Potential chemical and physical hazards related to demonstration activities are discussed below.

13.3.1 Chemical Hazards

Chemical hazards that may be encountered at the sites involve volatile organic compounds (VOC), inorganic compounds, and petroleum hydrocarbons. The results of previous site investigations indicate that VOCs and inorganic compounds are present in the three sites. A summary of site characteristics, including TPH concentrations, based on data collected during the predemonstration investigation is presented in Table 3-1 of Chapter 3. General information on VOCs and inorganic compounds at the sites is provided in Sections 13.3.1.1 and 13.3.1.2, respectively. A hazard evaluation of petroleum distillate fuel products is provided in Tetra Tech Safe Work Practice (SWP) 6-25, which will be available on site. These chemicals pose various physical, chemical, and toxicological hazards. Potential routes of exposure to these chemicals include dermal (skin) contact, inhalation, and ingestion. The chemicals may also contaminate equipment, vehicles, instruments, and personnel. The overall health threat associated with exposure to these chemicals is uncertain because (1) actual concentrations that personnel could be exposed to cannot be predicted, (2) the actual duration of exposure is unknown, and (3) the effects of low-level exposure to a mixture of chemicals cannot be predicted. However, Tetra Tech believes that the potential for high-level exposure is limited. Table 13-1 provides a task hazard analysis of the planned demonstration activities listed in Section 13.2.2.

Table 13-2 lists the materials, which may be brought to the sites by Tetra Tech or the developers. Material Safety Data Sheets (MSDS) summarize health and safety information for hazardous materials that will be brought to the Navy BVC site, such as laboratory reagents, decontamination solutions, and sample preservatives. Tetra Tech and the developers are responsible for making the MSDSs available on site as specified in Table 13-2.

13.3.1.1 Volatile Organic Compounds

Generally, VOCs are central nervous system depressants. Exposure to some VOCs may occur through skin absorption. General symptoms of VOC exposure, both acute and chronic, may include euphoria, headache, weakness, dizziness, nausea, narcosis, and possibly coma. Certain VOCs are also skin and eye irritants.

13.3.1.2 Inorganic Compounds

Inorganic compounds do not have carbon in their molecular structure. Heavy metals such as lead are inorganic compounds. The symptoms of acute exposure to inorganic compounds include but are not restricted to abdominal pain, hypertension, anemia, insomnia, and restrictive pulmonary function. Chronic exposure to some metals may lead to development of cancer.

13.3.2 Physical Hazards

Physical hazards associated with demonstration activities present a potential threat to on-site personnel. Dangers are posed by heavy equipment, utility and power lines, slippery surfaces, unseen obstacles, noise, heat, cold, and poor illumination.

Injuries may result, for example, from the following:

- Accidents caused by slipping, tripping, or falling
- Use of improper lifting techniques

Table 13-1. Task Hazard Analysis

Task	Potential Hazard	Control Measure	Initial Level of Protection	Upgraded Level of Protection
Oversight of soil sample collection using a subcontractor-operated Geoprobe® at the Kelly AFB and PC sites and in the FFA and NEX Service Station areas of the Navy BVC site	Physical injury	Exercise caution	Modified Level D as described in Section 13.5.1, including: • Tyvek® coveralls • Nitrile gloves	Level C as described in Section 13.5.1, including: • Tyvek® coveralls • Nitrile gloves • Full face air purifying respirator with combination organic vapor and particular cartridges (such as GMA-P100)
	Dermal exposure (contact with contaminated soil)	Wear proper PPE		
	Heat stress	Maintain proper hydration		
	Noise exposure	Use hearing protection		
	Buried utilities	Survey for buried utilities		
	Working near heavy equipment	Limit access to work areas		
Collection of soil samples by Tetra Tech in the PRA using a Split Core Sampler	Physical injury	Exercise caution		
	Dermal exposure (contact with contaminated soil)	Wear proper PPE		
	Heat stress	Maintain proper hydration		
Management of all demonstration soil samples collected at the Navy BVC, Kelly AFB, and PC sites, including sectioning core tube liners, characterizing soil samples, homogenizing samples, preparing samples for shipment to STL Tampa East, and transferring soil samples to the developers at the Navy BVC site	Physical injury	Exercise caution		
	Dermal exposure (contact with contaminated soil)	Wear proper PPE		
	Heat stress	Maintain proper hydration		
Oversight of TPH field measurement activities at the Navy BVC site	No task-specific potential hazards have been identified.	Follow all general safe work practices	Level D	No upgraded level of protection is expected to be needed.

Notes:

AFB = Air Force Base
BVC = Base Ventura County
FFA = Fuel Farm Area
NEX = Naval Exchange
PC = Petroleum company
PPE = Personal protective equipment
PRA = Phytoremediation area
STL Tampa East = Severn Trent Laboratories in Tampa, Florida
TPH = Total petroleum hydrocarbons

Table 13-2. Demonstration Participant Responsibilities for Providing Material Safety Data Sheets

Participant Responsible for Providing Material Safety Data Sheet	Chemical
CHEMetrics	Aluminum chloride, anhydrous Dichloromethane Sodium sulfate, anhydrous, crystalline
Wilks	Freon 113 Sodium sulfate, anhydrous, crystalline Vertrel® MCA, a hydrochlorofluorocarbon extraction solvent
Horiba	Chlorobenzene Diesel calibration standard Hexadecane Isooctane S-316 extraction solvent Sodium sulfate, anhydrous, crystalline Spiking material
Dexsil®	Developer solution Methanol-based extraction solvent Mineral oil
ESC	Anthracene Methanol Naphthalene
siteLAB®	Calibration standards Methanol
SDI	Detergent solution Hydrogen peroxide m-Xylene Methanol Phosphate buffer solution Sulfuric acid, 0.5 percent Tetramethylbenzidine
Tetra Tech EM Inc.	Alconox®

- Use of moving or rotating equipment
- Equipment mobilization and operation (such as electrocution from contact with overhead or underground power lines)
- Use of improperly maintained equipment

Injuries resulting from physical hazards can be avoided by using SWPs and employing caution when working with machinery. Specific SWPs applicable to the demonstration are listed in Section 13.8.5 and will be available on site during the demonstration. To ensure safe working conditions, the Tetra Tech SSC will conduct and document regular safety inspections and will make sure that all workers and visitors are informed of any potential physical hazards related to the sites. Additional physical hazards that have been identified at the sites include the following:

- Use of instruments powered by DC
- Use of instruments with ultraviolet or infrared light sources

13.4 Training Requirements

All Tetra Tech personnel who may be exposed to hazardous conditions on site will be required to meet training requirements outlined in 29 CFR 1910.120, “Hazardous Waste Operations and Emergency Response.” All personnel and visitors entering the sites will be required to sign the Daily Tailgate Safety Meeting form (Form HST-2 in Appendix C).

Before activities begin at the Navy BVC site, the Tetra Tech SSC will present a briefing for all personnel who will participate in on-site activities. The following topics will be addressed during the prework briefing:

- Names of the SSC and a designated alternate
- Site history
- Work tasks
- Hazardous chemicals that may be encountered on site
- Physical hazards that may be encountered on site
- PPE, including type or types of respiratory protection to be used for work tasks
- Training requirements
- Environmental surveillance (air monitoring) equipment use and maintenance
- Action levels and situations requiring upgrade or downgrade of level of protection
- Site control measures, including site communications, control zones, and SWPs
- Decontamination procedures
- Emergency communication signals and codes
- Environmental accident emergency procedures (in case contamination spreads outside the exclusion zone)
- Personnel exposure and accident emergency procedures (in case of falls, exposure to hazardous substances, and other hazardous situations)
- Fire and explosion emergency procedures

- Emergency telephone numbers
- Emergency routes

Any other health and safety-related issues that may arise before on-site activities begin will also be discussed during the prework briefing.

Issues that arise during implementation of on-site activities will be addressed during tailgate safety meetings to be held daily before the workday or shift begins. Such issues will be documented in the Daily Tailgate Safety Meeting form (Form HST-2 in Appendix C). Any changes in procedures or site-specific health and safety-related matters will be addressed during these meetings.

13.5 Personal Protection Requirements

The levels of personal protection to be used for work tasks at the Navy BVC, Kelly AFB, and PC sites have been selected based on known or anticipated physical hazards; types and concentrations of contaminants that may be encountered on site; and contaminant properties, toxicity, exposure routes, and matrixes. The following sections describe protective equipment and clothing; reassessment of protection levels; limitations of protective clothing; and respirator selection, use, and maintenance.

13.5.1 Protective Equipment and Clothing

Personnel will wear protective equipment when (1) demonstration activities involve known or suspected atmospheric contamination; (2) demonstration activities may generate vapors, gases, or particulates; or (3) direct contact with hazardous materials may occur. The anticipated levels of protection selected for use by field personnel during demonstration activities are listed in Table 13-1, Task Hazard Analysis. Based on the anticipated hazard level, personnel will initially perform field tasks in Level D or Modified Level D protection. If site conditions or the results of air monitoring performed during on-site activities warrant a higher level of protection, all field personnel will withdraw from the site, immediately notify the Tetra Tech SSC, and wait for further instructions. Descriptions of equipment and clothing required for Level D, Modified Level D, Level C, and Level B protection are provided below.

- Level D
  - Coveralls or work clothes, if applicable
  - Boots with steel-toe protection and steel shanks

  - A hard hat (face shield optional), if applicable
  - Disposable gloves (latex or nitrile), if applicable
  - Safety glasses or goggles
  - Hearing protection (for areas with a noise level exceeding 85 decibels on the A-weighted scale)
- Modified Level D
  - Coveralls or work clothes, if applicable
  - Chemical-resistant clothing (such as Tyvek® or Saranex® coveralls)
  - Outer gloves (neoprene, nitrile, or other), if applicable
  - Disposable inner gloves (latex or vinyl)
  - Boots with steel-toe protection and steel shanks
  - Disposable boot covers or chemical-resistant outer boots
  - Safety glasses or goggles
  - A hard hat (face shield optional)
  - Hearing protection (for areas with a noise level exceeding 85 decibels on the A-weighted scale)
- Level C
  - Coveralls or work clothes, if applicable
  - Chemical-resistant clothing (such as Tyvek® or Saranex® coveralls)
  - Outer gloves (neoprene, nitrile, or other), if applicable
  - Disposable inner gloves (latex or vinyl)
  - Boots with steel-toe protection and steel shanks
  - Disposable boot covers or chemical-resistant outer boots
  - A full- or half-face, air-purifying respirator with National Institute for Occupational Safety and Health (NIOSH)-approved cartridges to protect against organic vapors, dust, fumes, and mists (cartridges used for gas and vapors must be replaced in accordance with the change-out schedule described in the Respiratory Hazard Assessment form [Form RP-2 in Appendix C])
  - Safety glasses or goggles (with half-face respirator only)
  - A hard hat (face shield optional)

  - Hearing protection (for areas with a noise level exceeding 85 decibels on the A-weighted scale)

- Level B
  - Chemical-resistant clothing (such as Tyvek® or Saranex® coveralls)
  - Outer gloves (neoprene, nitrile, or other)
  - Disposable inner gloves (latex or vinyl)
  - Boots with steel-toe protection and steel shanks
  - Disposable boot covers or chemical-resistant outer boots
  - A NIOSH-approved, pressure-demand airline respirator with a 5-minute escape cylinder or self-contained breathing apparatus (SCBA)
  - A hard hat (face shield optional)
  - Hearing protection (for areas with a noise level exceeding 85 decibels on the A-weighted scale)

13.5.2 Reassessment of Protection Levels

PPE levels will be upgraded or downgraded based on a change in site conditions or investigation findings. When a significant change in site conditions occurs, hazards will be reassessed. Some indicators of the need for reassessment are as follows:

- Commencement of a new work phase, such as the start of a significantly different sampling activity or work that begins on a different portion of a site
- A change in job tasks during a work phase
- A change of season or weather
- Temperature extremes or individual medical considerations limiting the effectiveness of PPE
- Discovery of contaminants other than those previously identified
- A change in ambient levels of airborne contaminants (see the action levels listed in Table 13-3)
- A change in work scope that affects the degree of contact with contaminated media

13.5.3 Limitations of Protective Clothing

PPE clothing ensembles designated for use during demonstration activities have been selected to provide protection against contaminants at known or anticipated on-site concentrations and physical states. However, no protective garment, glove, or boot is entirely chemical-resistant, and no protective clothing provides protection against all types of chemicals. Permeation of a given chemical through PPE depends on the contaminant concentration, environmental conditions, physical condition of the protective garment, and resistance of the garment to the specific contaminant. Chemical permeation may continue even after the source of contamination has been removed from the garment.

All on-site personnel will use the procedures presented below to obtain optimum performance from PPE.

- When chemical-protective coveralls become contaminated, don a new, clean garment after each rest break or at the beginning of each shift.
- Inspect all clothing, gloves, and boots both before and during use for the following:
    - Imperfect seams
    - Nonuniform coatings
    - Tears
    - Poorly functioning closures
- Inspect reusable garments, boots, and gloves both before and during use for visible signs of chemical permeation, such as the following:
    - Swelling
    - Discoloration
    - Stiffness
    - Brittleness
    - Cracks
    - Any sign of puncture

Table 13-3. Site-Specific Air Monitoring Requirements and Action Levels

Contaminant or Hazard	Task	Monitoring Device	Action Level	Monitoring Frequency	Action[a]
Organic vapors	Collection of soil samples using Geoprobe® and management of samples	MiniRAE® or Photovac 2020 PID (or similar device)	Background to 5 ppm	At each new sampling location when using Geoprobe® and to evaluate exposure during sample management	Wear Modified Level D PPE
			5 to 100 ppm	Every 30 minutes	Implement engineering controls such as an exhaust fan
			> 100 ppm	Continuous	Use Level C PPE
Known compounds	NA	NA	<½ PEL or TLV	To be determined by Tetra Tech SSC	Continue work in Level D PPE with regular monitoring
			$½ PEL or TLV		Use Level C or B PPE, depending on concentration
Combustible atmospheres	Collection of soil samples using Geoprobe®	MSA MicroGard™ O$_2$-LEL indicator (or similar device)	<10% LEL	Every 15 minutes when PID readings are > 5 ppm	Continue work
			$10 to 25% LEL		Continue work with extreme caution
			>25% LEL		Stop work, evacuate area, and evaluate source
Oxygen-deficient or enriched atmosphere	NA	NA	$19.5 to #23.5% oxygen	Continuous	Continue work
			<19.5 or >23.5% oxygen		Stop work and evacuate area
External exposure to radiation	NA	NA	<2 mRem/hr	Continuous	Continue work and monitoring with caution
			$2 mRem/hr		Stop work, evacuate area, and re-evaluate source area
Particulates	NA	NA	<5 mg/m^3	Hourly or more frequently, as determined by Tetra Tech SSC	Use Modified Level D PPE
			5 to 10 mg/m^3	Continuous	Use Level C PPE and implement dust suppression activities
			>10 mg/m^3	NA	Stop work and implement dust suppression activities
Noise	To be determined by Tetra Tech SSC	Rule-of-thumb test	$85 dBA	To be determined by Tetra Tech SSC	Use hearing protection

Table 13-3. Site-Specific Air Monitoring Requirements and Action Levels (Continued)

Notes:

\> = Greater than
$ = Greater than or equal to
< = Less than
# = Less than or equal to
dBA = Decibel as measured on the A-weighted scale
LEL = Lower explosive limit
mg/m^3 = Milligram per cubic meter
mRem/hr = Millirem per hour
NA = Not applicable
PEL = Permissible exposure limit
PID = Photoionization detector
PPE = Personal protective equipment
ppm = Part per million
SSC = Site safety coordinator
TLV = Threshold limit value
Tetra Tech = Tetra Tech EM Inc.

[a] Refer to Table 13-1 for the specific types of gloves, chemical-protective clothing, respirators, and cartridges to be used.

- Any sign of abrasion

Reusable gloves, boots, or coveralls exhibiting any of the characteristics listed above must be discarded. Reusable PPE will be decontaminated in accordance with procedures described in Section 13.9 and will be neatly stored in the support zone away from work zones.

13.5.4 Respirator Selection, Use, and Maintenance

Tetra Tech personnel will be informed of the proper use, maintenance, and limitations of respirators during annual health and safety refresher training and the prework briefing. Any on-site personnel who will use a tight-fitting respirator must pass a qualitative fit test for the respirator that follows the fit testing protocol provided in Appendix A of the OSHA respirator standard (29 CFR 1910.134). Fit testing must be repeated annually or when a new type of respirator is used.

Respirator selection is based on assessment of the nature and extent of hazardous atmospheres anticipated during demonstration activities. This assessment will include a reasonable estimate of employee exposure to respiratory hazards and identification of each contaminant's anticipated chemical form and physical state.

For each work task requiring respirator use at the Navy BVC, Kelly AFB, and PC sites, a respiratory hazard assessment will be conducted. A blank Respiratory Hazard Assessment form (Form RP-2) is included in Appendix C. Amendments to the health and safety procedures described in this chapter and to Form RP-2 will be discussed during daily tailgate safety meetings and will be documented in the field logbook.

When an atmospheric contaminant is an identified gas or vapor and its concentration is known or can be reasonably estimated, respiratory protection options include the following:

- An atmosphere-supplying respirator (air-line or SCBA)
- An air-purifying respirator equipped with a NIOSH-certified, end-of-service-life indicator (ESLI) for the identified contaminant. If no ESLI is available, a change-out schedule for cartridges must be developed based on objective data or information. Respirator cartridge selection and change-out schedules will be evaluated by the HSR at the time of the respiratory hazard assessment. The Respiratory Hazard Assessment form (Form RP-2) will describe the data used as the basis for the cartridge change-out schedule and the proposed change schedule.

For protection against particulate contaminants, approved respirators can include the following:

- An atmosphere-supplying respirator
- A respirator equipped with a filter certified by NIOSH under 32 CFR Part 11 or 42 CFR Part 84 as a P100 filter (formerly known as a high-efficiency particulate [HEPA] air filter)

For any tasks performed in Level C PPE, a full- or half-face, air-purifying respirator equipped with NIOSH-approved cartridges or filters will be selected to protect against vapors, gases, and aerosols.

Air-purifying respirators will be used only in conjunction with breathing-space air monitoring, which must be conducted in adherence to the action levels outlined in Table 13-3. Air-purifying respirators will be used only when they can provide protection against the substances encountered on site.

Factors precluding use of Level C and air-purifying respirators are as follows:

- Oxygen-deficient atmosphere (less than 19.5 percent oxygen)
- Concentrations of substances that may be immediately dangerous to life and health
- Confined or unventilated areas that may contain airborne contaminants not yet characterized
- Unknown contaminant concentrations or concentrations that may exceed the maximum use levels for designated cartridges documented in the selected cartridge manufacturer's instructions
- Unidentified contaminants
- High relative humidity (more than 85 percent, which reduces the sorbent life of the cartridges)
- Respirator cartridges with an undetermined service life

Use, cleaning, and maintenance of respirators are described in Tetra Tech SWP 6-27, Respirator Cleaning Procedures, and SWP 6-28, Safe Work Practices for Use of Respirators.

13.6 Medical Surveillance

The following sections describe Tetra Tech's medical surveillance program, including health monitoring requirements, site-specific medical monitoring, and medical support and follow-up requirements. Procedures documented in these sections will be followed for all activities at the Navy BVC, Kelly AFB, and PC sites. Additional requirements are defined in the Tetra Tech, Inc., "Health and Safety Manual."

13.6.1 Health Monitoring Requirements

All Tetra Tech personnel involved in on-site activities at the Navy BVC, Kelly AFB, and PC sites must participate in a health monitoring program as required by 29 CFR 1910.120(f). Tetra Tech has established a health monitoring program with WorkCare, Inc., of Orange, California. Under this program, Tetra Tech personnel receive baseline and annual or biennial physical examinations consisting of the following:

- Complete medical and work history
- Physical examination
- Vision screening
- Audiometric screening
- Pulmonary function test
- Resting electrocardiogram
- Chest x-ray (required once every 3 years)
- Blood chemistry, including hematology and serum
- Urinalysis

For each employee, Tetra Tech receives a copy of the examining physician's written opinion after postexamination laboratory tests have been completed; the Tetra Tech employee also receives a copy of the written opinion. This opinion includes the following information (in accordance with 29 CFR 1910.120[f][7]):

- The results of the medical examination and tests

- The physician's opinion as to whether the employee has any medical conditions that would place the employee at an increased risk of health impairment from work involving hazardous waste operations or during an emergency response

- The physician's recommended limitations, if any, on the employee's assigned work; special emphasis is placed on fitness for duty, including the ability to wear any required PPE under conditions expected on site (for example, temperature extremes)

- A statement that the employee has been informed by the physician of the medical examination results and of any medical conditions that require further examination or treatment

All subcontractors must have health monitoring programs conducted by their own clinics in compliance with 29 CFR 1910.120(f). Any visitor or observer at the site will be required to provide records in compliance with 29 CFR 1910.120(f) before entering the site.

13.6.2 Site-Specific Medical Monitoring

For activities at the Navy BVC, Kelly AFB, and PC sites, no specific medical tests will be required before individuals enter the exclusion zone or decontamination zone (see Section 13.8.2, Site Control Zones).

13.6.3 Medical Support and Follow-Up Requirements

As a follow-up to an injury requiring care beyond basic first aid or to possible exposure above established exposure limits, all Tetra Tech employees are entitled to and encouraged to seek medical attention and physical testing. Such injuries and exposures must be reported to the Tetra Tech HSR. Depending on the type of injury or exposure, follow-up testing, if required, must be performed within 24 to 48 hours of the incident. It will be the responsibility of Tetra Tech's medical consultant to advise the type of test required to accurately monitor for exposure effects. The Accident and Illness Investigation Report (Form AR-1 in Appendix C) must be completed by the Tetra Tech SSC in the event of an accident, illness, or injury. A copy of this form must be forwarded to the HSR for use in determining the recordability of the incident and for inclusion in Tetra Tech's medical surveillance records.

13.7 Environmental Monitoring and Sampling

Environmental monitoring or sampling will be conducted to assess personnel exposure levels as well as site or ambient conditions and to determine appropriate levels of PPE for work tasks. The following sections discuss initial and background air monitoring, personal monitoring, ambient air monitoring, monitoring parameters and devices, use and maintenance of survey equipment, thermal stress monitoring, and noise monitoring. Site-specific air monitoring requirements and action levels are provided in Table 13-3.

13.7.1 Initial and Background Air Monitoring

Initial air monitoring of the work area will be performed before a work task begins. This monitoring will be performed using real-time field survey instrumentation. Air will also be monitored at the beginning of each workday to identify any potentially hazardous situation that might have developed during off-shift periods.

Operations at the sites may result in variable background levels of airborne compounds. Airborne compounds may be released from vehicles, blowing dust, material transfers, and so on. These sources can complicate evaluation of contaminant emissions during project tasks. Therefore, several upwind and prework measurements will be taken to assess contributions to airborne contamination by other potential sources.

13.7.2 Personal Monitoring

The employees working closest to a source of contamination have the highest likelihood of exposure to airborne contaminant concentrations that may exceed established exposure limits. Therefore, selective monitoring of the workers who are closest to a source of contaminant generation will be conducted during demonstration activities. Personal monitoring will be conducted in the breathing zone and, if a worker is wearing respiratory protective equipment, outside the face piece.

13.7.3 Ambient Air Monitoring

Most tasks will require monitoring of the general work area or ambient site conditions. Ambient monitoring will generally be conducted using direct-reading survey instrumentation or compound-specific instruments or detector tubes.

Initial ambient air monitoring will be performed as a minimum requirement when any of the situations listed below arise.

- Work begins on a different portion of a site.
- Contaminants other than those previously identified are encountered.
- A different type of operation is initiated
- Workers handle leaking containers or work in areas with obvious liquid contamination (for example, spill or lagoon areas).
- Obvious lithologic changes are noticed during drilling activities.
- Workers experience physical difficulties.

Periodic ambient air monitoring will be performed at the frequency listed in Table 13-3.

13.7.4 Monitoring Parameters and Devices

The following sections briefly describe the use and limitations of instruments used to monitor for organic vapors, known compounds, combustible atmospheres, percent oxygen, external exposure to radiation, and particulates. Site-specific air monitoring requirements and action levels are listed in Table 13-3.

All monitors will be calibrated in accordance with manufacturer recommendations at the beginning of every workday, if possible. Calibration results along with air monitoring data will be recorded in the field logbook.

13.7.4.1 Organic Vapors

A direct-reading organic vapor monitor, such as an FID or a PID, will be used to determine the presence of VOCs. Table 13-3 specifies the instrument that will be used for the demonstration. The concentrations of individual VOCs of concern cannot usually be determined using this instrument because the detector responds to the total VOC mixture.

13.7.4.2 Known Compounds

Compound-specific monitoring will not be conducted during the demonstration. However, if individual compounds must be identified, compound-specific instrumentation or colorimetric detector tubes may be required. Generally, action levels for known compounds are set at one-half the permissible exposure limit (PEL) or threshold limit value (TLV) of the compound with the lowest PEL or TLV.

13.7.4.3 Combustible Atmospheres

When a flammable compound reaches a certain concentration in air, it can become explosive when exposed to an ignition source. The lowest concentration able to support combustion is known as the lower explosive limit (LEL). Each flammable compound has its own LEL. Monitoring indicates how close to this limit the airborne concentration of a flammable compound is. Demonstration activities will cease when the airborne concentration of any flammable vapor or gas reaches 25 percent of its LEL (10 percent in a confined space). The monitoring device, monitoring frequency, and general action levels for combustible atmospheres to be used during demonstration activities are outlined in Table 13-3.

13.7.4.4 Percent Oxygen

Hazardous conditions exist whenever the oxygen level is too high or too low. Monitoring for percent oxygen is conducted to verify that a safe oxygen level is present for work activities. Workers must never enter or remain in low-oxygen atmospheres unless they are wearing supplied air respirators (air-line or SCBA). An oxygen-enriched atmosphere is hazardous because it causes an increased risk of fire. Based on the expected work conditions at the sites, percent oxygen monitoring is not required for the demonstration.

13.7.4.5 External Exposure to Radiation

Based on site conditions, no radiation hazard is expected to be encountered at any of the sites. Therefore, radiation monitoring will not be conducted during the demonstration. However, if the potential for external exposure to radiation exists, all personnel will be required to wear radiation exposure monitoring devices while working on site. Personal exposure monitoring devices may include a thermoluminescence detector (TLD), a standard film badge, or a pocket dosimeter. Monitoring devices are to be left on site at the end of each working day in a location removed from any source material. An outside vendor will supply TLDs, film badges, or dosimeters and will perform laboratory analyses on TLDs and film badges. The external radiation exposure limit for on-site personnel will be 1.25 Roentgen-equivalent man units (Rem) per 3 months with a 5-Rem maximum per year.

If necessary, radiation detectors will be used to determine the types and levels of radiation present on site. Appropriate instrumentation, such as alpha or gamma meters and Geiger counters, will be used.

13.7.4.6 Particulates

Based on site conditions, no particulate hazard is expected to be encountered at any of the sites. Therefore, particulate monitoring will not be conducted during the demonstration. Aerosols are a group of airborne materials that include particulates, fumes, mists, and smoke. Particulates are the primary aerosol of concern at hazardous waste sites. If climatic conditions, surface soil conditions, or site operations (such as excavation activities) adversely impact ambient air quality by increasing particulate matter for extended periods of time, air monitoring using a direct-reading instrument for particulates may become necessary. If elevated (visible) particulate matter conditions persist for 5 minutes or longer, the Tetra Tech SSC is responsible for sampling the breathing zone with a particulate monitor.

Generally, particulate monitors are capable of measuring both solid and liquid particulates within the size range of 0.1 to 10 µm (the respirable range). A monitor indicates the concentration of these particulates in units of mg per cubic meter of air.

Action levels for particulates will be based on the type of dust and hazardous materials that may contribute to the composition of the particulates and will be determined with the assistance of the Tetra Tech HSR or a designee.

13.7.5 Use and Maintenance of Survey Equipment

All personnel using field survey equipment must have training in its operation, limitations, and maintenance. Maintenance and internal or electronic calibration will be performed in accordance with manufacturer recommendations by individuals familiar with the equipment before its use on site. Repairs, maintenance, and internal or electronic calibration of the equipment will be recorded in equipment maintenance logbooks. The equipment maintenance logbook for each instrument will be kept in that instrument’s case. For rented monitoring equipment, repairs and maintenance will be conducted by the rental company. Results of routine calibration will be recorded in the field logbook.

Air monitoring equipment (such as combustible gas indicators, oxygen meters, and PIDs) will be calibrated before work begins. Only basic maintenance (such as changing batteries) will be performed by on-site personnel. Any additional maintenance or repairs will be performed by a trained service technician.

13.7.6 Thermal Stress Monitoring

Heat stress and cold stress are common and serious threats at hazardous waste sites. SWPs 6-15 and 6-16 discuss heat and cold stress, respectively, and include monitoring methods appropriate for the season and location of work. Based on anticipated site conditions during the demonstration, SWP 6-15 will be available on site.

13.7.7 Noise Monitoring

In most cases, high noise levels at a work site are caused by heavy equipment, such as drill rigs and backhoes, or sources associated with the work site, such as factory equipment and vehicles. When noise levels at the Navy BVC, Kelly AFB, or PC site are suspected to equal or exceed an 8-hour time-weighted average (TWA) of 85 decibels on an A-weighted scale in slow response mode (dBA), the Tetra Tech SSC will evaluate the work area to characterize the noise source and exposure levels. The SSC will use a simple rule-of-thumb test to determine whether noise levels exceed 85 dBA. The test requires the SSC to determine how loudly he must speak to be heard at an arm's length from another person. If the SSC must raise his voice to be heard, the average noise level likely exceeds 85 dBA.

If employees are exposed to noise levels that exceed the action level of 85 dBA, hearing protection must be worn. The protectors will be ear plugs or muffs that provide sufficient attenuation to limit noise exposure to less than 85 dBA. The SSC will supervise use of hearing protectors on site, as necessary. Table 13-3 lists the monitoring method and action level to be used.

13.8 Site Control

Site control is an essential component of implementing health and safety procedures. The following sections discuss measures and procedures for site control, including on-site communications, site control zones, site access control, site safety inspections, and SWPs.

13.8.1 On-Site Communications

Successful communication between field teams and personnel in the support zone is essential. The following communication systems will be available during demonstration activities:

- Cellular telephones
- Hard-wired telephones in the sample management trailer in the PRA at the Navy BVC site

The hand signals listed below will be used by on-site personnel in emergency situations or when verbal communication is difficult.

Signal	Definition
Hands clutching throat	Out of air or cannot breathe
Hands on top of head	Need assistance
Thumbs up	Okay, I am all right, or I understand
Thumbs down	No or negative
Arms waving upright	Send backup support
Gripping partner's wrist	Exit area immediately

13.8.2 Site Control Zones

To control the spread of contamination and employee exposures to chemical and physical hazards, on-site work areas may be divided into an exclusion zone, a decontamination zone, and a support zone. Access to the exclusion and decontamination zones will be restricted to authorized personnel. Any visitors to these areas must present proper identification and be authorized to be on site. The Tetra Tech SSC will identify work areas that visitors or personnel are authorized to enter and will enforce site control measures.

The following sections describe the exclusion zone, the decontamination zone, and the support zone as well as procedures to be followed in each.

13.8.2.1 Zone 1: Exclusion Zone

An exclusion zone includes areas where contamination is either known or likely to be present or, because of work activity, has the potential to cause harm to personnel. The perimeter of the exclusion zone and an appropriate radius around work task areas will be demarcated by a physical barrier, such as barricade tape or traffic cones, to restrict access. A daily roster with the date of each person’s entrance into the exclusion zone; the person’s name, signature, and organization; and the time of entry and exit will be kept for all personnel working in the zone. Visitors will not be permitted to enter the exclusion zone without proper qualifications, equipment, and Tetra Tech SSC authorization. Work tasks that may require establishment of an exclusion zone include the following:

- Collection of soil samples using a Geoprobe® or Split Core Sampler
- Sample management activities in the PRA

13.8.2.2 Zone 2: Decontamination Zone

A decontamination zone will be required at the Navy BVC, Kelly AFB, and PC sites. The decontamination zone will contain facilities to decontaminate personnel and portable equipment. The equipment decontamination procedures that Tetra Tech will follow are described in Section 7.3. Decontamination of Geoprobe® components will be performed by the Geoprobe® operators. Visitors will not be permitted to enter the decontamination zone without proper qualifications and Tetra Tech SSC authorization.

13.8.2.3 Zone 3: Support Zone

A support zone may consist of any uncontaminated and nonhazardous part of a site. The support zone should be situated in an area generally upwind of any exclusion zone and in a location where the chance of encountering hazardous materials or conditions is minimal. Site visitors not meeting the training, medical surveillance, and PPE requirements defined in this chapter must stay in the support zone.

13.8.3 Site Access Control

The PRA and FFA at the Navy BVC site are fully fenced, and access to this site is controlled by a security system that requires visitor passes to be issued before access is allowed. The NEX Service Station Area, however, is not fenced; site representatives will be present during sampling in this area to control visitor access.

The sampling area at the Kelly AFB site is bounded by a locked, chain-link fence. Tetra Tech and subcontractor personnel will be escorted by the demonstration representative while on site.

The sampling area at the PC site is generally bounded by fencing. Tetra Tech and subcontractor personnel will be escorted by the PC site environmental contractor at all times.

13.8.4 Site Safety Inspections

Periodic site safety inspections will be conducted by the Tetra Tech SSC to ensure safe work areas and compliance with the health and safety procedures described in this chapter. Results of the site safety inspections will be recorded in the field logbook or on a Field Audit Checklist (Form AF-1 in Appendix C).

13.8.5 Safe Work Practices

The following SWPs apply to the demonstration activities. These SWPs will be available on site.

- SWP 6-1, General Safe Work Practices
- SWP 6-14, Spill and Discharge Control Practices
- SWP 6-15, Heat Stress
- SWP 6-25, Oil and Petroleum Distillate Fuel Product Hazards

- SWP 6-27, Respirator Cleaning Procedures
- SWP 6-28, Safe Work Practices for Use of Respirators

13.9 Decontamination

Decontamination is the process of removing or neutralizing contaminants on personnel or equipment. When properly conducted, decontamination procedures protect workers from contaminants that may have accumulated on PPE, tools, and other equipment. Proper decontamination also prevents transport of potentially harmful materials to uncontaminated areas. Personnel and equipment decontamination procedures are described in the following sections.

13.9.1 Personnel Decontamination

Personnel decontamination at the demonstration sites will be limited by using disposable PPE whenever possible. Any personnel decontamination procedures will follow guidance in the *Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities* (NIOSH and others 1985). Personnel and PPE will be decontaminated with potable water or a mixture of detergent and water. Liquid and solid wastes generated during decontamination will be collected and drummed.

The decontamination procedures listed below will be conducted if personnel decontamination is required.

- Wash neoprene boots or disposable booties with a Liquinox® or Alconox® solution, and rinse them with water. Remove and retain neoprene boots for reuse, if possible. Place disposable booties in plastic bags for disposal.

- Wash outer gloves in a Liquinox® or Alconox® solution, and rinse them in water. Remove outer gloves, and place them in a plastic bag for disposal.

- Remove chemical-resistant clothing, and place it in a plastic bag for disposal.

- Remove the air-purifying respirator, if used, and place the spent filter in a plastic bag for disposal. Change the filter in accordance with the Respiratory Hazard Assessment form (Form RP-2 in Appendix C). Clean and disinfect the respirator in accordance with SWP 6-27, and place it in a plastic bag for storage.

- Remove inner gloves, and place them in a plastic bag for disposal.

- Thoroughly wash the hands and face with water and soap.

Used, disposable PPE will be collected in sealable containers and will be disposed of in accordance with procedures described in Section 7.3. Personnel decontamination procedures may be modified on site, if necessary.

13.9.2 Equipment Decontamination

Decontamination of all sampling and field monitoring equipment used during demonstration activities will be required. Decontamination of Geoprobe® components will be performed by the Geoprobe® operators.

Sampling equipment such as the Split Core Sampler will be decontaminated before and after each use as described below.

- Scrub the equipment with a brush in a bucket containing Liquinox® or Alconox® solution and distilled water.
- Triple-rinse the equipment with distilled water, and allow it to air-dry.
- Reassemble the equipment, and place it on plastic or aluminum foil in a clean area. If aluminum foil is used, wrap the equipment with the dull side of the aluminum foil toward the equipment.

13.10 Emergency Response Planning

This section describes emergency response planning procedures to be implemented for the demonstration. This section is consistent with local, state, and federal disaster and emergency management plans. The following sections discuss pre-emergency planning, personnel roles and lines of authority, emergency recognition and prevention, evacuation routes and procedures, emergency contacts and notifications, hospital route directions, emergency medical treatment procedures, protective equipment failure, fire or explosion, weather-related emergencies, spills or leaks, emergency equipment and facilities, and reporting.

13.10.1 Pre-Emergency Planning

During the prework briefing and daily tailgate safety meetings, all on-site employees will be trained in and reminded of the provisions of Section 13.10, site communication systems, and site evacuation routes. The emergency response provisions will be reviewed on a regular basis by the Tetra Tech SSC and will be revised, if necessary, to ensure that they are adequate and consistent with prevailing site conditions.

13.10.2 Personnel Roles and Lines of Authority

The Tetra Tech SSC has primary responsibility for responding to and correcting emergency situations and for taking appropriate measures to ensure the safety of on-site personnel and the public. Possible actions may include evacuation of personnel from site areas. The SSC is also responsible for ensuring that corrective measures have been implemented, appropriate authorities have been notified, and follow-up reports have been completed.

Individual subcontractors are required to cooperate with the SSC within the parameters of their scopes of work.

Personnel are required to report all injuries, illnesses, spills, fires, and property damage to the SSC. The SSC must be notified of any on-site emergencies and is responsible for ensuring that the appropriate emergency procedures described in this section are followed.

13.10.3 Emergency Recognition and Prevention

Table 13-1 provides information on the hazards associated with the different tasks planned for the demonstration sites. On-site personnel will be made familiar with this information and with techniques of hazard recognition through prework training and site-specific briefings.

13.10.4 Evacuation Routes and Procedures

In the event of an emergency that necessitates evacuation of a work task area or a site, the Tetra Tech SSC will contact all nearby personnel using the on-site communications discussed in Section 13.8.1 to advise the personnel of the emergency. The personnel will proceed along site roads to a safe area upwind from the hazard source. The personnel will remain in that area until the SSC or an authorized individual provides further instructions.

13.10.5 Emergency Contacts and Notifications

Appendixes B-1, B-2, and B-3 provide the names and telephone numbers of emergency contact personnel for the Navy BVC, Kelly AFB, and PC sites, respectively. The information in these appendixes must be posted on site or must be readily available at all times. In the event of a medical emergency, personnel will notify the appropriate emergency organization and will take direction from the Tetra Tech SSC. In the event of a fire, explosion, or spill at a site, the SSC will notify the appropriate local, state, and federal agencies and will follow procedures discussed in Section 13.10.9 or 13.10.11.

13.10.6 Hospital Route Directions

Before performing any demonstration activities at a site, Tetra Tech personnel will conduct a pre-emergency hospital run to familiarize themselves with the route to the local hospital. Maps showing the appropriate hospital routes are provided in Appendixes B-1 (Navy BVC site), B-2 (Kelly AFB site), and B-3 (PC site). These maps must be posted on site.

13.10.7 Emergency Medical Treatment Procedures

A person who becomes ill or injured during work tasks may require decontamination. If the illness or injury is minor, any decontamination necessary will be completed and first aid will be administered prior to patient transport. If the patient's condition is serious, partial decontamination will be completed at a minimum (such as complete disrobing of the person and redressing the person in clean coveralls or wrapping the person in a blanket). First aid will be administered until an ambulance or paramedics arrive. All injuries and illnesses must be immediately reported to the Tetra Tech project manager and HSR.

Any person transported to a clinic or hospital for chemical exposure treatment will be accompanied by information on the chemical that he or she has been exposed to at the site, if possible.

13.10.8 Protective Equipment Failure

If any worker in the exclusion zone experiences a failure of protective equipment (either engineering controls or PPE) that affects his or her personal protection, the worker and all coworkers will immediately leave the exclusion zone. Re-entry to the exclusion zone will not be permitted until (1) the protective equipment has been repaired or replaced, (2) the cause of the equipment failure has been determined, and (3) equipment failure is no longer considered to be a threat.

13.10.9 Fire or Explosion

In the event of a fire or explosion on site, the local fire department will be immediately summoned. The Tetra Tech SSC or a site representative will advise the fire department of the location and nature of any hazardous materials involved. Appropriate provisions of Section 13.10 will be implemented by on-site personnel.

13.10.10 Weather-Related Emergencies

Site work will not be conducted during severe weather conditions, including high-speed winds or lightning. In the event of severe weather, field personnel will stop work, secure and lower all equipment (for example, drilling masts), and leave the site.

Thermal stress caused by excessive heat may occur as a result of extreme temperatures, workload, or the PPE used. Heat stress treatment will be administered as described in SWP 6-15, which will be available on site. Cold stress is not anticipated during the demonstration.

13.10.11 Spills or Leaks

In the event of a severe spill or a leak, site personnel will follow the procedures listed below.

- Evacuate the affected area, and relocate personnel to an upwind location.
- Inform the Tetra Tech SSC, a Tetra Tech office, and a site representative immediately.
- Locate the source of the spill or leak, and stop the flow if it is safe to do so.
- Begin containment and recovery of spilled or leaked materials.
- Notify appropriate local, state, and federal agencies.

Additional information on spill and leak control is presented in SWP 6-14, which will be available on site.

13.10.12 *Emergency Equipment and Facilities*

The following emergency equipment and facilities will be available on site:

- First aid kit
- Eye wash (portable)
- Fire extinguisher (only in sample management trailer in PRA)
- Site telephone (only in sample management trailer in PRA)
- Cellular telephone
- Drums

13.10.13 *Reporting*

All emergency situations require follow-up and reporting. Appendix C contains the Tetra Tech Accident and Illness Investigation Report (Form AR-1). This report must be completed and submitted to the Tetra Tech project manager within 24 hours of an emergency situation. The project manager will review the report and then forward it to the Tetra Tech HSR for review. The report must include proposed actions to prevent similar incidents from occurring. The HSR must be fully informed of the corrective action process so that she may implement applicable elements of the process at other sites.

REVIEWS AND APPROVALS

CLIENT NAME: EPA

CONTRACT NO.: 68-C5-0037 WORK ASSIGNMENT NO.: 47

NAVY BVC SITE
PORT HUENEME, CALIFORNIA

KELLY AFB SITE
SAN ANTONIO, TEXAS

PC SITE
NORTH-CENTRAL INDIANA

We the undersigned have read and approve of the health and safety procedures presented in this chapter for on-site work activities at the Navy BVC, Kelly AFB, and PC sites.

Name	Signature	Date
Judith Wagner Tetra Tech HSR	Judith L. Wagner	2 June 2000
Kirankumar Topudurti Tetra Tech Project Manager	Sivan.	3 June 2000

This certifies that Tetra Tech has assessed the type, risk level, and severity of hazards for the project and has selected appropriate personal protective equipment for on-site personnel in accordance with OSHA Title 29 of the CFR, Section 1910.132.

Certified by

Judith Wagner Tetra Tech Technical Reviewer	Judith L. Wagner	2 June 2000

Chapter 14
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Appendix A
Review of Predemonstration Investigation Procedures and Results

Several findings of the predemonstration sampling and analysis investigation and the developers' review comments on the predemonstration investigation results were instrumental in developing the sampling and analysis procedures for the demonstration. This section discusses those findings and presents Tetra Tech's responses to developer comments on predemonstration investigation results.

A.1 Selected Predemonstration Investigation Findings

This section summarizes the findings of the predemonstration investigation that impacted the demonstration approach. Based on these findings, improvements were made to predemonstration investigation sampling and analysis procedures before the procedures were adapted for the demonstration.

A.1.1 Use of Nitrile Gloves During Sample Preparation

During the predemonstration investigation, nitrile gloves were used by the field sampling team for most sample preparation; however, when the supply of nitrile gloves was depleted, locally available plastic gloves were used. Use of plastic gloves resulted in phthalate contamination of predemonstration investigation samples. Therefore, as described in Section 7.1.3.1, only nitrile gloves will be used by the field sampling team during the demonstration.

A.1.2 Selection of Sampling Depth Intervals at Kelly AFB Site

During the demonstration, soil samples will be collected in the B-38 Area of the Kelly AFB Site at the four locations sampled during the predemonstration investigation. However, the depth intervals that will be sampled during the demonstration will differ from those sampled during the predemonstration investigation because the predemonstration investigation samples contained only trace concentrations of TPH. Based on discussions with the demonstration site representative after the predemonstration investigation, it was determined that most of the contamination in the B-38 Area can be found at or near the water table. Therefore, the depth intervals to be sampled during the demonstration will be 2 feet above and 2 feet below the water table. For the purposes of this demonstration plan, Tetra Tech assumes that the surface of the water table will be about 20 feet bgs during the demonstration. This assumption is based on previous groundwater-level data provided by the site representative. Using a water-level indicator, Tetra Tech will measure the exact depth to groundwater in four monitoring wells near the B-38 Area at the time of the demonstration. Tetra Tech will then calculate an average depth to groundwater and will collect soil samples 2 feet above and 2 feet below the average water table depth. Therefore, as described in Section 7.1.1.2, soil samples will likely be collected from two depth intervals—18 to 20 and 20 to 22 feet bgs—at the four sampling locations.

A.1.3 Spiking of Low- and Medium-Level PE Samples

During the predemonstration investigation, methanol and Freon 113 were used as carriers for low-level soil PE samples. After reviewing STL Tampa East results for these samples, Tetra Tech determined that the samples prepared using methanol as a carrier contained up to 40 percent lower levels of TPH than those prepared using Freon 113 as a carrier. According to the manufacturer of EnCores, using methanol as the carrier in an EnCore may have caused the sampler's viton ring to swell, opening the pores on the viton ring; as a result, some of the volatiles in the sample may have escaped. As described in Section 7.1.3.2, to address this potential loss of volatiles, ERA will spike the low- and medium-level PE samples prepared using methanol with 40 percent higher TPH levels than those actually requested by Tetra Tech.

A.2 Tetra Tech Responses to Developer Comments on Predemonstration Investigation Results

This section summarizes technical issues raised by developers based on their review of the predemonstration investigation results and presents Tetra Tech's responses to the developers' comments. In Sections A.2.1 through A.2.11, each developer comment is presented in italics and is followed by Tetra Tech's response. Improvements were made to predemonstration investigation sampling and analysis procedures based on developer comments before the procedures were adapted for the demonstration.

A.2.1 Field Sample Homogenization Procedure

Developer Comment. *When a given sample was divided into two aliquots, each aliquot extracted and analyzed separately, the analysis results for the two aliquots did not always match.*

Tetra Tech Response. STL Tampa East's blind field triplicate analytical results showed that (1) when samples did not contain clay, the RSD was only 5 percent (a high level of precision for soil samples), and (2) when samples contained a significant amount of clay, the RSD was about 25 percent (a typical level of precision for soil samples). Therefore, the field sample homogenization procedure produced acceptable results.

A.2.2 Interference of Complex Matrixes in Analysis

Developer Comment. *The nature of soil samples resulted in bubbling on acidification (perhaps because of high carbonate in soil), thick, gelatinous emulsion (perhaps because of high clay in soil), or precipitation of elemental sulfur (perhaps because of sulfide in soil). These complex matrixes might have caused some analytical problems.*

Tetra Tech Response. Tetra Tech acknowledges the validity of the comment. However, the environmental samples were collected in five different areas; depending on the analytical method used, such problems may not be uncommon in environmental sample analysis. The developers can make method modifications to address problems observed during the predemonstration investigation, if necessary.

A.2.3 Choosing Not to Analyze Samples from a Given Sampling Area

Developer Comment. *Would it be possible to exclude samples from the phytoremediation area because our kit is not designed [to] measure compounds that are heavier than diesel?*

Tetra Tech Response. A developer may choose not to analyze samples collected in a given area provided that the developer informs Tetra Tech of this choice in advance. To be fair to all the developers and to potential users of the field measurement devices, each ITVR will identify all the scenarios that are part of the demonstration approach (samples collected in five areas and PE samples) and will state that the developer chose not to analyze samples collected in a given area because its field measurement device is not designed to analyze such samples (for example, samples containing compounds heavier than diesel).

A.2.4 Low, Medium, and High Classification of Environmental Samples

Developer Comment. *For the actual demonstration, we would propose running samples greater than 200 ppm [mg/kg] because it appears that many samples will be above this level.*

Tetra Tech Response. The predemonstration investigation results show that no area's samples will all have TPH concentrations less than 200 mg/kg. To establish a reasonable starting point, samples will be identified as low-level (<100 mg/kg), medium-level (100 to 1,000 mg/kg) and high-level (>1,000 mg/kg). PE samples will be identified as such in their sample identification numbers, and their concentration levels will be identified as low, medium, or high. No backup environmental or PE samples will be provided.

A.2.5 STL Tampa East's Analytical Results for Blank and PE Samples

Developer Comment 1. *We noticed that STL Tampa East actually reported a higher value in the blank sample than for the triplicate series of reporting limits and wondered if their reporting limits needed adjusting and if this might have implications for their environmental sample results. In addition, we noted the apparent reporting discrepancy with the stated certified values for those samples. The blank sample has a certified value of <45 while an actual number, (26 mg/kg), is reported for the triplicate series.*

Developer Comment 2. *The fact that STL Tampa East failed in the PE blank analysis (reporting 52 ppm [mg/kg] for a certified value of <45 ppm [mg/kg]) is very disturbing. However, as a result, it could be reasoned that since the laboratory could not reasonably demonstrate accuracy at low concentrations, everything below 50 ppm [mg/kg] is void.*

Tetra Tech Response. STL Tampa East reported a higher value for the blank sample than for the triplicate series of reporting limit samples because STL Tampa East analyzed the blank sample for both GRO and EDRO but the triplicate reporting limit samples for GRO only. The triplicate reporting limit samples were analyzed for GRO only because it was not clear that they were spiked with weathered gasoline instead of fresh gasoline. Recent information provided by ERA indicates that the blank soil (processed garden soil) has low levels of EDRO. During the demonstration, these issues will be addressed as follows: (1) all PE samples containing GRO will be analyzed for both GRO and EDRO because weathered gasoline contains a significant quantity of TPH in the EDRO range and (2) low-level PE samples (<100 mg/kg) will be prepared using Ottawa sand, not processed garden soil.

ERA's certified values for the blank soil were <15 mg/kg gasoline and <45 mg/kg TPH. ERA has informed Tetra Tech that the certified TPH value was based on infrared analysis, not GC/FID analysis. According to ERA, should the blank soil be subjected to GC/FID analysis, the certified value will be greater, particularly in the EDRO range. Because a GC/FID method is the reference method for the project, ERA will attempt to provide the certified value based on the GC/FID method.

The reasons for the apparent reporting discrepancy associated with the stated certified value are as follows: (1) the blank soil result was based on infrared analysis, and (2) like STL Tampa East, ERA analyzed the reporting limit samples only for GRO. Tetra Tech will try to obtain an appropriate certified value for the blank soil and will have all GRO PE samples analyzed for both GRO and EDRO.

A.2.6 Comparing Field Measurement Device Results with Acceptance Limits

Developer Comment. *We understand that vendor results will be compared with the stated acceptance limits. We wanted to make a comment that if these limits were derived from the inter-laboratory results in which laboratories used Soxhlet/GC methods of analysis, it may not be appropriate to ~~hold~~ [compare] the field test results that rely on much less rigorous extraction methods [to the acceptance limits].*

Tetra Tech Response. The basis of the evaluation will be a comparison of the performance of a field measurement device with that of the reference method selected for the project. PE samples will be used primarily to verify that the laboratory's performance is acceptable. However, as stated in Section 4.2, for comparisons with field measurement device results, the laboratory results will not be adjusted based on the recoveries observed during PE sample analysis. Any consistent data trends will be discussed in each ITVR to explain the differences between the laboratory and field measurement device results. In practice, the recoveries observed for LCS/LCSD, MS/MSD, and PE samples are not used to correct investigative sample results. In addition, the soil characteristics of the PE samples will not be the same as those of all the investigative samples.

According to ERA, not all laboratories that contributed to the development of the acceptance limits used the same extraction method, but they did use a GC/FID for analysis. The reference method selected for the project is a method used for TPH measurement by about 75 percent of the states in the United States.

A.2.7 Acceptance Limits for PE Samples

Developer Comment. *How were the acceptance limits for PE samples determined? Why do you favor lower recovery concentrations to higher?*

Tetra Tech Response. The acceptance limits were derived by ERA based on historical mean recovery values for similar types of samples. The acceptance limits were evenly distributed about the historical mean recovery values. However, these acceptance limits were not evenly distributed about the certified values because the certified values were not corrected for the historical mean recovery values. Therefore, the acceptance limits may seem to have been biased low or high, depending on whether the certified values were higher or lower than the historical mean recovery values.

A.2.8 Quantification of TPH by STL Tampa East

Developer Comment. *There are some subtle details about the laboratory analysis that I think need to be considered. This consideration goes to the heart of the difficulty of this project and that is the fact that TPH is a method defined parameter. If the laboratory is measuring one thing and the field methods are measuring another they will never agree. The most obvious manifestation of this is the performance of the laboratory on the PE samples. As you can see the reference laboratory was at the low end in reporting the EDRO results. Does this mean that all of the laboratory results are low on all of the field samples as well?*

Tetra Tech Response. The laboratory procedure for TPH measurement is described in Section 9.1 so that it is clear what the laboratory will measure. Tetra Tech performed a detailed review of calibration data and observed that the average response factor for hydrocarbons in the C_{10} to C_{28} range was less than the average response factor for hydrocarbons in the C_{10} to C_{40} range. The reason for this decrease could not be defined with the limited data available. However, Tetra Tech believes that the decrease may have been due to a reduced area in the chromatogram for the early-eluting compounds in the DRO standard (C_{10} to C_{14}), possibly because of the use of the split/splitless injection system. This belief is based on Section 4.1.3 of SW-846 Method 8000, which indicates that the response for labile compounds may decrease in split/splitless injection systems.

To address the problem of low recoveries, the demonstration samples for EDRO analysis will be quantified over two ranges: (1) C_{10} (greater than n-decane) through C_{28} (including n-octacosane), which is the typical DRO range, and

(2) C_{28} (greater than n-octacosane) through C_{40} (n-tetracontane), which extends the DRO range. The sum of these two values will be reported as EDRO.

A.2.9 Solubility of C_{10} to C_{40} Standard in Methylene Chloride

Developer Comment. *Our laboratory people observed that C_{10} to C_{40} standard will not completely dissolve in methylene chloride. I think that this information on the methylene chloride and the low recoveries on the PE samples indicate that we should consider an alternative to the current method.*

Tetra Tech Response. Solubility constraints were not observed by STL Tampa East during initial calibration or sample analysis—that is, no precipitation was observed. The reason for the low recoveries and the proposed approach to address the issue are discussed in Section A.2.8. As stated in Section A.2.6, more than 75 percent of the states in the United States use a method based on SW-846 Method 8015B for GC/FID analysis.

A.2.10 Presentation of Demonstration Results on Wet Weight Basis

Developer Comment. *Regarding the certified values for PE samples, the wet weight and dry weight results have to be different from each other.*

Tetra Tech Response. ERA provided certified values on a dry weight basis. The PE sample certified values in the predemonstration investigation data summary tables provided to the developers are presented on a dry weight basis. For the demonstration, all analytical results will be presented on a wet weight basis.

A.2.11 Use of TPH Results for Field Measurement Devices

Developer Comment. *For the B38 PE series, shouldn't the field device GRO results (instead of TPH) be compared with STL Tampa East's GRO results? What are the GRO and EDRO concentrations for all confirmatory results?*

Tetra Tech Response. The TPH results were used because not all the field measurement devices can measure GRO and EDRO separately. During the demonstration, STL Tampa East will analyze all GRO-containing samples for both GRO and EDRO because weathered gasoline will have a significant portion of TPH that eludes in the early EDRO range. The GRO and EDRO results for all confirmatory samples were provided.

Appendix B
Emergency Information

- Appendix B-1, Navy Base Ventura County Site
- Appendix B-2, Kelly Air Force Base Site
- Appendix B-3, Petroleum Company Site

Appendix B-1
Emergency Information
Navy Base Ventura County Site

EMERGENCY INFORMATION
(POST ON SITE)

EMERGENCY CONTACTS AND ROUTE TO HOSPITAL
NAVY BASE VENTURA COUNTY SITE

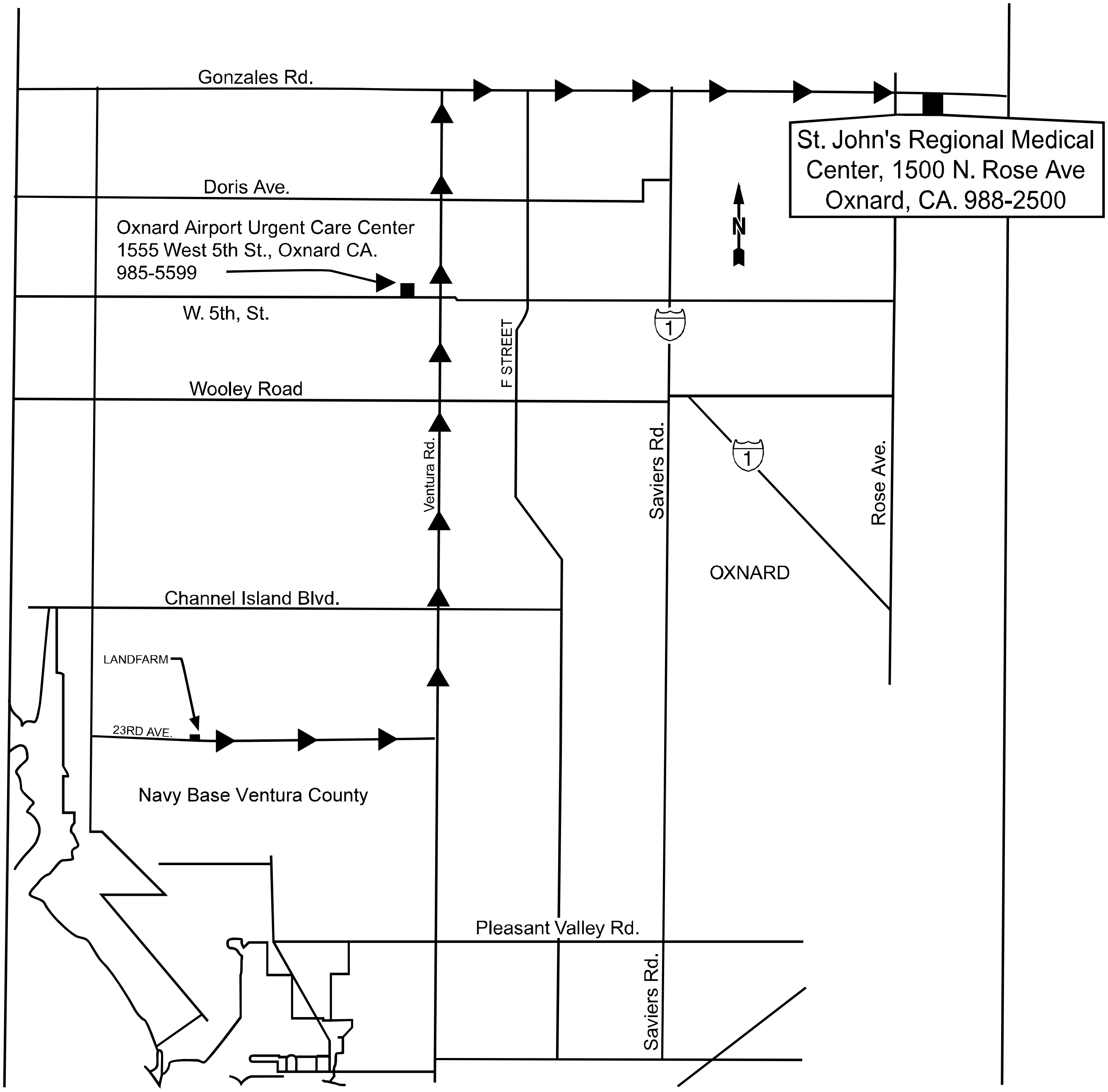
Emergency Contact	**Telephone No.**
U.S. Coast Guard National Response Center	(800) 424-8801
InfoTrac	(800) 535-5053
Fire Department	911
Police Department	911
Tetra Tech EM Inc. Personnel:	
Human Resources Representative: Norman Endlich	(703) 390-0626
Health and Safety Representative: Judith Wagner	(847) 818-7192
Office Health and Safety Coordinator: Carrie Haag	(312) 856-8748
Project Manager: Kirankumar Topudurti	(312) 856-8742
Site Safety Coordinator: Jill Ciraulo	(312) 946-6479
Client Contact:	
U.S. Environmental Protection Agency Project Manager: Stephen Billets	(702) 798-2232
Medical Emergency	
Hospital Name: St. John's Regional Medical Center	
Hospital Address: 1500 N. Rose Avenue Oxnard, CA 93030	
Hospital Telephone No.:	Emergency - 911 General - (805) 988-2500
Ambulance Telephone No.:	911

Route to Hospital: (see Page 238 for hospital route map)

From the site, drive north on Ventura Road to Gonzales Road. Turn east (right) on Gonzales Road, and proceed to Rose Avenue. Turn south (right) onto Rose Avenue. St. John's Regional Medical Center, will be on the left near the intersection of Gonzales Road and Rose Avenue.

EMERGENCY INFORMATION
(POST ON SITE)

HOSPITAL ROUTE MAP
NAVY BASE VENTURA COUNTY SITE



Appendix B-2
Emergency Information
Kelly Air Force Base Site

EMERGENCY INFORMATION
(POST ON SITE)

EMERGENCY CONTACTS AND ROUTE TO HOSPITAL
KELLY AIR FORCE BASE SITE

Emergency Contact	**Telephone No.**
U.S. Coast Guard National Response Center	(800) 424-8801
InfoTrac	(800) 535-5053
Fire Department	911
Police Department	911
Tetra Tech EM Inc. Personnel:	
Human Resources Representative: Norman Endlich	(703) 390-0626
Health and Safety Representative: Judith Wagner	(847) 818-7192
Office Health and Safety Coordinator: Carrie Haag	(312) 856-8748
Project Manager: Kirankumar Topudurti	(312) 856-8742
Site Safety Coordinator: Jill Ciraulo	(312) 946-6479
Client Contact:	
U.S. Environmental Protection Agency Project Manager: Stephen Billets	(702) 798-2232

Medical Emergency

Hospital Name: Wilford Hall Medical Center

Hospital Address: 2200 Bergquist Drive
San Antonio, TX 78236

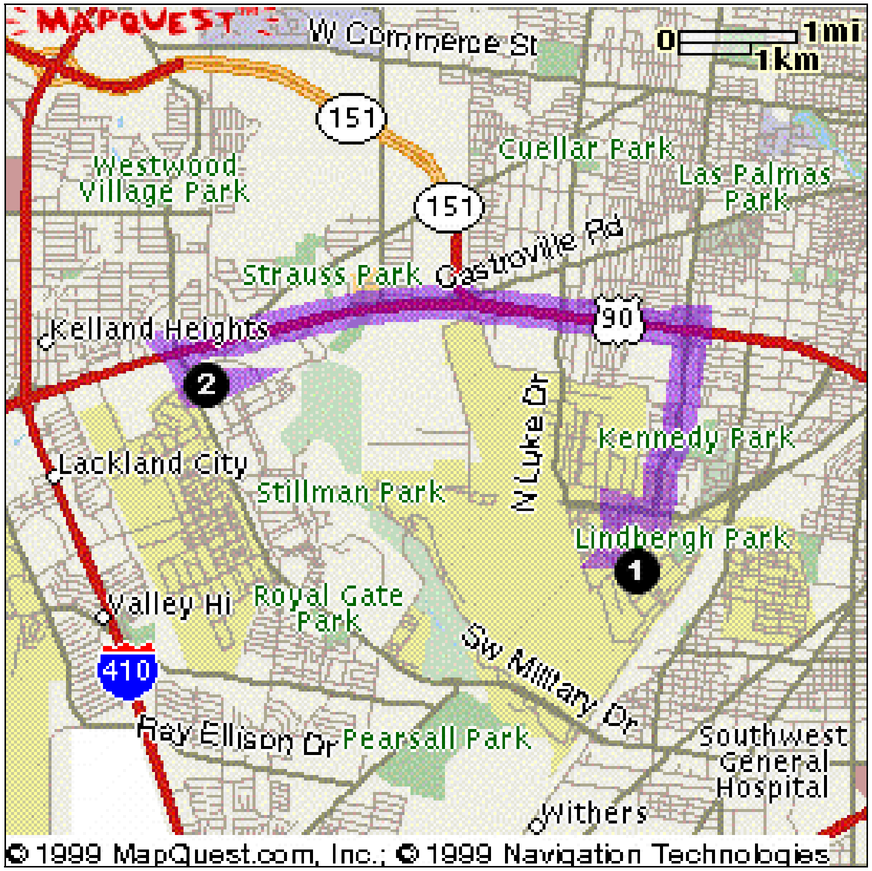
Hospital Telephone No.:	Emergency - 911 General - (210) 292-7331
Ambulance Telephone No.:	911

Route to Hospital: (see Page 241 for hospital route map)

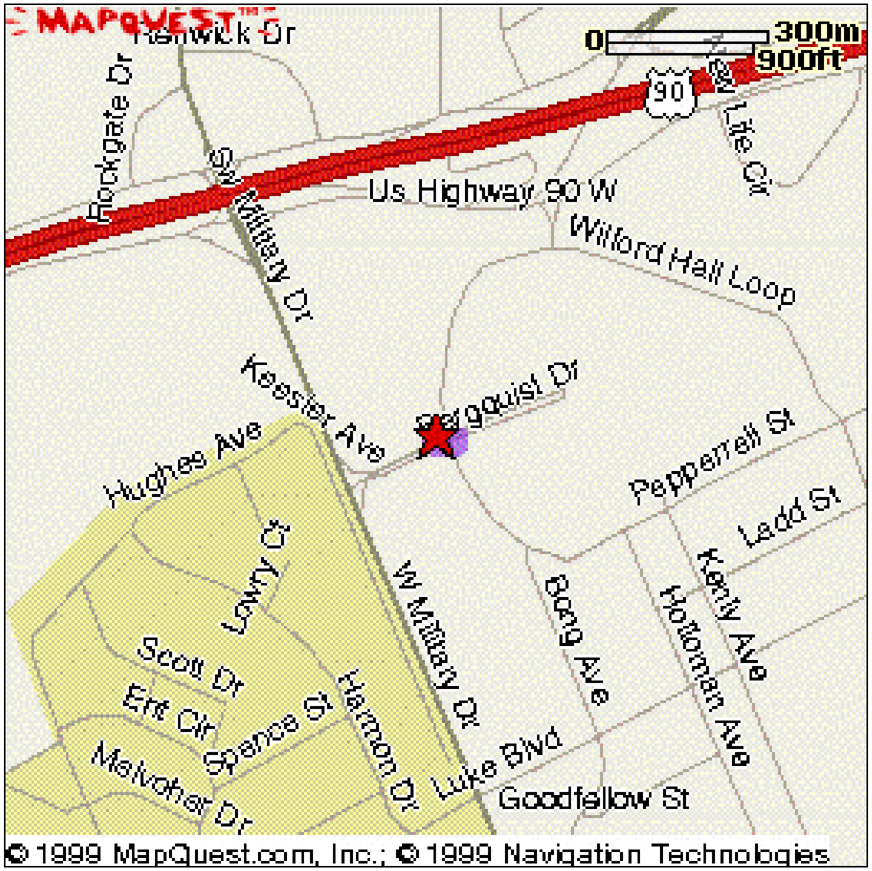
From the site, proceed to General McMullen Road, and head north to Highway 90. Turn left onto Highway 90, and head west to S.W. Military Drive. Turn left onto S.W. Military Drive, and head south to Wilford Hall Medical Center, which is on the left.

EMERGENCY INFORMATION
(POST ON SITE)

HOSPITAL ROUTE MAP
KELLY AIR FORCE BASE SITE



Full Route



Destination

Appendix B-3
Emergency Information
Petroleum Company Site

EMERGENCY INFORMATION
(POST ON SITE)

EMERGENCY CONTACTS AND ROUTE TO HOSPITAL
PETROLEUM COMPANY SITE

Emergency Contact	Telephone No.
U.S. Coast Guard National Response Center	(800) 424-8801
InfoTrac	(800) 535-5053
Fire Department	911
Police Department	911
Tetra Tech EM Inc. Personnel:	
Human Resources Representative: Norman Endlich	(703) 390-0626
Health and Safety Representative: Judith Wagner	(847) 818-7192
Office Health and Safety Coordinator: Carrie Haag	(312) 856-8748
Project Manager: Kirankumar Topudurti	(312) 856-8742
Site Safety Coordinator: Jill Ciraulo	(312) 946-6479
Client Contact:	
U.S. Environmental Protection Agency Project Manager: Stephen Billets	(702) 798-2232

Medical Emergency

Hospital Name: Home Hospital

Hospital Address: 2400 South Street
Lafayette, IN 47904

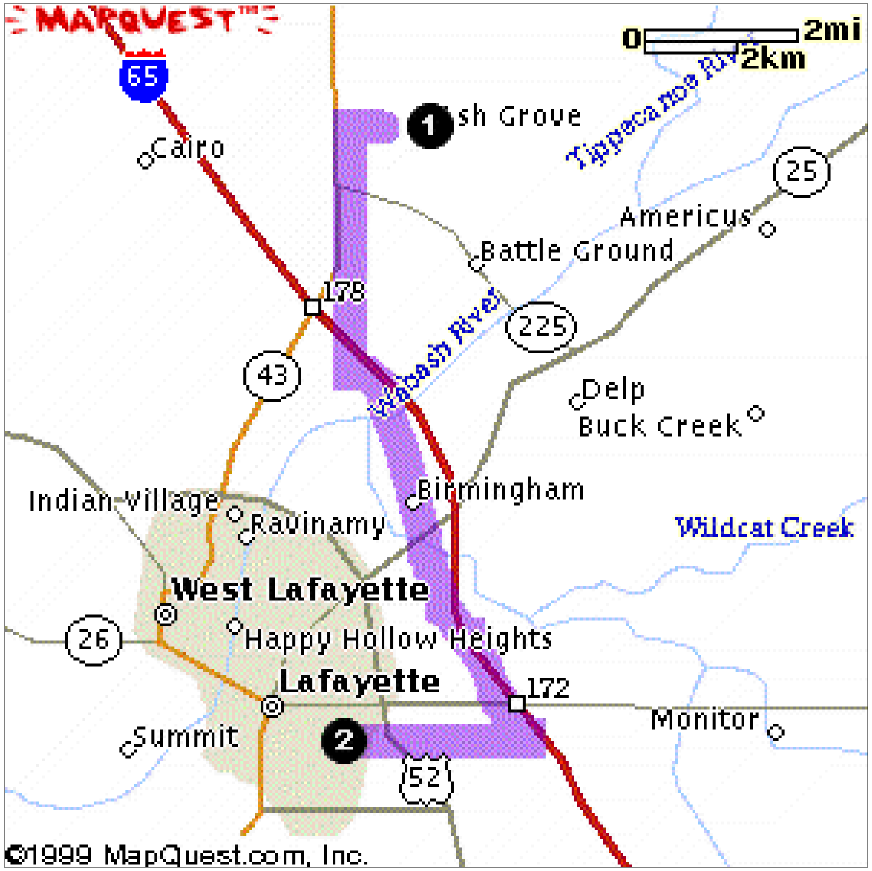
Hospital Telephone No.:	Emergency - 911 General - (317) 477-6811
Ambulance Telephone No.:	911

Route to Hospital: (see Page 244 for hospital route map)

From the site take State Route (SR) 43 south to Interstate 65. Take Interstate 65 South to SR 26 (South Street). Go west on SR 26. Home Hospital is on the north (right) side of SR 26.

EMERGENCY INFORMATION
(POST ON SITE)

HOSPITAL ROUTE MAP
PETROLEUM COMPANY SITE



Full Route

Destination

Appendix C
Tetra Tech, Inc., Forms

- Daily Tailgate Safety Meeting Form (Form HST-2)
- Daily Site Log (Form SSC-1)
- Accident and Illness Investigation Report (Form AR-1)
- Field Audit Checklist (Form AF-1)
- Respiratory Hazard Assessment (Form RP-2)

TETRA TECH, INC.

DAILY TAILGATE SAFETY MEETING FORM

Date: ______________ Time: ________________ Project No.: ________________

Client: __________________________________ Site Location: __________________________

Site Activities Planned for Today: ____

____

Safety Topics Discussed
Protective clothing and equipment:
Chemical hazards:
Physical hazards:
Environmental and biohazards:
Equipment hazards:
Decontamination procedures:
Other:
Review of emergency procedures:
Employee Questions or Comments:

TETRA TECH, INC.

DAILY TAILGATE SAFETY MEETING FORM (Continued)

Attendees	
Printed Name	**Signature**

Meeting Conducted by:

Name ______________________ Title ______________________

Signature ______________________

TETRA TECH, INC.

DAILY SITE LOG

Site Name: ______________________________ Date: ______________________

Name (print)	Company	Time	
		In	Out

Comments:

____

____

____

____

TETRA TECH, INC.

ACCIDENT AND ILLNESS INVESTIGATION REPORT

To: ______________________________
Regional or Subsidiary Health and Safety Representative

Cc: ______________________________
Workers Compensation Administrator

Project Name: ______________________________

Project No. ______________________________

Prepared by: ______________________________

Position: ______________________________

Office: ______________________________

Telephone: ______________________________

Fax: ______________________________

Information Regarding Injured or Ill Employee

Name: ______________________________

Home address: ______________________________

____

Home telephone: ______________________________

Occupation (regular job title): ______________________________

Department: ______________________________

Office: ______________________________

Gender: M ☐ F ☐ No. of dependents: __________

Marital status: ______________________________

Date of birth: ______________________________

Social Security Number: ______________________________

Date of Accident: ______________________________

Time of Accident: ______________________________

Location of Accident

Street address: ______________________________

City, state, and zip code: ______________________________

County: ______________________________

Was place of accident or exposure on employer's premises Yes ☐ No ☐

Narrative Description of How Accident Occurred: (Explain what the employee was doing and how the accident occurred.)

Did the employee die? Yes ☐ No ☐
Was employee performing regular job duties? Yes ☐ No ☐
Was safety equipment provided? Yes ☐ No ☐
Was safety equipment used? Yes ☐ No ☐
Note: Attach any police reports or related diagrams to this accident report.

TETRA TECH, INC.

ACCIDENT AND ILLNESS INVESTIGATION REPORT (Continued)

Witness(es):

Name:

Address:

Telephone:

Nature of Illness or Injury and Part of Body Affected:

Describe the Object or Substance which Directly Injured the Employee:

Medical Treatment Required:

☐ No ☐ Yes ☐ First Aid Only

Physician's Name: ____________________________

Hospital or Office Name: ______________________

Address: _________________________________

____

Telephone No.: ______________________________

Lost Work Days:

☐ No. of Lost Work Days ___________________

Last Date Worked ___________________________

Time Employee Left Work ___________________

Date Employee Returned to Work______________

☐ No. of Restricted Work Days ______________

☐ None

Corrective Action(s) Taken by Unit Reporting the Accident:

Corrective Action Still to be Taken (by whom and when):

Name of Tetra Tech employee the injury or illness was first reported to: ____________________________

Date of Report: ______________________________ **Time of Report:** ______________________________

TETRA TECH, INC.

ACCIDENT AND ILLNESS INVESTIGATION REPORT (Continued)

	Printed Name	Signature	Telephone No.	Date
Project or Office Manager				
Site Safety Coordinator				
Injured Employee				

To be completed by Human Resources:

SSN:

Date of hire: Hire date in current job:

Wage information: $ ____________ per __________ (hour, day, week, or month)

Position at time of hire:

Current position: Shift hours:

State in which employee was hired:

Status: ☐ Full-time ☐ Part-time Hours per week: ________ Days per week: _______

Temporary job end date:

To be completed during report to workers' compensation insurance carrier:

Date reported: Reported by:

Confirmation number:

Name of contact:

Field office of claims adjuster:

TETRA TECH, INC.

FIELD AUDIT CHECKLIST

Project Name: ______________________ Project No.: ______________________

Field Location: ______________________ Completed by: ______________________

Project Manager: ______________________ Site Safety Coordinator: ______________________

General Items		In Compliance?		
Health and Safety Plan Requirements		Yes	No	NA
1	Approved health and safety plan (HASP) on site or available			
2	Names of on-site personnel recorded in field logbook or daily log			
3	HASP compliance agreement form signed by all on-site personnel			
4	Material Safety Data Sheets on site or available			
5	Designated site safety coordinator present			
6	Daily tailgate safety meetings conducted and documented			
7	On-site personnel meet HASP requirements for medical examinations, fit testing, and training (including subcontractors)			
8	Compliance with specified safe work practices			
9	Documentation of training, medical examinations, and fit tests available from employer			
10	Exclusion, decontamination, and support zones delineated and enforced			
11	Windsock or ribbons in place to indicate wind direction			
12	Illness and injury prevention program reports completed (California only)			
Emergency Planning				
13	Emergency telephone numbers posted			
14	Emergency route to hospital posted			
15	Local emergency providers notified of site activities			
16	Adequate safety equipment inventory available			
17	First aid provider and supplies available			
18	Eyewash stations in place			
Air Monitoring				
19	Monitoring equipment specified in HASP available and in working order			
20	Monitoring equipment calibrated and calibration records available			
21	Personnel know how to operate monitoring equipment and equipment manuals available on site			
23	Environmental and personnel monitoring performed as specified in HASP			

TETRA TECH, INC.

FIELD AUDIT CHECKLIST (Continued)

Safety Items		In Compliance?		
Personal Protection		Yes	No	NA
1	Splash suit			
2	Chemical protective clothing			
3	Safety glasses or goggles			
4	Gloves			
5	Overboots			
6	Hard hat			
7	Dust mask			
8	Hearing protection			
9	Respirator			
Instrumentation				
10	Combustible gas meter			
11	Oxygen meter			
12	Organic vapor analyzer			
Supplies				
13	Decontamination equipment and supplies			
14	Fire extinguishers			
15	Spill cleanup supplies			
Corrective Action Taken During Audit:				
Corrective Action Still Needed:				

Note: NA = Not applicable

Auditor’s Signature

Site Safety Coordinator’s Signature

Date

TETRA TECH, INC.

RESPIRATORY HAZARD ASSESSMENT

Project Name:

Project No.:

Location:

Project Manager:

Type: ☐ Baseline ☐ Reassessment

Date:

Valid for _____ days

Job/Task Description:

☐ Routine
☐ Escape

Hazard Identification and Source:

Workplace Factors:
Temperature: ___________
Humidity: _____________
Other: ________________

User Factors:
Work rate: ______________________
Protective clothing: _______________
Other: _________________________

Chemical:					
PEL:					
ACGIH TLV:					
Form (part/gas/vapor):					
IDLH:					
Eye Irritant (Y/N):					
Skin Absorption(Y/N):					
Monitoring (Y/N) :*					
Frequency:					
Maximum Concentration Estimated:**					

* Monitoring Method:

☐ PID
☐ FID
☐ Detector tube: ___________
☐ NIOSH method: ___________
☐ Vapor badge: ______________
☐ Other: ___________________

Respirator Type:

☐ Half-face disposable Brand: ______________________
☐ Half-face reusable Brand: ______________________
☐ Full-face Brand: ______________________
☐ Air-supplied airline Brand: ______________________
☐ Air-supplied SCBA Brand: ______________________
☐ PAPR Brand: ______________________
☐ ESCBA Brand: ______________________

** If concentrations exceed the immediately dangerous to life and health (IDLH) value, use air-supplied systems.

Vapor and Gas Cartridge Exchange:

ESLI: ☐ Yes ☐ No

Exchange frequency: _________________________________

Cartridge/Filter Selection

☐ N100
☐ N99
☐ N95
☐ Organic vapor
☐ Ammonia
☐ R100
☐ R99
☐ R95
☐ Acid gas
☐ Mercury
☐ P100
☐ P99
☐ P95
☐ Formaldehyde
☐ Combo: ____
☐ Other: ____

Basis for Exchange Frequency

☐ Manufacturer's data
☐ Experimental methods
☐ Predictive modeling
☐ Workplace simulations
☐ AIHA "Rules of Thumb"
☐ Analogous chemical structure
☐ OSHA Regulation: ________________________________
☐ Other: ____

____________________________________ ______________
Completed by Date

____________________________________ ______________
Reviewed by Date

DEFINITIONS AND ACRONYMS

ACGIH	American Conference of Governmental Industrial Hygienists
AIHA	American Industrial Hygiene Association
ESLI	End of service life indicator
FID	Flame ionization detector
IDLH	Immediately dangerous to life and health
NIOSH	National Institute for Occupational Safety and Health
N100/99/95	Non-oil-proof particulate filter
OSHA	Occupational Safety and Health Administration
P100/99/95	Oil-proof particulate filter
PEL	Permissible exposure limit
PID	Photoionization detector
PPE	Personal protective equipment
R100/99/95	Oil-resistant particulate filter
SCBA	Self-contained breathing apparatus
TLV	Threshold limit value

Note: This form must be reviewed by a regional health and safety representative or subsidiary health and safety representative (or designee) only and must be attached to the site-specific health and safety plan once completed. A copy must also be placed in the project files.