
SELECTING FIELD ANALYTICAL METHODS

A DECISION-TREE APPROACH

HEALTH AND ENVIRONMENTAL SCIENCES DEPARTMENT
PUBLICATION NUMBER 4670
AUGUST 1998



American Petroleum Institute

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A DECISION-TREE APPROACH

Health and Environmental Sciences Department

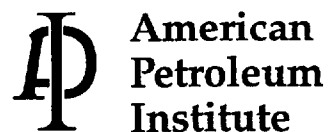
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ABSTRACT

A large number of portable instruments and techniques are available to perform on-site analyses of organic compounds in soil, groundwater, and soil gas samples at petroleum release sites. The appropriate selection and use of these methods can result in increased spatial site information in less time and with fewer phases of assessment than is typical using conventional sampling methods and off-site laboratories. There is some reluctance to using field analytical methods because of the lack of regulatory acceptance and the perception that field methods do not provide data of adequate quality for making decisions.

This report makes no recommendations, but presents a decision-tree approach for selecting and using field analytical methods. This approach will assist the user in the generation of higher-quality field analytical data by prompting selection of the appropriate method for the site's investigation goal. Quality assurance guidelines specific to the desired data quality level are also presented, to increase the credibility of the data by documenting method performance. The report also provides training suggestions and easy-to-use checklists for field quality control and formal documentation.

TABLE OF CONTENTS

EXECUTIVE SUMMARY	ES-1
1. INTRODUCTION	1-1
2. HOW TO USE THE DECISION TREE APPROACH	2-1
FLOW CHART STEP ONE: WHAT IS THE INVESTIGATION GOAL? ..	2-4
FLOW CHART STEP TWO: WHAT IS THE OPERATIONAL SCENARIO OF THE INVESTIGATION GOAL?	2-5
Operational Scenarios for Investigation Goal 1, Release Confirmation	2-5
Operational Scenarios for Investigation Goal 2, Contaminant Distribution Assessment	2-6
Operational Scenarios for Investigation Goal 3, Monitoring	2-7
Operational Scenarios for Investigation Goal 4, Closure	2-8
FLOW CHART STEP THREE: WHAT ARE THE TYPES OF PETROLEUM HYDROCARBONS BEING INVESTIGATED?	2-9
Product Types and Regulated Compounds	2-10
Gasoline	2-10
Diesel/Fuel Oil	2-10
Kerosene and Jet Fuel	2-11
FLOW CHART STEP FOUR: WHAT MEDIA WILL BE ANALYZED DURING THE INVESTIGATION?	2-13
FLOW CHART STEP FIVE: WHAT IS THE APPROPRIATE LEVEL OF DATA QUALITY PER INVESTIGATION GOAL?	2-14
Level of Data Quality 1	2-16
Level of Data Quality 2	2-17
Level of Data Quality 3	2-18
Level of Data Quality 4	2-18
FLOW CHART STEP SIX: WHAT ARE THE FIELD ANALYTICAL METHOD OPTIONS PER LEVEL OF DATA QUALITY?	2-19

	FLOW CHART STEP SEVEN: WHAT ARE THE QA/QC PROCEDURES PER SELECTED FIELD ANALYTICAL METHOD?	2-20
	OPTIONS INFORMATION BY DECISION TREE STEP	2-20
3.	ITERATIVE SAMPLING APPROACH	3-1
4.	TRAINING	4-1
	TRAINING SEQUENCE FOR TECHNICAL FIELD PERSONNEL	4-1
	Approach	4-1
5.	QUALITY ASSURANCE AND QUALITY CONTROL PROTOCOL	5-1
	ANALYTICAL PROCEDURES	5-1
	EPA-Approved Methods	5-1
	Non-Standard or Modified Methods	5-1
	DATA QUALITY VALIDATION	5-1
	Blanks	5-2
	Surrogate Standards	5-2
	Matrix Spikes and Matrix Spike Duplicates	5-3
	Duplicate Samples	5-3
	Precision	5-3
	Accuracy	5-4
	Method Detection Limit (MDL)	5-4
	CALIBRATION PROCEDURES	5-5
	Instrument Calibration	5-5
	Calibration Verification	5-6
	PROFICIENCY AND QUALITY CONTROL SAMPLES	5-6
	EPA Proficiency Evaluation Study	5-6
	Quality Control Standards	5-6
	DATA REDUCTION AND REPORTING	5-7
	Data Reduction	5-7
	Data Reporting	5-7
	REFERENCES	6-1

Appendix A

GLOSSARY OF TERMS	A-1
-------------------------	-----

Appendix B

DECISION TREE FLOW CHARTS	B-1
---------------------------------	-----

Appendix C

EXAMPLE DECISION TREE APPLICATIONS	C-1
------------------------------------------	-----

Appendix D

QUALITY ASSURANCE PACKAGES WITH LOG SHEETS	D-1
--------------------------------------------------	-----

LIST OF TABLES

Table 2-1	Commonly Regulated Compounds	2-11
Table 2-2	Gasoline Analytical Instruments	2-12
Table 2-3	Diesel and Fuel Oil Analytical Instruments	2-12
Table 2-4	Kerosene and Jet Fuel Analytical Instruments	2-12
Table 2-5	Levels of Data Quality	2-15
Table 2-6	Options Information	2-21
Table 4-1	Training Requirements	4-1
Table 5-1	Student's t Values for MDL Calculations	5-5

LIST OF FIGURES

Figure 3-1	Iterative Sampling Approach Schematic	3-2
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EXECUTIVE SUMMARY

Throughout the environmental remediation industry, there has been a drive to reconcile the time constraints and regimentation of "phased" investigations that occur in discrete steps, with a more fluid process of continuous sampling, analysis, and real-time decision making. This has been the case for petroleum product release sites, where a great deal is now known about the hydrocarbons involved and the specific indicator constituents of concern. This publication presents a different approach, through a *Decision Tree*, for the selection of appropriate field methods for the on-site testing of petroleum compounds in both soil and groundwater, which would be a key part of accelerated site characterizations (ASCs).

The manual addresses the primary constituents of petroleum fuels [benzene, toluene, ethylbenzene, and xylenes (BTEX); total petroleum hydrocarbons (TPH); polynuclear aromatic hydrocarbons (PAHs)]. The methods selected for inclusion in the flow charts are mature, off-the-shelf technologies that measure these constituents. There are other technologies more recently available for these constituents and for constituents not included (e.g., metals). However, alternative methods and other non-petroleum constituents can also be considered within the context of the *Decision Tree* approach.

A unique decision process flow chart (Section 2) is designed with "decision steps," to assist the site manager and regulatory remediation project managers with a tool to more efficiently manage the optimization of analytical data in the field, and to help determine the appropriate level of data quality (LDQ) needed for the job at hand. The analytical field method selection process is, therefore, job oriented and incorporates practical factors such as investigative goals, regulatory requirements, data quality needs, sampling media, and constituents of concern.

The *Decision Tree* charts are supported by quality control packages (Appendix D and Section 5). These packages contain 1) suggested quality controls in a checklist form, and 2) documentation

log sheets. This format is designed for ease-of-use in the field, and the forms can be attached to project reports.

An important part of the data optimization process, and a key advantage of on-site testing, is the opportunity for an iterative, or constant feedback approach, i.e., to repeat the sampling - evaluation process in the effort to get successively closer to desired results or goals. A version of an iterative sampling approach is illustrated in Section 3.

Finally, the minimum recommended training for personnel who will perform the testing in the field is described in Section 4.

Overall, the best approach to maximize the usefulness of the investigative data produced on site is to use this manual in combination with API's sister publication, *Compilation of Field Analytical Methods*, Publication No. 4635, and with other references noted in the Introduction of this manual. Additionally, the manufacturers are a worthwhile source of information on applications, background chemistry, and best practices for their products.

Section 1

INTRODUCTION

OBJECTIVE

This publication complements the API publication (December, 1996), *Compilation of Field Analytical Methods for Assessing Petroleum Product Releases*, in which operation, practical applications, and limitations of the most widely used field analytical methods are discussed. In this publication, a Decision Tree is developed to assist project or site managers with guidance for on-site investigations of suspected or confirmed hydrocarbon release sites, from an initial site assessment to site closure. The Decision Tree is complemented by method-specific quality assurance/quality control (QA/QC) protocols designed for the evaluation of quality, viability, and defensibility of field analytical data. Briefly, the objectives for this technology selection guidance document are:

1. To provide guidelines for field analytical technology selection and use on-site, through a Decision Tree or "Decision Flow" approach;
2. To assist in generating analytical data of known and consistent quality through method-specific QA/QC protocol packages;
3. To assure that the method sensitivity, accuracy, and precision meet the decision-making needs of the project or site manager, the client, and the regulatory agencies; and
4. To guide the regulated community in producing consistent and defensible field documentation and training for field personnel.

BACKGROUND

The use of field analytical methods for investigations of petroleum hydrocarbon release sites has gained broader attention in recent years as part of more cost-effective, single mobilization site assessments. This increasingly popular concept has been discussed as *Expedited Site Assessment* (ESA) by the U.S. Environmental Protection Agency's (EPA's) Office of Underground Storage Tanks (UST), as *Accelerated Site Characterization for Confirmed or Suspected Petroleum Releases* in the ASTM PS 3 guide, as *Expedited Site Characterization* by the Department of

Energy, or as *Expedited Site Closure Approach* (ESCA®) by Land Tech Remedial, Inc. All of these single mobilization site assessment concepts have in common the use of direct push technologies for rapid, minimally intrusive collection of soil and groundwater samples, and on-site data generation which permits the project or site manager to make crucial investigation decisions on-site.

In recent years technological developments and improvements of analytical instruments have led to a greater availability and affordability of "mobile" analytical equipment that can be used for on-site analysis. Particularly in the field of identification and quantification of petroleum hydrocarbons in environmental samples there is a wide range of field compatible tools at various degrees of sophistication (e.g., accuracy and precision) and analytical capabilities. Field analytical methods are currently reviewed and evaluated for use under the Resource Conservation and Recovery Act (RCRA), the Comprehensive Environmental Response Compensation and Liability Act (CERCLA), EPA's UST Office and Superfund Innovative Technologies Evaluation (SITE) Program, and by the Departments of Defense and Energy.

In spite of the apparent advancement of this technology, there still is some reluctance by the environmental community to use it to its full advantage. This conflict in attitudes arises partly from the lack of clear regulatory acceptance, and the perception that field-generated data may not be adequate for making regulatory or remedial decisions. A format for proper selection of field methods does not exist, nor does a framework for method selection that permits consistent validation and optimization of raw field data.

To help overcome reluctance, agencies and trade organizations have published technical information and guidance on the accelerated site characterization approach and the associated field technologies. Some of these publications are:

1. *EPA Expedited Site Assessment Guide* (March, 1997);
2. *New Jersey Guidelines for Field Analytical Technologies* (July, 1996);

3. *ASTM PS 3 Standard Guide for Accelerated Site Characterization* (January, 1996);
4. *ASTM E 1739 Guide for Risk-Based Corrective Action* (December, 1996); and
5. *API Compilation of Field Methods for Assessing Petroleum Product Releases* (December, 1996).

These publications provide technical information to characterize the available technologies in terms of their capabilities, advantages over non-field methods, and limitations, as well as provide general guidance on the accelerated site assessment approach.

This document is intended to fill information gaps by characterizing the decision-making process. The primary goal is to strengthen the users' and regulators' confidence in using field analytical data to make regulatory decisions. More specifically, the decision framework within this publication is designed to take the site or project manager through the sequence of questions that are critical to selecting field analytical methods appropriate for the particular monitoring objective at a petroleum release site. In that regard it is job oriented, using decision-making factors such as regulatory requirements, investigation goals, constituents of concern, and realistic data quality needs. Once the initial decisions are made, the document then provides information on optimizing data quality through unique field QC, and documenting those data through a rigorous documentation plan. To get the most out of this publication, it should be used in combination with the other publications mentioned above, and with equipment manufacturers' and engineering contractors' technical expertise on test procedures.

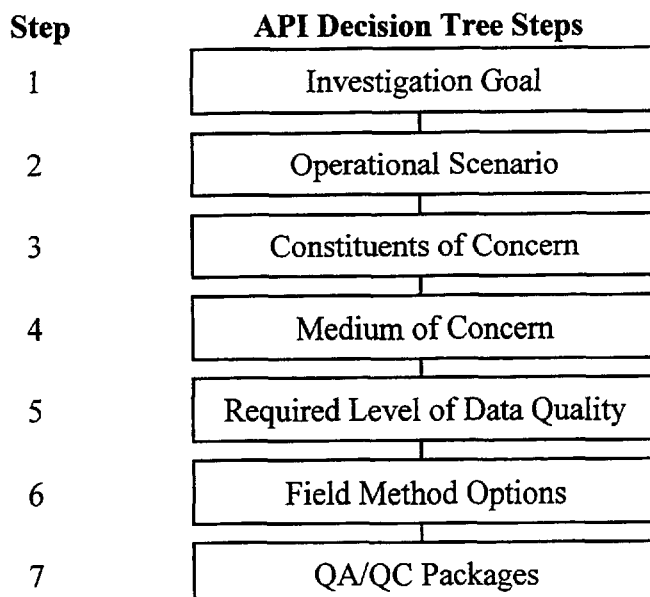
Section 2

HOW TO USE THE DECISION TREE APPROACH

This section is designed to guide the user through the Decision Tree flow charts (Appendix B), from the specification of investigation goals through the ultimate selection of a field analytical method or a group of methods to meet the goals specified. In this process, the user proceeds step-by-step through the following sequence of inquiries concerning the objectives of the monitoring effort:

- | | |
|--------|------------------------------------------------------------|
| Step 1 | What is the regulatory or investigative goal? |
| Step 2 | What remedial activities may be required? |
| Step 3 | What contaminants are present, or required to be measured? |
| Step 4 | What media are to be sampled? |
| Step 5 | What data quality is acceptable, or required? |
| Step 6 | What are the field test options? |
| Step 7 | What are the recommended QC procedures for that test? |

The flow chart dealing with the above decision-making questions is illustrated below. Refer to Appendix B (Decision Tree Flow Charts) for the detailed flow charts associated with each step.



Step 1 Investigation Goal:

This step is the broad-based plan for site evaluation and might correspond to the Executive Summary or Objectives section of a site's Project Plan. The investigation goal is usually driven by an agency program, as with a UST replacement, a Spill Act investigation, or ECRA/ISRA property transfer.

Step 2 Operational Scenario:

This step specifies the type of field work actually needed at the site. This might correspond to the Scope of Work section of a Project Plan, with activities ranging from tank pulls, to monitoring well installations, to comprehensive site assessments.

Step 3 Constituents of Concern:

The contaminants for analysis are identified, based on the history of the site and agency requirements. For petroleum release sites, they may be individual target constituents, such as benzene or BTEX; and/or general product identifications, such as gasoline-range organics (GRO), diesel-range organics (DRO), etc.

Step 4 Medium of Concern:

The impacted media are air, water, and soil. Field tests (especially test kits) are highly specialized, with variable performance capabilities by environmental medium.

Step 5 Level of Data Quality:

This step requires knowledge of how the data are to be reported and used. Key issues are the requirements for qualitative or quantitative data, the reporting limit desired [percent, parts per million (ppm), parts per billion (ppb)], and the types of constituents being reported (e.g., benzene vs. gasoline hydrocarbons).

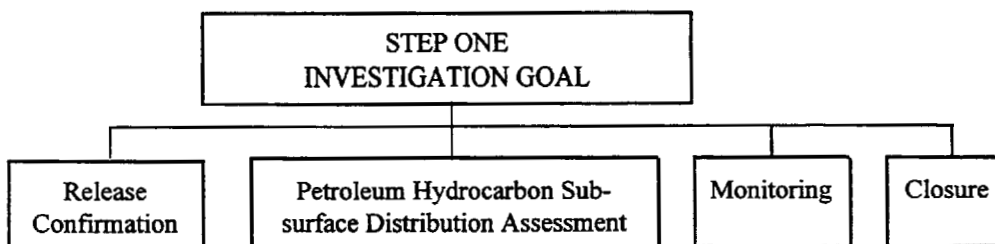
Step 6 Field Method Options:

The final test options are listed at this step. Flexibility is important, and one method, or several in combination, may be used to achieve the best results for each medium being investigated. One may also use this decision step to refine the field needs, gather more specific information from vendors, and review agency requirements.

Step 7 QA/QC Packages:

There are four packages, each related to the type of method employed. The QC packages contain 1) a QC protocol list containing the suggested minimum calibration and QC sampling in the field; and 2) a field log containing spaces for basic documentary evidence, a calibration schedule, and a QC sample data log section. These are in checklist form for easy use in the field and attachment to project reports. They may need expansion for some types of activities at the site.

FLOW CHART STEP ONE: WHAT IS THE INVESTIGATION GOAL?



There are four options within Step One, each of which is a broad-based investigation goal frequently encountered during the investigation and management of confirmed or suspected petroleum hydrocarbon releases. The four options are:

1. **Release Confirmation**: The investigation of site media (generally soil and/or groundwater) to confirm that the subsurface has been impacted by the release of petroleum hydrocarbons from a primary source (i.e., a container used for the storage of petroleum hydrocarbons or its associated piping, pumps, or other appurtenances). The level of investigation is limited to the confirmation of such a release, and not its geometrical extent or magnitude.
2. **Petroleum Hydrocarbon Subsurface Distribution Assessment**: The investigation of site media (generally soil and/or groundwater) to determine the geometrical extent and magnitude of a petroleum hydrocarbon release.
3. **Monitoring**: Site analytical activities associated with one-time or routine periodic measurement of petroleum hydrocarbon concentrations within site media (generally water or air, but occasionally soil) to evaluate a specific process (remediation progress, compliance with discharge permits, etc.).
4. **Closure**: Site analytical activities associated with the measurement of petroleum hydrocarbon concentrations within site media (generally soil and/or groundwater) to document that environmental impact at the site does not warrant further action.

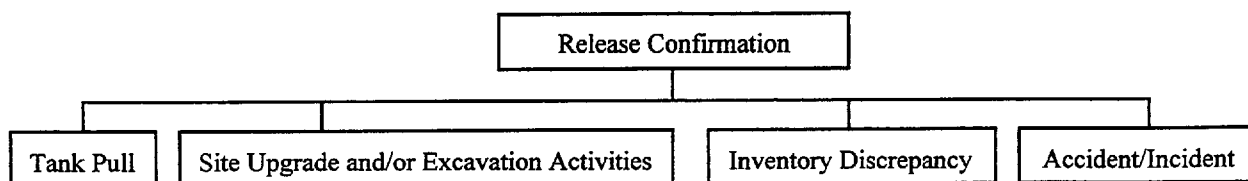
Generally, most site activities requiring field analyses of petroleum hydrocarbons can be classified as one of these four categories. Once the user has broadly categorized the subject investigation in Step One, the user narrows the scope of the subject investigation by specifying further detail in Step Two of the Decision Tree.

FLOW CHART STEP TWO: WHAT IS THE OPERATIONAL SCENARIO OF THE INVESTIGATION GOAL?



Within Step Two, the broad investigation goal identified in Step One is narrowed to more specifically determine the scope of the subject investigation. Each of the broad categories within Step One is broken down into two to four more detailed investigation sub-categories, called "Operational Scenarios." The user is required to match the details of the subject investigation with one of the Operational Scenarios listed below. While each investigation will be unique, and any specific investigation may not perfectly coincide with the listed Operational Scenarios, the user should attempt a "best fit" between the subject investigation and one of the Operational Scenarios, considering probable data quality requirements needed to meet the investigation goals.

Operational Scenarios for Investigation Goal 1, Release Confirmation

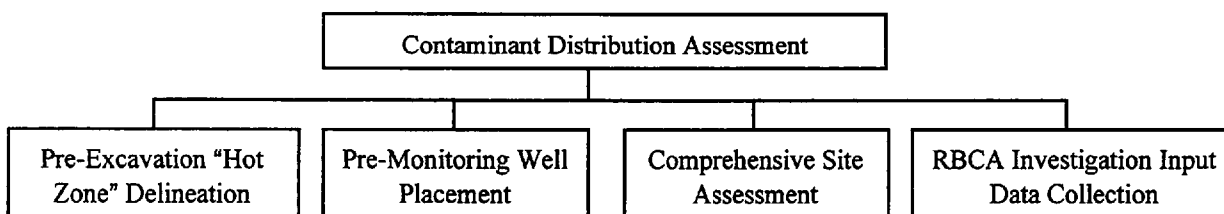


1. **Tank Pull:** Organoleptic analyses suggest petroleum hydrocarbon impact to soils and/or groundwater surrounding the UST, or below a UST in the excavated area. The goal of the investigation is to confirm the organoleptic analyses using field analytical techniques. Constituent-specific analyses generally will not be required.
2. **Site Upgrade and/or Excavation Activities:** During the excavation and/or upgrade, organoleptic analyses suggest petroleum hydrocarbon impact to soils and/or groundwater surrounding affected site features (footings, pumps, utility trenches, etc.). The goal of the investigation is to confirm the organoleptic analyses using field analytical techniques. Constituent-specific analyses generally will not be required.
3. **Inventory Discrepancy:** During the routine operation of a site used for petroleum hydrocarbon distribution, (e.g., petroleum retail service stations or bulk storage facilities), a

discrepancy between product volume delivered and product volume sold is noticed. The goal of the investigation is to determine whether the inventory discrepancy is due to a release of petroleum hydrocarbons to the subsurface. Constituent-specific analyses generally will not be required.

4. Accident/Incident: During the routine operation of a site used for petroleum hydrocarbon distribution, an accident or incident occurs (e.g., a vehicle collides with a petroleum dispenser). The goal of the investigation is to determine whether the accident/incident resulted in the release of hydrocarbons to the subsurface.

Operational Scenarios for Investigation Goal 2, Contaminant Distribution Assessment

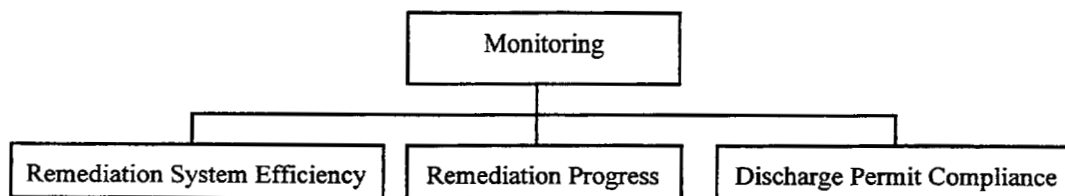


1. Pre-Excavation "Hot Zone" Delineation: A petroleum hydrocarbon release to subsurface soils has been confirmed, and the petroleum hydrocarbon impacted soils are to be excavated for *ex situ* treatment and/or disposal. In this case, the investigation goal is impact determination (hot/not hot) to ensure that all impacted soils are successfully excavated, and that unnecessary disposal is avoided. Constituent-specific data generally will not be required.
2. Pre-Monitoring Well Placement: A petroleum hydrocarbon release to site groundwater has been confirmed, and monitoring wells are to be installed for subsequent constituent-specific groundwater quality analyses. In this case, the investigation goal is impact determination (hot/not hot) to ensure that monitoring wells are installed in locations which will allow efficient monitoring. Constituent-specific data generally will not be required.
3. Comprehensive Site Assessment: A petroleum hydrocarbon release to subsurface soils and/or groundwater has been confirmed or is suspected, and the nature and extent of the subsurface impact is to be determined. The potential goals of a comprehensive site assessment include, but are not limited to:
 - The evaluation of site conditions for the efficient application of a range of remediation technologies.
 - Baseline evaluation of site conditions for property transfer purposes.
 - The evaluation of site conditions within a risk-based corrective action (RBCA) framework (see RBCA Investigation Input Data Collection, below).

In general these considerations as well as the individual physical characteristics (mobility, volatility, solubility, etc.) and health risks (carcinogenic or non-carcinogenic) associated with different petroleum hydrocarbon compounds, dictate the use of constituent-specific analytical methods.

4. **RBCA Investigation Input Data Collection:** This Operational Scenario involves the investigation of site media (generally soil and/or groundwater) specifically for the purpose of evaluating the site within an RBCA framework. The goal of the RBCA evaluation is the identification of exposure pathways. Therefore, high-level, constituent-specific data quality analyses will be focused on exposure pathway evaluations ("decision points") as opposed to broader, more arbitrary sampling strategies at sites where all site media are required to meet numerical maximum containment levels (MCLs).

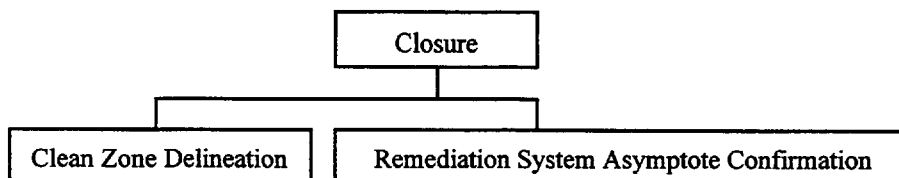
Operational Scenarios for Investigation Goal 3, Monitoring



1. **Remediation System Efficiency:** The periodic measurement of petroleum hydrocarbon concentrations in remediation system influent and effluent streams in order to assess treatment performance and the rate of removal of petroleum hydrocarbons from the subsurface as a function of time. These measurements generally are made to determine either the total removed mass of one or more petroleum hydrocarbon constituents, or the rate of TPH removed relative to previous monitoring events. In the case of the former, constituent-specific concentration data are required.
2. **Discharge Permit Compliance:** The regular, periodic determination of whether remediation system effluent streams, stormwater runoff, etc., are meeting regulatory discharge limits as specified in county, state or federally issued discharge permits. In these cases, relatively high levels of constituent-specific analytical data quality are required, since inaccurate discharge concentration data, and therefore permit non-compliance, may result in fines and other penalties.
3. **Remediation Progress:** The measurement of petroleum hydrocarbon concentrations in site media (usually groundwater) in order to show that petroleum hydrocarbon impact to the site is diminishing as a function of time. This can be accomplished through periodic measurement of petroleum hydrocarbon concentrations in site media during the remediation phase. Since the remediation of different petroleum hydrocarbon constituents proceed at different rates, these data should be constituent-specific. Additionally, since the progress of

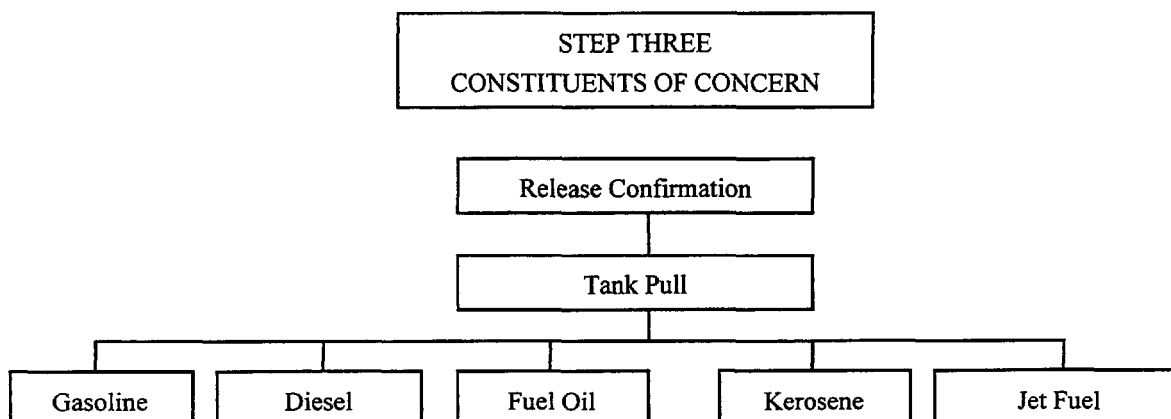
remediation at a site often will be subject to regulatory scrutiny, a percentage of the collected data should be of sufficiently high quality that trends observed in the monitoring data can be relied upon.

Operational Scenarios for Investigation Goal 4, Closure



1. Clean Zone Delineation: Following remediation activities, site media samples (generally soil and groundwater) are analyzed to determine if petroleum hydrocarbon concentrations have been reduced to meet maximum containment levels (MCLs), risk-based screening levels (RBSLs), or site-specific target levels (SSTLs). These data are necessarily constituent-specific, and of high quality due to the regulatory scrutiny of the analytical results, and the potential liabilities involved with inaccurate analyses.
2. Remediation System Asymptote Confirmation: Site closure is predicated upon a performance-based goal for a remediation system, and documentation of asymptotic petroleum hydrocarbon removal rates is required. Many regulatory agencies will require two or more monitoring rounds to obtain high-quality analytical data for asymptote confirmation. These data are necessarily constituent-specific, and of high quality due to the regulatory scrutiny of the analytical results, and the potential liabilities involved with inaccurate analyses.

FLOW CHART STEP THREE: WHAT ARE THE TYPES OF PETROLEUM HYDROCARBONS BEING INVESTIGATED?



Investigations and management of subsurface impact can involve the evaluation of many different types of petroleum hydrocarbons. Step Three allows the selection of the most common types of petroleum hydrocarbons:

1. Gasoline
2. Diesel
3. Fuel Oil
4. Kerosene
5. Jet Fuel.

If the petroleum hydrocarbon type is unknown, contaminant fingerprinting may become necessary before the investigation can proceed. While there are additional petroleum hydrocarbon types which may need to be evaluated during any specific investigation, the Decision Tree is limited to the most commonly encountered petroleum hydrocarbon types. Additional petroleum hydrocarbon types (e.g., waste oils) are beyond the scope of this manual.

The selection of more than one petroleum hydrocarbon type is also possible. At Step Three, if there is more than one type of petroleum hydrocarbon selected, the user potentially will be following two separate "branches" of the Decision Tree in parallel, possibly resulting in the selection of an individual field analytical method for each type of petroleum hydrocarbon selected, once the bottom of the Decision Tree is reached. Alternatively, within some Operational Scenarios, depending on the petroleum hydrocarbon types selected, two or more different types of

petroleum hydrocarbons can be analyzed by the same field analytical method, and the user may proceed along a single "branch" of the Decision Tree.

Product Types and Regulated Compounds

Although in the majority of spill scenarios a point source hydrocarbon product release represents the origin of contaminated soil or groundwater, it is the water-soluble hydrocarbon fraction transported in groundwater that poses the greatest risk to the environment and human health. Of the water-soluble fraction, those compounds with the highest confirmed or suspected toxicity require the highest degree of attention and more precise and sophisticated analytical tools. Standards for these compounds are in the ppb to sub-ppb range. In Table 2-1 commonly regulated constituents of several product types are summarized.

Gasoline

Chemically, gasoline is predominantly a mixture of hydrocarbons containing 4 to 10 carbon atoms. Gasoline has a boiling point range of 40° to 180°C. Analytically, the total gasoline range concentration of an environmental sample is most commonly determined by its content of hydrocarbons from C₆ to C₁₀ (GRO methods—Gasoline-Range Organics) inclusive. Constituents of concern in gasoline include benzene, toluene, ethylbenzene and xylenes, commonly reported as BTEX, and methyl-tertiary butyl ether (MTBE). Several field analytical instruments can be used to detect and quantify gasoline-derived hydrocarbons in environmental samples as summarized in Table 2-2.

Diesel/Fuel Oil

Chemically, the average composition of commercial diesel or No. 2 fuel oil consists of a mixture of petroleum hydrocarbons having 13 to 25 carbon atoms. Diesel fuel has a boiling point range of 220° to 350°C. Analytically, the total diesel-range organics (DRO) concentration of an environmental sample is most commonly determined by its content of hydrocarbons from C₁₀ to C₂₈, inclusive. Constituents of concern of diesel and fuel oil origin include naphthalene and lower molecular weight polynuclear aromatic hydrocarbons (PAHs). Several field analytical

instruments can be used to detect and quantify diesel- or fuel oil-derived hydrocarbons in environmental samples as summarized in Table 2-3.

Table 2-1. Commonly regulated compounds.

Chemical	Gasoline	Diesel	Jet Fuel	Kerosene	Fuel Oil	Used Oil
Volatiles						
Benzene	x	x	x	x	x	x
1,2-Dibromoethane	x					x
Ethylbenzene	x	x	x	x	x	x
MTBE	x					x
Toluene	x	x	x	x	x	x
1,2,4-Trimethylbenzene	x	x	x	x	x	x
m-Xylene	x	x	x	x	x	x
o-Xylene	x	x	x	x	x	x
p-Xylene	x	x	x	x	x	x
Xylenes (mixed)	x	x	x	x	x	x
PAHs						
Acenaphthylene						x
Anthracene						x
Benz(a)anthracene						x
Benzo(a)pyrene						x
Chrysene						x
Dibenz(a,h)anthracene						x
Fluorene	x			x	x	x
Naphthalene	x	x	x	x	x	x
Pyrene		x		x	x	x

Kerosene and Jet Fuel

Kerosene and jet fuel are petroleum hydrocarbon mixtures with a boiling point range of 160° to 250°C, slightly lower than diesel and fuel oil. Analytically, the total medium hydrocarbon range concentration of an environmental sample is most commonly determined by its content of hydrocarbons from C₁₀ to C₂₅, inclusive. Constituents of concern in kerosene include benzene, toluene, ethylbenzene and xylenes (BTEX), naphthalene, and other PAHs. Several field analytical instruments can be used to detect and quantify kerosene- and jet fuel-derived hydrocarbons in environmental samples as summarized in Table 2-4.

Table 2-2. Gasoline analytical instruments.

Analytical Instrument	Detected Gasoline Constituents	
	Range or Compound Groups	Constituent-Specific
TOV Detectors: PID/FID	Volatile organic vapors	
Immunoassay	Total BTEX, TPH	¹ Benzene
Infra-Red/Turbidimetric	TPH	
GC with PID and/or FID	TPH, gasoline-range organics	BTEX, MTBE

¹ Please consult manufacturers of immunoassay test kits for availability of other constituent-specific tests.

Table 2-3. Diesel and fuel oil analytical instruments.

Analytical Instrument	Detected Diesel or Fuel Oil Constituents	
	Range or Compound Groups	Constituent-Specific
TOV Detectors: PID/FID	¹ Volatile organic vapors	
Immunoassay	TPH, PAHs	
Infra-Red/Turbidimetric	TPH	
GC with PID and/or FID	TPH, diesel-range organics	BTEX, PAH

¹ It is important to note that even "fresh" diesel or fuel oil is primarily composed of semi-volatile organics, and only a small fraction of volatile organic compounds (VOCs). The presence of old and/or weathered diesel or fuel oil may not be detected using a total organic vapor (TOV) detector.

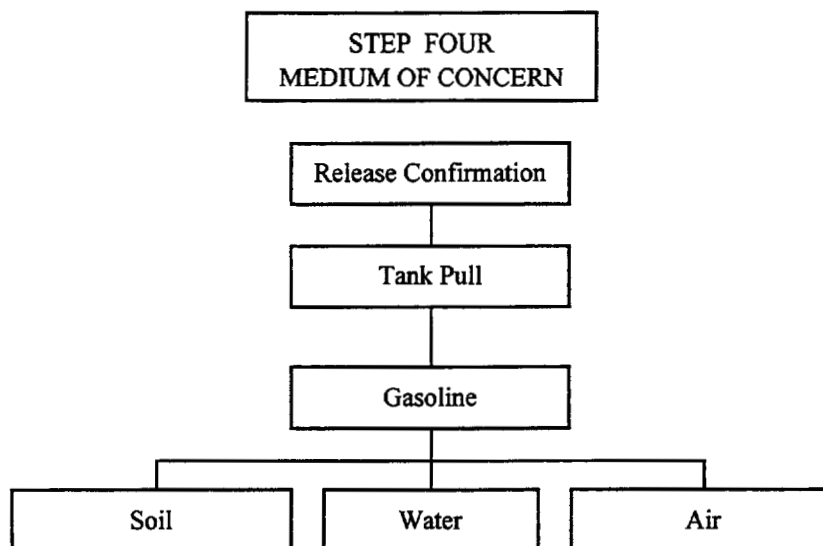
Table 2-4. Kerosene and jet fuel analytical instruments.

Analytical Instrument	Detected Kerosene or Jet Fuel Constituents	
	Range or Compound Groups	Constituent-Specific
TOV Detectors: PID/FID	¹ Volatile organic vapors	
Immunoassay	Total BTEX, TPH, PAH	² Benzene
Infra-Red/Turbidimetric	TPH	
GC with PID and/or FID	TPH, medium range organics	BTEX, PAH

¹ It is important to note that kerosene and jet fuel contain more semi-volatile than volatile organic compounds. The presence of old and/or weathered kerosene may not be detected using a TOV detector.

² Please consult manufacturers of immunoassay test kits for availability of other constituent-specific tests.

FLOW CHART STEP FOUR: WHAT MEDIA WILL BE ANALYZED DURING THE INVESTIGATION?

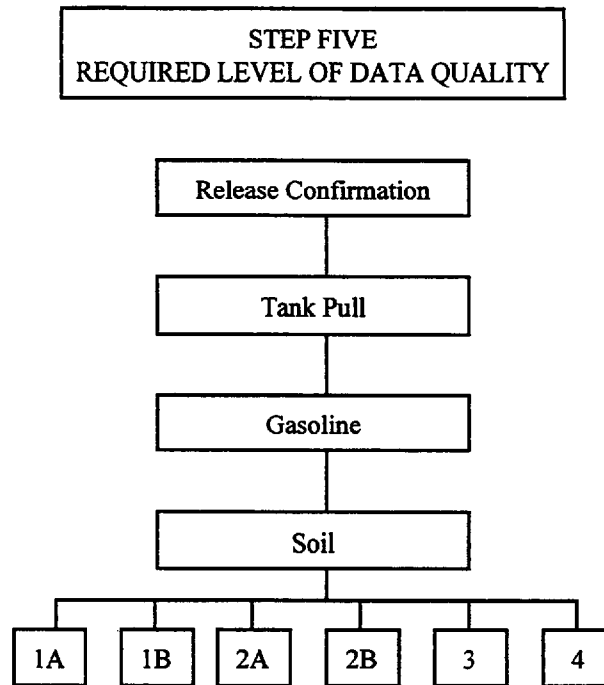


The majority of the field analytical methods described within the Decision Tree have their greatest applicability to a limited number of media. Subsurface impacts to soils, groundwater, or both, may be monitored. In some scenarios, air may also be monitored. Accordingly, Step Four allows the selection of the following media:

1. Soil
2. Water (e.g., groundwater, effluent waste streams)
3. Air.

The selection of more than one medium is possible. Depending on the selected Operational Scenario, this may result in the user proceeding along separate, parallel "branches" of the Decision Tree, leading to the selection of more than one field analytical method.

FLOW CHART STEP FIVE: WHAT IS THE APPROPRIATE LEVEL OF DATA QUALITY PER INVESTIGATION GOAL?



By this point in the Decision Tree process, the user has determined within Flow Chart Step Two whether he/she will be selecting constituent-specific, or non-constituent specific field analytical methods. However, within these broad classes of field analytical methods there exists a wide variety of specific methodologies, with varying degrees of precision and accuracy (data quality). In Step Five, the broad classes of constituent-specific and non-constituent specific analytical methods are further refined into four categories, called Levels of Data Quality (LDQs).

LDQ is defined as the degree of sophistication of analytical data. LDQ components are:

1. The method of analysis;
2. The selected analytical instrument; and
3. The QA/QC protocol employed to validate the desired data quality.

In any given LDQ, field analytical methods and instruments are designed for similar field applications and possess comparable analytical capabilities. Factors determining the quality of analytical data for a given LDQ include the method design, the intrinsic instrument capabilities

and limitations, and the recommended QA/QC protocol. Field analytical data at any given LDQ must represent a known, reproducible, and documented data quality that can be used for on-site decision-making during assessments of petroleum hydrocarbon releases.

To facilitate the use of LDQ terminology major levels are assigned a LDQ number— LDQ 1, 2, 3 and 4—where a higher number represents a more sophisticated means of analysis. Subdivisions within a group of methods are distinguished using letters A and B. Although slightly modified, the definition of LDQ used in this document is based on the previous definition of Data Quality Levels in the *Field Analysis Manual* of the New Jersey Department of Environmental Protection (NJDEP, 7/1994). The LDQs are summarized in Table 2-5, and discussed more thoroughly below.

Table 2-5. Levels of data quality.

Level of Data Quality	Description	Sensitivity
1A	Indirect analysis of organic vapors to determine the presence/absence of petroleum hydrocarbon compounds	Percent or parts per million
1B	Semi-quantitative analysis, accurate to within an order of magnitude	Parts per million
2A	Quantitative analysis of hydrocarbon groups or ranges	Parts per million to parts per billion
2B	Quantitative analysis of specific petroleum hydrocarbon constituents	Parts per billion
3	Quantitative analysis of specific petroleum hydrocarbon constituents by modified methods with EPA QA/QC documentation	Parts per billion
4	Quantitative analysis of specific petroleum hydrocarbon constituents utilizing EPA SW-846 methods.	Parts per billion

The selection of field analytical methods used to generate data of a given LDQ should be based on the analytical capabilities of methods. API's *Compilation of Field Analytical Methods for Assessing Petroleum Product Releases* (API, 1996) presents a compilation of the most widely used field analytical methods for assessing petroleum product releases including total organic vapor analyzers, field gas chromatographs, immunoassays, and infrared analyzers. Practical applications and limitations of each method are discussed along with a "job-" or objective-oriented Data Quality Classification Scheme to assist in selecting the appropriate method for the

task. The compilation also lists other field analytical techniques that are not as widely used but show promise for future application.

As the LDQ increases, the field analytical methods assigned to these levels provide data with increased precision and accuracy. Furthermore, as the LDQ increases, the field analytical method chosen will generally increase in sophistication and decrease in ease of use. Field analytical methods in LDQ 3 will require more time for analyses, generally be more cost-intensive, and may require additional operator training than field analytical methods in LDQ 1. The recommended percentage of analyses at a particular LDQ is dictated by Investigation Goal.

The specified LDQs within the Decision Tree have been selected to provide the *minimum* data quality sufficient to meet the requirements of a selected Investigation Goal. The user has the option to change the specified LDQ, resulting in increased accuracy and precision. For example, a change may be made to provide increased data quality due to site-specific regulatory requirements. However, substituting lower LDQs may result in data which are inadequate to fulfill the Investigation Goals. At best, such a downward substitution will result in lower quality site investigations and at worst may result in regulatory penalties. In scenarios which include provisions for field analytical methods from more than one LDQ, the user may choose to run all collected samples at the highest specified LDQ; however it is not recommended to analyze less than the suggested minimum at the highest specified LDQ. For example, if a given scenario suggests 20 percent of the samples to be run at LDQ 3 and 80 percent at LDQ 1A, it is acceptable to run all samples at LDQ 3, yet not recommended to run more than 80% of the samples at LDQ 1A.

Level of Data Quality 1

LDQ 1A requires field analytical methods with which the presence or absence of petroleum hydrocarbons can be determined qualitatively (percent or ppm level) at moderate to high levels of contamination (hot/not hot). The qualitative data represent an indirect measurement of total organic volatile vapors that originated from hydrocarbon contaminated soil or groundwater.

Methods that are used to qualitatively analyze for hydrocarbons require **QA/QC Protocol 1** (See Flow Chart Step 7 below, and Appendix D).

LDQ 1B requires methods with which moderate to high levels of petroleum hydrocarbon contamination can be measured semi quantitatively. Semi quantitative data provide an order-of-magnitude estimate of contamination at the ppm level. Methods that provide semi quantitative data determine the concentration of total organic volatile vapors originating from contaminated soil or groundwater, or ranges of petroleum hydrocarbons (TPH) in soil and groundwater.

Methods that are used to semi quantitatively screen for hydrocarbons require **QA/QC Protocol 1**.

Level of Data Quality 2

LDQ 2 is subdivided into 2A and 2B based on the capability of a field analytical method and instrument to analyze for hydrocarbon (TPH) ranges (2A), or give constituent-specific results (2B). Methods at this LDQ measure hydrocarbons from the lower ppb level to the lower ppm level.

LDQ 2A requires methods that can be used to analyze hydrocarbon ranges (TPH or total BTEX) directly in soil or groundwater. Compared to methods and instruments that may also be used at LDQ 1B, **QA/QC Protocol 2** for LDQ 2A is more sophisticated.

LDQ 2B requires the use of a portable, low precision Gas Chromatograph (GC) or a transportable lab-grade, high precision GC for *constituent-specific* analysis of hydrocarbons. Methods at this LDQ are supposed to measure individual hydrocarbons reliably from the lower ppb level (i.e., at the regulatory level) to the lower ppm level. **QA/QC Protocol 3** at this LDQ must permit the identification and evaluation of contaminant concentration levels at the ppb level within known analytical uncertainties.

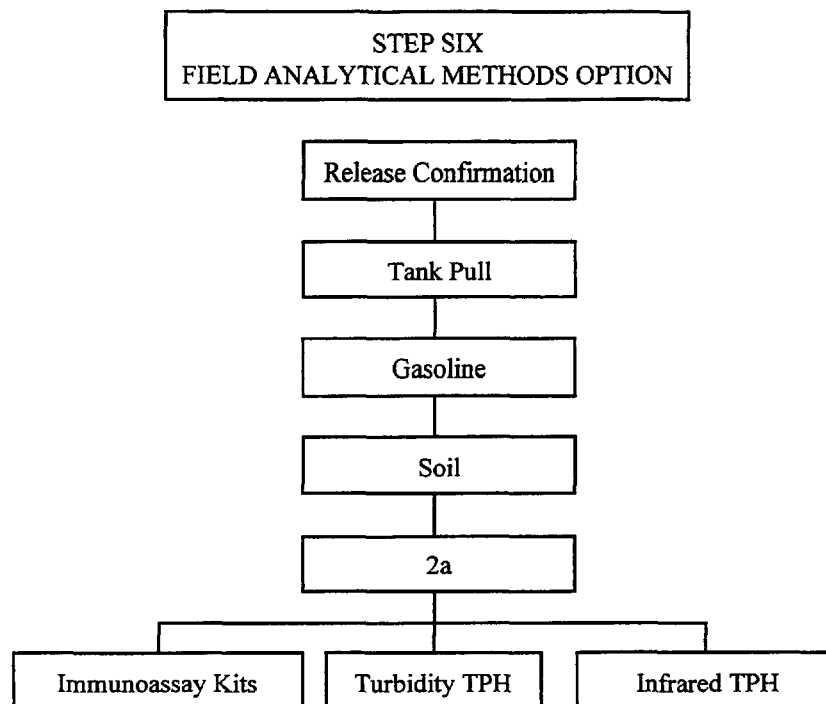
Level of Data Quality 3

LDQ 3 requires the use of a laboratory-grade, high-precision GC and a rigorous **QA/QC Protocol 4** to generate defensible analytical data at or below regulatory drinking water standards. Data at this LDQ can be generated using methods and instruments that deliver precision and accuracy equivalent to EPA SW-846 methods 602/8020 [e.g., modified EPA Method 3810 (headspace)].

Level of Data Quality 4

LDQ 4 requires the use of a laboratory-grade, high-precision GC and a **QA/QC Protocol 5** to generate defensible analytical data at or below regulatory drinking water standards. Data at this LDQ can be generated only using EPA SW-846 methods by approved analysts of state-certified mobile laboratories.

FLOW CHART STEP SIX: WHAT ARE THE FIELD ANALYTICAL METHOD OPTIONS PER LEVEL OF DATA QUALITY?



Each of the LDQs specified by the Decision Tree in Flow Chart Step Five contain one or more field analytical methods. Within Flow Chart Step Six, all of the field analytical methods covered by the Decision Tree are listed. The selection of field analytical methods to generate data appropriate for a given LDQ is based on the analytical capabilities of methods discussed in the *Compilation of Field Analytical Methods for Assessing Petroleum Product Releases* (API, 1996). This report presents a compilation of the most widely used field analytical methods for assessing petroleum product releases, including:

- Total organic vapor analyzers
- Immunoassay kits
- Infrared analyzers
- Field gas chromatographs
- Turbidimetric THP kits.

The Compilation presents the practical applications and limitations of each method, along with a “job-” or objective-oriented Data Quality Classification Scheme to assist in the selection of appropriate methods for any given task. Within Flow Chart Step Six, field analytical methods are applied to the specified LDQ following the general selection criteria developed in the API Compilation report (API, 1996).

FLOW CHART STEP SEVEN: WHAT ARE THE QUALITY ASSURANCE PROCEDURES PER SELECTED FIELD ANALYTICAL METHOD?

STEP SEVEN QUALITY ASSURANCE PROCEDURES

Flow Chart Step Seven provides the user with specific quality assurance procedures for each of the field analytical methods selected in Flow Chart Step Six. The quality assurance procedures are method-specific, and are consistent with the tiered LDQs. By following the provided quality assurance procedures, the user can be assured that the data generated by the selected field analytical method will be of sufficient quality to satisfy the requirements of the initially selected Investigation Goal. Furthermore, adherence to the quality assurance procedures will permit the results of the investigation to bear scrutiny regarding the quality, viability, and defensibility of field analytical data.

Individual quality assurance procedure packages, per field analytical method within a specific LDQ, are provided in Appendix D.

OPTIONS INFORMATION BY DECISION TREE STEP

As discussed in this section, each Step of the Decision Tree Flow Chart requires the user to process existing site information in such a way that results in the selection of one or more “branches” of the Decision Tree, which in turn, ultimately specifies field analytical methodology and data quality. Table 2-6 summarizes some of the types of site information which commonly will drive the selection of individual “branches” within the Decision Tree, and which should be evaluated prior to initiating any given investigation in order to make efficient use of the Decision Tree framework.

More complete documentation of the rationale behind the selection of any given “branch” within the Decision Tree is given in the examples provided in Appendix C.

Table 2-6. Options information.

Flow Chart Step	Site Information/Branch Selection Criteria
Step One: Investigation Goal → Release confirmation → Petroleum hydrocarbon subsurface distribution assessment → Monitoring → Closure	<ul style="list-style-type: none"> • Site environmental "phase" <ul style="list-style-type: none"> * New site (recently reported or suspected, previously undocumented accident/incident, hydrocarbon impact or spill) <ul style="list-style-type: none"> ◇ impact documentation ◇ impact characterization * Existing site <ul style="list-style-type: none"> ◇ previously documented hydrocarbon impact needing further characterization ◇ support of on-going site remediation activities (media and/or process monitoring) ◇ documentation of remedial goal attainment following site remediation activities (closure analyses) • Regulatory requirements <ul style="list-style-type: none"> * Mandated level/detail of investigation * Mandated remedial goals * Data requirements within risk-based corrective action program
Step Two: Operational Scenarios	<ul style="list-style-type: none"> • Site Environmental Activities <ul style="list-style-type: none"> * Release Confirmation Scenarios <ul style="list-style-type: none"> ◇ excavation ◇ site/facility upgrade ◇ tank pull ◇ incident/accident evaluation * Hydrocarbon distribution assessment objectives <ul style="list-style-type: none"> ◇ targeting areas for excavation ◇ evaluating remediation options and requirements ◇ data collection for risk-based corrective action program * Monitoring objectives <ul style="list-style-type: none"> ◇ evaluation of remediation system efficiency ◇ documenting discharge permit compliance ◇ evaluation of remediation system progress * Closure documentation requirements <ul style="list-style-type: none"> ◇ attainment of performance-based remedial goals ◇ attainment of numerical remediation goals ◇ site media clean-zone documentation
Step Three: Petroleum Types/Constituents of Concern	<ul style="list-style-type: none"> • Gasoline • Diesel • Fuel Oil • Kerosene • Jet Fuel
Step Four: Media Being Analyzed	<ul style="list-style-type: none"> • Water • Air • Soil • Combination of two or more of the above

(continued)

Table 2-6 (continued)

Flow Chart Step	Site Information/Branch Selection Criteria
<p>Step Five: Level of Data Quality</p> <ul style="list-style-type: none"> → 1A (qualitative, hot/not hot) → 1B (semi quantitative, order of magnitude) → 2A (quantitative, non-constituent specific) → 2B (quantitative, constituent-specific) → 3 (quantitative, constituent-specific, EPA QA/QC) → 4 (quantitative, constituent-specific, EPA SW-846 methods) 	<ul style="list-style-type: none"> • Overriding regulatory requirements <ul style="list-style-type: none"> * Acceptance of field methods * Required analysis of specific constituents of concern * Detection limits/regulatory clean-up levels * Certification requirements * Confirmatory (fixed-lab) analyses • Data requirements <ul style="list-style-type: none"> * Constituent-specific * Non-constituent specific * Expected concentration ranges <ul style="list-style-type: none"> ◇ detection limits ◇ closure criteria/regulatory cleanup levels ◇ risk/health-based levels • Cost
<p>Step Six: Field Analytical Method</p> <ul style="list-style-type: none"> → Total Organic Vapor Analyzers → Immunoassay Kits → Infrared/Turbidimetric Analyzers → Field Gas Chromatographs 	<ul style="list-style-type: none"> • Practicality/analytical efficiency <ul style="list-style-type: none"> * Portability/power requirements * Ease of use/operator training requirements * Analytical timeframes/turnaround times * Availability * Cost per analysis * Capital costs
<p>Step Seven: Quality Assurance Procedures</p>	<ul style="list-style-type: none"> • Selected field analytical method • Required level of data quality

Section 3

ITERATIVE SAMPLING APPROACH

The objective of the iterative sampling approach is to guide the team of project/site manager and field analyst through an accelerated site assessment. By following the flow chart segments in Figure 3-1, the use of appropriate field methods will be optimized through careful scrutiny of instrument performance and field analytical data, using the recommended quality assurance (QA) packages (Appendix D) selected from the flow charts in the Decision Tree (Appendix B).

1. Prior to the field mobilization, a sampling and analysis plan (SAP) with the selection of the target compounds of concern and matrix should be generated based on site history, characteristics, and the chosen Investigation Goal.
2. Using the flow chart for the chosen Investigation Goal in the Decision Tree (Appendix B), the appropriate field analytical method(s) and the recommended QA packages (Appendix D) can be selected.
3. Upon arrival at the investigation site and prior to sample analysis, the performance of the field analytical instrument(s) should be tested through blank(s) and standards analyses. If repeated calibration attempts do not result in satisfactory instrument performance, it should be repaired or replaced.
4. Any receipt of unknown environmental or QA samples must be appropriately recorded by following the requirements detailed in the QA packages (Appendix D).
5. Analysis of any environmental samples must follow EPA guidelines if an SW-846 method is used, or follow standard operating procedures (SOPs) detailed in a SOP manual if modified methods are used. All field analyses must be conducted by an experienced and qualified operator (see Section 4, Training).
6. The review of analytical data entails determining whether 1) the generated data lie within the capabilities and performance criteria of the analytical instrument used for analysis (e.g., calibration range); and 2) all QA sampling results are within acceptable limits (e.g., surrogate recovery). Data are considered acceptable only when all required elements of the respective QA protocol are within their recommended limits.

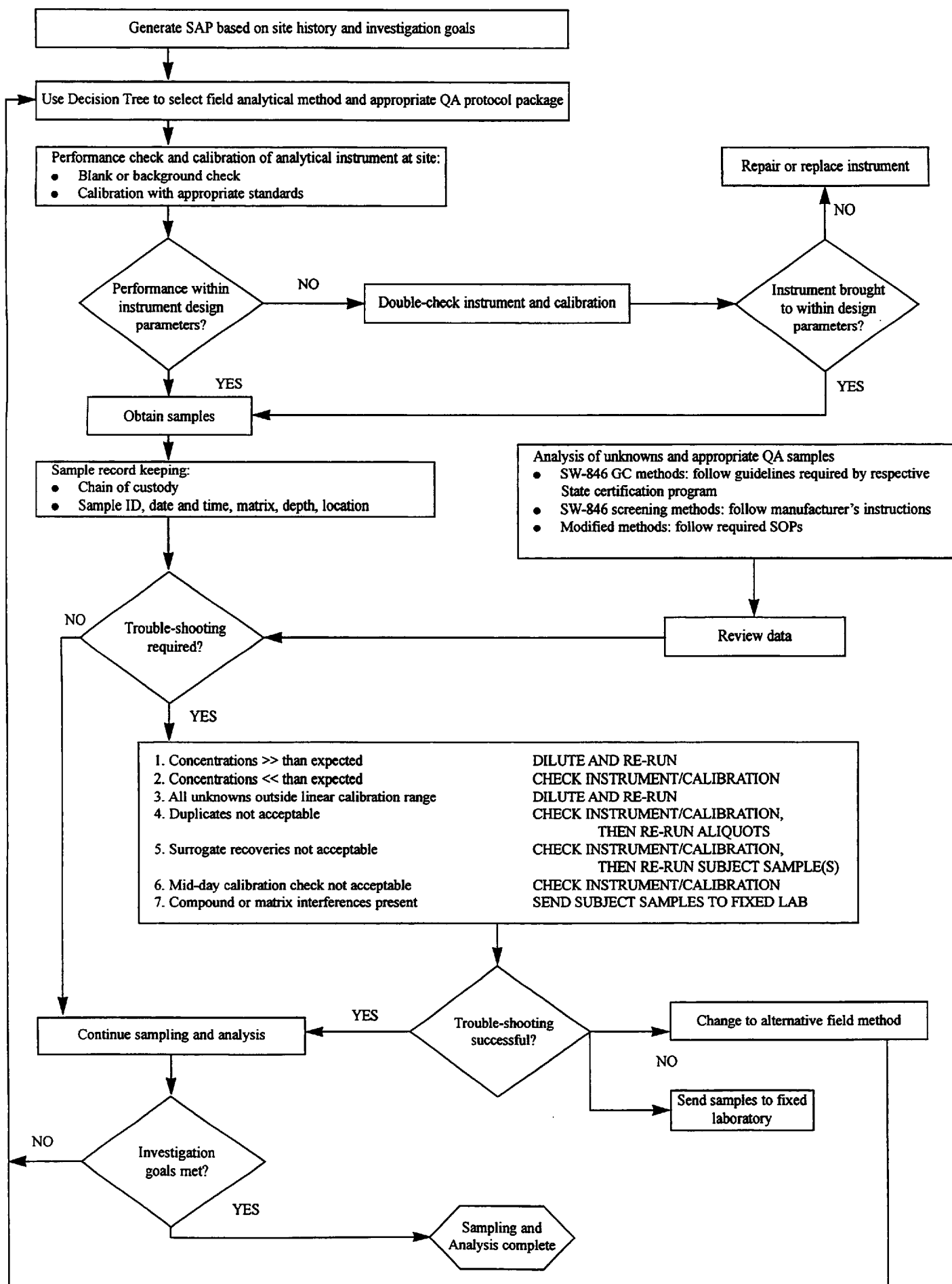


Figure 3-1. Iterative sampling approach flow chart.

Section 4

TRAINING

Training procedures for field analytical instruments vary significantly depending on the degree of sophistication of the instrument or method employed. Table 4-1 summarizes training requirements that should be completed before a person is permitted to operate a specific instrument.

Table 4-1. Training requirements.

Instrument/Method	Degree	Experience	Instrument Training
TOV Analyzer	None	One passive session	By experienced operator with instrument manual
Immunoassay Kits	None	One passive and one supervised active session	By experienced operator with instrument manual
IR TPH	None	Two passive and one supervised active session	By experienced operator with instrument manual
Turbidimetric TPH	None	Two passive and one supervised active session	By experienced operator with instrument manual
Portable GC	B.S. degree or equivalent*	At least three months with specific instrument	By experienced analyst with >1 year experience
Transportable GC	B.S. degree or equivalent*	At least three months with specific instrument	By experienced analyst with >1 year experience

* Degree in physical science.

TRAINING SEQUENCE FOR TECHNICAL FIELD PERSONNEL

Approach

Obtain as much training from the manufacturer of the equipment or test kit as possible. The manufacturer has performed research and has technical support staff and backup technical information. Combine this API publication with others on the subject, such as the ones mentioned in Section 1 (Introduction). This will set the basis of knowledge, prior to the operator checkout sequence noted below.

1. Attend a manufacturers' training course. Obtain formal training, if available, on all of the methods covered in this guidance manual, plus any other methods that could possibly be used for petroleum release work. The course, ideally, should cover the basics in the following areas:

- Equipment use, including calibration
- Preparation and use of QA standards
- Trouble-shooting in the field
- Chemistry which dictates best uses and limitations of the instrument.

2. Obtain supervised field experience. Table 4-1 summarizes the recommended *minimum* degree of supervised sessions one may require. The following activities are recommended:

- Instrument manual review
- Instrument/test kit trouble-shooting
- Data log-in procedures (see example log-in sheets in Appendix D)
- Field QA (blanks, duplicates, spikes, and standards, as appropriate)
- Performance of iterative sampling approach.

3. Pass a proficiency certification. Prior to unsupervised use of the field technology, an operator should pass agreed-to acceptability guidelines for performance evaluation samples. Generally, a blank and a low and high sample or standard can be analyzed in duplicate. The results can be compared with the range of values of a purchased standard (TPH in soil, TPH in water, etc.), compared against a set of values generated by multiple analyses in an off-site laboratory, or compared against a set of values generated by a second field technician who has a higher quality control level.

Section 5

QUALITY ASSURANCE PROTOCOL

Quality Assurance/Quality Control (QA/QC) protocols are specific quality-assurance and quality-control guidelines required for data evaluation and validation for a given field analytical instrument at a specified LDQ. QA elements are specifically selected for each method and LDQ to assure that the field analytical data are adequate for the intended Investigation Goal, and to permit the evaluation of quality, viability, and defensibility of analytical data. The following section details the various elements that can be specified for individual field analytical methods selected through the use of the Decision Tree. The required QA elements for each field analytical method and LDQ are specified in the individual QA/QC Protocol Checklists, included in Appendix D. These checklists are presented in appendix form so that the user can easily reproduce the checklists and use the protocols as a separate reference in the field.

ANALYTICAL PROCEDURES

EPA-Approved Methods

EPA-approved or other validated, standard methods should be referenced by their method number. Generally, states require that only state-certified laboratories with a qualified and state-approved laboratory director may generate analytical data for regulatory purposes.

Non-Standard or Modified Methods

Non-standard methods should be described in detail in SOPs as needed to support the use of the analytical data.

DATA QUALITY VALIDATION

Data quality validation is an evaluation of the ability of the analytical procedures and methods to withstand external scrutiny. Data precision and accuracy must be defensible. Documentation of the appropriate use of QA/QC protocol packages in verifying the accuracy of testing procedures,

instrument calibrations, and sample preparation must be maintained. In order to validate individual sample results, documentation must track the samples from the field, to the laboratory, through analytical testing, to reporting of analytical results.

Blanks

Method Blank. Method blanks are analyzed to determine the level of background contamination. Method blank runs should be performed at least once daily prior to standard and sample analysis. Method blanks should be recorded with each set of sample results for organic analyses. Method blank results are acceptable only if values for target compounds are less than 50 percent of their respective detection limits.

Reagent Blank. Reagent blanks are samples of analyte-free water or other reagents used in sample preparation and analysis. Reagent blanks are prepared following the guidelines for sample preparation of each method. Reagent blank results are acceptable only if values for target compounds are less than 50 percent of their respective detection limits.

Rinsate or Field Blank. A rinsate or field blank is defined as the rinsing of decontaminated sampling equipment (bailer, tubing of peristaltic pump, etc.) with analyte-free water and subsequent analysis. This blank analysis serves to indicate acceptable decontamination procedures for sampling equipment.

Surrogate Standards

Surrogate standards are organic compounds which are similar to the analytes of interest in chemical behavior but which are not normally found in environmental samples. Surrogates are added to samples to monitor the effect of the matrix on the accuracy and precision of the organic analysis. Results are reported in percent recovery.

Matrix Spikes and Matrix Spike Duplicates

Matrix spikes and matrix spike duplicates are two aliquots of the same environmental sample to which known amounts of organic analytes have been added and are subjected to the entire analytical procedure. The results of matrix spike and matrix spike duplicate analysis are reported as percent recovery, and are used to evaluate the effect of the sample matrix and the method on the accuracy and precision (reproducibility) of the results.

Duplicate Samples

The reproducibility of standards and samples can be determined by the analysis of two aliquots of the same standard or sample. Duplicate analyses serve as an internal check on sampling techniques, sample homogeneity, analytical accuracy, and precision.

Precision

Precision is a statistically calculated value, expressed in percent (%), that describes the reproducibility of analytical results for aliquots of the same environmental sample.

Analytical Precision calculated from duplicate measurements

$$RPD = \frac{(C_1 - C_2) \times 100\%}{(C_1 + C_2)/2} \quad \text{Equation (5-1)}$$

where: RPD = relative percent difference
 C_1 = larger of the two observed values
 C_2 = smaller of the two observed values

Analytical Precision calculated from the average relative standard deviation (RSD) using three or more replicate analyses

$$RSD = \frac{\text{Standard deviation}}{\text{Mean of replicate analysis}} \times 100\% \quad \text{Equation (5-2)}$$

Where standard deviation, S , is defined as follows:

$$S = \sqrt{\frac{\sum_{i=1}^n (Y_i - \bar{y})^2}{n - 1}}$$

Equation (5-3)

where: S = standard deviation
 Y_i = measured value of the i th replicate
 \bar{y} = mean of the replicate measurements
 n = number of replicates

Accuracy

Accuracy is a statistically calculated value, expressed in percent (%), that describes the ability of an analytical instrument to detect the true concentration of a compound in an environmental sample. The accuracy of field analytical instruments should be calculated as percent recovery defined as follows:

$$\% \text{ Recovery} \times 100\% \times \frac{S - U}{C_{SA}}$$

Equation (5-4)

where: S = the measured concentration in the spiked aliquot
 U = the measured concentration in the unspiked aliquot
 C_{SA} = the actual concentration of spike added

Method Detection Limit (MDL)

The MDL is the minimum concentration of a compound that can be determined at the 99 percent confidence level using a specified analytical method. MDLs for pre-calibrated instruments such as the TPH analyzer or immunoassay test kits can be found in the respective operating manuals, whereas MDLs for instruments such as gas chromatographs are statistically determined, both for EPA and modified methods.

For statistical MDL calculation, between five and nine consecutive replicate standard runs at about two to five times the expected detection limit are required. MDLs of individual analytes are calculated by:

$$MDL = t_{(n-1, 1-\alpha=0.99)} \times S_1 \quad \text{Equation (5-5)}$$

where:

MDL	=	the detection limit
S	=	the standard deviation of the replicate analyses
$t_{(n-1, 1-\alpha=0.99)}$	=	the Student's t value appropriate at the 99 percent probability level (Table 5-1), and a standard deviation estimate with n-1 degrees of freedom

Table 5-1. Student's t values for MDL calculations.

Number of Results	Student's t Values at the 99% Probability Level
5	4.6
6	4.03
7	3.71
8	3.5
9	3.35

Source: (Pecsok *et al.*, 1976)

CALIBRATION PROCEDURES

Instrument Calibration

Instruments and equipment used for analysis of petroleum hydrocarbons should be controlled by a formal calibration program. The purpose of such a program is to verify that equipment is of the proper type, range, accuracy, and precision to provide data compatible with specified requirements.

For factory pre-calibrated instruments such as TPH analyzers and immunoassay test kits, the manufacturers' manuals should be followed in detail. For analytical instruments that are calibrated on an operational basis (e.g., gas chromatographs), the calibration consists of determining the instrumental response to standards of known composition and concentration. Calibrations must

be performed by trained personnel using reference standards, or externally by calibration agencies or equipment manufacturers. Records of these calibrations should be prepared and maintained for each piece of equipment subject to calibration. Records demonstrating accuracy of preparation, stability, and proof of continuity of reference standards used for calibrations also should be maintained.

Calibration Verification

A calibration check, which covers the routine working range of the instrument, should be conducted periodically during analysis. Calibration verification standards that are appropriate for the Investigation Goal and the analytical instrument should be prepared at or near the estimated MDL, and the expected upper linear range.

PROFICIENCY AND QUALITY CONTROL SAMPLES

EPA Proficiency Evaluation Study

Field laboratories may participate in the analysis of Proficiency Evaluation (PE) samples which contain parameters of interest that are monitored under a regulatory program to establish and maintain proficiency. The EPA Drinking Water Program provides PE samples that are designed to evaluate the analytical performance of participating laboratories. Environmental standards vendors can provide water and many soil matrix standards of analytes mentioned in this publication. Use PE samples freely in training and QC programs, for continuous feedback on performance.

Quality Control Standards

Certified environmental QC standards are commercially available for organic analytes (BTEX, GRO, TPH, etc.) and should be analyzed routinely to evaluate the data quality of field analytical instruments used at LDQs of 2 or higher.

DATA REDUCTION AND REPORTING

Data Reduction

Data reduction is the process of converting raw data to a usable format beginning with data processing and continuing through data review and reporting of results. Data reduction should be performed by the analyst/operator who obtained the data on site.

Data Reporting

All raw data for a given sample must be traceable throughout all testing. The operator/laboratory record keeping system must include, but is not limited to, the following parameters:

- Unique sample ID number
- Sample identification and matrix
- Date, time of analysis, and name of analyst/operator
- Method of analysis
- Records of sample preparation (extraction technique, dilution factor, etc.)
- Raw analytical data (e.g., chromatogram)
- Signature of analyst
- Analytical results table.

Section 6

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Appendix A

GLOSSARY OF TERMS

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Usage of the following terms in this document implies the definitions specified:

Accuracy: A statistically calculated value that defines the ability of an analytical instrument to detect the true concentration of a compound in an environmental sample.

Aliquot: A representative well-mixed portion of a sample.

Bias: A measure of systematic error with two components: error due to the method, and error due to a laboratory's use of the method.

BTEX: Acronym for group of volatile aromatic compounds: benzene, toluene, ethylbenzene, and total xylenes (meta, para, and ortho-xylenes).

Constituents of Concern: Specific petroleum constituents that are identified as posing a potential risk to human health or the environment.

Diesel-Range Organics (DRO): Diesel range organics are commonly defined as all chromatographic peaks eluting between decane ($n\text{-C}_{10}$) and octacosane ($n\text{-C}_{28}$).

Dissolved Oxygen: Molecular oxygen dissolved in water. The solubility of oxygen is a function of O_2 partial pressure, temperature, and water composition.

Field Analytical Methods: Methods or techniques that measure physical properties or chemical presence in soil, soil vapor, and groundwater in the field, within a relatively short period of time.

Gas Chromatograph, portable: Compact, isothermally operated instruments which contain internal batteries and operating gas supplies. Measurement depends on elution of separated compounds from a packed or capillary column.

Gas Chromatograph, transportable: Laboratory-grade instruments that require a temperature-controlled environment with external power and gas supplies (e.g., a mobile laboratory).

Gasoline-Range Organics (GRO): Gasoline range organics are commonly defined as all chromatographic peaks eluting between and including pentane (C_5) and 1,2,4-trimethylbenzene (C_{10}).

Headspace Gas Chromatography: Static headspace analysis involves the partitioning of volatile compounds between the aqueous and gas phases enclosed in a gas-tight vial. The static headspace method requires that chemical and thermal equilibrium are achieved within the sample vial.

Immunoassay: An analytical technique that uses an antibody molecule as a binding agent in the detection and quantification of substances in an environmental sample.

Level of Data Quality (LDQ): The degree of sophistication of analytical data. LDQ considers 1) the method of analysis, 2) the selected analytical instrument, and 3) the QA/QC protocol employed to validate the desired quality of the data.

Matrix: Medium that is suspected to contain contamination as a dissolved or suspended phase in water, adsorbed phase on soil particles, or gas phase in air.

Maximum Contaminant Level (MCL): A standard for drinking water established by U.S. EPA under the Safe Drinking Water Act. The maximum permissible level of a chemical(s) of concern in water delivered to any user of a public water supply.

Method Detection Limit (MDL): Minimum concentration of a compound that can be determined at the 99 percent confidence level.

Methyl tertiary-butyl ether (MTBE): A synthetic compound derived from methanol and isobutylene. MTBE is added to gasoline in quantities up to 15 percent by volume as an octane enhancer and oxygenate (oxygen-bearing compound).

Organoleptic: Sensory means utilized by an investigator for determining sample (soil, groundwater, air, etc.) characteristics, including, but not limited to, olfactory and visual inspection.

Petroleum: Crude oil or any fraction thereof that is liquid at standard conditions of temperature and pressure. The term includes petroleum-based substances comprised of a complex blend of hydrocarbons derived from crude oil by fractional distillation and refinery processing to yield refinery gas, gasoline, kerosene/jet fuel, and diesel/fuel oil.

pH: A logarithmic scale for expressing the acidic intensity of a solution. To a first approximation, the pH of a solution can be defined as the negative logarithm ($-\log_{10}$) of the concentration of hydrogen ions in solution.

Point of Compliance: A location selected between the source area(s) and potential points of exposure where concentrations of constituents of concern must be at or below the determined groundwater target levels.

Polynuclear Aromatic Hydrocarbons (PAHs): These often are byproducts of petroleum processing or combustion. PAHs include: acenaphthene, acenaphthylene, anthracene, benz(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(ghi)perylene, benzo(k)fluoranthene, chrysene, dibenz(a,h)anthracene, fluoranthene, fluorene, indeno(1,2,3-cd)pyrene, naphthalene, phenanthrene, and pyrene. PAHs are relatively insoluble in water and many of these compounds are highly carcinogenic at relatively low levels (ppb to ppt levels).

Precision: A statistically calculated value that defines the reproducibility of analytical results for aliquots of the same environmental sample.

Quality Assurance & Quality Control (QA/QC): The use of standards and procedures to ensure that samples collected and data generated are reliable, reproducible, and verifiable.

REDOX: Oxidation-reduction (REDOX) reactions describe the loss of electrons (oxidation) and the gain of electrons (reduction) during chemical reactions. This definition of oxidation and reduction applies only to reactions in which electron transfer occurs.

Release: Any spilling, leaking, emitting, discharging, escaping, leaching, or disposing of petroleum products into groundwater, surface water, soils, or air.

Risk-Based Corrective Action (RBCA): A consistent decision-making process for the assessment and response to a petroleum release, based on the protection of human health and the environment.

Risk-Based Screening Level (RBSL): Risk-based corrective action levels for chemical(s) of concern developed under a Risk-Based Corrective Action (RBCA) Tier I evaluation.

Site Assessment/Characterization: An evaluation of subsurface geology, hydrology, and surface characteristics to determine if a release has occurred, the concentrations of chemical(s) of concern, and the extent of migration of the chemical(s) of concern. The site assessment/characterization collects data on soils and groundwater quality and potential receptors and generates information to support remedial action decisions.

Site-Specific Target Level (SSTL): Risk-based corrective action target levels for chemical(s) of concern developed for a particular site under a Risk-Based Corrective Action (RBCA) Tier II or Tier III evaluation.

Standard Operating Procedure (SOP) Manual: Includes step-by-step instructions for all procedures and operations involved with each method of analysis of a laboratory or a person engaged in analysis of environmental samples.

Surrogate Standards: Organic compounds which are similar to the analytes of interest in chemical behavior but which are not normally found in environmental samples.

Total Petroleum Hydrocarbons (TPH): The sum of all petroleum-derived hydrocarbons present in an environmental sample.

TPH by Infrared Spectroscopy: Method for TPH analysis (EPA SW-846 Method 418.1) that requires hydrocarbon extraction from environmental samples using Freon-113 and analysis of the extract by IR spectroscopy at a wavelength of 3.4 μm .

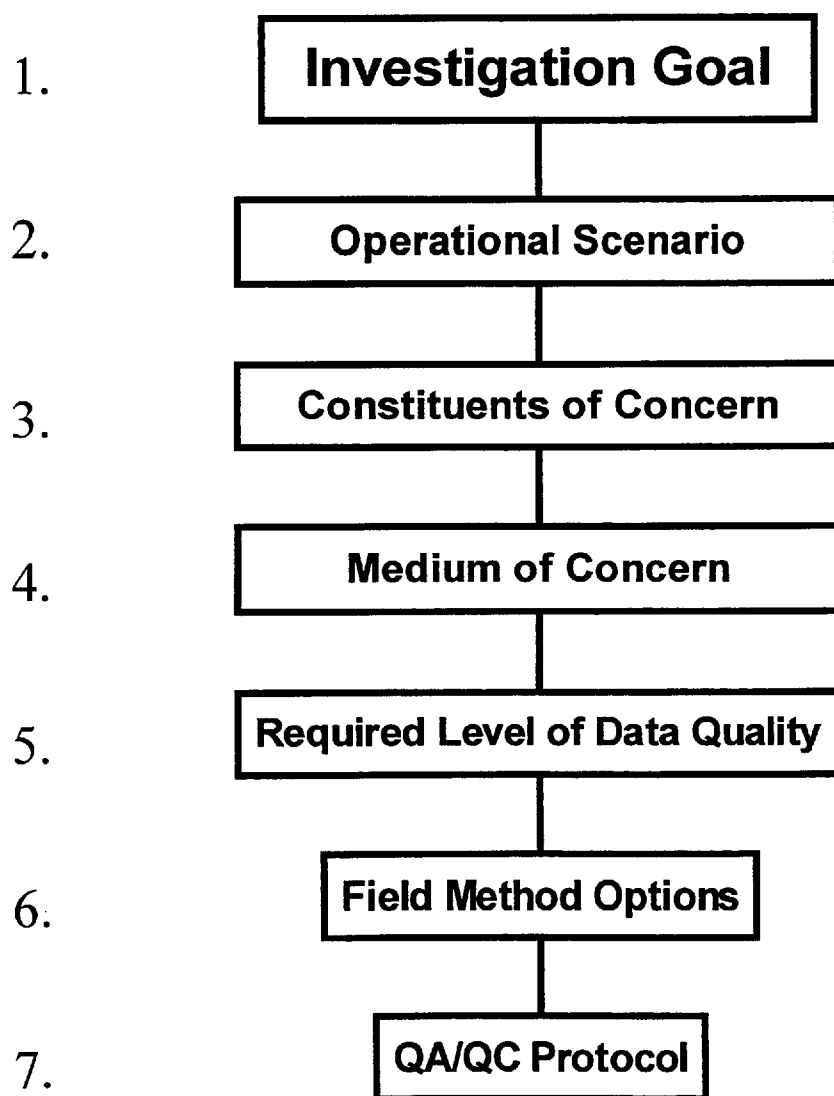
TPH by Turbidimetric Analysis: Method for TPH analysis (EPA SW-846 Draft Method 9074) that is based on hydrocarbon extraction from sediments and soils with a methanol-like solvent, and mixing of the extracted hydrocarbons with a developing agent which keeps hydrocarbons in suspension. The degree of turbidity that is analyzed with a spectrophotometer is proportional to the TPH concentration in the sample.

Appendix B

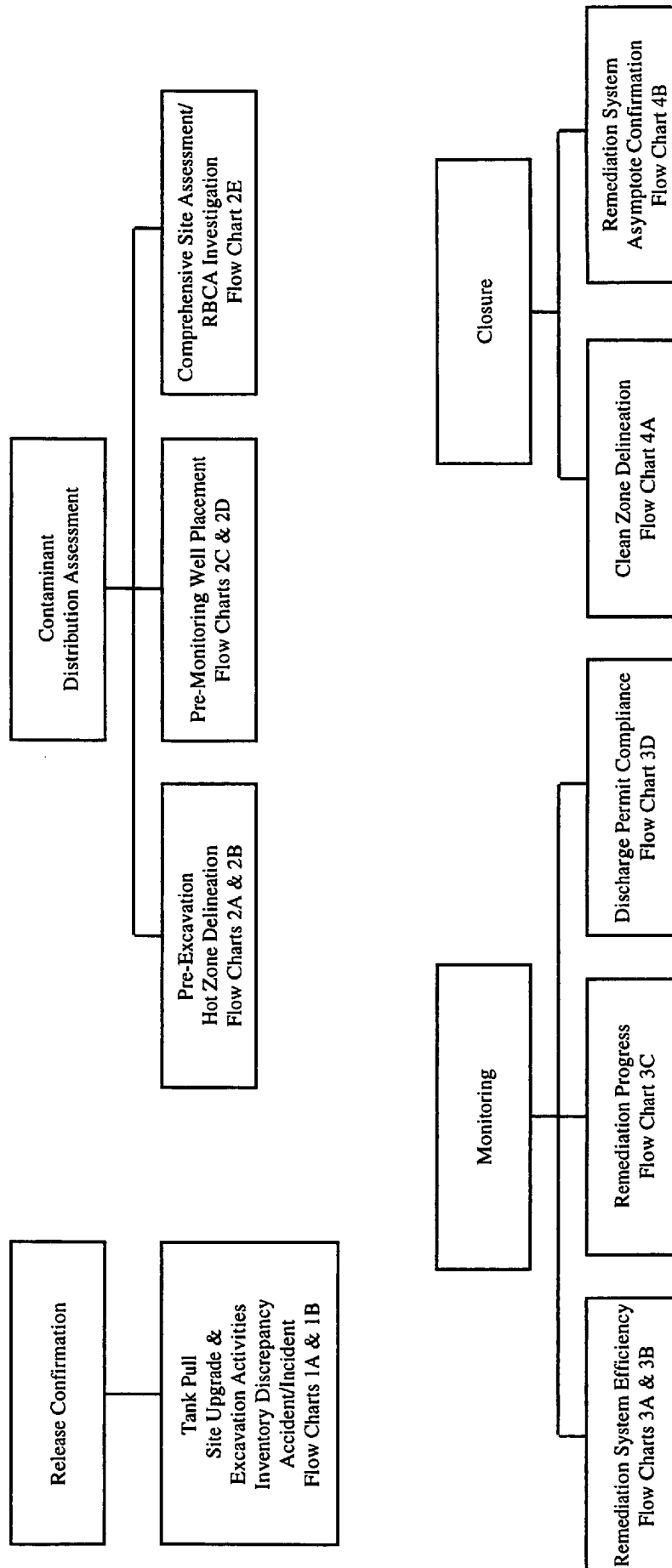
DECISION TREE FLOW CHARTS

Decision Tree Flow Steps

Step

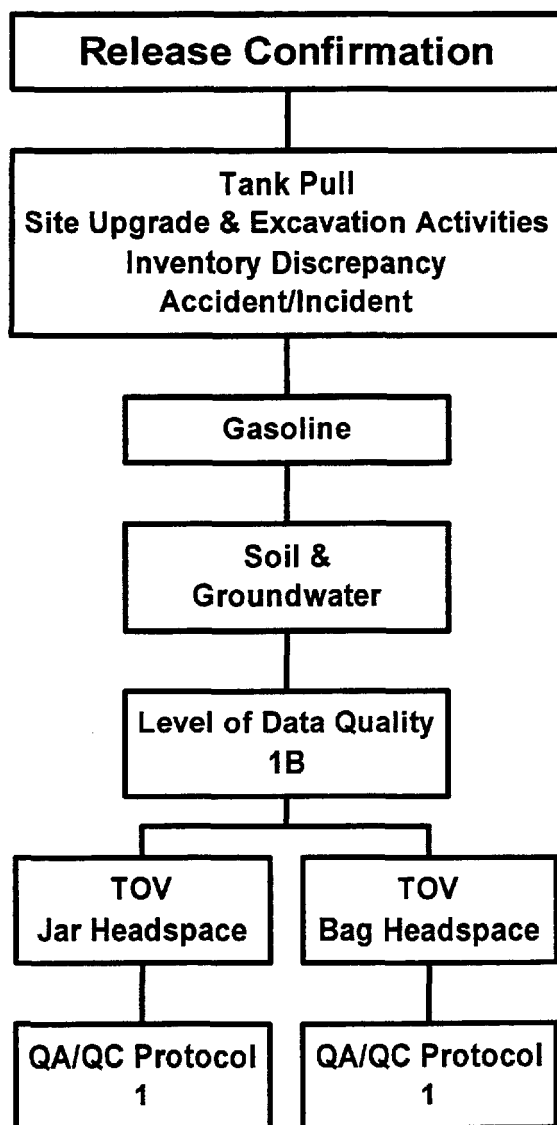


Decision Tree Summary



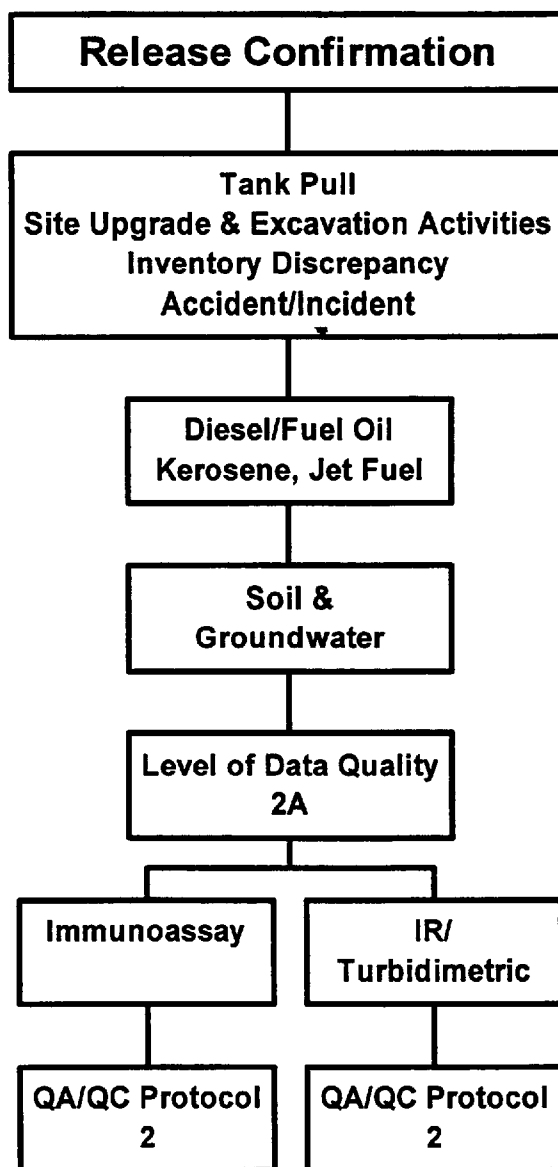
B-2

Flow Chart 1A



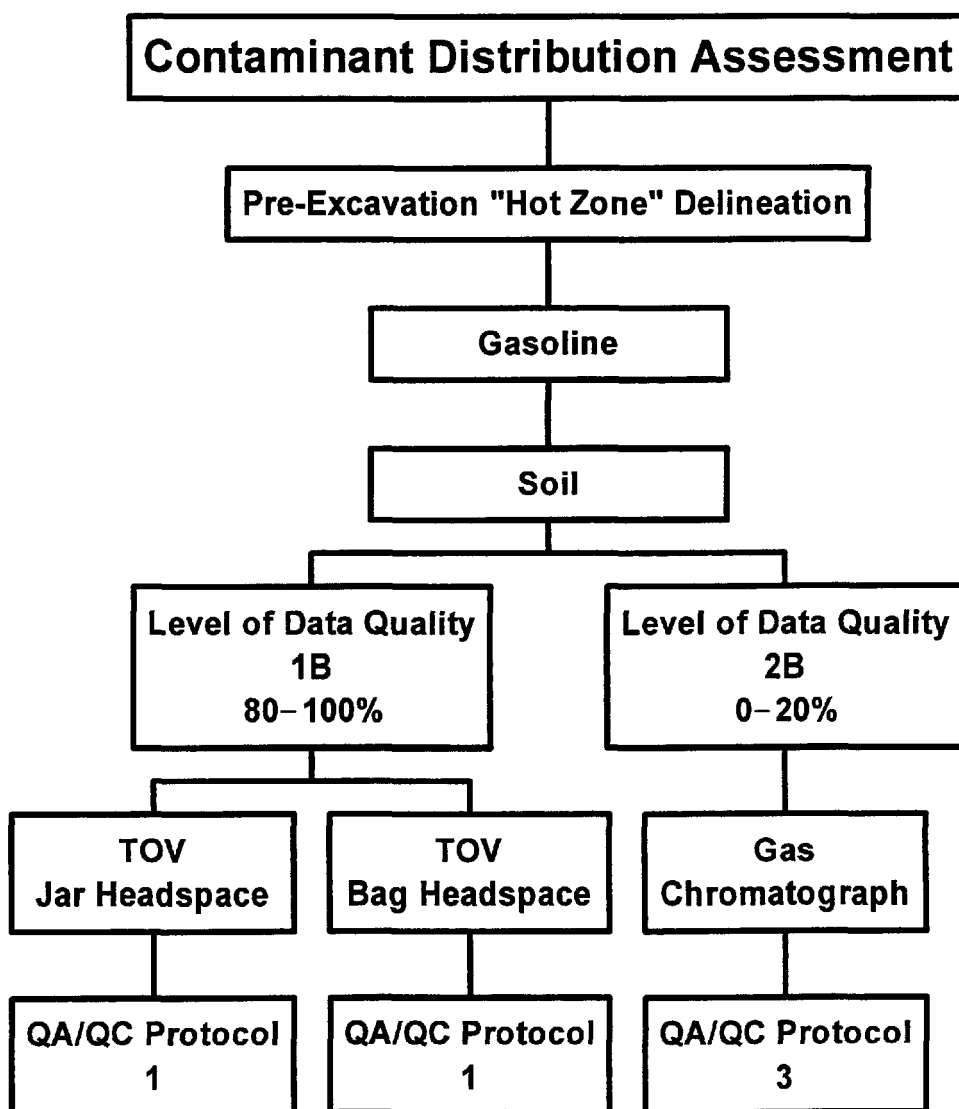
B-3

Flow Chart 1B



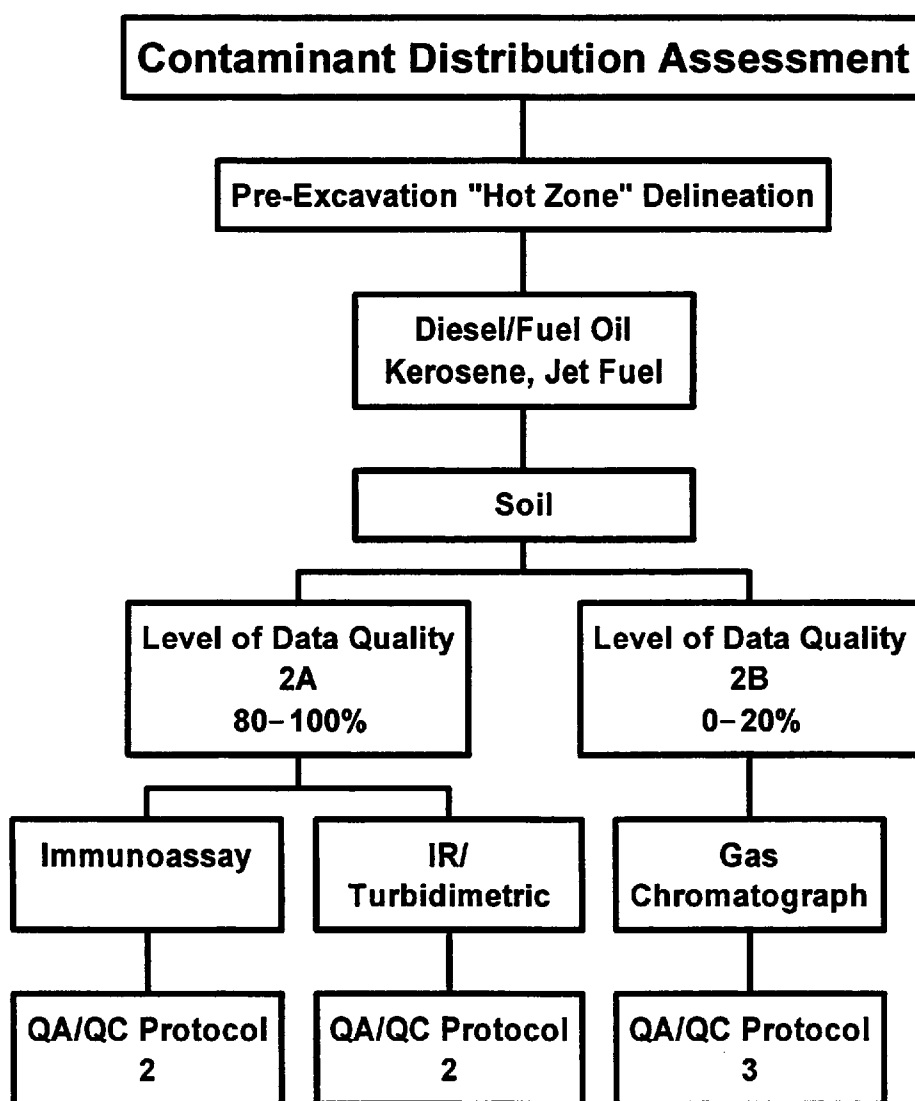
B-4

Flow Chart 2A



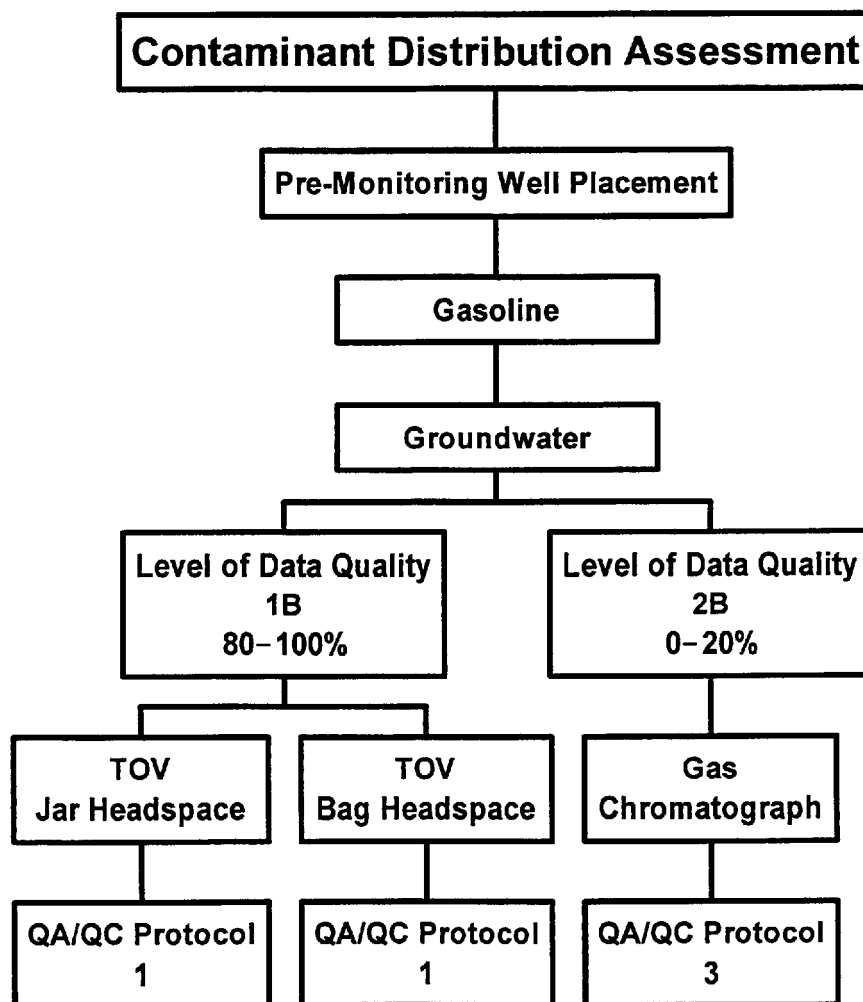
B-5

Flow Chart 2B



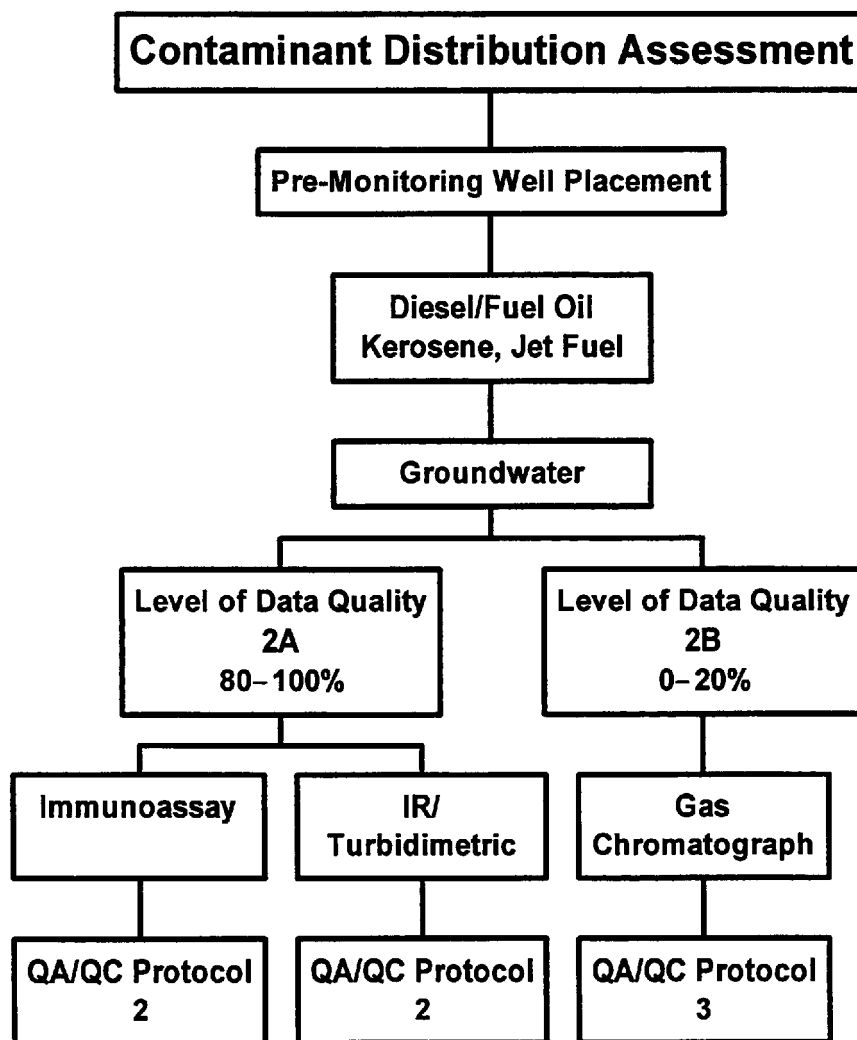
B-6

Flow Chart 2C

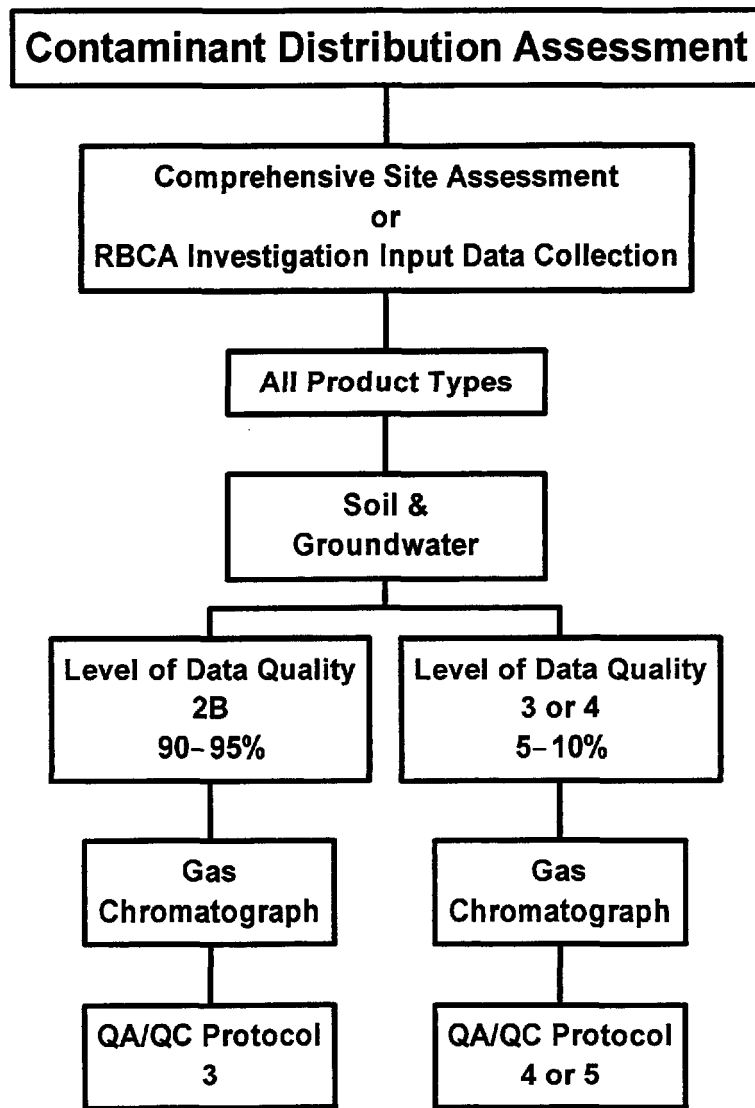


B-7

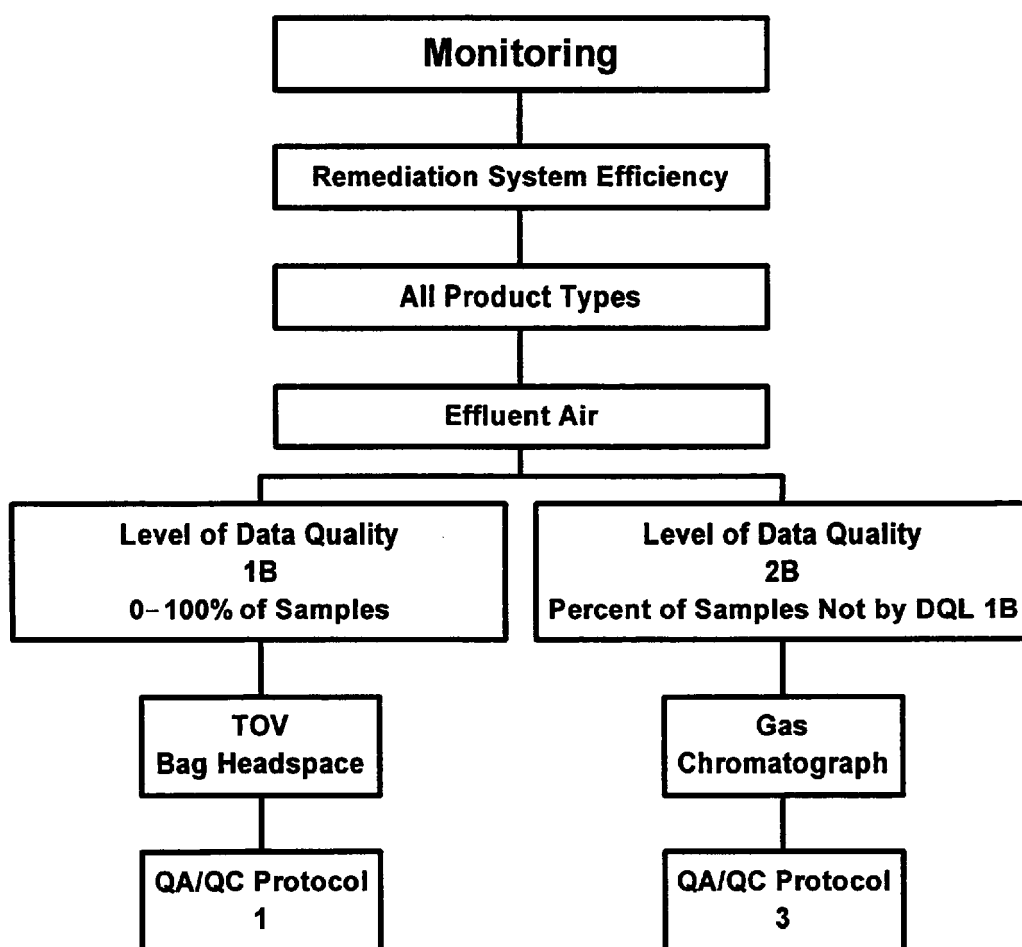
Flow Chart 2D



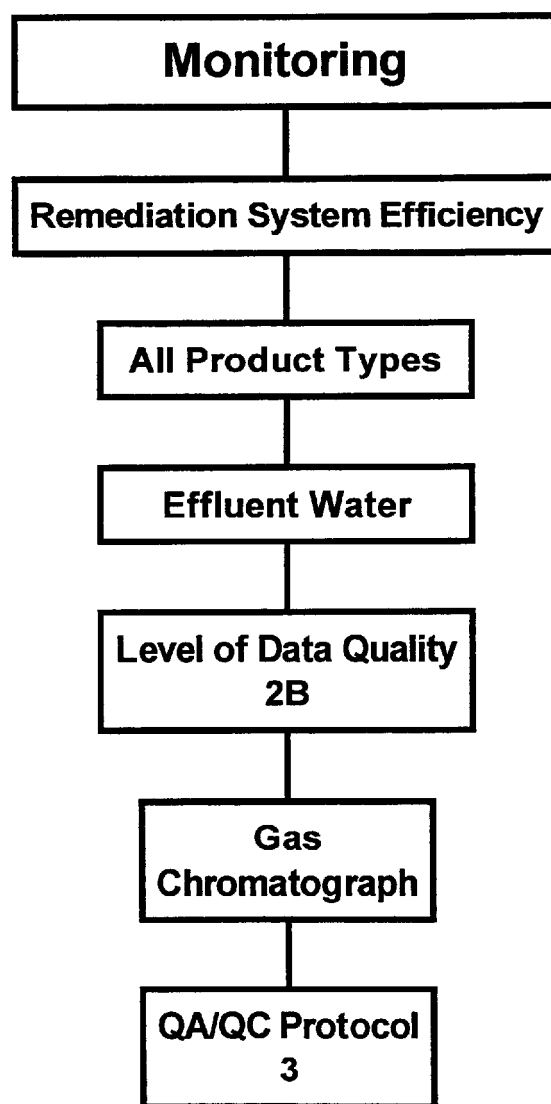
Flow Chart 2E



Flow Chart 3A

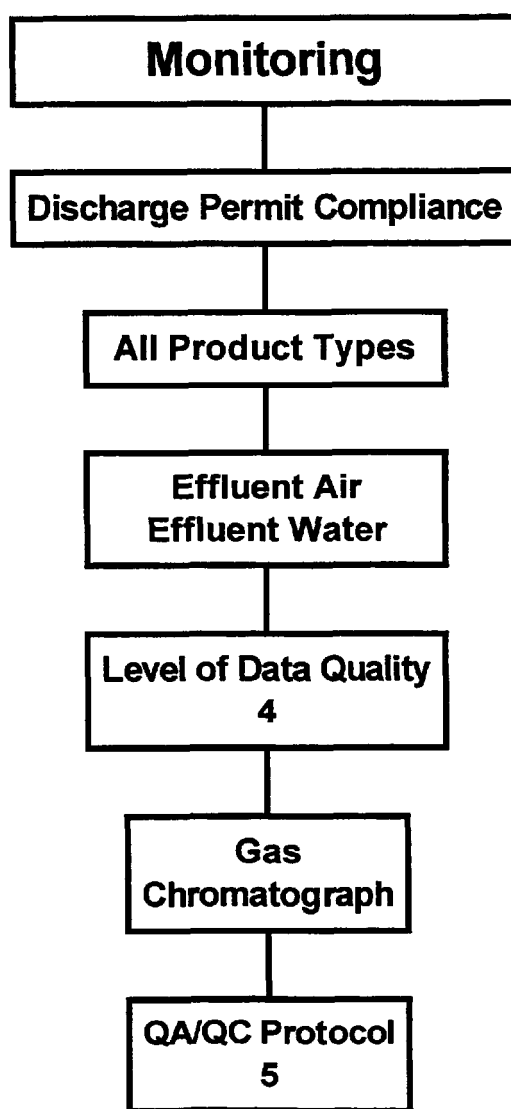


Flow Chart 3B



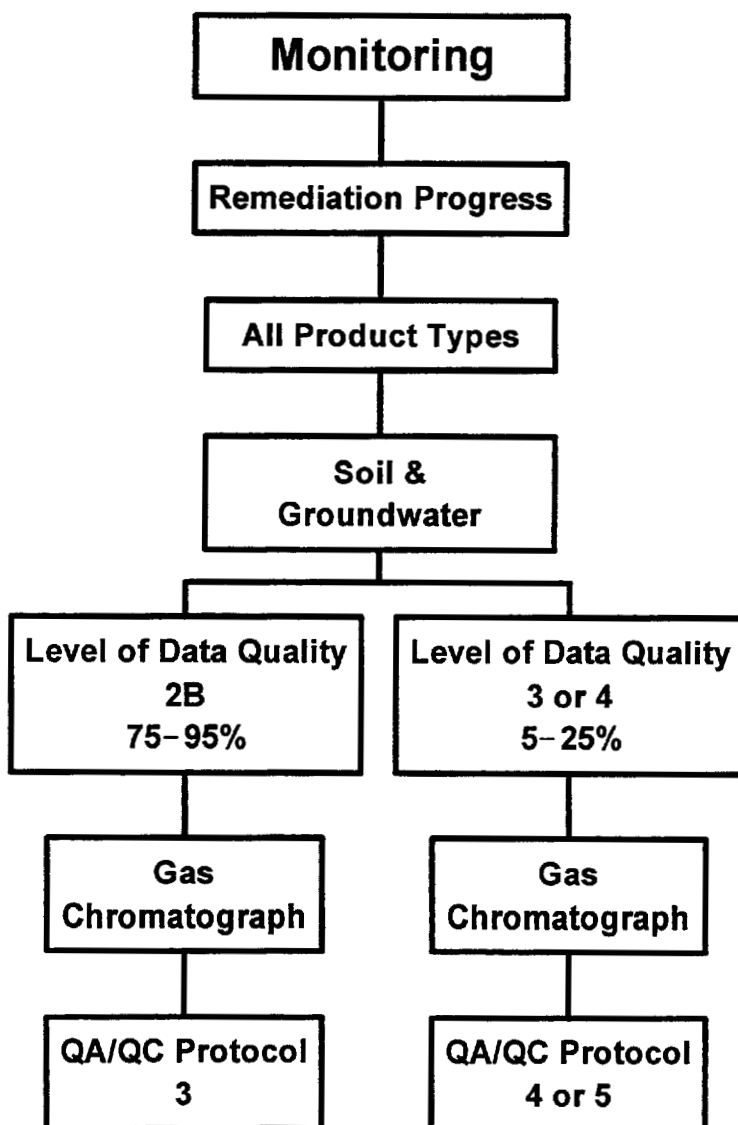
B-11

Flow Chart 3C



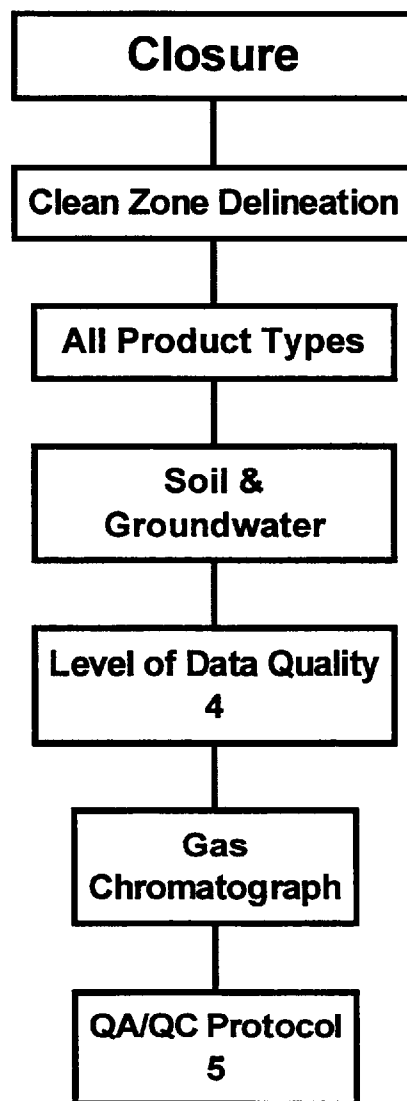
B-12

Flow Chart 3D



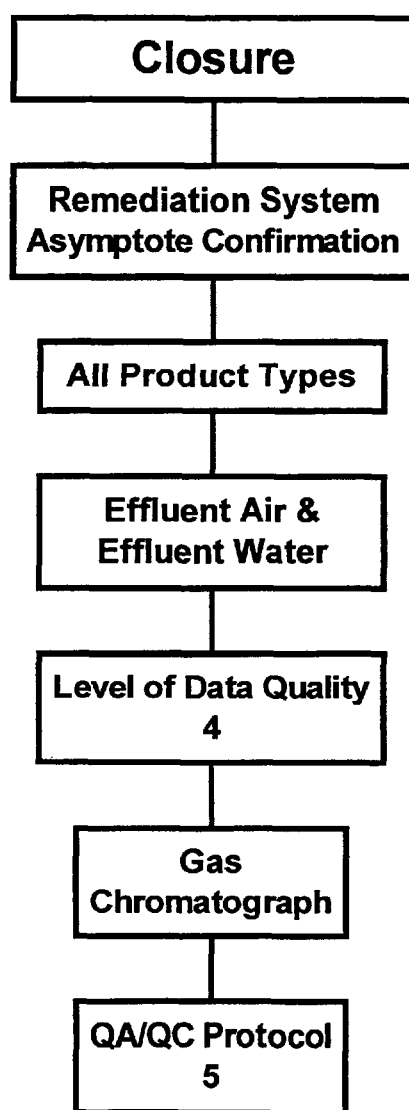
B-13

Flow Chart 4A



B-14

Flow Chart 4B



B-15

Appendix C

EXAMPLE DECISION TREE APPLICATION

EXAMPLE DECISION TREE APPLICATION

The following presents an example of the Decision Tree process applied to two phases of a gasoline underground storage tank removal program:

1. Release confirmation
2. Remediation by excavation.

Example Site Information:

- New site, no previously documented subsurface impacts
- A gasoline underground storage tank removal/upgrade program is scheduled
- Regulatory requirements are to screen soils during excavation to ensure that any potential subsurface hydrocarbon impact is identified
- Any hydrocarbon-impacted soils are to be removed from the site prior to the installation of new tanks
- Depth to water is 130 feet below grade.

Phase 1

In Phase 1 of the underground storage tank removal program, the user selects "Release Confirmation" as the Step One Investigation Goal (Flow Charts 1A and 1B, Appendix B). In Step Two, the flow charts show that all operational scenarios are covered, and that no additional decisions are required. In Step Three, the Decision Tree requires that the user select either

- a) Gasoline (Flow Chart 1A), or
- b) One or more of: diesel/fuel oil, kerosene, jet fuel (Flow Chart 1B)

Since this is a gasoline underground storage tank removal/upgrade program, Flow Chart 1A is selected. In Decision Tree Step Four, the medium to be analyzed is selected. At this site, depth to groundwater is 130 feet below grade, and therefore it is expected that only soil will be encountered during the excavation program. In all cases within the Step One "Release Confirmation" Investigation Goal for gasoline, the Decision Tree recommends the use of Level of Data Quality LDQ 1B (semiquantitative, non-constituent specific, order-of-magnitude analyses) in the Decision Tree Step Five. Within the Level of Data Quality 1b for the analysis of gasoline in soils, the Decision Tree "branch" specifies the use of one of the following field analytical methods in Flow Chart Step Six:

- a) Total Organic Volatiles (TOV), Jar Headspace
- b) TOV, Bag Headspace

The user is invited to select one of the above field methods depending on site-specific and user-specific criteria such as ease of use, availability, and cost. For any of the selected methods, the flow chart identifies the appropriate method-specific QA/QC protocol (Appendix D) which will

guide the user through the field analytical procedures required to produce data commensurate with the nature of the Investigation Goal. Note that had there been any diesel, kerosene or jet fuel at the site (Flow Chart 1B), higher LDQs would have been specified since these petroleum compounds are only poorly detected with TOV analytical instruments.

Phase 2

Based on Phase 1 of the gasoline underground storage tank removal/upgrade program, it is determined that some subsurface hydrocarbon impact has occurred. Organoleptic analysis has identified soils with hydrocarbon staining and odors, and the TOV Jar Headspace field analytical method has returned maximum values of approximately 6,000 ppm. Phase 2 of the program consists of removing all hydrocarbon-impacted soils from the site through excavation.

Additional Example Site Information

- Due to the depth to water at the site, regulation requires that all “grossly contaminated” soils be removed from the site.
- Installation of the new tanks is scheduled for the same day that the remedial excavation is taking place.

Given the above, in Step One of the Decision Tree the user selects “Contaminant Distribution Assessment” (Flow Charts 2A, 2B, 2C, 2D and 2E) as the Investigation Goal since the extent of the “grossly contaminated” soil must be delineated and removed. In this case, the delineation and removal will occur concurrently. The Step Two options are:

1. Pre-Excavation “Hot Zone” Delineation
2. Pre-Monitoring Well Placement
3. Comprehensive Site Assessment
4. RBCA Investigation Input Data Collection.

The user selects “Pre-Excavation ‘Hot Zone’ Delineation” as the Investigation Goal’s Operational Scenario in Step Two. In a manner identical to the “branch” selection process used in Phase 1 of the project, the user selects gasoline as the hydrocarbon compound to be analyzed (Step Three) and soil as the impacted media to be evaluated (Step Four), which leads the user to the selection of the Decision Tree Branch represented by Flow Chart 2A. In this “branch” of the Decision Tree, Step Five of the flow chart recommends between 80% and 100% of the samples be analyzed via LDQ 1b (semiquantitative, non-constituent specific, order-of-magnitude analyses), with the remaining 0% to 20% of the samples be analyzed for “confirmatory” purposes via LDQ 2B (quantitative analysis of specific petroleum hydrocarbon constituents). Decision Tree Step Six again provides the choice between the following field analytical methods within LDQ 1B:

- a) TOV, Jar Headspace
- b) TOV, Bag Headspace.

Additionally, for the confirmatory samples analyzed via LDQ 2B, the Step Six recommendation is the use of a gas chromatograph.

The user should then choose the appropriate field method(s), again depending on site-specific factors such as the regulatory definition of "grossly contaminated" soils¹, ease of use, availability and cost. The field QA/QC protocols for each method are then provided in the referenced QA/QC packages, Appendix D.

¹ **Note:** In excavation scenarios, regulatory requirements often mandate the collection of endpoint soil samples for fixed-lab certified analyses. In these cases, it is important to obtain regulatory concurrence with the site's scope of work, particularly in regard to "field endpoints" which will trigger the end of excavation activities. If instead of an arbitrary definition of "grossly contaminated" (e.g., 1000 ppm headspace VOCs), the regulatory requirement is excavation to below detection limits via EPA 8240, then targeted on-site confirmation of the lower LDQ analytical methods used to delineate the "hot-zone" may be required using higher LDQ methodologies. While these issues should be considered prior to any investigation, they will be site- and regulator-specific, and are therefore out of the primary scope of the Decision Tree framework.

Appendix D

QUALITY ASSURANCE PACKAGES

- LDQ Protocol Checklists—by Method
- Calibration and Sampling Log

QA/QC Protocol 1

TOV Analysis

QA/QC Protocol 1	Checked if Required	Frequency & Acceptance Criteria
Analytical Method (EPA No. or SOP)	U	record instrument and method used
Data Quality Validation		
Method Blank	U	run background blank with instrument check
Reagent Blank		
Rinsate/Field Blank		
Surrogate Standard Recovery		
Matrix Spike		
Matrix Spike Duplicate		
Duplicate Sample		
Method Detection Limit (MDL)		
Precision		
Accuracy		
Calibration Procedures		
Instrument Calibration	U	morning calibration check
Calibration Verification	U	mid-day calibration check
Calibration Range		
Calibration Correlation		
Proficiency & Quality Control Samples		
Quality Control Standards		
EPA Proficiency Evaluation Study		
Data Reduction & Reporting		
Data Reduction		
Chain of Custody	U	log in each sample
Unique Sample ID No.	U	identify sample ID, depth, and location
Matrix	U	record sample matrix
Date & Time of Analysis	U	record date and time of analysis
Name of Analyst/Operator	U	record name of analyst/operator
Records of Sample Preparation (e.g., dilution)		
Raw Analytical Data (e.g., chromatograms)		
Analytical Results Table	U	present results in table
Signature of Analyst/Operator	U	sign analytical report

TOV Field Log[illegible]

QA/QC Protocol 2

Immunoassay Test

QA/QC Protocol 2 Immunoassay Tests	Checked if Required	Frequency & Acceptance Criteria
Analytical Method (EPA No. or SOP)	U	record instrument and method used
Data Quality Validation		
Method Blank	U	analyze method blank with each batch
Reagent Blank		
Rinsate/Field Blank	U	analyze one (1) per day
Surrogate Standard Recovery		
Matrix Spike		
Matrix Spike Duplicate		
Duplicate Sample	U	analyze 5% or at least 1 per day
Method Detection Limit (MDL)		
Precision		
Accuracy		
Calibration Procedures		
Instrument Calibration	U	run standards with each batch
Calibration Verification	U	run standards with each batch
Calibration Range		
Calibration Correlation		
Proficiency & Quality Control Samples		
Quality Control Standards		
EPA Proficiency Evaluation Study		
Data Reduction & Reporting		
Data Reduction		
Chain of Custody	U	log in each sample
Unique Sample ID No.	U	identify sample ID, depth, and location
Matrix	U	record sample matrix
Date & Time of Analysis	U	record date and time of analysis
Name of Analyst/Operator	U	record name of analyst/operator
Records of Sample Preparation (e.g., dilution)	U	record dilutions
Raw Analytical Data (e.g., chromatograms)		
Analytical Results Table	U	present results in table
Signature of Analyst/Operator	U	sign analytical report

Immunoassay Log

Client:			Date:		Job ID #:		
Location:			Operator:				
Instrument:			Signature:				
Method:			Calibration Temperature:				
		Soil	Dilution		Photometer	Concentration	
Sample ID	Depth	Weight	Factor	Time	Reading	Range	Units
Blank	N/A	N/A	N/A				
Calibrator 1	N/A	N/A	N/A				
Calibrator 2	N/A	N/A	N/A				
1)							
2)							
3)							
4)							
5)							
6)							
7)							
8)							
9)							
Duplicate							
Blank	N/A	N/A	N/A				
Calibrator 1	N/A	N/A	N/A				
Calibrator 2	N/A	N/A	N/A				
Field Blank							
12)							
13)							
14)							
15)							
16)							
17)							
18)							
19)							
20)							

QA/QC Protocol 2

Turbidimetric TPH Analyzer

QA/QC Protocol 2 Turbidimetric TPH Analyzer	Checked if Required	Frequency & Acceptance Criteria
Analytical Method (EPA No. or SOP)	U	record instrument and method used
Data Quality Validation		
Method Blank	U	analyze method blank with each batch
Reagent Blank		
Rinsate/Field Blank	U	analyze one (1) per day
Surrogate Standard Recovery		
Matrix Spike		
Matrix Spike Duplicate		
Duplicate Sample	U	analyze 5% or at least 1 per day
Method Detection Limit (MDL)		
Precision		
Accuracy		
Calibration Procedures		
Instrument Calibration	U	run standards with each batch
Calibration Verification	U	run standards with each batch
Calibration Range		
Calibration Correlation		
Proficiency & Quality Control Samples		
Quality Control Standards		
EPA Proficiency Evaluation Study		
Data Reduction & Reporting		
Data Reduction		
Chain of Custody	U	log in each sample
Unique Sample ID No.	U	identify sample ID, depth, and location
Matrix	U	record sample matrix
Date & Time of Analysis	U	record date and time of analysis
Name of Analyst/Operator	U	record name of analyst/operator
Records of Sample Preparation (e.g., dilution)	U	record dilutions
Raw Analytical Data (e.g., chromatograms)		
Analytical Results Table	U	present results in table
Signature of Analyst/Operator	U	sign analytical report

Turbidimetric TPH Log

Client:			Date:			Job ID #	
Location			Operator				
Instrument:			Signature:				
Method:			Calibration Temperature:				
		Soil	Dilution	Response		TPH Reading	TPH
Sample ID	Depth	Weight	Factor	Factor	Time	(uncorrected)	ppm
Blank	N/A	N/A	N/A				
Standard	N/A	N/A	N/A				
1)							
2)							
3)							
4)							
5)							
6)							
7)							
8)							
9)							
Duplicate							
Blank	N/A	N/A	N/A				
Standard	N/A	N/A	N/A				
Field Blank							
12)							
13)							
14)							
15)							
16)							
17)							
18)							
19)							
20)							

QA/QC Protocol 2

IR TPH Analyzer

QA/QC Protocol 2 IR TPH Analyzer	Checked if Required	Frequency & Acceptance Criteria
Analytical Method (EPA No. or SOP)	U	record instrument and method used
Data Quality Validation		
Method Blank	U	analyze method blank with instrument check
Reagent Blank		
Rinsate/Field Blank	U	analyze one (1) per day
Surrogate Standard Recovery		
Matrix Spike		
Matrix Spike Duplicate		
Duplicate Sample	U	analyze 5% or at least 1 per day
Method Detection Limit (MDL)		
Precision		
Accuracy		
Calibration Procedures		
Instrument Calibration	U	run high/low standards with instrument check
Calibration Verification	U	run mid standard mid-day
Calibration Range		
Calibration Correlation		
Proficiency & Quality Control Samples		
Quality Control Standards		
EPA Proficiency Evaluation Study		
Data Reduction & Reporting		
Data Reduction		
Chain of Custody	U	log in each sample
Unique Sample ID No.	U	identify sample ID, depth, and location
Matrix	U	record sample matrix
Date & Time of Analysis	U	record date and time of analysis
Name of Analyst/Operator	U	record name of analyst/operator
Records of Sample Preparation (e.g., dilution)	U	record dilutions
Raw Analytical Data (e.g., chromatograms)		
Analytical Results Table	U	present results in table
Signature of Analyst/Operator	U	sign analytical report

IR TPH Log

Client:			Date:		Job ID #:	
Location:			Operator:			
Instrument:			Signature:			
Method:			Temperature:			
				Time	TPH Reading	TPH (ppm)
	Method Blank (before sample analysis)					
	Low STD (before sample analysis)					
	High STD (before sample analysis)					
	Mid STD (mid-day)					
	Field Blank					
		Soil	Dilution		TPH Reading	TPH
Sample ID	Depth	Weight	Factor	Time	(uncorrected)	ppm
1)						
2)						
3)						
4)						
5)						
6)						
7)						
8)						
9)						
10)						
11)						
12)						
13)						
14)						
15)						
16)						
17)						
18)						
19)						
Duplicate						
20)						

QA/QC Protocol 3

Gas Chromatograph Analysis

QA/QC Protocol 3 GC Analysis	Checked if Required	Frequency & Acceptance Criteria
Analytical Method (EPA No. or SOP)	U	record instrument and method used
Data Quality Validation		
Method Blank	U	analyze method blank with instrument check
Reagent Blank	U	analyze reagent blank with instrument check
Rinsate/Field Blank	U	analyze one (1) per day
Surrogate Standard Recovery	U	surrogate recovery must be between 80-120%
Matrix Spike		
Matrix Spike Duplicate		
Duplicate Sample	U	analyze 5% or at least 1 per day
Method Detection Limit (MDL)	U	MDL study for each analyte must be available
Precision	U	must be statistically determined
Accuracy	U	must be statistically determined
Calibration Procedures		
Instrument Calibration	U	run high/low standards with instrument check
Calibration Verification	U	run mid-level standard mid-day
Calibration Range	U	linear calibration range must be documented
Calibration Correlation	U	correlation coefficient must be ≥ 0.95
Proficiency & Quality Control Samples		
Quality Control Standards	U	must be analyzed yearly
EPA Proficiency Evaluation Study	U	must be analyzed yearly
Data Reduction & Reporting		
Data Reduction	U	chromatographic results should be evaluated
Chain of Custody	U	log in each sample
Unique Sample ID No.	U	identify sample ID, depth, and location
Matrix	U	record sample matrix
Date & Time of Analysis	U	record date and time of analysis
Name of Analyst/Operator	U	record name of analyst/operator
Records of Sample Preparation (e.g., dilution)	U	record dilutions
Raw Analytical Data (e.g., chromatograms)	U	available upon request
Analytical Results Table	U	present results in table
Signature of Analyst/Operator	U	sign analytical report

GC Log for QA/QC Protocol 3

Client:		Date:		Analyst:		Instrument:	
Location:		Job ID #:		Signature:		Method:	
Sample ID	Matrix	Depth	Sample ID No.	Time	Dilution Factor	Recovery	ppb
Instrument Blank	N/A	N/A			N/A		ppb
Method Blank	N/A	N/A			N/A		ppb
Low STD	N/A	N/A			N/A		ppb
Retention Time							
High STD	N/A	N/A			N/A		ppb
Retention Time							
1)							
2)							
3)							
4)							
5)							
6)							
7)							
8)							
9)							
Duplicate							
Mid STD	N/A	N/A			N/A		ppb
Retention Time							
Field Blank							
12)							
13)							

QA/QC Protocol 4

Gas Chromatograph Analysis

QA/QC Protocol 3 GC Analysis	Checked if Required	Frequency & Acceptance Criteria
Analytical Method (EPA No. or SOP)	U	record instrument and method used
Data Quality Validation		
Method Blank	U	analyze method blank with instrument check
Reagent Blank	U	analyze reagent blank with instrument check
Rinsate/Field Blank	U	analyze one (1) per day
Surrogate Standard Recovery	U	surrogate recovery must be between 80-120%
Matrix Spike	U	analyze 5% or at least one (1) per day
Matrix Spike Duplicate	U	analyze 5% or at least one (1) per day
Duplicate Sample	U	analyze 10% or at least two (2) per day
Method Detection Limit (MDL)	U	MDL study for each analyte must be available
Precision	U	must be statistically determined
Accuracy	U	must be statistically determined
Calibration Procedures		
Instrument Calibration	U	run high/low standards with instrument check
Calibration Verification	U	run mid-level standard mid-day
Calibration Range	U	linear calibration range must be documented
Calibration Correlation	U	correlation coefficient must be 0.99
Proficiency & Quality Control Samples		
Quality Control Standards	U	must be analyzed quarterly
EPA Proficiency Evaluation Study	U	must be analyzed semi-annually
Data Reduction & Reporting		
Data Reduction	U	chromatographic results should be evaluated
Chain of Custody	U	log in each sample
Unique Sample ID No.	U	identify sample ID, depth, and location
Matrix	U	record sample matrix
Date & Time of Analysis	U	record date and time of analysis
Name of Analyst/Operator	U	record name of analyst/operator
Records of Sample Preparation (e.g., dilution)	U	record dilutions
Raw Analytical Data (e.g., chromatograms)	U	available upon request
Analytical Results Table	U	present results in table
Signature of Analyst/Operator	U	sign analytical report

GC Log for QA/QC Protocol 4

Client:				Date:		Analyst:		Instrument:			
Location:				Job ID #:		Signature:		Method:			
Sample ID	Matrix	Depth	Sample ID No.	Time	Dilution Factor	Surrogate Recovery	ppb	ppb	ppb	ppb	ppb
Instrument Blank	N/A	N/A			N/A						
Method Blank	N/A	N/A			N/A						
Low STD	N/A	N/A									
Retention Time											
High STD	N/A	N/A			N/A						
Retention Time											
1)											
2)											
3)											
4)											
5)											
6)											
7)											
8)											
9)											
Duplicate											
Mid STD	N/A	N/A			N/A						
Retention Time											
Field Blank											
12)											
13)											

MS: Matrix Spike
MS Dup.: Matrix Spike Duplicate

QA/QC Protocol 5

Gas Chromatograph Analysis Using SW 846 Methods

Follow the QA/QC protocol mandated by the State Agency which certified the laboratory being used for the analysis of the subject samples.

Fixed-laboratory methods in this category, for volatile organics analysis, include (parentheses specify SW-846 Edition III replacement methods):

- EPA 8020A (8021B) Aromatic Volatiles by GC
- EPA 8240B (8260B) Volatile Organic Compounds by GC/MS
- EPA 8030A (8260B) Volatile Organic Compounds by GC/MS

Source: Test Methods for Evaluating Solid Waste: Physical/Chemical Methods (SW-846), Updates II and III, United States Environmental Protection Agency Office of Solid Waste and Emergency Response, Washington, DC.



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