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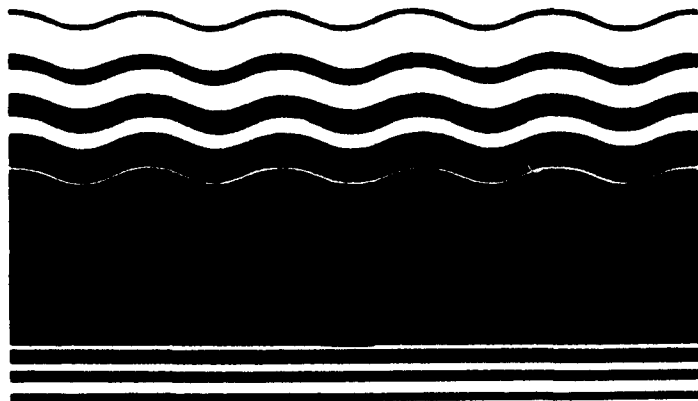
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Protocol for a Chemical Treatment Demonstration Plan

SITE

***SUPERFUND INNOVATIVE
TECHNOLOGY EVALUATION***



PROTOCOL FOR A CHEMICAL TREATMENT
DEMONSTRATION PLAN

U.S. ENVIRONMENTAL PROTECTION AGENCY
26 WEST MARTIN LUTHER KING DRIVE
CINCINNATI, OHIO 45268

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PREFACE

This document was developed for the U.S. Environmental Protection Agency by PEI Associates, Inc., under Contract No. 68-03-3413, Work Assignment No. 0-1. The document is intended to provide guidance for the development of demonstration plans for chemical treatment under the Superfund Innovative Technologies Evaluation (SITE) Program. Its purpose is to provide direction to the EPA and SITE Evaluation Contractor on the content, general format, and kinds of information that must be developed for each chemical technology demonstration, so that the demonstration can proceed in a technically sound manner.

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SECTION 1

INTRODUCTION

1.1 OVERVIEW OF SITE PROGRAM

The Congress and EPA have expressed concern over the use of land-based disposal and containment technologies to mitigate problems caused by releases of hazardous substances at Superfund sites. Through legislation, Congress has mandated a program entitled Superfund Innovative Technologies Evaluation (SITE Program) to accelerate the development and demonstration of new or innovative technologies. The emphasis of the SITE Program is on technologies that deal with the treatment or destruction of hazardous substances.

The SITE Program has four objectives:

- ° To identify and, where possible, remove impediments to the development and commercial use of alternative technologies.
- ° To conduct a demonstration program of the more promising innovative technologies to establish reliable performance and cost information for site characterization and decision making regarding technology applicability.
- ° To develop procedures and policies that encourage selection of available alternative treatment remedies at Superfund sites.
- ° To structure a development program that nurtures emerging technologies.

The protocol guidance provided by this document is offered to assist those involved in the SITE demonstration program and the development program.

Demonstration Program

The focus of the demonstration portion of the SITE Program is on pilot-scale or full-scale tests of new or innovative technologies. The technologies the EPA selects for demonstration in the SITE Program will already have undergone laboratory bench-scale testing. The main objective of the demonstration program is to collect reliable performance and cost data on

innovative alternative technologies to permit their consideration by Superfund decision-making personnel. Because the emphasis is on the collection of quality performance data, sampling and analysis procedures are of primary importance. Therefore, quality assurance/quality control (QA/QC) procedures must be strictly followed throughout the demonstration program.

The demonstration program will provide sufficient data on performance, cost-effectiveness, and reliability to allow potential users to make sound judgments regarding the applicability of a technology for a specific site and to compare its effectiveness with that of other alternatives. An evaluation of the technology demonstration will include characterizing the performance, determining the possible need for pre- and post-processing of wastes, identifying the site-specific wastes and media to which the process can be applied, determining potential operating problems and solutions, and establishing the approximate capital and operating costs. The evaluation will identify governmental policy and regulatory requirements applicable to the technology and the hazardous substances being treated/destroyed. The demonstration will permit some evaluation of long-term operating and maintenance costs.

Demonstrations will take place either at actual hazardous waste or Superfund locations or at locations that duplicate or closely simulate the wastes and conditions found at Superfund sites. This will enhance reliability of the information collected and its acceptance by the user community.

Development Program

Although the emphasis of the SITE Program is on demonstration of full-scale technologies, there is a need for further development of technologies that are not yet ready for full-scale demonstration. Such technologies may currently be undergoing continued development in the private sector; they may also include some on which further development is halted because of a limited market, inadequate funds, or perceived institutional impediments. The SITE Program will focus on those developing technologies that would meet a need at Superfund locations because of the inadequacy of existing technologies. The EPA will provide technical and financial assistance to further the development of selected promising technologies to ensure their continuing progress toward commercialization.

1.2 DEFINITION OF RESPONSIBILITIES OF EPA AND THE DEVELOPER

Under the SITE Program, private firms that have developed a technology to a point where it is ready for pilot- or full-scale demonstration may apply to EPA for participation in the SITE Program. A private firm that EPA has selected to participate in the SITE Program is referred to as the "Developer."

The EPA will also select a consulting firm to assist the Agency in conducting the SITE Program; this firm is referred to as the Contractor. The EPA, the Contractor, and the Developer will work together as a team in conducting the technology demonstrations.

The EPA's responsibility in carrying out the SITE Program entails a broad scope of work. The EPA Project Manager (PM) is responsible for overseeing the technology demonstration and for completing individual tasks as described here. Many of these tasks may be delegated to the Contractor, but the EPA PM will still have ultimate responsibility for them. The EPA PM will assign the Contractor to develop the Test Plan, Health and Safety Plan, Sampling and Analysis Plan, and Quality Assurance Project Plan for each technology demonstration. The EPA PM will review these plans and will approve them only after determining that they provide sufficient and appropriate guidance to assure the collection of quality data on technology performance and cost. The plans will be based on information the Contractor obtains from the Developer. Through the SITE Contractor, the EPA PM will document the experimental conditions and the results obtained during the demonstration and will verify that the approved plans were followed.

The Contractor will prepare a comprehensive report that includes a description of the technology, copies of the plans mentioned in the preceding paragraph, the test conditions, the performance data, cost estimates, test results, and the conclusions drawn. The Contractor will submit the report to the EPA PM for approval and revisions before it is issued as a final report. Additional details regarding EPA's and the Contractor's scope of work are addressed, as appropriate, in other sections of this document.

The Developer's responsibilities will encompass all the tasks necessary to conduct a demonstration of the technology at the selected location and

with the selected wastes. The Developer is also responsible for decontamination of equipment and personnel in accordance with EPA guidelines.

Table 1-1 lists some, but not all, of the typical responsibilities of the Developer and the EPA PM. These are described in more detail in Section 2.2 and throughout this document.

1.3 PURPOSE AND USE OF THIS PROTOCOL DOCUMENT

This document provides protocol guidance for the Test Plan, Quality Assurance Project Plan (which must include a Sampling and Analysis Plan), and Health and Safety Plan that the Contractor must prepare for each chemical technology demonstrated under the EPA SITE Program. Its purpose is to provide guidance on the content, general format, and kinds of information that must be developed for each technology demonstration. These plans are prepared to ensure that verifiable performance and cost data are collected for each technology and that the field work is conducted in accordance with recommended protocols for health and safety, sampling and analysis, and quality assurance/quality control (QA/QC). Each plan is prepared by the Contractor and submitted to the EPA PM for review. The EPA PM must approve the plans before the demonstration can proceed. This guidance document has been prepared to facilitate the preparation, review, and approval process so that the demonstration can proceed in a technically sound manner.

The generic plans presented herein are intended to serve as guides. The user will need to modify the contents to fit the specific technology involved. EPA recognizes that, especially with regard to chemical technologies, even a single process may be configured in several different ways to meet the diverse requirements for treatment in different hazardous waste scenarios. The examples provided throughout this document do not collectively represent any single technology.

This protocol guidance is organized into three main sections following Section 2, which covers generic factors affecting a technology demonstration. Section 3 presents a sample Test Plan, Section 4 a sample Health and Safety Plan, and Section 5 a sample Quality Assurance Project Plan.

The Test Plan describes the technology, scope of work, and technical approach, and outlines the schedule, management and staffing, and budget. The Health and Safety Plan addresses protocols for personnel protection, the levels of protection required, and decontamination procedures. The Quality

TABLE 1-1. AREAS OF RESPONSIBILITY OF THE EPA PM
AND THE DEVELOPER UNDER THE SITE PROGRAM

EPA	Developer
<u>Initial work items</u>	Provide technology description
Organize kickoff meeting	Conduct initial site visit
Take baseline samples for site characterization or pretreatment needs	Attend kickoff meeting
Assist Developer in acquiring required permits	Supply "list of needs" to EPA
<u>Demonstration plan</u>	Meet permitting and other regulatory requirements
Coordinate, review, and approve Sampling & Analysis (S&A) Plan	Implement H&S program for Developer's personnel
Develop Health & Safety (H&S) Plan for EPA personnel	Pretreat feed material
Coordinate overall H&S program	Bear costs of operating the technology during the demonstration
Develop and approve QA/QC program	
<u>Site preparation</u>	Decontaminate and demobilize equipment
Prepare site in accordance with Developer's "List of Needs"	
Excavate waste material and bring to process developers	
<u>Demonstration</u>	
Carry out S&A in accordance with QAPP	
Provide H&S training	
Arrange for QA/QC audit	
Supply site H&S officer	
<u>Post demonstration site cleanup</u>	
Arrange for disposal of waste material	
<u>Report preparation</u>	
Prepare S&A report	
Prepare final technology evaluation report	

Assurance Project Plan, which includes a Sampling and Analysis Plan, addresses the essential elements for the collection of representative samples by acceptable reproducible methods; the selection of standard methods for analysis, chain-of-custody, and sample preservation; and Category II QA/QC requirements (blanks, matrix spike, matrix spike duplicates, and other required protocols). The Quality Assurance Project Plans are of utmost importance in the SITE Program technology demonstrations. Strict adherence to the approved plans is essential to obtaining quality performance data on each technology.

For the convenience of the user, the three plans are presented in a manner that permits the use of one plan at a time. Users should follow the protocol guidance presented for the general organization and scope and then adapt specific details to meet their own particular technology requirements.

SECTION 2

FACTORS AFFECTING TECHNOLOGY DEMONSTRATION

Several factors affect the planning and execution of a technology demonstration. The EPA and the Developer must recognize and consider these factors in the development of the Test, Health and Safety, and Quality Assurance Project Plans. The factors are briefly discussed in this section and referred to throughout this document.

2.1 PROCESS CHARACTERISTICS

Several process characteristics affect the duration, cost, and complexity of a technology demonstration. For example, one such characteristic is whether the process operates in a batch or continuous mode. For batch processes, data from several consistently operated runs must be collected to provide enough data for comprehensive statistical analysis. For continuous processes, some initial run time is required to allow the system to reach equilibrium. Thereafter, the testing begins and continues until a predetermined number of samples of various types have been collected to provide statistically significant data. If a process upset occurs, the process must be restarted and allowed to reach equilibrium again before more sampling can be conducted.

The size, type, and configuration of equipment affect implementation of the technology demonstration. For example, the use of a large mixer might require the construction of a concrete foundation, pad, and berms before the mixer can be moved to the site. Heavy equipment (cranes, etc.) may also be required. Sufficient time and resources must be provided to accommodate such preparatory work. Demobilization of large equipment can also be complex if heavy equipment is required.

The variability of process performance should be defined and considered in designing the test plans. The greater the variability in a process, the greater the duration of the test and the number of samples required to obtain scientifically valid data.

The effect of variations in the process should be assessed. For example, if changes in the waste feed material will significantly affect performance, some pretreatment in the form of sizing, crushing, blending, drying, chemical addition, or other steps may be warranted. The Developer will advise EPA of the need for pretreatment of waste feed.

Another factor that will affect the technology demonstration is the Developer's budget. The Test Plan and QA Project Plan (Sampling and Analysis) will define the number and duration of runs and the required number of samples (based on the amount of data needed to conduct a scientifically valid evaluation). If the cost of conducting the demonstration according to the EPA-approved plans exceeds budget, the Developer may want to decrease the test duration. The EPA must ensure, however, that plan modifications to meet budget constraints will still result in a valid demonstration that complies with SITE Program objectives.

2.2 TESTING SITE ACCEPTABILITY

In addition to the technology requirements of a suitable location for a SITE Program technology demonstration (discussed in Section 3), several nontechnical site characteristics must be considered early in the planning. These relate to the political acceptability of conducting experiments at a particular site and whether permits must be obtained.

As part of the SITE Program, the SITE personnel will work with the Regional Community Relations person to meet with the community before a demonstration. The SITE personnel or Regional Community Relations person will explain the scope, purpose, and applications of the technology to be demonstrated, as well as the need for the overall program.

In some cases, it may be necessary to obtain permits to conduct the demonstration. The EPA will determine the need for permits; however, the Developer will be responsible for obtaining them. The applications for any required permits should be prepared and submitted early in the planning. No permitting will be necessary for demonstrations carried out at NPL Superfund sites. Nevertheless, the Office of Solid Waste and Emergency Response

(OSWER) will conform to the functional equivalent of any applicable or relevant and appropriate laws and regulations, as required by the Superfund Amendments and Reauthorization Act (SARA) of 1986 or by the National Oil and Hazardous Substances Pollution Contingency Plan (NCP)(40 CFR Part 300).

Permits will be required for demonstrations conducted either at offsite private facilities owned by the Developer (e.g., at its manufacturing or research facility) or at a commercial offsite facility. In these cases, the Developer or facility owner will be responsible for obtaining the permit; however, OSWER may provide some assistance.

Occasionally, demonstrations will be conducted at EPA Office of Research and Development (ORD) Test and Evaluation (T&E) facilities. This will be determined by the safety requirements, the need for specialized equipment or facilities, the QA/QC needs, or cost considerations. T&E facilities should already have basic permits that will require, at most, only modification for demonstration activities. In these cases, ORD will be responsible for obtaining any permit modifications. Such facilities also have modern pollution control and safety equipment in place, which means that technologies can be tested for full determination of their capabilities without fear of pollutant releases. These facilities therefore would be readily available for moderate-sized demonstration activities under safe and controlled conditions and at a lower cost. In some cases, EPA believes that tests at a T&E facility may be necessary prior to a field demonstration to determine appropriate design details or operating conditions. Such tests can also be used as a post-test, followup study to determine the flexibility of a technology to treat additional wastes and/or media. These tests might be conducted with the demonstration equipment or with pilot-scale equipment. In any event, the demonstration will normally be conducted on waste obtained from a hazardous waste site. The results of these demonstrations must be shown to be applicable to "real world" situations at actual Superfund sites regardless of where the demonstration is performed.

The Office of Research and Development is currently considering several facilities for possible development as T&E facilities for SITE demonstrations, e.g., the Mill Creek T&E Facility and the Center Hill T&E Facility, Cincinnati, Ohio; the Combustion Research Facility (CRF), Pine Bluff, Arkansas; the Edison, New Jersey, facility; and the Air and Energy Engineering Research Laboratory, Research Triangle Park, North Carolina.

The permits that may be required include both Federal and State permits under RCRA (RCRA manifests, RD&D permits, TSDF permits, generator permits), NPDES permits, and air permits. The Developer will provide auxiliary equipment or modify the experimental system as necessary to meet permit requirements.

SECTION 3

TEST PLAN

3.1 TECHNOLOGY DESCRIPTION

The EPA PM and the Contractor should review the plans for the technology demonstration with the Developer and prepare a list of specific objectives for the demonstration design. After defining the project objectives, the EPA PM should review the objectives of the SITE Program to ensure that the project goals are consistent with the overall SITE Program goals. The specific project objectives must further the objectives of the SITE Program.

The Developer will develop the technology description, including an engineering design for the field demonstration, and the EPA PM will review the design for completeness, technical adequacy, and applicability.

The Developer should review existing performance data on the technology to provide information enabling rational selection of design assumptions. For example, previous work on a chemical treatment process may have indicated a pH range necessary for the process to proceed; thus, the design would include a determination of the required pH adjustments.

The EPA PM, the Contractor, and the Developer should determine the need for presampling and analysis to characterize the site more fully before the demonstration is begun. The EPA will conduct all sampling and analysis to determine the Developer's need to pretreat any material and for site characterization. Developers are encouraged to conduct any additional sampling and analysis for their own benefit. If these sampling efforts indicate that waste pretreatment is required, EPA will conduct the pretreatment work.

The operating conditions, which will be addressed in the design, include waste characterization. For example, a particular chemical dechlorination process may be applicable to a wide variety of halogenated organic compounds, including PCBs, ethylene dibromide, chlorinated dioxins, and chlorinated dibenzofurans; however, the same process may not adequately treat hydroxylated aromatic compounds such as pentachlorophenol, hexachlorophene, and phenols.

The Developer should define the operating parameters, which will be reviewed carefully by EPA. In a study of a chemical degradation process for PCBs, the following daily operational parameters were determined:^{*}

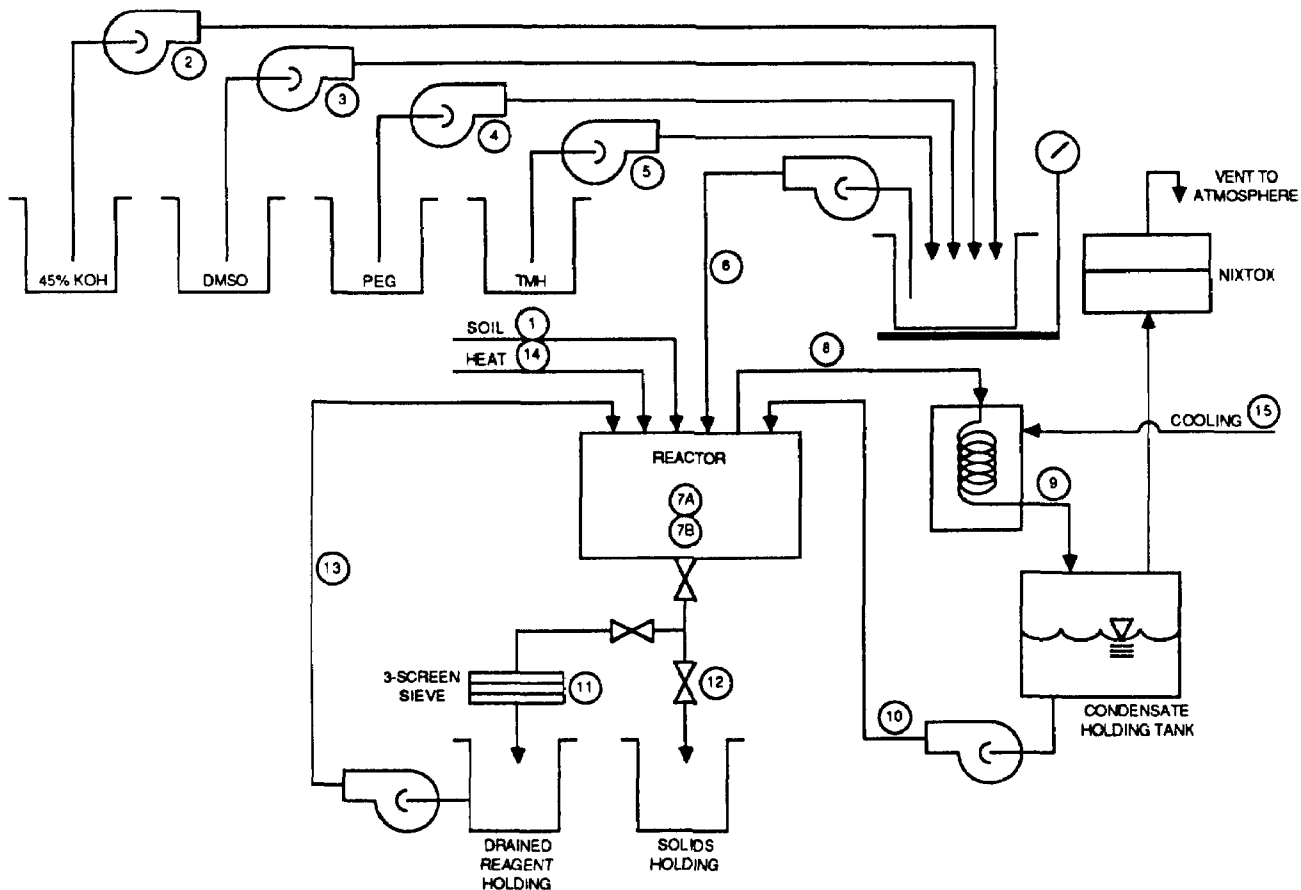
- 1) Weight of contaminated soil charged to the mixer.
- 2) Weight of each reagent chemical charged to the mixer.
- 3) The pH of soil and reagent mixture in the reactor before and after heat-up.
- 4) Temperature of mixer contents monitored with time during heat-up, hold, and cool-down.
- 5) Volume of condensate collected during process.
- 6) Volume of reagent recovered from treated soil.
- 7) Volume of acid added to soil to adjust pH to pH 9.
- 8) Weight of treated soil discharged from mixer.

The development of mass and energy balances around a process is essential to the design because it provides the database needed for selection and sizing of the equipment and instrumentation. Figure 3-1 is a schematic showing the unit operations in a treatment process. The tabular information at the bottom of the figure shows the amounts of soil and reagents that go into and out of each unit.

A flow diagram that includes mass and energy balances allows the design engineer to specify instrumentation. These specifications should be detailed enough to include types and capacities of pumps and gauges and other instruments. Piping and instrumentation diagrams should show the configuration, sizing, and materials of construction (PVC, carbon steel, stainless steel, etc.) of the piping.

The equipment specifications should address the various types of equipment, the size of each unit, the material of construction, and the location

^{*} Copa, W. M., Ph.D., et al. Powdered Activated Carbon Treatment (PACTTM) of Leachate from the Stringfellow Quarry. Taken From Incineration and Treatment of Hazardous Waste. Proceedings of the Eleventh Annual Research Symposium. EPA 600/9-85-028. September 1985.



NOTE: ITEM NO. 7A REPRESENTS REACTOR CONTENTS PRIOR TO HEATING
ITEM NO. 7B REPRESENTS REACTOR CONTENTS AFTER HEATING

TOTAL REACTOR CAPACITY: 4.6 ft³
WORKING REACTOR CAPACITY: 2.76 ft³

	(1)	(2)	(3)	(4)	(5)	(6)	(7A)	(7B)	(8)	(9)	(10)	(11) & (13)	(12)	HEATING UNIT (14)
TOTAL SOIL	107.7	-	-	-	-	-	107.7	96.9	-	-	-	-	96.9	50,000 Btu/hr
DRY SOIL	96.9	-	-	-	-	-	96.9	-	-	-	-	-	-	50 lb/hr STEAM
WATER IN SOIL	10.8	-	-	-	-	-	10.8	-	10.8	-	-	-	-	CONDENSER UNIT (15)
TOTAL KOH	-	35.9	-	-	-	35.9	35.9	18.2	-	-	-	8.1	8.1	30,000 Btu/hr
DRY KOH	-	18.2	-	-	-	16.2	16.2	-	-	-	-	-	-	
WATER IN KOH	-	19.7	-	-	-	19.7	19.7	-	19.7	-	-	-	-	
DMSO	-	-	35.9	-	-	35.9	35.9	35.9	-	-	-	17.8	17.8	
PEG	-	-	-	18.0	-	18.0	18.0	18.0	-	-	-	9.0	9.0	
TMH	-	-	-	-	18.0	18.0	18.0	18.0	-	-	-	9.0	9.0	
TOTAL WATER	10.8	19.7	-	-	-	19.7	30.5	0	30.5	-	-	15.3	15.3	

ALL VALUES ARE pounds PER BATCH REACTOR

Figure 3-1. Schematic of unit operations in a treatment process.

of each piece in the overall process. They should also include the manufacturer (supplier).

The equipment specifications usually include a waste feed system unless the feed is manual or the technology involves an in situ process. If a waste feed system does exist, the Developer should characterize it similarly to the rest of the process (i.e., supply unit dimensions, method of operation, construction materials, etc.). The EPA PM should check all piping, equipment, and instrumentation specifications for waste, reagent, and product compatibility.

A pollution control system may be required to control volatile emissions, particulates, runoff, seepage, releases, etc. The complexity of such controls varies with the process. An in situ soil treatment might require control of runoff in the event of a heavy rain. Such control might consist of a proper slope of the soil plot and a runoff collection system. Pollution controls on a pressurized pilot-scale reactor, however, may include a condenser followed by a molecular sieve and activated carbon to collect and treat emissions. The EPA PM should review the pollution controls to ensure compliance with applicable regulations.

The utilities necessary to conduct the demonstration should be specified in the design so that the site can be adequately prepared for the demonstration. Specific requirements for water, electricity, steam, etc, must be addressed in the actual equipment design, such as specific voltage and wat-tage requirements, or required water supply flow rates.

The Developer's design should include a site layout showing the location and approximate size of each piece of equipment, as well as support equipment and supplies (e.g., a lab trailer, office trailer, decontamination station, waste storage area, drums, tanks, parking area). The layout also should include the contaminated and the uncontaminated zones, if they are sufficiently defined at this stage.

While demonstrating performance of a treatment technology is the primary objective of the demonstration program, another important objective is to estimate the cost of the technology. The summary in Table 3-1 is an example of a cost estimate for an electric arc furnace (EAF) recycling process. This estimate presents both capital and operating costs for three plant sizes.

TABLE 3-1. CAPITAL AND OPERATING COSTS FOR EAF RECYCLING PROCESS^a

Cost component	Small facility costs	Medium facility costs	Large facility costs
<u>Capital (Installed) Costs</u>			
Fixed capital investment (FCI)	\$70,000	\$130,000	\$200,000
Working capital costs (15% of FCI)	10,500	19,500	30,000
TOTAL CAPITAL COST	\$80,500	\$149,500	\$230,000
<u>Annualized Costs</u>			
Annualized capital cost (5 yr at 12%)	\$22,500	\$ 41,700	\$ 64,220
<u>Operating costs</u>			
Raw material	0	0	0
O&M labor ^b	33,280	33,280	66,560
Electricity	2,000	6,000	18,000
Water	250	750	2,250
O&M supplies (10% of FCI)	7,000	13,000	20,000
Lab charges (\$1/ton)	1,000	3,000	9,000
<u>Miscellaneous costs/credits</u>			
Local taxes and insurance (3% of FCI)	2,100	3,900	6,000
Plant overhead (15% of operating costs)	6,500	8,400	17,370
Home office management and administration (25% of operating costs)	10,900	14,000	28,950
Credit for sale of high zinc dust (\$10/ton)	(10,000)	(30,000)	(90,000)
Transport of high zinc dust	10,000	30,000	90,000
TOTAL ANNUALIZED COST	\$85,530/yr	\$124,030/yr	\$232,350/yr
Unit cost for processed dust	\$85.50/ton	\$41.34/ton	\$25.82/ton

^a Adapted from E. R. Krishnan et al. PEI Associates, Inc.^b Includes onsite supervision and clerical, 2080 h/yr/person at \$16/h.

Capital costs generally include engineering design, equipment purchase, and installation/fabrication; operating costs include operation and maintenance. Where possible, life-cycle cost estimates should be included.

The following is an example of a technology description:

"Chemical Treatment of Chlorinated Organics in Soils Using Potassium Polyethylene Glycol (KPEG)"

Chlorinated dioxins (such as TCDD) and polychlorinated biphenyl (PCB) wastes and contamination of water and soils are a serious concern in many areas of the country because of the high toxicity of these compounds and the very limited treatment or disposal options currently available for them. Some laboratory-scale research has been completed on a chemical destruction technique to dechlorinate dioxins and PCBs and render them far less hazardous. The chemical decontamination process has been successfully demonstrated on a small scale on contaminated soils. The process involves the addition of potassium polyethylene glycol (KPEG) to contaminated soils. The chlorinated dioxins or PCBs dechlorinate to form water-soluble compounds under relatively mild conditions of temperature and pressure. The soils are then washed counter currently with water to remove the soluble dechlorinated products and to recover the reagents.

The basics of chemical soil decontamination are straightforward. Contaminated soil is mixed with an alkaline reagent consisting of potassium hydroxide in a solution of mixed polyethylene glycol and dimethyl sulfide. The reagent mixture dechlorinates the aryl halide to form a PEG ether, which may further degrade to form a totally dechlorinated species. This mechanism is shown in Figure 3-2. A schematic diagram of the process is shown in Figure 3-3.

3.2 TESTING APPROACH

The Test Plan should identify all the controls and variables in the process and indicate any variables to be tested during the demonstration. In a chemical treatment technology, reagent ratios and pressure may be held constant while reaction vessel temperature is varied. Or the reagent mixture may be changed while all other variables (temperature, pressure, mixing time, etc.) are held constant. By manipulating only one variable at a time and collecting sufficient performance data for that particular scenario before it is changed, reliable data on system performance will be generated.

The Test Plan should define the range of performance for each variable tested; if temperature is a variable, the plan should state (for example) that Runs 1 through 10 will be conducted at 100°C, Runs 11 through 20 will be

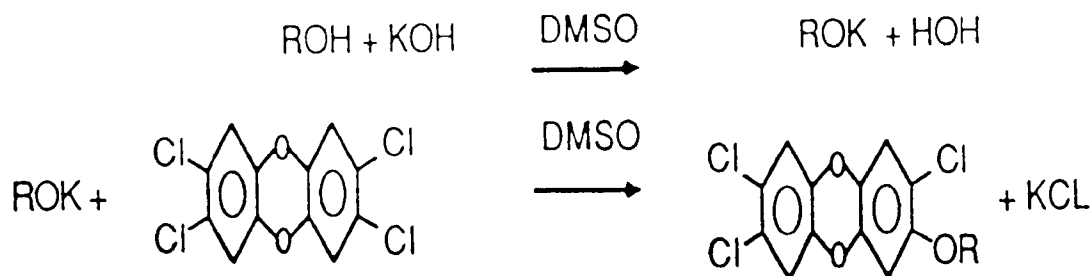


FIGURE 3-2. PROPOSED REACTION MECHANISM.

The soil/reagent mixture is heated to 30°C to 150°C, and mixing takes place until the reaction has been completed. At the end of the reaction, reagent is recovered by decantation and washing the soil with several volumes of water. The decontaminated soil is then discharged and the reagent is recycled for reuse, as shown in the process diagram in Figure 3-3.

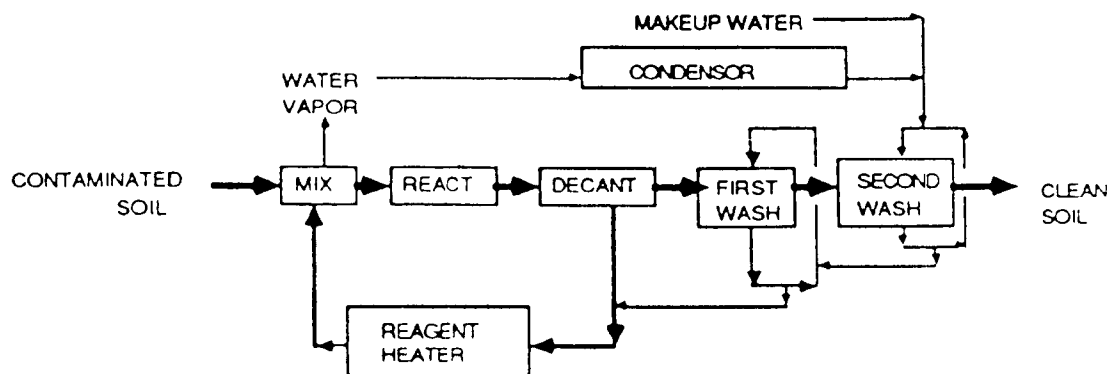


FIGURE 3-3. PROCESS DIAGRAM.

The KPEG treatment is capable of detoxifying or destroying a wide variety of halogenated organic compounds, including PCBs, ethylene dibromide, chlorinated dioxins, and chlorinated dibenzofurans; exceptions include hydroxylated aromatic compounds such as pentachlorophenol, hexachlorophene, and phenols and related compounds such as 2,4-D and 2,4,5-T.

conducted at 140°C, and Runs 21 through 30 will be conducted at 180°C. Ranges for control parameters also should be defined. If pressure is identified as a constant, the plan should state for example that the process will be run at a slight pressure of 5 to 10 psi. It should also state the basis for concluding that operation within a defined pressure range does not significantly affect the process so that the EPA PM can judge whether or not such a factor is indeed a control parameter.

The Test Plan should list the controls, the variable to be changed, and the expected range of performance for each test run in the field demonstration.

Sampling and analysis are key steps in the SITE Program demonstrations. Every unit operation input and output should be sampled so that changes in the process can be characterized. Figure 3-4 presents a simplified example. Samples would be taken at each point identified by a number. In some cases, the reactor must be sampled many times. If a reaction occurs in steps or over a period of time, it may be advisable to take samples at periodic intervals (e.g., every 15 minutes or every hour) to provide sufficient data for development of a reaction curve. If progressive dechlorination occurs, analysis of the concentrations of each of the subsequent dechlorination intermediates would provide valuable data on the kinetics of the process that might be used later for further optimization of the process. Operating parameters (i.e., temperature, pressure, mixing speed, etc.) also should be monitored so that data on all controls and variables are recorded and documented. Ambient air monitoring at most field demonstrations is also recommended as a safety measure for determining any potential exposure of the field crew to air toxics. The specific parameters monitored will vary with the technology; however, the main contaminants generally should be measured, as well as the more general indicators such as volatile organic compounds (VOCs) or particulates.

3.3 FIELD DEMONSTRATION PREPARATION

The Developer has primary responsibility for the field demonstration, and the EPA PM is responsible for overseeing these efforts. The Developer will identify the resources required for conducting the field demonstration

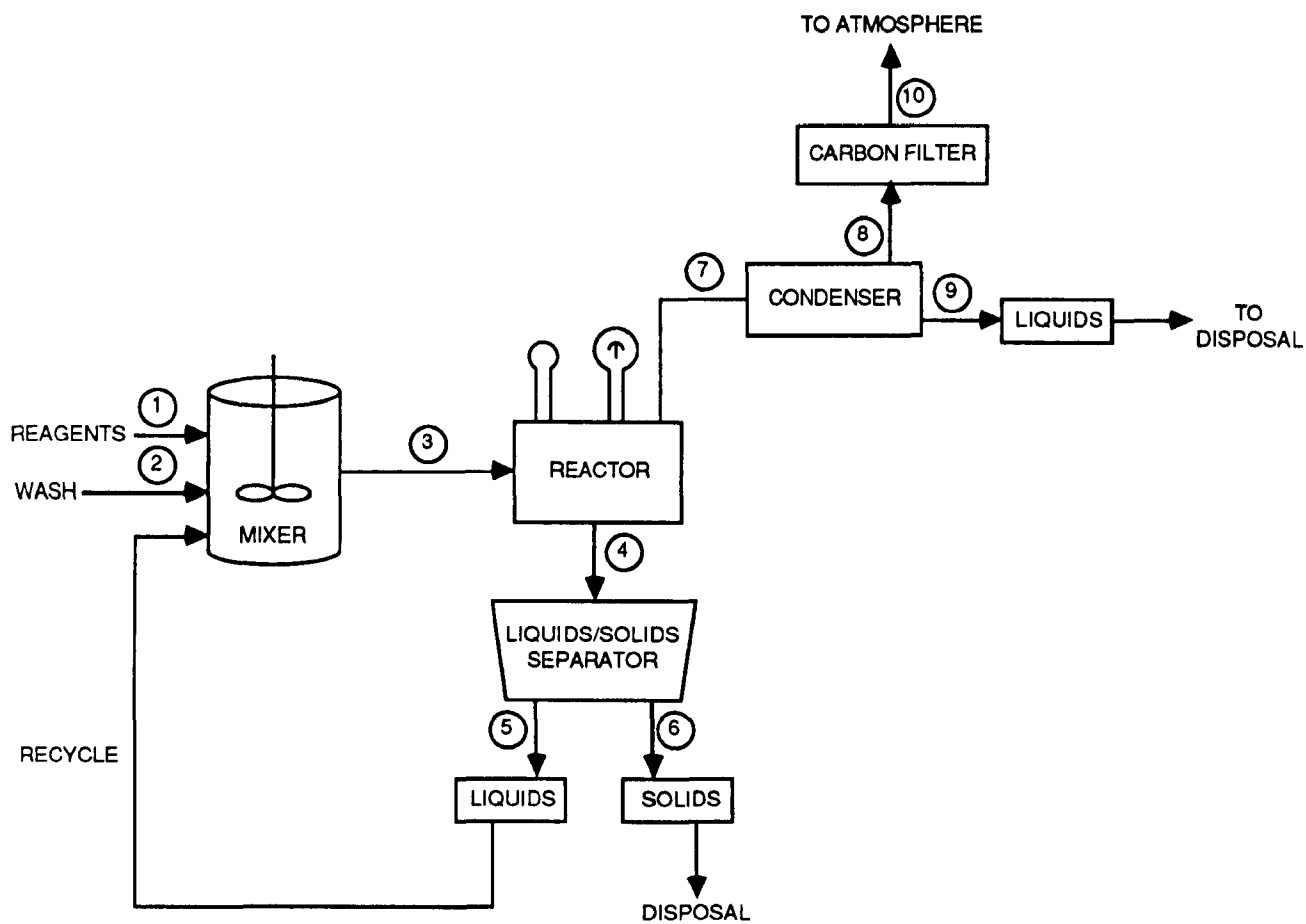


Figure 3-4. Location of sampling points in a hypothetical chemical treatment process.

and will document them in the form of a "needs list." The needs list can be divided into site-preparation needs and technology-demonstration needs.

The site preparation needs list should identify the items or site characteristics necessary for the performance of a technology demonstration. The list might include such items as:

- 1) Suitable waste supply. Quantity and quality of waste needed and any seasonal restrictions on obtaining or using the waste should be defined.
- 2) Access roads sufficient for the equipment.
- 3) A firm and level site for all equipment.
- 4) Availability and proximity of necessary support services or skills.
- 5) Site security to prevent accidents and to minimize vandalism.

The technology-demonstration needs list can be developed under the main categories of utilities, facilities, equipment, and supplies. The technology demonstration needs list includes items such as the following:

- 1) Utilities
 - Fuel (liquid propane, natural gas, diesel oil, gasoline, kerosene, etc.)
 - Water (city, well, or bottled drinking water; deionized water; softened water; etc.)
 - Electricity (utility-supplied or generator-supplied, 110V, 220V, or 440V, etc.)
 - Telephone (portable or stationary)
- 2) Facilities
 - Waste storage and handling areas (sheds, bermed/lined pits, etc.)
 - Residual storage area (bermed/lined pits, etc.)
 - Mobile or stationary laboratory
 - Personnel changing, eating, and showering areas (lockers, showers, etc.)
 - Office
 - Decontamination station
 - Secure storage area for equipment and supplies
 - Appropriate on- or offsite disposal facilities (landfills, incinerators, etc.)

3) Equipment:

- Heavy equipment (bulldozers, cranes, backhoes, drum grapplers, forklifts, etc.)
- Experiment-related equipment (reactors, agitators, tanks, drums, dumpsters, conveyors, scaffolding, scales, etc.)
- Sampling and analytical equipment (samplers, gas chromatograph, atomic absorption spectrophotometer, pH meter, balance, etc.)
- Safety equipment (shower, eye-wash station, fire extinguisher, etc.)

4) Supplies:

- Reagents
- Chemicals
- Personal protective equipment
- Sampling bottles, labels, forms, notebooks, etc.
- Office supplies

The EPA is responsible for all site preparation, including but not limited to such things as gaining permission to enter the site, arranging for physical access to the site, utility hookups, secure storage areas for equipment, providing a cleared and level work site, and conducting any necessary excavations to bring contaminated waste to the technology demonstration unit. In addition, the EPA must obtain waste for pretreatment and conduct sampling and analysis to characterize the waste. The Developer will estimate types and quantities of wastes and byproducts for which EPA must arrange disposal. EPA will arrange for suitable storage, treatment, and disposal of all residues or byproducts.

3.4 FIELD DEMONSTRATION IMPLEMENTATION

The Developer is responsible for the performance and staffing of this task, and the SITE Contractor is responsible for overseeing it for the EPA. The task includes setup, shakedown, test runs, decontamination, and demobilization of the field demonstration equipment. The EPA will provide the staff for monitoring and evaluating the demonstration.

When the Developer's team arrives at the site, the EPA PM will establish routines for implementing the health and safety, sampling and analysis, QA/QC, and documentation procedures as explained in the plans, and review them with the field crew. This review by the EPA PM is conducted in addition to OSHA-required training.

The Contractor will set up a decontamination station and provide appropriate supplies, the Developer will be responsible for proper decontamination of its personnel and equipment, and the EPA will supervise decontamination operations.

The final aspect of the field demonstration will be demobilization of equipment and personnel. Rented supplies and equipment will be returned by appropriate parties. EPA will return their own rental equipment. A checklist should be developed for all major equipment to ensure protection of the equipment during demobilization.

3.5 DOCUMENT TESTS AND PREPARE REPORTS

Inasmuch as the overall objective of the SITE Program is to provide valid performance and cost data on the field demonstrations, the written documentation and final reports will serve as the "product" of the Program.

The EPA has primary responsibility for documentation of the technology demonstration. The EPA Contractor will prepare data sheets specific to the tests to be run. Monitoring data sheets, sampling data sheets, and analytical results sheets be prepared and filled out for each run. In addition, each member of the field team will be issued a numbered daily logbook in which to document test conditions, samples taken, problems encountered, corrective actions taken, results obtained (if known at the time), and procedures. The EPA will document the extent to which all provisions in the Test Plan, Health and Safety Plan, and QA Project Plan are followed. Any deviations from the approved plans must be documented, along with the reasons for these deviations and any known or likely impacts on the process. The daily logbooks should describe any equipment or instrumentation failures or operating difficulties and document all instruments readings during process operation. Pictures, slides, and/or video tapes will be prepared as appropriate, and will be made part of the permanent documentation file.

The EPA will prepare data summaries with all monitoring and analysis results to be used ultimately in the comprehensive reports prepared by the EPA. The technical report should address, at a minimum, the following topics:

- Description of technology
- Objectives of field demonstration
- Design
- Operations
- Results - performance
- Results - costs
- Conclusions and recommendations
- Appendices
 - Test Plan
 - Quality Assurance Project Plan

The Technical Report will be reviewed by the EPA PM and (as directed) others in EPA Headquarters, Regional offices, State agencies, the Developer's firm, or other involved parties. In some instances, EPA may request a peer review.

The review comments of various parties will be submitted to the EPA PM, who will direct the EPA Contractor to respond to them as appropriate. The EPA Contractor will then submit a revised final report to the EPA PM.

In addition to the technical report, EPA intends to develop an Application Analysis Report describing the types of wastes that were treated during the technology demonstration and the types of wastes that might be treated. The objective is to provide information about applications of the technology to other waste/matrix scenarios.

3.6 MANAGEMENT AND SCHEDULE

The EPA PM, the EPA Contractor, and the Developer will all participate in the SITE Program technology demonstrations. Each party has specific responsibilities, as summarized in Figure 3-5. Overall responsibility for management lies with the EPA PM; however, EPA will delegate the execution of many of the task areas to the SITE Contractor. The EPA will also be responsible for approving all plans, procedures, and reports.

The Developer will design the equipment, prepare it for the field demonstration, and operate the process. Responsibilities also include decontamination and demobilization after the demonstration is completed.

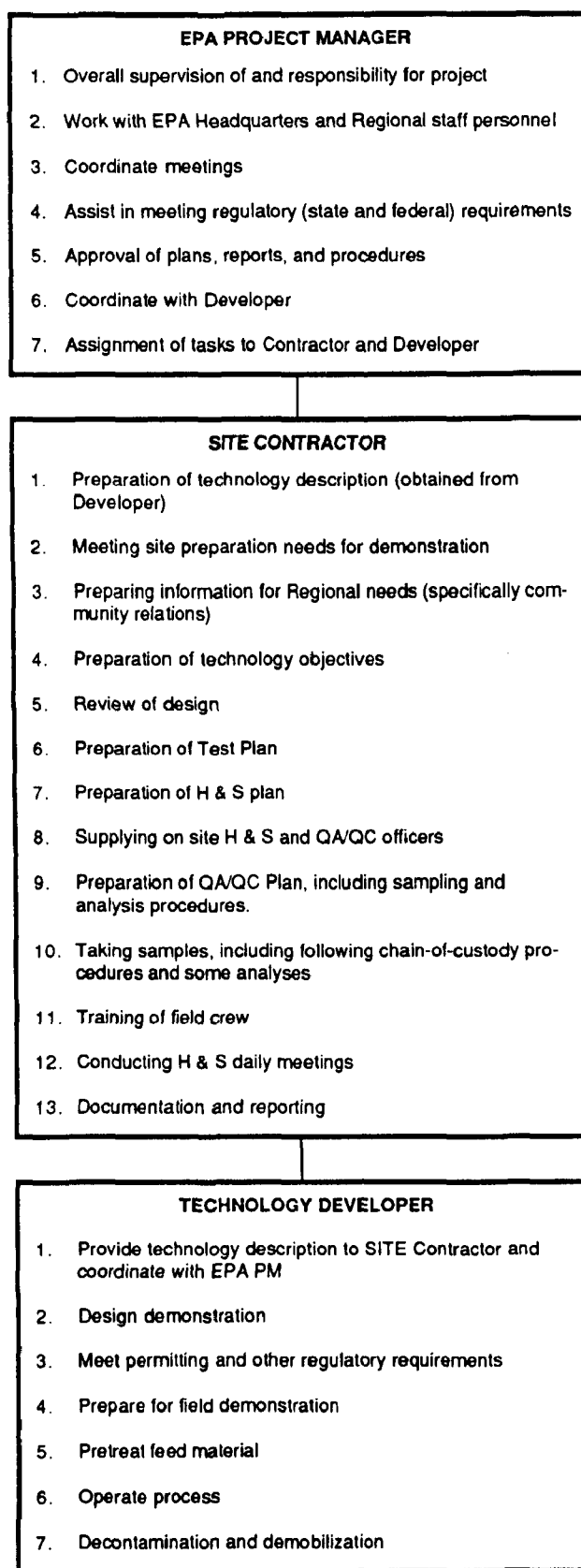


Figure 3-5. Organization of responsibilities for field demonstrations conducted under the EPA SITE Program.

Figure 3-6 is a sample schedule to serve as a guide for developing the specific schedule for a given demonstration. An individual project may take more or less time than that shown in the figure, depending on such factors as the complexity of the technology, the reaction time of the process, and the need to obtain permits.

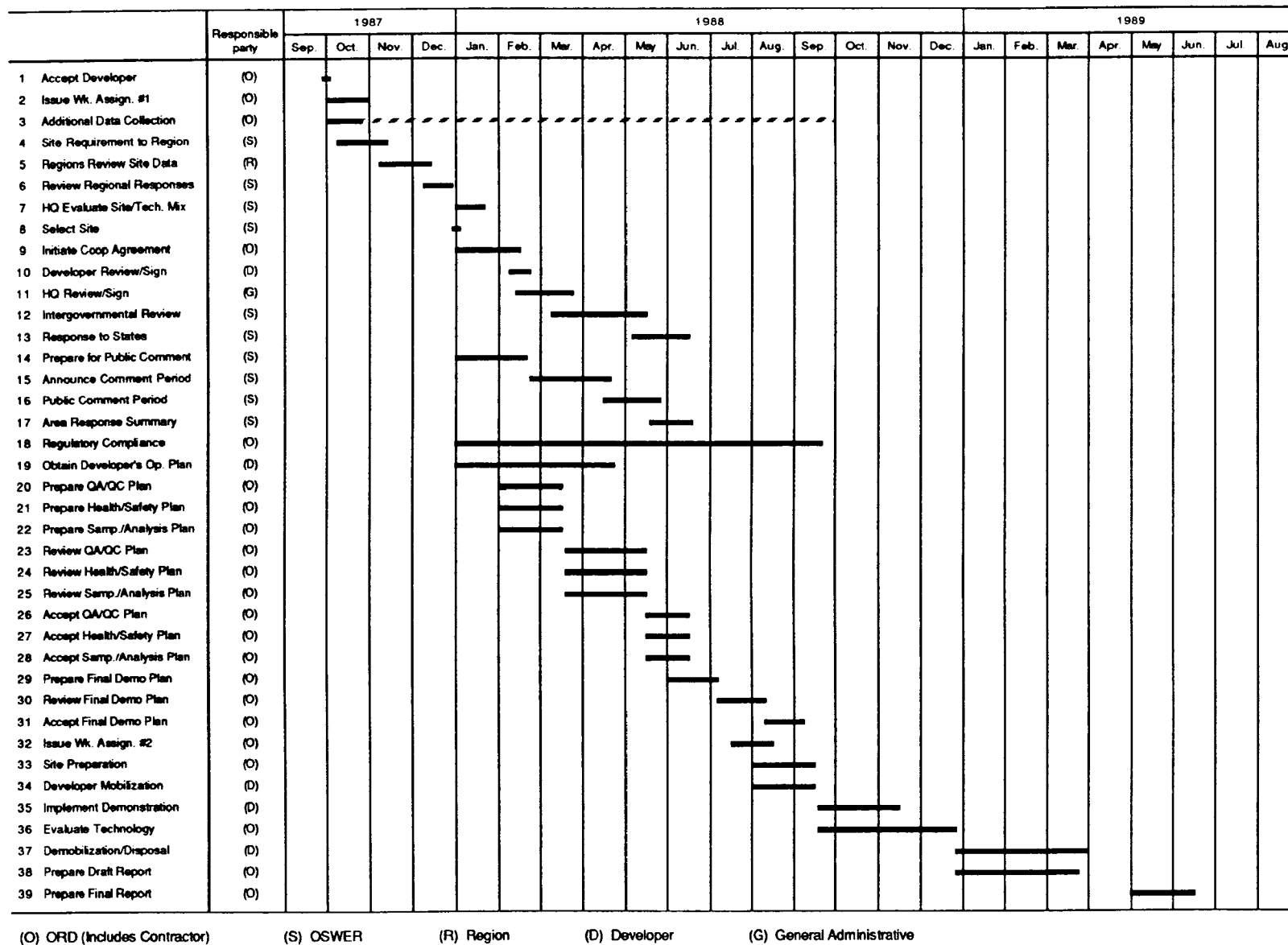


Figure 3-6. Project schedule.

SECTION 4

GUIDELINE DOCUMENT FOR HEALTH AND SAFETY PLAN FOR EVALUATION OF CHEMICAL TECHNOLOGIES

4.1 INTRODUCTION

The contents of a Health and Safety (H&S) Plan will vary from project to project; however, it must include key elements to enable the preparation of a complete health and safety program. The purpose of this generic H&S Plan is to provide a framework for the formulation of a site- or project-specific H&S Plan. It is important to note that any contractor is responsible for the health and safety of his/her employees; this includes medical monitoring.

4.2 PROJECT DESCRIPTION

Location

The H&S Plan should include a detailed description of the location of the technology demonstration and the surrounding geographic area. In addition to a written description, it should include maps showing the demonstration area and the general surrounding area. These maps should highlight roads, railroads, airports, hospitals, fire departments, and the locations of other emergency equipment.

If the demonstration is housed indoors, the square footage of the building(s) involved in the project also must be described. If the demonstration is slated for a specific site (i.e., a landfill), the affected area must be designated. The description should also note such site-specific characteristics as:

- Adjacent buildings (or rooms).
- Presence of structures other than buildings (towers, etc.).
- Presence of other wastes or reagents (not a part of the demonstration).

- ° Identification of contamination and decontamination zones.
- ° Presence of heavy equipment dedicated to the site.
- ° Location of the waste to be treated.

In addition to site-specific information, the H&S Plan should consider the surrounding area and its population. In a worst-case accident scenario, local residents and property will be affected. This point is especially important in an outdoor demonstration.

Process Description

A chemical process is a series of unit operations and/or unit processes, the products (and byproducts) of which result from the following:

1. One or more chemical or physiochemical changes.
2. The extraction, separation, or purification of a product with or without the aid of chemical reactions.
3. The preparation of a specific product from a mixture of materials (either natural or synthetic in origin).

For the SITE Program, the chemical technology will use the waste as a feed stream and transform the hazardous constituents of the waste into a more desirable form.

The type of process used in the technology will affect its operation and inherent hazards. A chemical process may be operated in either a batch or continuous mode. In a batch process, the feed (the waste) is charged into the system at the beginning of the process and the products are removed simultaneously some time later.¹ In a continuous process, the inputs and outputs flow continuously throughout the duration of the processing operation.¹

Either type of process may be operated safely; however, a continuous process is more difficult to control than a batch process. The lessened degree of control can tend to compound hazards during the processing of waste. The designated Safety Officer should note this tendency and be familiar with the chemical process and its operations before the onsite testing is begun.

A chemical process may use one or more of the following pieces of equipment:²

- Columns
- Vessels
- Reactor
- Heat Exchangers
- Pumps and Compressors
- Process Instrumentation

The Safety Officer should identify all process equipment used in a given chemical technology and define any associated hazards.

4.3 PROJECT OBJECTIVES

In this section, the H&S Plan will outline the project and its objectives. The project objectives should be clearly summarized early in the H&S Plan so that users can review the H&S procedures with the objectives in mind. All onsite personnel should be aware of the process and its operating characteristics (heat, odors, noise, etc.); the number (and length) of trial runs in the project; specific waste components; waste byproducts produced by processing; project milestones; and the impact of this project on waste treatment. Each worker should be oriented to the health and safety factors pertaining to each phase of the project. All onsite personnel should be made aware of any changes to project outlines or scope and the effect of the changes on the health and safety procedures. Having been educated on all aspects of the project, a worker will be better equipped to make an intelligent decision in the event of an accident or emergency.

4.4 SAFE WORK PRACTICES

The major goal of a H&S Plan is to inform personnel of the techniques and/or equipment that will guard them from site-specific hazards. Common safe work practices are equally important during day-to-day operations. Such safe work practices, as described in OSHA/EPA's "Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities," include, but are not limited to the following:

- 1) A daily safety meeting should be held to inform and review with workers the activities planned for the day and the associated safety issues.

- 2) Eating, drinking, chewing gum or tobacco, smoking, or any practice that increases the probability of hand-to-mouth transfer and ingestion of material is prohibited in any area designated as contaminated.
- 3) At a minimum, personnel must wash their hands and faces thoroughly upon leaving the work area. For those undergoing higher degrees of potential exposure, full showers (or baths) are required.
- 4) Whenever decontamination procedures for outer garments are in effect, the entire body should be thoroughly washed as soon as possible after the protective garment is removed and disposed of in a proper manner.
- 5) No facial hair that interferes with a satisfactory fit of the mask-to-face seal is allowed on personnel required to wear respirators.
- 6) Contact with contaminated or suspected contaminated surfaces should be avoided when possible. Personnel should avoid walking through puddles, leachate, or discolored surfaces; kneeling on ground; and leaning, sitting, or placing equipment on drums, process equipment, or the ground/flooring.
- 7) Because some medicines and alcohol can worsen the effects from exposure to toxic chemicals, prescribed drugs must not be taken by personnel on duty where the potential for absorption, inhalation, or ingestion of toxic substances exists unless specifically approved by a qualified physician. The intake of alcoholic beverage/illegal substance is prohibited during work hours.
- 8) All field personnel must be trained and have medical examination verification. They also must be familiar with standard operating procedures and any site-specific instructions and information contained in the SITE Safety Plan.
- 9) All personnel must follow the instructions contained in the SITE Safety Plan.
- 10) All personnel assigned to a site must be adequately trained and thoroughly briefed on anticipated hazards, equipment to be worn, safety practices to be followed, emergency procedures, and communications.
- 11) Any necessary respiratory protective devices and clothing must be worn by all personnel going into areas designated as requiring such protective equipment.

- 12) Onsite personnel must use the buddy system when wearing respiratory protective equipment. In the event of an accident, a third person, suitably equipped as a safety backup, is required during initial entries.
- 13) Visual contact should be maintained between pairs on site and with safety personnel.
- 14) During continual operations, onsite workers should act as safety backup to each other. Offsite personnel provide emergency assistance.
- 15) Personnel should practice unfamiliar operations before actually performing them.
- 16) Entrance and exit locations must be designated and emergency escape routes delineated. Warning signals for site evacuation must be established.
- 17) Communications (by radios, hand signals, signs, or other means) must be maintained between initial entry members at all times. Emergency communications should be prearranged in case of radio failure, necessity for evacuation of site, or other reasons.
- 18) For outside demonstrations, wind indicators visible to all personnel should be strategically located throughout the site.
- 19) The number of personnel and the amount of equipment in the contaminated area should be held to a minimum consistent with effective site operation.
- 20) Procedures for leaving a contaminated area must be planned and implemented before the site is entered. Work areas and decontamination procedures must be established based on expected site conditions.
- 21) Disposable time devices should be used to document and reference times during such events as sampling, trial runs, and emergency situations.

4.5 CONTROL

For a demonstration to proceed safely, it must be performed in a controlled environment. A controlled environment means that the site is operated by a defined staff of professionals and skilled workers, including a designated Safety Officer. Further, all activities must be organized and implemented from a centralized command location. Finally, all site boundaries and perimeters must be accurately defined, and access to these areas

must be tightly regulated. Enforcement of the criteria described in the following subsections should help to ensure the completion of a safe demonstration.

Key Safety Personnel

During the demonstration, safety is the responsibility of the Safety Officer. This individual has the authority to suspend the demonstration temporarily if there appears to be a threat to health or safety. The Safety Officer (who will have been selected by the EPA or its designated representatives) has the authority and responsibility for the following:

- Upgrading the level of protection when necessary.
- Requiring additional monitoring.
- Suspending operations for any health and/or safety reason.

The Safety Officer, or his/her appointed representative, will be responsible for planning and execution of the overall site safety policy. He/she will be responsible for making project-level decisions regarding safety rules and operations.

The Safety Officer will also have primary responsibility for the following:

- 1) Assuring that appropriate personal protective equipment is available and properly utilized by all site personnel.
- 2) Assuring that site personnel are aware of the provisions of the H&S Plan, are instructed in the work practices necessary to ensure safety, and are trained in planned procedures for dealing with emergencies.
- 3) Assuring that personnel are aware of the potential hazards associated with the demonstration and the site.
- 4) Supervising the monitoring of safety performance by all personnel to ensure that required work practices are employed.
- 5) Correcting any work practices or conditions that may result in injury to personnel or exposure to hazardous substances.

The Safety Officer or an appointed representative will plan and supervise specific safety activities in support of the work performed at the project demonstration in accordance with the H&S Plan. The Safety Officer will

have the authority to make changes to the H&S Plan where required by unforeseen, site-specific conditions.

At the demonstration site, the Safety Officer shall:

- 1) Conduct site monitoring of personnel hazards to determine the degree of hazard present.
- 2) Determine personnel protection levels and necessary clothing and equipment to ensure the safety of personnel.
- 3) Evaluate chemical hazard and weather (if applicable) information, and recommend to the EPA PM any necessary modifications to work plans and personnel protection levels to maintain personnel safety.
- 4) Monitor the safety performance of all personnel to ensure that the required practices are adhered to.

Site Boundaries/Perimeters

The information presented here has been paraphrased from Section 6 of the Hazardous Materials Incident Response Manual established by EPA's Office of Emergency and Remedial Response. These guidelines were established for investigation and cleanup of uncontrolled hazardous waste sites; however, the same precepts are applicable to demonstrations operated under the SITE Program. The Safety Officer must determine the applicability of these principles based on the site and type of technology involved.

One important means of preventing or reducing the migration of contaminants is to delineate zones on the site where the prescribed operations are to occur. Movement of personnel and equipment between zones and onto the site itself should be limited by access control points. Three contiguous zones are recommended: Zone 1: Exclusion Zone, Zone 2: Contamination Reduction Zone, and Zone 3: Support Zone. Each is discussed in the following sections.

Zone 1: Exclusion Zone³--

The Exclusion Zone, the innermost of the three zones, is the area where contamination does or could occur. All people entering the Exclusion Zone must wear prescribed levels of protection. An entry and exit check point must be established at the periphery of this zone to regulate the flow of personnel and equipment into and out of the zone and to verify that the established entry and exit procedures are followed.

The outer boundary of Zone 1, called the Hotline, is established initially through a visual survey of the immediate environs of the site and a determination of where the hazardous substances are located or where any material has leaked or spilled. The Safety Officer also should provide guidance for determining the boundaries and should indicate the presence of any organic or inorganic vapors/gases or particulates in the air, combustible gases, and radiation resulting from water and soil contamination.

Additional factors that should be considered include how much distance is required to prevent fire or an explosion from affecting personnel outside the zone, the physical area necessary to conduct the demonstration, and the potential for contaminants to be blown from the area. Once the Hotline has been determined, it should be physically secured, fenced, or well defined by landmarks. During subsequent site operations, the boundary may be modified or adjusted as indicated by the Safety Officer.

All personnel within the Exclusion Zone must wear the required level of protection. Personal protective equipment is stipulated on the basis of site-specific conditions, including the type of work to be done and the hazards that might be encountered. Frequently, different levels of protection are justified within the Exclusion Zone. Subareas should be specified and conspicuously marked as to whether Level A, B, or C protection is required. The level of protection is determined by the measured concentration of substances in air, the potential for contamination, and the known or suspected presence of highly toxic substances.

Job assignments also may influence the level of protection required in the Exclusion Zone. For example, collecting samples from open containers might require Level B protection, whereas Level C protection might be sufficient for walk-through ambient air monitoring. The assignment of different levels of protection within the Exclusion Zone, as appropriate, generally allows a more flexible, more effective, and less costly operation while still maintaining a high degree of safety.

Zone 2: Contamination Reduction Zone³--

The Contamination Reduction Zone, which lies between the Exclusion Zone and the Support Zone, provides a transition between contaminated and clean

zones. By serving as a buffer, this intermediate zone reduces the probability of the clean zone becoming contaminated or being affected by other existing hazards. It provides additional assurance that the physical transfer of contaminating substances on people, equipment, or in the air is limited through a combination of factors, including decontamination, distance between Exclusion and Support Zones, air dilution, zone restrictions, and work functions.

Initially, the Contamination Reduction Zone is considered an uncontaminated area. At the boundary between the Exclusion and Contamination Reduction Zones, Contamination Reduction Corridors (decontamination stations) are established, one for personnel and one for heavy equipment. Depending on the size of the operation, more than two corridors may be necessary. Exit from the Exclusion Zone is through a Contamination Reduction Corridor. As operations proceed, however, the area around the decontamination station may become contaminated, although to a much lesser degree than the Exclusion Zone. The amount of contaminants present should decrease from the Hotline to the Support Zone because of the distance involved and the decontamination procedures used.

Zone 3: Support Zone³--

The Support Zone (at the outermost part of the site), is considered a contamination-free or clean area. Support equipment (command post, equipment trailer, etc.) is located in this zone. Traffic is restricted to authorized response personnel. Normal work clothes are appropriate within this zone, but potentially contaminated personal clothing, equipment, and samples must be left in the Contamination Reduction Zone until they are decontaminated.

The location of the command post and other support facilities in the Support Zone depends on a number of factors, including:

- ° Accessibility: Open space available, topography, location of roads, or other limitations.
- ° Wind direction: The preferred location of support facilities is upwind of the Exclusion Zone; however, shifts in wind direction and other conditions may be such that an ideal location cannot be based on wind direction alone.

The boundary between the Support Zone and the Contamination Reduction Zone, called the Contamination Control Line, separates the possibly low-contamination area from the clean Support Zone. Access to the Contamination Reduction Zone from the Support Zone is through a control point. Personnel entering this zone might be required to wear the prescribed personal protective equipment for persons working in the Contamination Reduction Zone. Anyone entering the Support Zone is required to remove any protective equipment worn in the Contamination Reduction Zone.

Command Post³--

The Command Post should be located in the clean work area. Operational activities in the office and command post include:

- 1) Supervision of demonstration operations.
- 2) Maintenance of communication, including emergencies of communication.
- 3) Recordkeeping (e.g., chain-of-custody records, daily logbooks, accident reports, equipment records, H&S Plan, etc).
- 4) Interfacing with government agencies, medical personnel, the media, and other interested parties.

Discussion of Boundaries and Work Areas³--

The use of a three-zone system, access control points, and exacting decontamination procedures provides reasonable assurance against the translocation of contaminating substances. This site control system is based on a worst-case situation. Site control and decontamination procedures may be less stringent if more definitive information is available on the types of substances involved and the hazards they present. This information can be obtained through air monitoring, instrument survey, and sampling, and from available technical data concerning the characteristics and behavior of material present.

The distance between the Hotline, the Contamination Control Line, and the Command Post, and the size and shape of each zone must be based on site-specific considerations. Assuring that the distances between zone boundaries

are sufficient to allow room for the necessary operations, to provide adequate distances to prevent the spread of contaminants, and to eliminate the possibility of injury due to explosion or fire requires considerable judgment. For long-term operations, reasonable methods (for example, air surveillance and visible deterioration) would have to be developed to determine if material is being transferred between zones and to assist in modifying site boundaries.

The following criteria should be considered in establishing area dimensions and boundaries:

- 1) Physical and topographical features of the site.
- 2) Weather conditions (if applicable).
- 3) Field/laboratory measurements of air contaminants and environmental samples.
- 4) Air dispersion calculations.
- 5) Potential for explosion and flying debris.
- 6) Physical, chemical, toxicological, and other characteristics of the substances present.
- 7) Cleanup activities required.
- 8) Potential for fire.
- 9) Area needed for conducting operations.
- 10) Potential for exposure.
- 11) Proximity to residential or industrial areas.

4.6 HAZARD EVALUATION

The evaluation of potential hazards is an important first step in securing a safe demonstration site. The exposure to one or more hazardous materials represents a significant threat to the health of both onsite personnel and neighboring residents. After identifying all hazardous materials, the Safety Officer must assemble pertinent toxicological, physical, and chemical

data. In addition to exposure to hazardous materials, the more traditional hazards (such as explosion, fire, and heavy equipment accidents) must be identified and minimized to the extent possible.

Hazardous Materials

During the planning stages of a demonstration, the hazardous materials that may be involved with the project must be inventoried. The inventory should include every hazardous material used in the technology plus those within the waste involved in the demonstration. The list of substances that might be involved in a technology demonstration could number less than 10 to more than 100. These hazardous substances may be organic or inorganic in nature, and they may exist as a single compound or as a component in a mixture of compounds. The chemical compounds used may exist in one or more phases (solid, liquid, or gas). All of these factors must be considered when assembling a list of chemical compounds of concern.

Each substance also must be properly identified with a label. Symbols, numbers, and colors can be used to provide the following important information:

- ° Hazard identification
- ° Degree of danger it posed by exposure or contact
- ° Required actions should an accident or overexposure occur

If a substance is transferred from a large container to a small container, the smaller container also must be properly labeled.

Concentrations

After completing the list of all hazardous substances involved in a specific demonstration, the Safety Officer must establish the likely range of concentration of each substance. Such information is necessary for designating levels of personal protection during normal operations, cleanup, and emergency situations. In addition to concentration data, a definition of regulatory exposure limits is needed. These limits should include, but not be limited to, the following:

- 1) The OSHA permissible exposure limits (PELs) representing the time-weighted average (TWA) exposures during an 8-hour working day in a 40-hour workweek and allowable excursions for designated periods of

time at specified concentrations above the TWA concentrations (providing the TWA is not exceeded during the 8-hour day). The PELs are set forth in OSHA regulations in 29 CFR Part 1910, Subpart Z.

- 2) Exposure limits recommended by the National Institute for Occupational Safety and Health (NIOSH) based on criteria documents and the January 1981 publication "Occupational Health Guidelines for Chemical Hazards." The values by the Standards Completion Program (SCP) recommended concentrations for IDLH (Immediately Dangerous to Life and Health) which represent the maximum level from which an individual could escape within 30 minutes without any escape-impairing symptoms or irreversible health effects. Other TWA values and excursion limits may also be referenced when OSHA PEL values are not available and NIOSH has no recommended values.
- 3) Threshold Limit Values (TLVs) recommended by the American Conference of Governmental Industrial Hygienists (ACGIH) for TWA and excursion limits (current edition). Where available, these values may be used when OSHA and NIOSH have no established or recommended values.

Primary Hazards⁴

It is important that all primary hazards be identified during the planning stages of a technology demonstration. An initial site survey and review of demonstration work scope should be useful in determining hazardous or potentially hazardous conditions. The main effort should focus on rapid identification of the primary hazards that may affect the public, site personnel, and the environment. Of major concern are the real or potential dangers from fire, explosion, airborne contaminants, and (to a lesser degree) radiation and oxygen-deficient atmospheres.

Organic Vapors and Gases⁴--

If known organic substances are involved and the materials could be volatile or become airborne, measurements for organics in the air should be made with one or more appropriate and properly calibrated survey instruments.

If it is not known whether any organic vapors/gases are present, instruments such as a photoionizer (e.g., HNU Systems) or a portable gas chromatograph (e.g., a Foxboro Systems OVA) should be operated in the total readout mode to detect organic vapors. If the constituents can be identified, the readout indicates the total airborne substances to which the instrument is responding. Identification of the individual vapor/gas constituents permits the instruments to be calibrated to these substances and used for more specific and accurate analysis.

Sufficient data should be obtained during the initial entry to map or screen the site for various levels of organic vapors. These gross measurements may be used on a preliminary basis to 1) to determine levels of personal protection, 2) establish site work zones, and 3) select candidate areas for more thorough qualitative and quantitative studies. Very high readings on the HNU or OVA also may indicate the displacement of oxygen or the presence of combustible vapors.

Inorganic Vapors and Gases⁴--

Very few direct-reading instruments are capable of detecting and quantifying nonspecific inorganic vapors and gases. Currently, the HNU photo-ionizer has very limited detection capability, whereas the Foxboro OVA has none. If specific inorganics are known or suspected to be present, measurements should be made with appropriate instruments, if available. Colorimetric tubes are practical only if the substances involved are known or can be narrowed to a few.

Radiation⁴--

Although many sites will not require radiation monitoring, this type of monitoring should be incorporated into the Site Plan where radioactive materials could be present (e.g., fires at warehouses or hazardous material storage facilities, transportation incidents involving unknown materials, or abandoned waste sites).

Oxygen Deficiency⁴--

Normal air contains about 20.5 percent by volume of oxygen. At or below 19.5 percent oxygen air-supplied respiratory protective equipment is needed. Oxygen measurements are particularly important when work is performed in enclosed spaces, low-lying areas, or in the vicinity of accidents that have produced heavier-than-air vapors that could displace ambient air. These oxygen-deficient areas are also prime locations for taking further organic vapor and combustible gas measurements because the air has been displaced by other substances. Oxygen-enriched atmospheres increase the potential for fires.

Combustible Gases⁴--

The presence or absence of combustible vapors or gases must be determined. If readings approach or exceed 10 percent of the lower explosive limit (LEL), extreme caution should be exercised in continuing the investigation. If readings approach or exceed 25 percent of the LEL, personnel should be withdrawn immediately. Before any onsite activities are resumed, project personnel (in consultation with experts in fire or explosion prevention) must develop procedures for continuing operations.

Visual Observations⁴--

While on site, the project team should make visual observations to assist in the evaluation of site hazards (e.g., dead fish or animals; land features; wind direction; labels on containers indicating explosive, flammable, toxic, or corrosive materials; conditions conducive to splash or other contact with unconfined liquids, sludges, or solids; and other general conditions).

Direct-Reading Instruments⁴--

Because direct-reading field instruments will not detect or measure all organic vapors, inorganic vapors, gases, or particulates, negative readings should not be interpreted as indications of the complete absence of airborne toxic substances. Negative results can only be verified by collecting air samples and analyzing them in a laboratory.

Hazardous Substance Information Forms

After all hazardous substances and situations have been identified, this information must be organized into a useful form. Use of a hazardous substance data sheet (HSDS) will ensure that all important information for a given substance has been gathered. This sheet should include physical/chemical properties, hazardous characteristics, safety information, and site-specific notes. Figure 4-1 is a sample hazardous substance data sheet.

Process Safety Audit

A process safety audit should be an integral part of any hazard evaluation program. The process safety audit comprises an evaluation of the design, construction, and operation of a process from a safety point of view. One or more engineers (not involved with the demonstration) should review both process drawings and equipment in a pipe-by-pipe, valve-by-valve inspection that includes:

NAME OF SUBSTANCE: _____

COMMON: _____

CHEMICAL: _____

1. PHYSICAL/CHEMICAL PROPERTIES:

			SOURCE
Normal Physical State:	Gas _____	Liquid _____	Solid _____
Molecular Weight	_____		_____
Density	_____ gm/ml		_____
Specific Gravity	_____ F / C		_____
Solubility: Water	_____ F / C		_____
Solubility: _____	_____ F / C		_____
Boiling Point	_____ F / C		_____
Melting Point	_____ F / C		_____
Vapor Pressure (mmHg)	_____ @ _____	F / C	_____
Vapor Density	_____ @ _____	F / C	_____
Flash Point OC/CC	_____ F / C		_____
Other: _____	_____		_____
_____	_____		_____

2. HAZARDOUS CHARACTERISTICS

A. TOXICOLOGICAL HAZARD	HAZARD	CONCENTRATIONS	SOURCE
Inhalation	Yes No	_____	_____
Ingestion	Yes No	_____	_____
Skin/Eye Absorption	Yes No	_____	_____
Skin/Eye Contact	Yes No	_____	_____
Carcinogenic	Yes No	_____	_____
Teratogenic	Yes No	_____	_____
Mutagenic	Yes No	_____	_____
Aquatic	Yes No	_____	_____
Other: _____	Yes No	_____	_____
B. FIRE HAZARD	HAZARD	CONCENTRATIONS	SOURCE
Combustibility	Yes No	_____	_____
Toxic Byproducts	Yes No	_____	_____
Other: _____	Yes No	_____	_____
_____	Yes No	_____	_____
Flammable/Explosive	Yes No	_____	_____
LFL/LEL	_____	_____	_____
UFL/UEL	_____	_____	_____
C. REACTIVITY HAZARD	HAZARD	CONCENTRATIONS	SOURCE
Water	Yes No	_____	_____
Other: _____	Yes No	_____	_____
_____	Yes No	_____	_____
_____	Yes No	_____	_____

Figure 4-1. Hazardous Substance Data Sheet.

NAME OF SUBSTANCE: _____

D. CORROSIVE HAZARD	HAZARD	pH	SOURCE
Acid	Yes No	_____	_____
Base	Yes No	_____	_____
Neutralizing Agent:		_____	_____

E. RADIOACTIVE HAZARD	HAZARD	EXPOSURE RATE	SOURCE
Background	Yes No	_____	_____
Alpha Particles	Yes No	_____	_____
Beta Particles	Yes No	_____	_____
Gamma Radiation	Yes No	_____	_____

3. INCIDENT RELATED:

Quantity Involved: _____

Release Information: _____

Monitoring/Sampling Recommended: _____

4. RECOMMENDED PROTECTION:

Public: _____

Environment: _____

Worker: _____

5. RECOMMENDED SITE CONTROL:

Exclusion Zone: _____

Contamination Reduction Zone: _____

Support Zone: _____

Figure 4-1 (continued)

1. Checking the proper sizing of piping, valves, fittings, etc.
2. Checking to ensure the proper use of materials for construction.
3. Checking to ensure that all conditions described on the as-built drawings actually exist on site.
4. Checking to ensure the proper installation of all process equipment.
5. Checking to ensure that process equipment is properly plumbed (i.e., inlet piping is connected to pump inlet and outlet piping is connected to pump outlet).
6. Checking to ensure that all Federal, State, and local regulations are met concerning safety, fire, and other codes.

Safety audits should be tailored to meet the individual needs of a given technology. They should be conducted on a regular basis, and records from each audit should be kept on file.

4.7 PERSONAL PROTECTION EQUIPMENT

All onsite personnel must wear protective equipment when involved in activities in areas where known or suspected atmospheric contamination, vapors, gases, or particulates may be generated or when such activities could result in direct contact with skin-affecting substances. Full-face-piece respirators will protect lungs, gastrointestinal tract, and eyes against airborne toxicants. Chemical-resistant clothing will protect the skin from possible contact with skin-destructive and/or absorbable chemicals. As always, good personal hygiene will limit or eliminate the ingestion of materials by unsuspecting workers.

The Safety Officer (or his designated representative) is responsible for ensuring the health, safety, and efficiency of the team. He/she will determine the level of personal protection necessary for the health and safety of the team based on many criteria, some of which are also used in boundary determinations. These factors include characteristics of the demonstration process, types and amounts of hazardous waste present at the site, surface air and wind characteristics, the location of the site relative to human traffic, and overt signs of hazards to life and health. Any team member can seek to upgrade the level of protection through consultation with the Safety

Officer, and an agreement regarding the desired level will be reached before the team member enters the exclusion area.

All personal protection equipment will be stored and maintained in the Support Zone (Zone 3). In the case of an outside demonstration, equipment may be stored in the command post to protect it against weather, vandalism, or theft.

Levels of Protection⁴

Personal protection equipment for the protection of team members against exposure to hazardous materials is divided into four categories. The EPA endorses the philosophy of providing a higher-than-required degree of protection until support data prove this degree is not needed, rather than providing a lower degree of protection only to find that the support data substantiate the need for upgraded protection. The four categories of protection are designated A, B, C, and D. Level A provides the highest degree of protection, including the self-contained breathing apparatus (SCBA). The degree of protection decreases from Level A to Level D. The decision to use a given level of protection is based on the total atmospheric vapor/gas concentration for a specific area. Table 4-1 gives a general outline of personal protective clothing and accessories. The following subsections present criteria for the use of this clothing and equipment for each level of protection.

Level A Protection--

Level A protection provides the highest degree of protection of the respiratory tract, skin, and eyes, provided the inherent limitations of the equipment are not exceeded. Level A protection must be used when the concentration of total vapors/gases in air is in the range of 500 to 1000 parts per million (ppm) based on the following criteria:

- 1) Although Level A provides protection against air concentrations greater than 1000 ppm for most substances, an operational restriction of 1000 ppm is established as a warning flag for the following reasons:
 - ° To evaluate the need to enter environments with unknown concentrations greater than 1000 ppm.
 - ° To identify the specific constituents contributing to the total concentration and their associated toxic properties.

TABLE 4-1. PROTECTIVE CLOTHING AND ACCESSORIES

BODY PART PROTECTED	TYPE OF CLOTHING OR ACCESSORY	DESCRIPTION	TYPE OF PROTECTION	USE CONSIDERATIONS
Full Body	Fully-encapsulating suit	One-piece garment. Boots and gloves may be integral, attached and replaceable, or separate.	Protects against splashes, dust, gases, and vapors.	Does not allow body heat to escape. May contribute to heat stress in wearer, particularly if worn in conjunction with a closed-circuit SCBA; a cooling garment may be needed. Impairs worker mobility, vision, and communication.
	Non-encapsulating suit	Jacket, hood, pants, or bib overalls, and one-piece coveralls.	Protects against splashes, dust, and other materials but not against gases and vapors. Does not protect parts of head or neck.	Do not use where gas-tight or pervasive splashing protection is required. May contribute to heat stress in wearer. Tape-seal connections between pant cuffs and boots and between gloves and sleeves.
	Aprons, leggings, and sleeve protectors	Fully sleeved and gloved apron. Separate coverings for arms and legs. Commonly worn over non-encapsulating suit.	Provides additional splash protection of chest, forearms, and legs.	Whenever possible, should be used over a non-encapsulating suit (instead of using a fully-encapsulating suit) to minimize potential for heat stress. Useful for sampling, labeling, and analysis operations. Should be used only when there is a low probability of total body contact with contaminants.
Firefighters' protective clothing		Gloves, helmet, running or bunker coat, running or bunker pants (NFPA No. 1971, 1972, 1973), and boots.	Protects against heat, hot water, and some particles. Does not protect against gases and vapors, or chemical permeation or degradation. NFPA Standard No. 1971 specifies that a garment consist of an outer shell, an inner liner, and a vapor barrier with a minimum water penetration of 25 lbs/in ² (1.8 kg/cm ²) to prevent the passage of hot water.	Decontamination is difficult. Should not be worn in areas where protection against gases, vapors, chemical splashes, or permeation is required.
Proximity garment (approach suit)		One- or two-piece overgarment with boot covers, gloves, and hood of aluminized nylon or cotton fabric. Normally worn over other protective clothing, such as chemical-protective clothing, firefighters' bunker gear, or flame-retardant coveralls.	Protects against brief exposure to radiant heat. Does not protect against chemical permeation or degradation. Can be custom-manufactured to protect against some chemical contaminants.	Auxiliary cooling and an SCBA should be used if the wearer may be exposed to a toxic atmosphere or needs more than 2 or 3 minutes of protection.
Blast and fragmentation suit		Blast and fragmentation vests and clothing, bomb blankets, and bomb carriers.	Provides some protection against very small detonations. Bomb blankets and baskets can help redirect a blast.	Does not provide hearing protection.

(continued)

TABLE 4-1 (continued)

BODY PART PROTECTED	TYPE OF CLOTHING OR ACCESSORY	DESCRIPTION	TYPE OF PROTECTION	USE CONSIDERATIONS
Full Body (cont.)	Radiation-contamination protective suit	Various types of protective clothing designed to prevent contamination of the body by radioactive particles.	Protects against alpha and beta particles. <i>Does NOT protect against gamma radiation.</i>	Designed to prevent skin contamination. If radiation is detected on site, consult an experienced radiation expert and evacuate personnel until the radiation hazard has been evaluated.
	Flame/fire retardant coveralls	Normally worn as an undergarment.	Provides protection from flash fires.	Adds bulk and may exacerbate heat stress problems and impair mobility.
	Flotation gear	Life jackets or work vests. (Commonly worn underneath chemical protective clothing to prevent flotation gear degradation by chemicals.)	Adds 15.5 to 25 lbs (7 to 11.3 kg) of buoyancy to personnel working in or around water.	Adds bulk and restricts mobility. Must meet USCG standards (46 CFR Part 160).
	Cooling garment	One of three methods: (1) A pump circulates cool dry air throughout the suit or portions of it via an air line. Cooling may be enhanced by use of a vortex cooler, refrigeration coils, or a heat exchanger. (2) A jacket or vest having pockets into which packets of ice are inserted. (3) A pump circulates chilled water from a water/ice reservoir and through circulating tubes, which cover part of the body (generally the upper torso only).	Removes excess heat generated by worker activity, the equipment, or the environment.	(1) Pumps circulating cool air require 10 to 20 ft ³ (0.3 to 0.6 m ³) of respirable air per minute, so they are often uneconomical for use at a waste site. (2) Jackets or vests pose ice storage and recharge problems. (3) Pumps circulating chilled water pose ice storage problems. The pump and battery add bulk and weight.
Head	Safety helmet (hard hat)	For example, a hard plastic or rubber helmet.	Protects the head from blows.	Helmet shall meet OSHA standard 29 CFR Part 1910.135.
	Helmet liner		Insulates against cold. Does not protect against chemical splashes.	
	Hood	Commonly worn with a helmet.	Protects against chemical splashes, particulates, and rain.	
	Protective hair covering		Protects against chemical contamination of hair. Prevents the entanglement of hair in machinery or equipment. Prevents hair from interfering with vision and with the functioning of respiratory protective devices.	Particularly important for workers with long hair.
Eyes and Face*	Face shield	Full-face coverage, eight-inch minimum.	Protects against chemical splashes. Does not protect adequately against projectiles.	Face shields and splash hoods must be suitably supported to prevent them from shifting and exposing portions of the face or obscuring vision. Provides limited eye protection.

*All eye and face protection must meet OSHA standard 29 CFR Part 1910.133.

(continued)

TABLE 4-1 (continued)

BODY PART PROTECTED	TYPE OF CLOTHING OR ACCESSORY	DESCRIPTION	TYPE OF PROTECTION	USE CONSIDERATIONS
Eyes and Face (cont.)	Splash hood		Protects against chemical splashes. Does not protect adequately against projectiles.	
	Safety glasses		Protect eyes against large particles and projectiles.	If lasers are used to survey a site, workers should wear special protective lenses.
	Goggles		Depending on their construction, goggles can protect against vaporized chemicals, splashes, large particles, and projectiles (if constructed with impact-resistant lenses).	
	Sweat bands		Prevents sweat-induced eye irritation and vision impairment.	
Ears	Ear plugs and muffs		Protect against physiological damage and psychological disturbance.	Must comply with OSHA regulation 29 CFR Part 1910.95. Can interfere with communication. Use of ear plugs should be carefully reviewed by a health and safety professional because chemical contaminants could be introduced into the ear.
	Headphones	Radio headset with throat microphone.	Provide some hearing protection while enabling communication.	Highly desirable, particularly if emergency conditions arise.
Hands and Arms	Gloves and sleeves	May be integral, attached, or separate from other protective clothing.	Protect hands and arms from chemical contact.	Wear jacket cuffs over glove cuffs to prevent liquid from entering the glove. Tape-seal gloves to sleeves to provide additional protection.
		Overgloves.	Provide supplemental protection to the wearer and protect more expensive undergarments from abrasions, tears, and contamination.	
		Disposable gloves.	Should be used whenever possible to reduce decontamination needs.	
Foot	Safety boots	Boots constructed of chemical-resistant material.	Protect feet from contact with chemicals.	
		Boots constructed with some steel materials (e.g., toes, shanks, insoles).	Protect feet from compression, crushing, or puncture by falling, moving, or sharp objects.	All boots must at least meet the specifications required under OSHA 29 CFR Part 1910.136 and should provide good traction.
		Boots constructed from nonconductive, spark-resistant materials or coatings.	Protect the wearer against electrical hazards and prevent ignition of combustible gases or vapors.	

(continued)

TABLE 4-1 (continued)

BODY PART PROTECTED	TYPE OF CLOTHING OR ACCESSORY	DESCRIPTION	TYPE OF PROTECTION	USE CONSIDERATIONS
Foot (cont.)	Disposable shoe or boot covers	Made of a variety of materials. Slip over the shoe or boot.	Protect safety boots from contamination. Protect feet from contact with chemicals.	Covers may be disposed of after use, facilitating decontamination.
General	Knife		Allows a person in a fully-encapsulating suit to cut his or her way out of the suit in the event of an emergency or equipment failure.	Should be carried and used with caution to avoid puncturing the suit.
	Flashlight or lantern		Enhances visibility in buildings, enclosed spaces, and the dark.	Must be intrinsically safe or explosion-proof for use in combustible atmospheres. Sealing the flashlight in a plastic bag facilitates decontamination. Only electrical equipment approved as intrinsically safe, or approved for the class and group of hazard as defined in Article 500 of the National Electrical Code, may be used.
	Personal dosimeter		Measures worker exposure to ionizing radiation and to certain chemicals.	To estimate actual body exposure, the dosimeter should be placed inside the fully-encapsulating suit.
	Personal locator beacon	Operated by sound, radio, or light.	Enables emergency personnel to locate victim.	
	Two-way radio		Enables field workers of communicate with personnel in the Support Zone.	
	Safety belts, harnesses, and lifelines		Enable personnel to work in elevated areas or enter confined areas and prevent falls. Belts may be used to carry tools and equipment.	Must be constructed of spark-free hardware and chemical-resistant materials to provide proper protection. Must meet OSHA standards in 29 CFR Part 1926.104.

- ° To make more precise determinations of concentrations of constituents.
 - ° To evaluate the calibration and/or sensitivity error associated with the instrument(s).
 - ° To evaluate instrument sensitivity to wind velocity, humidity, temperature, etc.
- 2) A limit of 500 ppm total vapors/gases in air was selected as the value at which upgrading from Level B to Level A should be considered. This concentration was selected to provide full protection of the skin until the constituents can be identified and measured and substances affecting the skin are excluded.
 - 3) The range of 500 to 1000 ppm is sufficiently conservative to provide a safe margin of protection if readings should be low because of instrument error, calibration, and sensitivity; if higher than anticipated concentrations were to occur; and if substances that are highly toxic to the skin should be present.
 - 4) A range of concentrations that would provide adequate protection from exposure to particulates has not been strictly defined. The opportunity for exposure to high levels of airborne particulate matter is low for most SITE demonstrations. Guidance for exposure to airborne particulates can be found in "Threshold Limit Value for Chemical Substances in the Work Environment," by American Conference of Governmental Industrial Hygienists, 1987-88.

Typically, high ambient air contaminant concentrations have been found only in closed buildings when containers were being opened, when personnel were working in the spilled contaminants, or when organic vapors/ gases were released in transportation accidents.

In a decision concerning the requirement for Level A protection, the negative aspects also should be considered; e.g., the higher probability of accidents due to cumbersome equipment and, more important, the physical stress caused by heat buildup in fully encapsulating suits. The use of Level A protection will also increase the time required to perform most work tasks.

Level A personal protective equipment includes:

- 1) SCBA
- 2) Fully encapsulated suit
- 3) Coveralls, cotton, white
- 4) Underwear, cotton

- 5) Gloves, surgical
- 6) Boots, neoprene, steel toe and shank
- 7) Booties, butyl rubber or PVC
- 8) Gloves, disposable (optional)
- 9) Booties, disposable (optional)
- 10) Hard hat (optional)

Level B Protection--

Level B protection represents the minimum protection recommended for initial entry to an open site where the type, concentration, and presence of airborne vapors are unknown. This level of protection provides a high degree of respiratory protection. Skin and eyes are also protected, but a small portion of the body (neck and sides of head) may be exposed. The use of a separate hood or hooded, chemical-resistant jacket would further reduce the potential for exposure to this area of the body. Level B impermeable protective clothing also increases the probability of heat stress.

A limit of 500 ppm has been selected as a decision point for a careful evaluation of the risks associated with higher concentrations. The following factors should be weighed carefully when downgrading to Level B is being considered:

- 1) Whether a person wearing Level B protection may be required to enter areas with unknown concentrations that could exceed 500 ppm.
- 2) The probability that the substance present are severe skin hazards.
- 3) The work involved and the increased probability of exposure.
- 4) The need for qualitative and quantitative identification of the specific components.
- 5) Inherent limitations of the instruments used for air monitoring.
- 6) Instrument sensitivity to winds, humidity, temperature, and other factors.

Level B personal protective equipment includes:

- 1) Pressure-demand airline respirator
- 2) Apron or long-sleeved jacket and pants, butyl rubber or neoprene.
- 3) Gloves, butyl rubber or neoprene
- 4) Gloves, surgical
- 5) Boots, butyl rubber or neoprene, steel toe and shank

- 6) Booties, butyl rubber or neoprene
- 7) Coveralls, chemical-resistant (optional)
- 8) Underwear, cotton (optional)
- 9) Booties, disposable (optional)
- 10) Gloves, disposable (optional)
- 11) Hard hat (with face shield - optional)
- 12) Escape mask (5 minutes)

Level C Protection--

Level C skin protection is identical to Level B protection, assuming the same type of chemical protective clothing is worn, but Level C provides less protection against inhalation hazards. A range of background to 5 ppm above ambient background concentrations of vapors/gases in the atmosphere has been established as guidance for the selection of Level C protection. Air concentrations of unidentified vapors/gases approaching or exceeding 5 ppm would warrant upgrading respiratory protection to a self-contained breathing apparatus.

A full-face, air-purifying mask equipped with an organic vapor canister (or a combined organic vapor/particulate canister) provides protection against low concentrations of most common organic vapors/gases. Among the substances against which full-face, canister-equipped masks will not protect are substances that have very low TLV or IDLH concentrations. Every effort should be made to identify the individual constituents and the presence of particulates contributing to the total vapor readings of a few parts per million before respiratory protective equipment is selected. It is exceedingly difficult, however, to obtain a constant, real-time identification of all components in a vapor cloud with concentrations of a few parts per million at a site where ambient concentrations are constantly changing. If highly toxic substances have been ruled out, but ambient levels of a few parts per million persist, one should not assume that the wearing of only a self-contained breathing apparatus is sufficient. The continuous use of air-purifying masks in vapor/gas concentrations of a few parts per million provides reasonable assurance that the respiratory tract is protected, as long as the absence of highly toxic substances has been confirmed.

Level C personal protective equipment includes:

- 1) Full-face respirator (appropriate cartridge must be selected)
- 2) Escape mask (optional)
- 3) Gloves, butyl rubber or neoprene
- 4) Gloves, surgical
- 5) Boot, butyl rubber or neoprene, steel toe and shank
- 6) Booties, butyl rubber or neoprene (optional)
- 7) Coveralls, chemical-resistant (optional)
- 8) Underwear, cotton (optional)
- 9) Booties, disposable (optional)
- 10) Gloves, disposable (optional)
- 11) Hard hat (with face shield - optional)
- 12) Splash-proof goggles (optional)

Level D Protection--

Level D protection should be used only in ambient settings, and should be chosen only after laboratory analysis has verified that ambient or background conditions exist.

Level D equipment includes:

- 1) Boots/shoes, safety leather or chemical-resistant, with steel toes
- 2) Safety glasses (optional)
- 3) Hard hat (optional)
- 4) Full-face respirator (readily available) (optional)
- 5) Escape mask (optional)
- 6) Work gloves (optional)

Basic Personal Protective Equipment for Chemical Technologies⁵

The preceding subsections have covered the use of personal protective equipment for scenarios where chemicals are or have been released to the environment in an uncontrolled manner. Numerous chemical technologies involve processes that operate in a closed loop (closed to the environment) format. Just as a chemical processing area is different from a hazardous waste site, the needs for personal protective equipment are also different.

The general types of protection for a chemical processing area include:

- 1) Ear plugs and ear muffs
- 2) Spectacles, goggles, and face shields
- 3) Safety helmets (hard hats)
- 4) Gloves
- 5) Boots

Hearing Protection--

Hearing loss is one of the least obvious health problems associated with chemical processing. All site personnel must be made aware of the hazards of high noise levels from the operations of pumps, compressors, mixers, etc. Regulations established by OSHA require that workplace noise exposure levels not exceed 85 dB.

Eye and Face Protection--

Protective eye wear is probably the most important of all the personal protective equipment. Glasses worn for everyday use are not suited for a processing environment. Regulations established by OSHA require that appropriate safety eye wear conform to ANSI test standards. Materials of construction include heat-treated glass or thermoplastics (e.g., polycarbonate).

Head Protection--

Safety helmets protect the head from injuries caused by the impact or penetration of falling objects. They also provide some degree of protection from high voltage shock and burns. A safety helmet consists of a hard resilient shell and a suspension system. Safety helmet shells may be constructed of plastic, fiberglass, polyester resins, or aluminum. The material selected will depend on the working environment. For example, a polyethylene helmet will offer a reasonable degree of protection from attack by various solvents. Most suspension systems are made of a woven nylon webbing and plastic system.

Safety helmets are classified by ANSI as providing A, B, or C safety protection. Class A helmets reduce the impact of falling objects and offer limited voltage protection. Class B helmets are designed to reduce the danger of exposure to high voltages. Class C helmets offer no electrical protection and should only be used in situations where the hazard potential is minimal.

Hand Protection--

Hand protection (gloves), one of the oldest forms of personal protection equipment, falls into two major categories: 1) that which protects the worker from hazards, and 2) that which protects the process from outside contamination. The gloves must be able to protect workers from chemical exposure, cuts, abrasion, vibration, and electrical hazards.

Foot Protection--

Safety boots (or shoes) are used to protect personnel from injuries resulting from falling objects, stepping on sharp objects, and objects rolling over the foot. They can also provide protection from exposure to biological/chemical hazards, slipping, heat (or cold), and electrical (or static electricity) hazards. Protective footwear must be used in wet environments, especially where asbestos or known carcinogens may be encountered.

4.8 EDUCATION AND TRAINING

OSHA requires that all hazardous waste operators receive from 24 to 48 hours of training before being permitted to engage in hazardous waste operations that could expose them to safety and health hazards (29 CFR Part 1910, Federal Register, Volume 51, No. 244, December 19, 1986). The Developer must ensure that its own field staff are properly trained and that they participate in a medical surveillance program. The SITE Contractor is responsible for training its own field staff and for ensuring the adequacy of the training of all demonstration participants.

An additional part of the training will involve the EPA PM's review of the following procedure with the SITE Program technology evaluation field staff: health and safety; sampling and analysis; QA/QC and operating procedures; and equipment assembly, startup, shakedown, and disassembly procedures.

Equipment

Primary personnel with an alternate backup should be trained in the use of any and all task-related equipment. This type of equipment may include:

- 1) Material-handling devices
- 2) Process instrumentation

- 3) Monitoring devices
- 4) Power and hand tools
- 5) Heavy equipment (i.e., forklifts, skid movers, etc.)

In addition to work-related training, all personnel should be trained in the use of personal protective equipment, in the performance of primary first-aid functions, and in site evacuation procedures.

Safe Practices/Procedures

The topic of safe practices/procedures is site- and task-specific. Personnel working in proximity to chemical processing equipment should be sensitive to the following conditions:

- 1) Unusual buildup of localized heat. This situation is often a precursor to explosion or fire. Supervisory personnel should be notified of this condition immediately.
- 2) Unusual or strained sound from pumps or motors. This may indicate the clogging or freezing of equipment. Supervisors should be notified immediately.
- 3) Unusual or strong odors. Although chemical processes may be responsible for mild odors, unusual or stronger-than-normal odors may indicate that a reaction vessel, pipe, pump seal, etc. is leaking.
- 4) Leaking pipes. Chemical processes may require the use of overhead process or utility piping. Thus, leaking pipes may drip hazardous materials on unsuspecting site personnel.
- 5) Unusual hot or cold surfaces. Large amounts of heat may have to be added or removed from some chemical processes. Extreme care should be used around hot or cold surfaces.
- 6) Pressurized vessels. In some chemical processes, reaction vessels may be required for operation at pressures in excess of 100 psi. Extreme care should be used when work is done near pressurized vessels or pipes.

Advanced First Aid

Situations may arise that require the use of advanced first aid. The Safety Officer or his representative should be trained in these procedures. Site personnel should be discouraged from performing advanced first aid techniques unless they demonstrate prior knowledge and training in this area. The misapplication of advanced techniques can cause further harm to an injured person.

Chemical Education

Chemical education for onsite personnel should be developed on the basis of those chemicals involved in the technology demonstrated. The extent of individual training should vary with the degree of potential hazard or exposure. All new employees must be trained promptly, and all personnel (new and old) should be retrained at regular intervals.

Training sessions (or meetings) must address the following issues:

- 1) A list of all chemicals an individual is likely to encounter in the course of a technology demonstration.
- 2) A brief discussion of the chemicals and their properties.
- 3) How to identify each of the chemicals.
- 4) The effects of uncontrolled exposure and how to identify uncontrolled exposure.
- 5) Procedures for reporting a chemical exposure.
- 6) Protective measures that must be followed to prevent exposure.

Onsite personnel must be trained to read and understand labels and material safety data sheets (MSDSs). The training sessions should outline the basic points of any applicable Federal, State, or local right-to-know laws. Each program should be tailored so that workers learn about the chemicals with which they will work directly.

4.9 COMMUNICATION PROCEDURES

This section outlines communication needs and procedures during normal operations and during emergency conditions. In the event of an emergency, the command post must request outside assistance, regardless of site conditions.

Radio Communications

Radio communication between work areas enables instantaneous transfer information during trial runs of the technology. Process workers can also use the radio to request additional waste or processing reagents, consult with engineers in the command post, ask for emergency aid, monitor safety and progress, and solve minor problems.

Alarm Systems

The alarm system should provide warning to personnel in all areas. Warning devices, such as hand-held horns, should be placed in all work areas. Chemical processes for which such process alarms should be considered might exhibit one or more of the following characteristics:

- 1) The potential for rapid heat generation, resulting in explosion or fire.
- 2) Processes that are sensitive to changes in pH. Changes in pH might change the process chemistry and thereby generate unwanted or dangerous products.
- 3) Processes that require thorough mixing. The lack of mixing might lead to conditions similar to those above.
- 4) Processes using reagents that are extremely corrosive or reactive.

In addition, automatic alarms should be assigned to those process parameters (temperature, pH, etc.) that have an impact on normal operations in order to warn onsite personnel of operational problems as well as emergency conditions.

Hand Signal

Hand signals should be established as a backup measure in the event of radio failure. All site personnel should be trained in the use of sight-specific hand signals, to be presented and reviewed regularly by the Safety Officer.

Disposable Timepieces

Advances in computer chip technology have enabled the manufacture of small, lightweight, disposable timepieces. These timepieces should be placed on logbooks and personal protection suits so that site personnel can document the time that specific events (sampling, etc.) take place and be aware of workshift changes or breaks. The use of such timepieces should increase the efficiency of a project.

4.10 DECONTAMINATION PROCEDURES⁴

Standard Level and Appropriate Decontamination Protocols

All site personnel must go through appropriate decontamination procedures before leaving the site. Decontamination areas should be set up in appropriate zones, and receptacles should be provided for all disposable clothing. Conventional trash cans lined with heavy-duty polyethylene trash bags should be used for this purpose. Wash tubs containing a detergent-water solution or an appropriate decontamination solution and soft-bristle brushes should be used to decontaminate reusable personal protective clothing and boots. After the detergent-water washing, an intermediate rinse is applied when applicable. Clean potable water should be used for the final rinsing.

Monitoring equipment should be brushed to remove any obvious contamination. All heavy equipment must be decontaminated before it is removed from the site. This should include manual removal of gross contamination with shovels, etc., followed by a steam or high-pressure wash; particular attention should be given to tracks, wheels, and undercarriages.

The wash waters used and all disposable items should be collected for disposal in accordance with applicable State and Federal regulations and policies.

Decontamination procedures are based on the level of personal protection equipment being worn and the degree of exposure. As a general rule, the personnel who perform decontamination activities are outfitted in one level of protection below that worn by the personnel being decontaminated; i.e., if personnel entering the decontamination line are outfitted with Level B protection equipment the decontamination personnel would normally wear Level C equipment. Higher or lower levels of protection for decontamination personnel may be determined in the field based on the types of contaminants (if any) found, air monitoring, results, etc.

Decontamination Solutions

Decontamination solutions are prepared to react with, neutralize, or remove physically specific contaminants at a site. All such solutions should be individually marked and coded. Only trained personnel should administer decontamination procedures. All decontamination solutions and rinse waters should be collected for proper disposal. The following are four examples of

decontamination solutions:

- 1) Light contamination: A detergent-based solution.
- 2) Organic contamination: A detergent-based solution.
- 3) Acid and alkali contamination: A trisodium phosphate-based solution.
- 4) Organophosphate or cyanide contamination: A calcium hypochlorite-based solution.

Emergency Decontamination

It is difficult to outline procedures that deal specifically with emergency situations because the responses of man and machine during such conditions cannot be predicted. A high percentage of emergencies will be medical in nature; thus, decontamination must be improvised to adapt to the emergency.

The decontamination effort may aggravate or cause more serious health effects. If prompt life-saving first aid and medical treatment are required, decontamination procedures should be omitted. Whenever possible, site personnel should accompany contaminated victims to the medical facility to advise on matters involving decontamination.

Physical injuries can range from a sprained ankle to a compound fracture, from a minor cut to massive bleeding. Depending on the seriousness of the injury, trained response personnel may give treatment at the site. For more serious injuries, additional assistance may be required at the site or the victim may have to be treated at a medical facility.

Lifesaving care should be instituted immediately with no consideration being given to decontamination. The injured person's outside garments may be removed (depending on the weather) if such removal does not cause a delay, interfere with treatment, or aggravate the problem. Respirators and backpack assemblies must always be removed. Fully encapsulating suits or chemical-resistant clothing can be cut away. If the outer contaminated garments cannot be safely removed, the individual should be wrapped in plastic, rubber, or blankets to prevent contamination of the inside of an ambulance and the medical personnel. In this case, outside garments would be removed at the medical facility. No attempt should be made to wash or rinse the victim at the site unless the individual is known to have been contaminated with an

extremely toxic or corrosive material that could also cause severe injury or loss of life. In the case of minor medical problems or injuries, the normal decontamination procedure should be followed.

Heat-related illnesses range from heat fatigue to heat stroke. When heat stroke (the most serious of these illnesses) occurs, treatment must be prompt to prevent irreversible damage or death. Protective clothing may have to be cut off. Less serious forms of heat stress also require prompt attention, or they may lead to a heat stroke. Unless the victim is obviously contaminated, decontamination should be omitted or minimized and treatment begun immediately.

Injuries resulting from exposure to chemicals can be divided into two categories:

- 1) Injuries from direct contact, such as acid burns or inhalation of toxic chemicals.
- 2) Potential injury due to gross contamination of clothing or equipment.

If a person has inhaled contaminants, only qualified physicians can render treatment. If the contaminant is on the skin or in the eyes, immediate measures must be taken to counteract the substance's effect. First aid treatment usually consists of flooding the affected area with water; however, for a few chemicals, water may cause more severe problems.

When protective clothing is grossly contaminated, contaminants may be transferred to treatment personnel or the wearer and cause injuries. Unless severe medical problems have occurred simultaneously with the splashes, protective clothing should be washed off as rapidly as possible and carefully removed.

General Decontamination Protocol and Equipment

Decontamination procedures may have to be adapted to meet conditions found at the site. Certain conditions can intensify or lessen the degree of decontamination. Adaptation of decontamination procedures should be based on the following factors:

- 1) Type of Contaminant. The extent of personnel decontamination depends on the effects the particular contaminant involved has on the body. All contaminants do not exhibit the same degree of toxicity (or other hazard). Whenever it is known or suspected that personnel could become contaminated with highly toxic or skin-destructive

substances, a full decontamination procedure should be followed. If less hazardous materials are involved, the procedure can be downgraded.

- 2) Amount of Contamination. The amount of contamination on protective clothing usually can be determined visually. If the clothing is badly contaminated, thorough decontamination is generally required.
- 3) Level of Protection. The level of protection and specific pieces of clothing worn determine, on a preliminary basis, the layout of the decontamination line. Each level of protection introduces different problems in the decontamination and doffing of the equipment.
- 4) Work Function. The work each person does determines the potential for contact with hazardous materials and, in turn, dictates the layout of the decontamination line.
- 5) Location of Contamination. Contamination on the upper areas of protective clothing poses a greater risk to the worker because volatile compounds may generate a hazardous breathing concentration both for the worker and for the decontamination personnel. The probability of contact with skin when doffing the upper part of clothing is also greater.
- 6) Reason for Leaving Site. The reason for leaving the Exclusion Zone also determines the need for and extent of decontamination. A worker leaving the Exclusion Zone to pick up or drop off tools or instruments and immediately returning may not require decontamination. A worker leaving to get a new air cylinder or to change a respirator or canister, however, may require some degree of decontamination. Individuals departing the Exclusion Zone for a break, lunch, or at the end of the day, must be thoroughly decontaminated.
- 7) Equipment. Selection of decontamination equipment, materials, and supplies is generally based on availability. Other considerations are ease of equipment decontamination or disposability. Most equipment and supplies can be easily procured. Decontamination equipment can include soft-bristle scrub brushes, long-handled brushes, buckets, garden sprayers, large galvanized wash tubs, stock tanks, children's wading pools, large plastic garbage cans, plastic bags, and paper or cloth towels.

4.11 SITE PLAN

The site specific H&S Plan should contain the following information.

Designated Safety Officer

The site plan must contain the name, address, and phone number(s) of the Safety Officer.

Site-specific Information

- 1) Maps/directions to all areas of the demonstration site.
- 2) Details regarding local fire departments, including the name of the chief, phone numbers, etc. The Safety Officer should notify local authorities prior to starting the first test run.
- 3) The presence and requirements of local ambulance services. The Safety Officer should notify the ambulance services prior to the first test run.
- 4) A list of the full complement of first aid equipment kept on site.
- 5) Emergency medical information, including hospitals, doctors, ambulance services, emergency phone numbers, including medical facilities, fire departments, utilities, etc.
- 6) A complete list of monitoring instruments required by site conditions. This list would include explosimeters, organic vapor meters, pH meters, etc.
- 7) Site-specific emergency procedures.

Medical Monitoring

All site personnel should be involved in a personal monitoring program. This program should include a baseline physical and routine followup exams, including:

- Occupational and general medical history
- Physical examination
- CBC and differential
- Methemoglobin
- Blood chemistry screen
- Urinalysis
- Blood lead
- Heavy metal screen (Cd, Hg, As)
- Stool for occult blood
- Chest x-ray
- EKG
- Spirometry (screening)
- Vision screen tonometer
- Audiogram

- ° Urine phenol
- ° Dicubaine cholinesterase

Emergency Medical Care

During each technology demonstration, the Safety Officer should ensure that one or more onsite personnel are trained as an Emergency Medical Technician.

4.12 REFERENCES FOR SECTION 4

1. Felder and Rousseau. Elementary Principles of Chemical Processes. J. Wiley and Son, Inc., New York. 1978.
2. Peters and Timmerhaus. Plant Design and Economics for Chemical Engineers. McGraw-Hill Co., New York. 1980.
3. U.S. Environmental Protection Agency. Office of Emergency and Remedial Response Hazardous Materials Incident Response Manual. October 1987.
4. National Institute for Occupational Safety and Health. Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities. 1985.
5. Plant Engineering. Personal Protective Equipment, A Basic Selection and Application Guide. Jeanie Katzel, Sr. Editor. Vol. 39, Page 48-56, October 1985.

SECTION 5

GUIDELINE DOCUMENT FOR QUALITY ASSURANCE PROJECT PLAN FOR EVALUATION OF CHEMICAL TECHNOLOGIES

The intent of this plan is to assist the Project Manager in preparing a Quality Assurance Project Plan (QAPP) for a specific chemical technology SITE demonstration. Because it is structured to encompass all aspects of "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans" (QAMS-005/80), it should allow for the development of a QAPP that will satisfy all Category II requirements.

In addition to standard QAPP requirements, this plan addresses sampling and analysis (S&A) requirements. Sampling and analyzing raw waste, intermediate products, final products (treated waste), and byproducts are critical functions in a technology demonstration. These S&A guidelines should provide a framework for the formulation of specific S&A goals.

During the development of this plan, the Developer must work closely with the EPA (or its representative). Ultimately, the EPA (or the Contractor) will be responsible for both the sampling and analysis functions and the quality assurance aspects of the demonstration.

Figure 5-1 shows an example QAPP approval form for a Category II project. This form must contain the names, signature, and date of signature of appropriate managerial and QA personnel. In addition to this approval form, the table of contents should contain a distribution list containing the names and titles of all pertinent managerial and QA personnel who receive the QAPP.

5.1 PROJECT DESCRIPTION

A QAPP must reflect a complete understanding of the project and its objective. This section of the plan should provide a general description of the project that includes the following:

QUALITY ASSURANCE PROJECT PLAN APPROVAL FORM
for
HWERL Contracts/IAG's/Cooperative Agreements/In-house Projects

Lab Workplan No: _____ Task Start Date: _____
(for measurement, data gathering, and/or data generation
Project Category: _____ activities)
QA ID No: _____ Date QAPP Received: _____
Task Title: _____

Technical Project Officer: _____
Contractor: _____

APPROVALS:

_____ Contractor Project/Task Manager	_____ Signature	_____ Date
_____ Contractor QA Manager	_____ Signature	_____ Date
_____ Affiliate Task Manager*	_____ Signature	_____ Date
_____ Other (as appropriate)	_____ Signature	_____ Date
_____ HWERL Technical Project Officer	_____ Signature	_____ Date
_____ HWERL Branch or Staff Chief**	_____ Signature	_____ Date
_____ HWERL Quality Assurance Officer	_____ Signature	_____ Date

* Approval signature is required for any ancillary sampling, analytical, or data gathering support provided by a subcontractor or HWERL principal investigator.

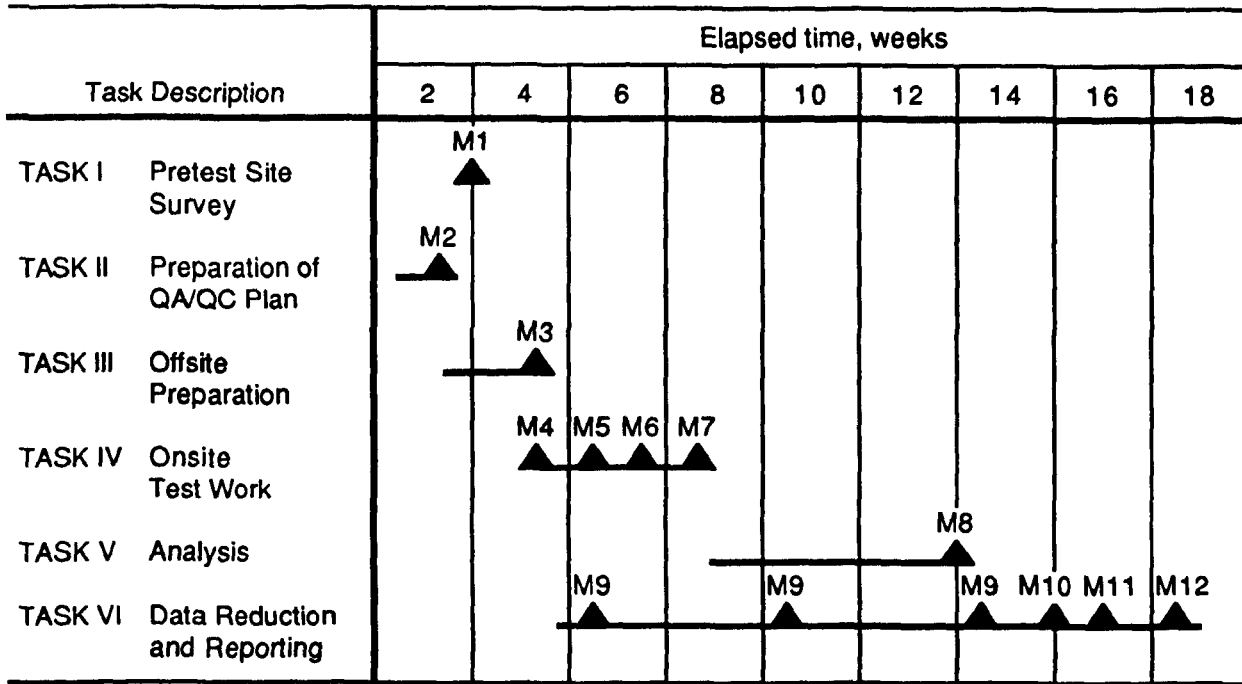
** Approval signature from the HWERL Branch or Staff Chief is required for Category I, II, and III extramural projects and for all in-house projects.

HWERL (QAPP AF)
(October 1986)

Figure 5-1. Quality Assurance Project Plan Approval Form.

- 1) Project background. This should include a brief outline of the project history and all parties involved with the project.
- 2) Experimental design of the process. The plan should include a brief review of the demonstration process, equipment, and chemical reagents that will be used.
- 3) Project data base. The plan should cover the intended use and ultimate application of the project data. The project description should clearly define each type of critical measurement to be made, each type of matrix (air, water, soil, biota, etc.) to be sampled for measurement, and each type of system condition (e.g., facility or process controls and operating parameters) to be monitored on a routine basis. The project description should clearly distinguish between critical measurements and system conditions. Critical measurements are those that have a direct impact on the technical objectives of a project (see Quality Assurance Procedures for HWERL, Reference 1). Sampling objectives should encompass the collection of data required for the performance of a mass and energy balance. Project hypotheses also should be presented.
- 4) Project timing. The plan should include a flow diagram or table showing the sequence of events and the estimated completion time of project tasks. Management tools, such as Critical Path Charts, may be included in this section.
- 5) Startup and completion dates. In addition to Item 4 above, the plan should include anticipated project startup and completion dates. Task-specific startup and completion dates also should be included in this section. Figure 5-2 shows a sample project schedule highlighting project milestones.

The S&A Plan for a technology demonstration must address the operation of a chemical unit to which one or more reagents are added, heat is added or withdrawn, and mixing or agitation is applied. Each of these actions will initiate a physical or chemical change in the waste being treated. The real-time measurement and monitoring of these changes will provide the project team with information regarding the success or failure of a given trial run. For example, a sudden drop in pH to very low levels might negate or alter subsequent process reactions. The following list presents examples of parameters that could be monitored during a chemical process demonstration. This list is not inclusive; monitoring parameters should be developed to apply to the specific process being demonstrated.



▲ M00 – Project-Specific Milestone

Figure 5-2. Sample project schedule.

- pH
- Temperature
- Oxygen production
- By-product production
- Carbon dioxide production

By monitoring changes in the operating parameters, the project team may be able to mitigate or correct a problem with the process demonstration. Also, a review of monitoring information can provide a basis for the solution of the problem. The inclusion of process measurements should be considered in the development of specific sampling and analysis objectives.

5.2 ORGANIZATIONAL CHART AND DELINEATION OF QA/QC RESPONSIBILITIES

The QAPP should list and briefly describe the responsibilities of all personnel whose task it is to ensure the collection of valid measurement data and to make routine assessments of measurement systems for precision and accuracy. This group of persons, which should be listed in a table or on a chart showing project organization and lines of authority (see Figure 5-3), would include the EPA Project Manager and Quality Assurance Officer. It is important that the specific QA/QC responsibilities within this group be delineated, as shown in Table 5-1. It is highly desirable for the QA Officer to work independently of the rest of the project team to minimize the risk of conflict of interest within the project team.

If subcontractors are used, particular attention should be given to how QA/QC activities will be monitored throughout the subcontractor's portion of the project. The subcontractor should be included in the organization chart, along with the appropriate line of authority. The specific functions (e.g., analysis, engineering) performed by the subcontractor also should be listed.

5.3 QUALITY ASSURANCE OBJECTIVES

The quality assurance objectives of a demonstration will depend on the types of sampling and analysis involved. Although sampling and analysis functions will vary widely throughout the program, the S&A Plan must consider and list each critical measurement. Critical measurements are defined as all measuring, data gathering, or data generation activities that have a direct impact on the technical objectives. Sampling must provide information on the

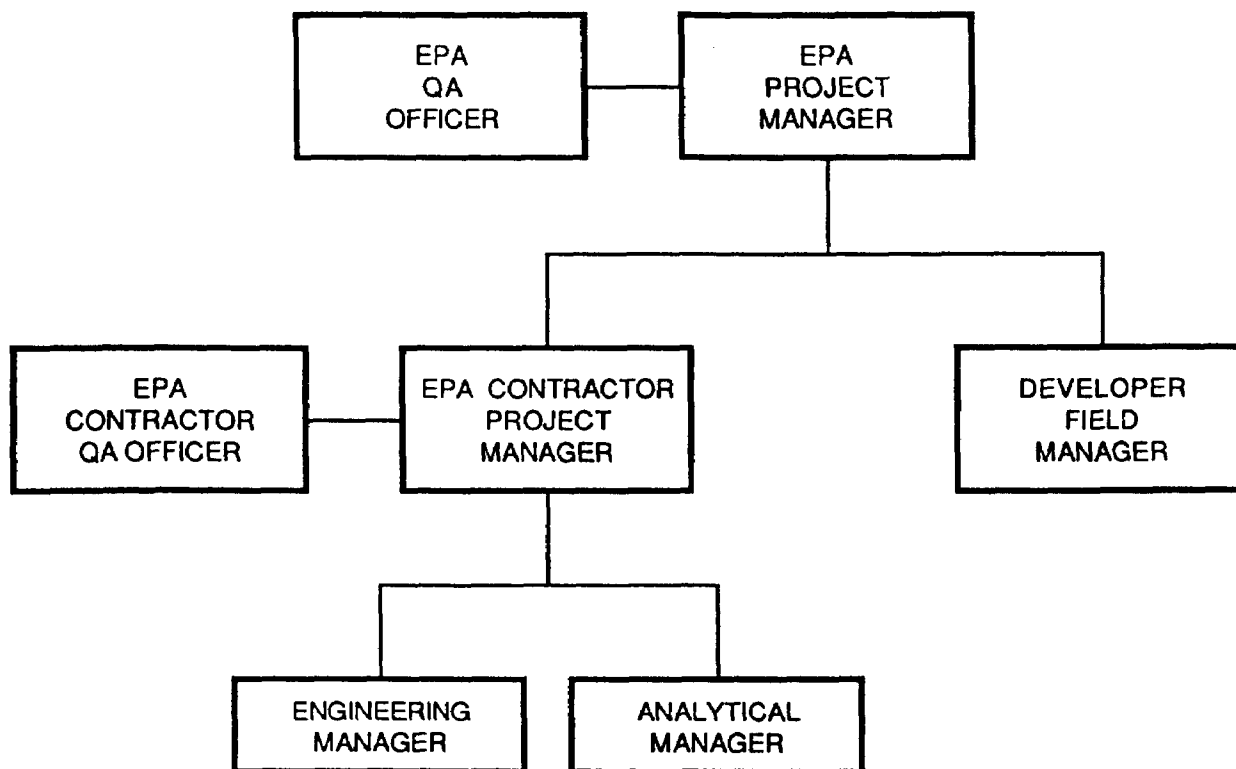


Figure 5-3. Sample project organization scheme.

TABLE 5-1. EXAMPLE OF DEFINITION OF QA/QC RESPONSIBILITIES

EPA Contractor QA Officer

- ° Collection of valid measurement data
- ° Routine assessment of measurement systems for precision and accuracy

EPA Contractor Project Manager

- ° Documentation
- ° Reporting

EPA Contractor Analytical Manager

- ° Sampling
 - ° Sample custody
 - ° Sample preparation
 - ° Sample analysis
 - ° Data reduction
 - ° Data storage and retrieval
-

characteristics of the waste prior to, during, and following treatment.

Sampling must also characterize intermediate products and byproducts.

In conjunction with each parameter, the QAPP must assign QA objectives to ensure that each of the project's technical goals is met. In general, the QA objectives established for each type of critical measurement should be based on prior knowledge of the measurement system being used; method validation studies in which replicates, spikes, standards, calibrations, recovery studies, etc., are used; and the requirements of the specific project. Where possible, these objectives should be expressed in terms of precision, accuracy, completeness, representativeness, comparability, and, where applicable, method detection limit.

The two estimators of precision are relative percent difference (RPD) and relative standard deviation (RSD). The former is the appropriate estimator when duplicate observations are used to determine precision; the latter is the more appropriate estimator of precision where more than two replicate observations are made. Precision objectives for most listed measurements (except pH) are presented as RPD of field duplicates. Precision objectives for pH are listed in pH units and expressed as limits for field duplicates.

Accuracy objectives for organic and metals determinations are given as percentage recovery range of laboratory matrix spikes. Accuracy objectives

for cation exchange capacity and alkalinity measurements are given as percentage bias as determined by analysis of standard reference materials (e.g., EPA QC check samples). Accuracy objectives for pH are given in pH units and expressed as bias limits as determined by analysis of standard reference materials (e.g., EPA QC check samples).

For a Category II project, completeness entails a comparison of the amount of valid data obtained with the amount of valid data planned to be obtained. Completeness must be defined for a Category II project. Completeness is affected by both controllable and uncontrollable factors. Total completeness addresses dropped samples, mechanical failures, holidays, no sample available, etc. In other words, any time a sample is extracted, but for any reason does not produce a result, it is "not completed." For Category II projects, completeness entails an assessment of the amount of valid data obtained compared to the amount of data that was planned to be obtained to achieve a particular statistical level of confidence. Generally, a level of 90 to 95 percent completeness is used as an objective.

For certain kinds of critical measurements (such as determination of overall process efficiencies, rates, mass balances, etc.) or the characterization of certain kinds of physical properties, some data quality indicators may not be quantifiable. To cover this possibility, the QAPP should include a qualitative statement of data quality requirements. For example, consider a project in which a chemical treatment technology is designed to field-test the rate of a dechlorination of PCBs in soil media. In such a project, it may be necessary to establish a data acceptability criterion for a total chlorine mass balance, in addition to individual method precision and accuracy objectives to ensure an appropriate level of significance for the dechlorination process. A qualitative statement of data quality requirements for total chlorine mass balance might be written as follows:

The acceptability of treatment efficiency data will be determined by total chlorine mass balance. Data will be acceptable if at least 75 percent of the total organic chlorine component initially present can be accounted for in the total chlorine mass balance at the end of each sampling period after control sample effects have been taken into account.

The QA objectives for representativeness and comparability are generally not quantifiable; therefore, these should be discussed qualitatively in the text.

All critical measurements must be such that results are representative of the media (air, water, etc.) and conditions being measured. Any deviation from approved procedures must be documented and factored into the process before conclusions are drawn. Unless otherwise specified, all data must be calculated and reported in units consistent with other demonstrations reporting similar data so that data bases can be compared. The use of officially approved EPA methods (or equivalent) also aids in the relative comparison of data from one project to another. The QA objectives established for each critical measurement parameter should be based on prior knowledge of the measurement being used.

In chemical method validation studies, replicates, spikes, standards, calibrations, recovery studies, etc., should be used with particular emphasis on the requirements of the specific project. The QAPP also should define a subset of samples that require analysis by a referee lab. When quantification is impossible, this section should include a qualitative statement of data quality requirements for the affected parameter.

Table 5-2 presents an example of QA objectives for measuring parameters likely to be of interest in aqueous, solid, and sludge samples. Table 5-3 presents QA objectives for determining selected chlorinated organics. The QAPP should provide project-specific tables for each aspect of the demonstration.

The Plan should include a statement explaining the overall impact on the project should one or more QA objectives not be met, even after corrective action measures have been taken.

For a Category II QAPP, the overall project impact of not meeting the specified QA objectives is usually a reduction in the ranges of validity (e.g., confidence level) and the applicability of the data below the level needed to achieve the technical or regulatory project goals. Data that fail to meet QA objectives usually must be accompanied by detailed qualifying statements that explain the reasons for the failure and describe the limitations on the validity and use of the data. Satisfactory qualifying statements are typically predicated on a comprehensive set of corrective actions.

TABLE 5-2. QA OBJECTIVES FOR PRECISION, ACCURACY, COMPLETENESS,
AND METHOD DETECTION LIMIT (MDL)

Critical measurement	Matrix type	Method reference	Units	MDL	Precision ^a	Accuracy ^b	Completeness, %
Volatile chlorinated hydrocarbons	Water	EPA Method 601 (Ref. 5)	µg/liter	c	c	c	90
	Soil	SW-846 Method 8010 (Ref. 4)	µg/kg	c	c	c	90
Purge-and-trap	Water/soil	SW-846 Method 5030 (Ref. 4)	-	-	-	-	90
Semivolatile chlorinated hydrocarbons	Water	EPA Method 612 (Ref. 6)	µg/liter	c	c	c	90
	Soil	SW-846 Method 8120 (Ref. 4)	µg/kg	c	c	c	90
Sonication extraction	Soil	SW-846 Method 3550 (Ref. 4)	-	-	-	-	85
Metals							
Antimony	Water/soil	SW-846 Method 7040 (Ref. 4)	d	800	40	40-115	90
Arsenic	Water/soil	SW-846 Method 7060 (Ref. 4)	d	5	30	65-130	90
Barium	Water/soil	SW-846 Method 7080 (Ref. 4)	d	500	20	75-125	90
Beryllium	Water/soil	SW-846 Method 7090 (Ref. 4)	d	40	20	80-120	90
Cadmium	Water/soil	SW-846 Method 7130 (Ref. 4)	d	20	20	80-120	90
Chromium	Water/soil	SW-846 Method 7190 (Ref. 4)	d	200	30	70-130	90
Copper	Water/soil	SW-846 Method 7210 (Ref. 4)	d	100	20	80-120	90
Lead	Water/soil	SW-846 Method 7420 (Ref. 4)	d	500	30	70-120	90
Mercury	Water/soil	SW-846 Method 7470 (Ref. 4)	d	0.5	35	75-120	90
Nickel	Water/soil	SW-846 Method 7520 (Ref. 4)	d	200	25	80-120	90
Selenium	Water/soil	SW-846 Method 7740 (Ref. 4)	d	10	50	35-125	90
Silver	Water/soil	SW-846 Method 7760 (Ref. 4)	d	50	30	55-130	90
Thallium	Water/soil	SW-846 Method 7841 (Ref. 4)	d	20	50	40-150	90
Zinc	Water/soil	SW-846 Method 7950 (Ref. 4)	d	20	35	60-140	90
Digestion	Water	SW-846 Method 3005 (Ref. 4)	-	-	-	-	90
Digestion	Water	SW-846 Method 3010 (Ref. 4)	-	-	-	-	90

(continued)

TABLE 5-2. (continued)

Critical measurement	Matrix type	Method reference	Units	MDL	Precision ^a	Accuracy ^b	Completeness, %
Digestion	Water	SW-846 Method 3020 (Ref. 4)	-	-	-	-	90
Digestion	Soil	SW-846 Method 3050 (Ref. 4)	-	-	-	-	90
Chloride	Water/soil	EPA Method 325.1 (Ref. 6)	e	1.0	15	80-120	90
Chloride residual (RsCl ₂)	Water/soil	EPA Method 330.2 (Ref. 6)	e	0.2	20	70-130	90
pH	Water	EPA Method 150.1 (Ref. 6)	pH units	-	±0.02 ^f	±0.04 ^g	90
	Soil	SW-846 Method 9045 (Ref. 4)	pH units	-	±0.02 ^f	±0.04 ^g	90
Cation exchange capacity (CEC)	Soil	SW-846 Method 9080 (Ref. 4)	meg/100 g	0.05	30	±40 ^h	80
Acidity	Water	EPA Method 305.1 (Ref. 6)	mg/liter	5	20	i	90
Alkalinity	Water	EPA Method 310.1 (Ref. 6)	mg/liter	5	20	±25 ^h	90
Total dissolved solids (TDS)	Water	EPA Method 160.3 (Ref. 6)	mg/liter	5	30	i	90

^a As Relative Percent Difference (RPD) of field duplicates, unless otherwise noted.

^b As Percent Recovery Range of laboratory matrix spikes, unless otherwise noted.

^c QA objectives or organic analyte measurements are presented in Table 5-3.

^d As µg/liter for water samples; as µg/kg for soil samples.

^e As mg/liter for water samples; as mg/kg for soil samples.

^f Expressed in pH units as limits for field duplicates.

^g Expressed in pH units as bias for measurement of standard QC check sample.

^h As percent bias for measurement of standard QC check sample.

ⁱ Not available for method.

TABLE 5-3. PRECISION, ACCURACY, AND METHOD DETECTION LIMIT
QA OBJECTIVES FOR PROJECT-SPECIFIC CHLORINATED ORGANICS

Analyte	MDL ^a	Precision RPD	Accuracy, % recovery
<u>Volatile Chlorinated Hydrocarbons</u>			
Carbon tetrachloride	0.20	45	40-150
Chlorobenzene	0.35	40	35-155
Chloroethane	0.90	35	40-145
2-Chloroethylvinyl ether	0.30	60	10-190
Chloroform	0.10	35	45-140
Chloromethane	0.20	60	>0-200
Dibromochloromethane	0.20	50	20-195
1,2-Dichlorobenzene	0.30	45	>0-210
1,3-Dichlorobenzene	0.65	70	5-190
1-4-Dichlorobenzene	0.50	45	35-150
1,1-Dichloroethane	0.20	25	40-140
1,2-Dichloroethane	0.10	40	45-150
1,1-Dichloroethene	0.25	50	25-170
trans-1,2-Dichloroethene	0.25	50	35-160
1,2-Dichloropropane	0.15	40	40-165
cis-1,3-Dichloropropene	0.90	60	20-180
trans-1,3-Dichloropropene	0.75	60	20-180
Methylene chloride	0.50	35	20-175
1,1,2,2-Tetrachloroethane	0.10	70	5-190
Tetrachloroethane	0.10	45	20-170
1,1,1-Trichloroethane	0.10	40	35-145
1,1,2-Trichloroethane	0.10	30	30-145
Trichloroethene	0.25	35	30-150
Trichlorofluoromethane	1.00	50	15-165
Vinyl chloride	0.40	45	20-170
<u>Semivolatile Chlorinated Hydrocarbons</u>			
2-Chloronaphthalene	1.80	50	5-155
1,2-Dichlorobenzene	2.20	40	5-175
1,3-Dichlorobenzene	2.30	35	>0-160
1,4-Dichlorobenzene	2.50	30	10-145
Hexachlorobenzene	0.10	35	10-165
Hexachlorobutadiene	0.70	30	>0-145
Hexachloroethane	0.80	35	>0-120
1,2,4-Trichlorobenzene	0.10	45	5-145

^a MDL units are µg/liter for water samples, µg/kg for low-level soils.

These usually include a verification or demonstration that the analytical measurement system was and is in control, a complete reparation and re-analysis of the affected samples, a thorough validation check of data from both original and reanalyzed samples, and full documentation of results for both original and reanalysis data. Statements outlining a mechanism for reporting the additional requirements to meet the specified QA objectives and to achieve project goals also should be included.

5.4 SITE SELECTION AND SAMPLING PROCEDURES

This section should include a scientifically credible plan for site selection and sampling. First, the scientific, regulatory, and program objectives must be defined, and then a sampling plan should be developed to meet those objectives.

Scientific, Regulatory, and Program Objectives

The scientific, regulatory, and program objectives for sampling should be clearly stated. For example, the sampling objectives for a PCB treatment technology might be to demonstrate that PCBs in soil can be reduced to less than 2 parts per million. Thus, the sampling scheme should be designed primarily to obtain initial and final PCB concentrations in soil. Additional sampling to monitor PCB levels over time of the treatment probably also should be included.

Regulatory objectives generally relate to applicable, relevant, and appropriate Federal or State environmental standards. Scientific objectives may encompass any other critical measurement data. For example, the level of oxygen in a system or the system temperature are critical to a reaction; the objectives for monitoring such parameters would include the range of allowable values.

The major objective of the SITE Program is to provide treatment technologies necessary to implement cleanup standards that require a greater reliance on permanent remedies at Superfund sites. The demonstration program is designed to provide sound engineering and cost data on selected technologies. Monitoring objectives should focus on providing information on system performance and reliability.

Selection of Sampling Locations

The techniques or guidelines used to select sampling locations should be described. The complexity of selecting sampling locations varies with the type of technology and whether it is conducted in vessels or in situ, is a continuous or a batch operation, etc. Sampling locations for a treatment technology conducted in a vessel are usually collected from one or more sampling ports. Selection of sampling locations for an in situ process requires that a grid be developed with designated sampling points. Monitoring a batch process requires that each batch be sampled before, after, and usually during the treatment process. A continuous process is usually sampled at constant time intervals. Including a description of the procedure for selecting sampling locations in the QAPP will promote consistency in the approach.

Sample Types

The sampling plan should include all types of samples relevant to a chemical technology. These may include the following:

<u>Gas samples:</u>	<u>Liquid samples:</u>	<u>Solid samples:</u>
Ambient air	Reagents (new)	Waste (untreated)
Emissions	Reagents (used)	Waste (treated)
	Waste (untreated)	Sludges/residues (i.e., filter cake, etc.)
	Waste (treated)	Soil (untreated)
	Condensate	Soil (treated)
	Effluents	
	Groundwater	
	Surface water	

Sampling Strategy

The sampling strategy is key to a demonstration evaluation program. It must be well conceived so that all critical measurements are made and sufficient samples are taken to be representative. The sampling strategy should address the following:

- ° Types of strategy (e.g., simple, stratified, or systematic random sampling).
- ° Sampling frequencies or sampling counts for each sample type.

Types of Strategy--

The characteristics of the technology determine the types of samples

taken. For example, grab samples taken at a sampling port would be appropriate for the well-mixed liquid contents of a vessel equipped with an operating agitator and operating in the batch mode. Sampling a stratified lagoon, however, might require obtaining a sample profile by collecting samples from layers in the waste at various depths. The sampling strategy for a continuous operation might entail taking samples at specified time intervals.

Sampling strategies are presented in Section 1.1.3 of "Test Methods for Evaluating Solid Waste," SW-846, 1986. Each strategy includes instructions on how to choose the necessary number of samples based on available preliminary information about the item that is to be sampled. Such information can be obtained through an initial round of sampling, or it can be estimated. The informational requirements of the sampling strategy should be kept in mind during the design of the sampling plan.

In the case of dynamic matrices (such as reactors, piping, etc.), the use of grab samples may be appropriate. The plan must address both the frequency and number of grab samples that should be taken in a given situation. A deep groundwater well that has been in service for some time may require only a single grab sample, whereas a batch manufacturing process may require many and frequent grab samples (e.g., one sample every 15 minutes over an 8-hour period). Whenever possible, charts, maps, sampling grids, flow diagrams, and/or tables delineating sampling program operations should be included in the plan.

Sampling Frequencies or Counts--

The sampling frequencies or counts of various sample types are determined by the characteristics of the technology. Generally, sampling schemes are designed to sample every stream going into a system and all streams coming out of a system, with emphasis on the waste(s) in and the waste(s) out. Table 5-4 is an example of a proposed sampling program for a treatment process. A similar table should be prepared for each QAPP developed under the SITE Program.

Sampling Procedures and Volumes

Sampling procedures should be taken from SW-846, 3rd Edition, 1986, if applicable; additional procedures should be based on the guidelines and recommendations provided in the following bibliography:

1) Water

Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, Federal Register, Volume 49, Number 209, October 26, 1984.

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

Handbook for Sampling and Sample Preservation of Water and Wastewater, EPA-600/4-82-029, September 1982.

Handbook for Analytical Quality Control in Water and Wastewater Laboratories, EPA-600/4-79-019, March 1979.

2) Water, Solids, Slurries

Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition, November 1986.

3) Air

Quality Assurance Handbook for Air Pollution Measurement Systems:

Volume I - Principles, EPA-600/9-76-005, March 1976.

Volume II - Ambient Air-Specific Methods, EPA-600/4-77-027a, May 1977.

Volume III - Stationary Source-Specific Methods, EPA-600/4-77-027b, August 1977.

TABLE 5-4. SUMMARY OF PROPOSED SAMPLING PROGRAM

Sampling location	Classification ^a	No. of samples
Raw feed tank	RF	6
Oxidized liquor filtrate	OF	3
Oxidized liquor filter cake	OC	3
Filter cake	FC	6
Effluent	EF	6
Blanks	BL	3
Total		27

^a RF = Raw feed or influent FC = Filter cake
OF = Oxidized liquor filtrate EF = Effluent
OC = Oxidized liquor filter cake BL = Blank

When standard EPA-approved procedures are used, the standard method should be referenced, usually in a table. When nonstandard procedures are used, the entire sampling procedure should be described in the text.

Solid and liquid samples may be taken under a variety of situations. Samples are most likely to be taken from containers and reaction vessels (or tanks) during a SITE project. The following subsections provide some sampling guidelines.

Sampling From Containers--

As used here, the term container refers to receptacles designed for transporting materials (e.g., drums and other smaller receptacles as opposed to stationary tanks). Weighted bottles, Coliwasas, drum thieves, or triers are the suggested sampling devices for the sampling of containers. (These devices are discussed in later sections.)

The sampling strategy for containers varies according to 1) the number of containers to be sampled, and 2) access to the containers. If the waste is contained in several containers, ideally every container will be sampled. If the large number of containers or cost factors makes this impossible, a subset of individual containers must be randomly selected for sampling. This can be done by assigning each container a number and then randomly choosing a set of numbers for sampling.

Access to a container will affect the number of samples that can be taken from the container and the location within the container from which samples can be taken. Ideally, several samples should be taken from both vertical and horizontal locations throughout the waste container. The number of samples required for reliable sampling will vary depending on the distribution of the waste components in the container. When the content of the waste is unknown, a sufficient number and distribution of samples should be taken to address any possible vertical anomalies in the waste. Containerized wastes tend to be nonrandomly heterogeneous in a vertical rather than a horizontal direction because of 1) settling of the solids and the denser phases of liquids, and 2) variation in the content of the waste as it entered the container. Bags, paper drums, and open-headed steel drums (the entire top of which can be removed) generally do not restrict access to the waste and therefore do not limit sampling.

When access to a container is unlimited, a three-dimensional, simple, random-sampling strategy, in which the container is divided by constructing an imaginary three-dimensional grid, can be used to obtain a representative set of samples. This strategy involves the following steps:

- 1) The top surface of the waste is divided into a grid whose sections either approximate the size of the sampling device or are larger than the sampling device if the container is large. (Cylindrical containers can be divided into imaginary concentric circles, which are then further divided into grids of equal size.)
- 2) Each section is assigned a number.
- 3) The height of the container is then divided into imaginary levels that are at least as large as the vertical space required by the chosen sampling device.
- 4) Each of these imaginary levels is assigned a number.
- 5) Specific levels and grid locations are then selected for sampling by using a random number table or random number generator.

Another appropriate sampling approach is the two-dimensional, simple, random-sampling strategy, which can usually yield a more precise sampling when fewer samples are collected. This strategy involves:

- 1) Dividing the top surface of the waste into an imaginary grid as in the three-dimensional strategy.
- 2) Selecting grid sections for sampling by using random number tables or random number generators.
- 3) Sampling each selected grid point in a vertical manner along the entire length from top to bottom with a sampling device such as a Drum Thief or a Coliwas.

Some containers (e.g., drums with bung openings) limit access to the contained waste and restrict sampling to a single vertical plane. Samples taken in this manner can be considered representative of the entire container only if the waste is known to be homogeneous. Precautions must be taken during the sampling of any type of steel drum because the drum may explode or expel gases or pressurized liquids.

Sampling From Reactors and Tanks--

Reactors and tanks are essentially large containers. Reactors are usually equipped for agitation and may have heating or cooling sources. Tanks are used for "holding" or storing process mixtures. Because of their

similarity, the considerations involved in sampling these vessels are much the same as those for sampling containers. As with containers, the goal is to acquire a sufficient number of samples from different locations within the waste to provide analytical data that are representative of the contents of the entire vessel. If the vessel is operating under mixed conditions, it is considered a homogeneous system; however, if the mixing conditions are observed to be operating poorly, this assumption does not apply. Poor mixing conditions are indicated by an inadequate number of mixing blades, inadequate power input, or obstructions within the vessel that permit the development of pockets of unmixed fluid.

The accessibility of the vessel contents also will affect the sampling methodology. If the vessel is an open one allowing unrestricted access, a representative set of samples is usually best obtained by use of the three-dimensional, simple, random-sampling strategy described in Section 1.4.1 of Publication SW-846, which was described earlier.

A less comprehensive sampling approach may be appropriate if information regarding the distribution of waste components is known or assumed (e.g., vertical compositing will yield a representative sample). In such cases, a two-dimensional simple random sampling strategy may be appropriate. This strategy was also described earlier. If the waste components are known to consist of two or more discrete strata, a more precise representation of the tank contents can be obtained by using a stratified random sampling strategy (i.e., sampling each stratum separately by using the two- or three-dimensional simple random sampling strategy).

Some vessels permit only limited access to their contents, which restricts the locations within the vessel from which samples can be taken. If sampling is restricted, the sampling strategy must be to take at least sufficient samples to address the potential vertical anomalies in the waste so sampling can be considered representative. Contained wastes tend to display vertical rather than horizontal nonrandom heterogeneity as a result of the settling of suspended solids or denser liquid phases. If access restricts the sampling of a portion of the vessel contents (e.g., in an open vessel, the size of the vessel may restrict sampling to the perimeter of the vessel;

in a closed vessel, the only access to the waste may be through inspection ports), the resulting analytical data will only be considered representative of the accessed area, not of the entire vessel contents unless the vessel contents are known to be homogeneous.

The most appropriate type of sampling device for vessels depends on the vessel's parameters. In general, subsurface samplers (i.e., pond samplers) are used to sample shallow vessels, whereas weighted bottles are normally used to sample vessels deeper than 5 ft. Dippers are useful for sampling pipe effluents.

Sampling of In Situ Treatments--

Potential SITE projects may address the in situ treatment of contaminated soils or sludges. In this case, materials sampled may either be solid or semisolid in nature.

A random three-dimensional sampling approach is best suited for in situ treatments. A three-dimensional sampling approach involves establishing an imaginary three-dimensional grid of sampling points in the contaminated area. Sampling points are then selected by using a random number generator or table. The sampling grid is generated by assembling maps and general soil information for the area. The map is divided into 2 two-dimensional grids with sections of equal size. These sections are then assigned numbers sequentially. Next, the depth to which sampling will take place is determined and subdivided into equal levels, which also are sequentially numbered. The horizontal and vertical sampling coordinates are then selected.

Hollow-stem augers combined with split-spoon samplers are used when sampling to depths greater than 5 ft. Shallow sampling devices are discussed in succeeding subsections.

Air Sampling Procedures--

This subsection outlines the various air sampling procedures that may be required for a SITE demonstration. Each technology has its own sampling requirements. Air sampling can be simplified by generating a complete list of compounds and estimates of their concentrations that may be emitted during

treatment with a specific technology. An example might be the monitoring of emissions of methylene chloride from an in situ treatment of contaminated soil. In this case, the compound is known and the amount of methylene chloride may be estimated. The sampling and analysis may be performed to verify actual conditions. Air sampling data will be critical to the performance of a material balance. The protocol presented herein is meant to serve as an example from which a specific air sampling program may be designed.

Air sampling may take many different forms, including sampling gases, particulate emissions, and ambient air. A specific chemical technology may not require all of these; however, an overview of the method required for each is presented.

The sampling of gases for organic constituents entails the use of EPA Method 25, which is described in the Federal Register, Volume 44, No. 195, pages 57808-57822. This method applies to the measurement of total gaseous nonmethane organics from source emissions. The procedure involves drawing a sample through a chilled condensate trap into a gas collection tank. A simplified diagram of the sampling apparatus is shown in Figure 5-4.

The sampling of particulate emissions entails the use of EPA Method 5, which is described in the Federal Register, Volume 42, No. 160, pages 41777 through 41782. This method applies to the determination of particulate emissions from a stationary source. This procedure provides for the measurement of particulate matter in air as it is collected on a glass fiber filter. The amount of particulates is determined gravimetrically after the removal of uncombined water. A diagram of a Method 5 sampling train configured for stack testing is presented in Figure 5-5.

Ambient air sampling encompasses several sampling procedures. The following points must be considered in the design of an ambient air sampling program:

- Sampling locations
- Frequency of sampling
- Pollutant to be measured
- Specific procedures

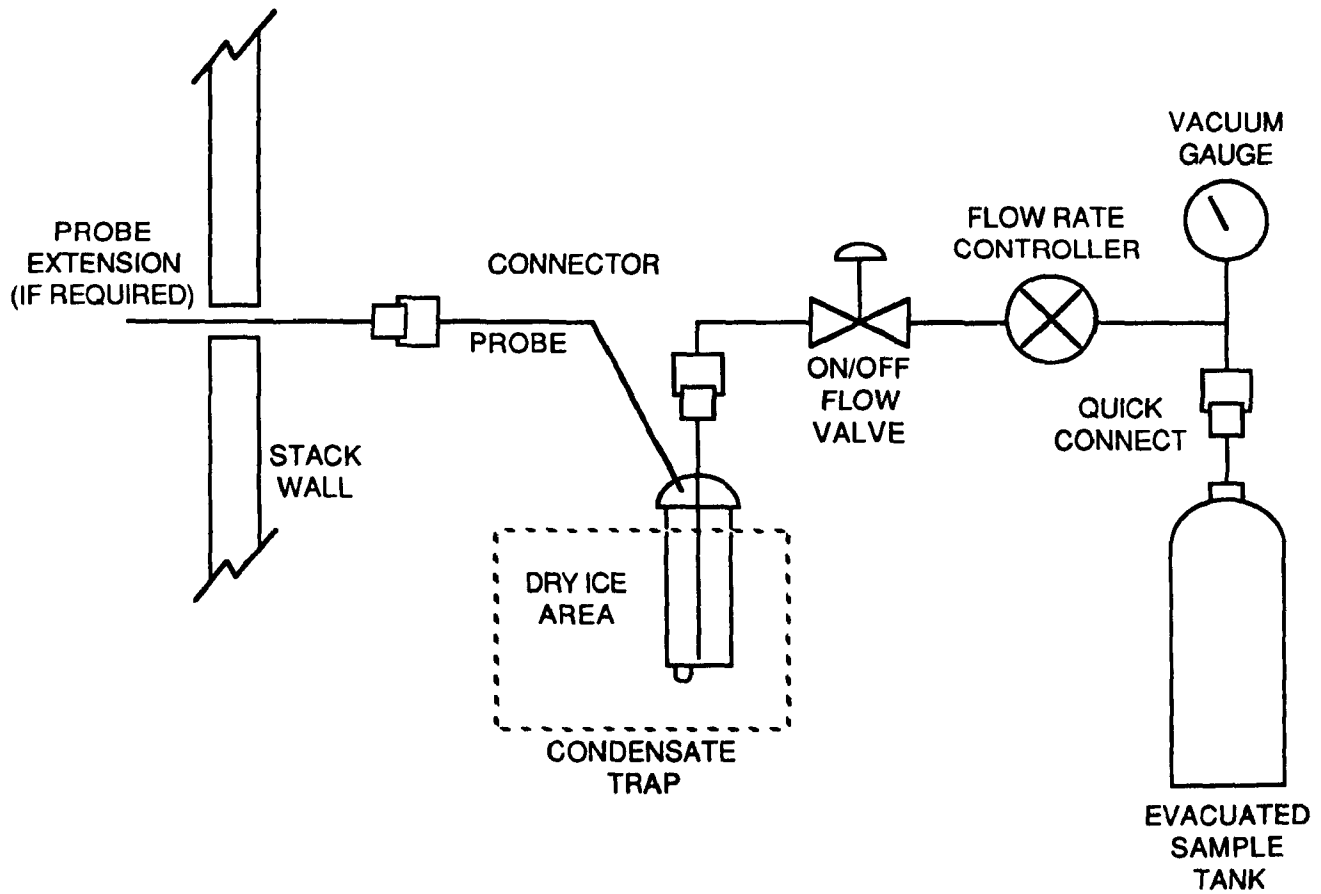


Figure 5-4. Air sampling apparatus.

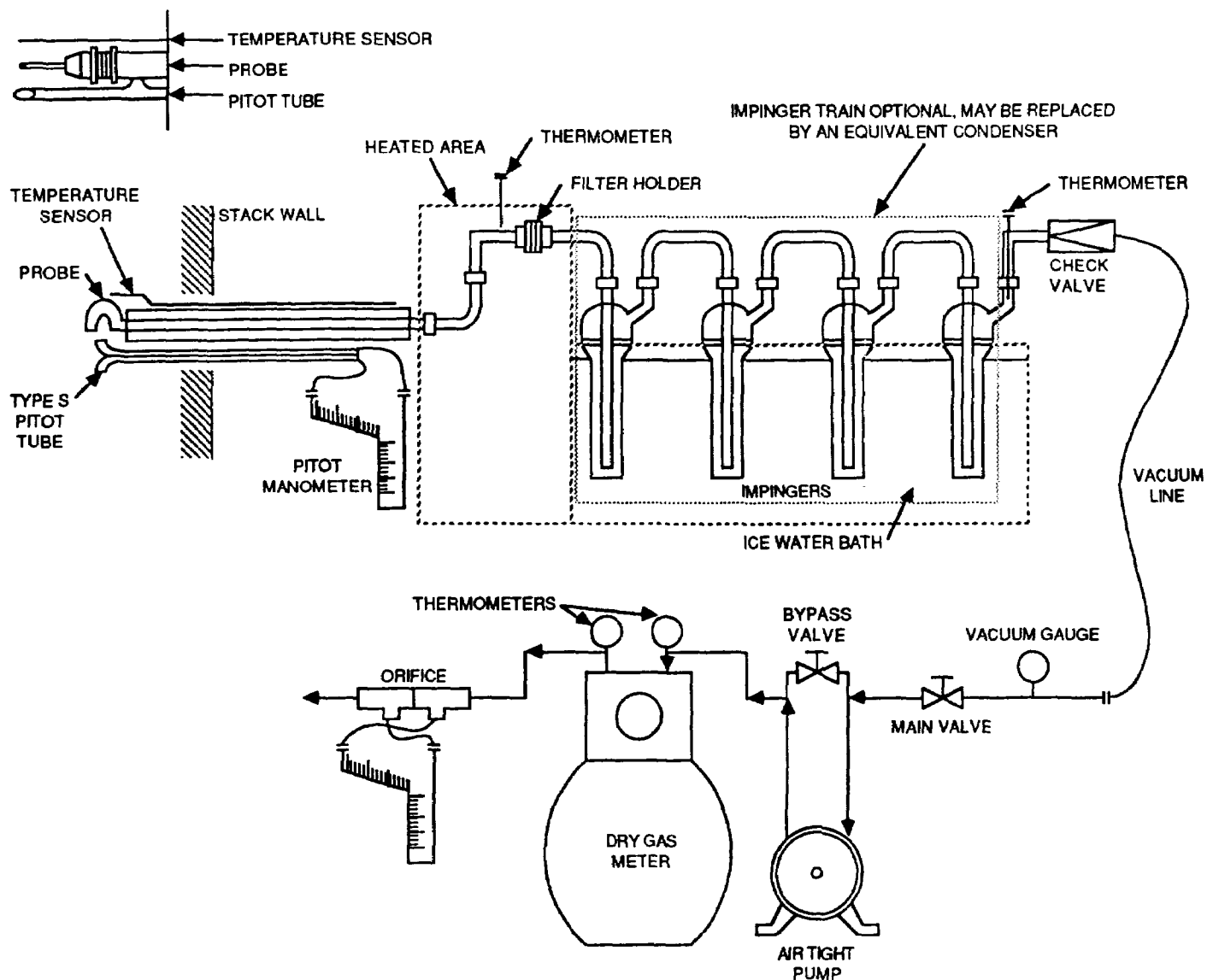


Figure 5-5. Method 5 sampling train.

The bibliography presented in Table 5-5 should assist in the assembly of an ambient air sampling program.

Sampling Volumes--

The volume of a sample for analysis should be sufficient to carry out all of the tests required. Test methods and laboratories can usually specify the necessary volume of the samples.

TABLE 5-5. BIBLIOGRAPHY FOR AMBIENT AIR SAMPLING PROCEDURES

Condition	Reference
Airborne particulates	Clements, J. B. Extraction of Airborne Particulates with Benzene, Ca-1. Research Triangle Park, NC. 1972.
Hydrocarbons	<u>Federal Register</u> , Vol. 36, No. 84, April 30, 1971.
Fugitive particulates	Cowhens, C., et al. Development of Emission Factors for Fugitive Dust Sources. EPA-450/3-74-037, 1974.
General	Jarke, F. H., Ambient Air Monitoring at Hazardous Waste Facilities. In: Proceedings of 78th Meeting of Air Pollution Control Association, 1985.
Modeling	Thibodeaux, J., and S.T. Hwang. Landfarming of Petroleum Waste--Modeling the Air Emission Problem. Environmental Progress. 1982.

The sampling volumes required depend on the number of different sample preparation procedures needed for analysis. Such techniques include graphite-furnace atomic adsorption spectrometry (GFAA), flame atomic absorption spectrometry (FLAA), inductively coupled argon plasma emission spectrometry (ICP), hydride-generation atomic absorption spectrometry (HGAA), and cold-vapor atomic absorption spectrometry (CVAA), each of which entails a different digestion procedure. The volumes shown in Table 5-6 represent those required for individual digestion procedures and recommended sample collection volumes for metals determinations.

TABLE 5-6. RECOMMENDED COLLECTION VOLUMES FOR METAL DETERMINATIONS

Measurement	Digestion volume required, ^a ml	Collection volume, ml
Metals (except hexavalent chromium and mercury)		
Total recoverable	100	600
Dissolved	100	600
Suspended	100	600
Total	100	600
Chromium IV	100	400
Mercury		
Total	100	400
Dissolved	100	400

^a Solid samples must be at least 200 g, and they usually require no preservation other than storing at 4°C until analyzed.

Sampling Containers and Equipment

The sampling equipment and the preparation and cleaning procedures should be discussed in the text. The containers used for sample collection, transportation, and storage for each sample type should also be described.

Sampling Equipment for Solids--

Organization of solid waste sampling equipment is based on the size of the solids being sampled, their moisture content, and their degree of compaction. Sampling equipment for solids includes a drum thief, a trier, an auger, a scoop, and a shovel. This equipment is referenced in EPA SW-846, "Testing Methods for Evaluating Solid Waste." Each piece is briefly described in this subsection.

Drum Thief--A drum thief consists of two slotted concentric tubes, usually made of stainless steel or brass. The outer tube has a conical pointed tip that permits the sampler to penetrate the material being sampled. The inner tube is rotated to open and close the sampler (see Figure 5-6).

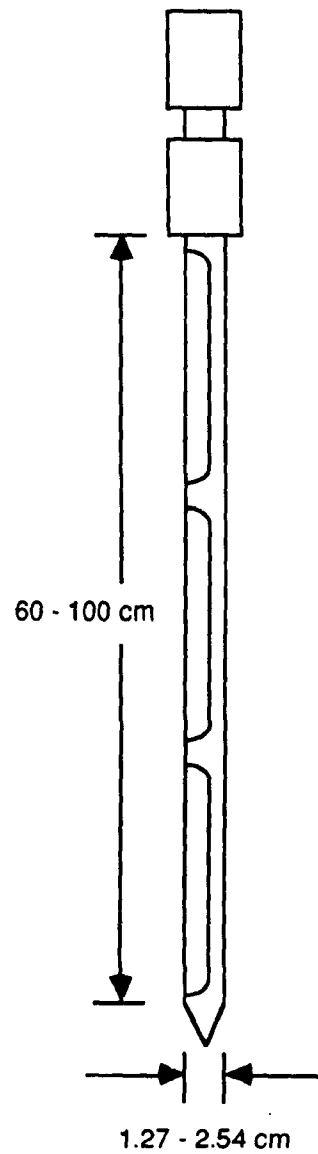


Figure 5-6. Thief sampler.

Trier--A trier consists of a tube cut in half lengthwise and having a sharpened tip that allows the sampler to cut into sticky solids and to loosen soil (see Figure 5-7). A trier samples moist or sticky solids with a particle diameter of less than one-half the diameter of the trier.

Auger--An auger, which consists of sharpened spiral blades attached to a hard metal central shaft, is used to sample hard or packed solid wastes or soil.

Scoop and shovel--Scoops and shovels are used to sample granular or powdered material in bins, shallow containers, and conveyer belts.

Sampling Equipment for Liquids--

Organization of liquid-sampling equipment depends on the type of container in which the liquid is stored. Liquid sampling will apply to many aspects of a biological technology, including:

- 1) Sampling of containerized waste prior to treatment.
- 2) Sampling of liquid-based treatment schemes.
- 3) Sampling of holding vessels that contain process byproducts.

Liquid-sampling equipment includes a composite liquid sampler (Coli-wasa), a weighted bottle, and a dipper. This equipment is referenced from EPA SW-846, "Testing Methods for Evaluating Solid Waste." Each of these three equipment items is briefly described in this subsection.

Composite liquid waste sampler (Coliwasa)--The Coliwasa is used to sample free-flowing liquids and slurries in drums, shallow open-top tanks, pits, and similar containers. It is especially useful for sampling wastes that comprise several immiscible liquid phases.

The Coliwasa consists of a glass, plastic, or metal tube equipped with an end closure that can be opened and closed while the tube is submerged in the material to be sampled.

A more detailed discussion of the Coliwasa can be found in the U.S. EPA 600/2-80-018 report entitled "Samplers and Sampling Procedures for Hazardous Waste Streams." A modification of the device is described in "Evaluation of

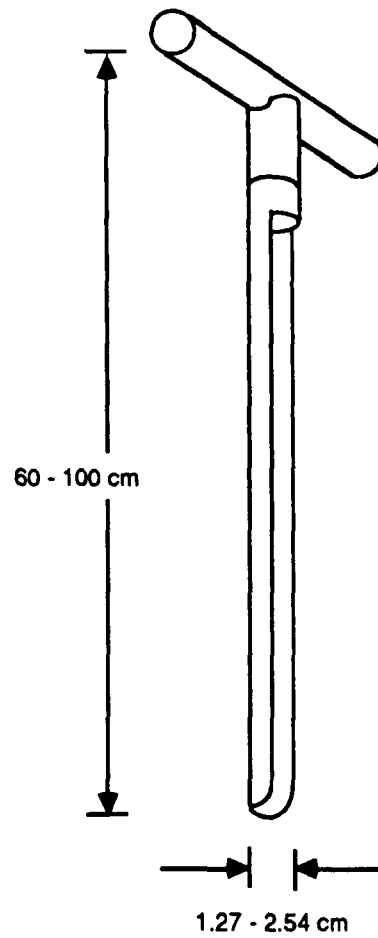
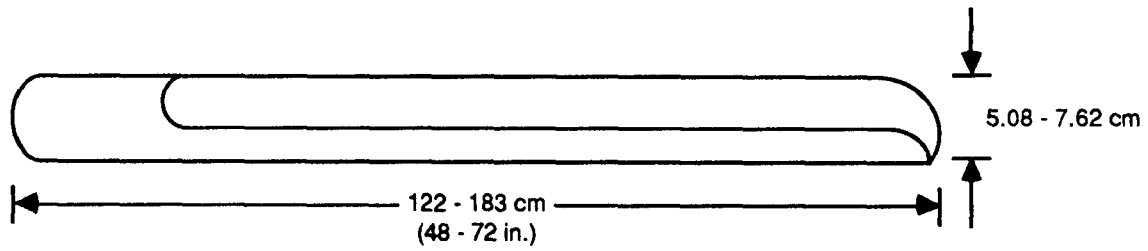


Figure 5-7. Sampling triers.

the Procedures for Identification of Hazardous Wastes," by L. R. Williams, et al. (EPA 600/4-81-027 April 1981).

Some experienced sampling personnel find the Coliwasa cumbersome and difficult to clean or dispose of after use.

Weighted bottle--This sampler consists of a glass or plastic bottle, sinker, stopper, and a line that is used to lower, raise, and open the bottle. The weighted bottle, which is used to sample liquids and free-flowing slurries, is built to the specifications in ASTM Methods D 270 and E 300. Figure 5-8 shows the configuration of this type of sampler.

Dipper--The dipper, shown in Figure 5-9, consists of a glass or plastic beaker clamped to the end of a two- or three-piece telescoping aluminum or fiberglass pole that serves as the handle. A dipper is used to sample liquids and free-flowing slurries. Dippers are not available commercially and must be fabricated to conform to individual specifications.

Sample Containers--

In the measurement of trace metals, containers can introduce either positive or negative errors by 1) contributing contaminants through leaching or surface desorption, and 2) depleting concentrations through adsorption. Therefore, particular attention must be given to the collection and treatment of a sample prior to its analysis. The following cleaning treatment sequence has been determined to be adequate to minimize contamination in the sample bottle, whether it is borosilicate glass, linear polyethylene, polypropylene, or Teflon: detergent (tap water), 1:1 nitric acid (tap water), 1:1 hydrochloric acid (tap water), and deionized water.

Standard 40-ml, screw-cap, glass volatile organics analysis (VOA) vials with Teflon-faced silicone septa may be used for both liquid and solid matrices. The vials and septa should be washed with soap and water and rinsed with distilled, deionized water. After the vials and septa have been thoroughly cleaned, they should be placed in a muffle furnace and dried at 105°C for approximately 1 hour. (Note: The septa must not be heated for an extended period of time (i.e., more than 1 hour) because the silicone begins to degrade at 105°C.)

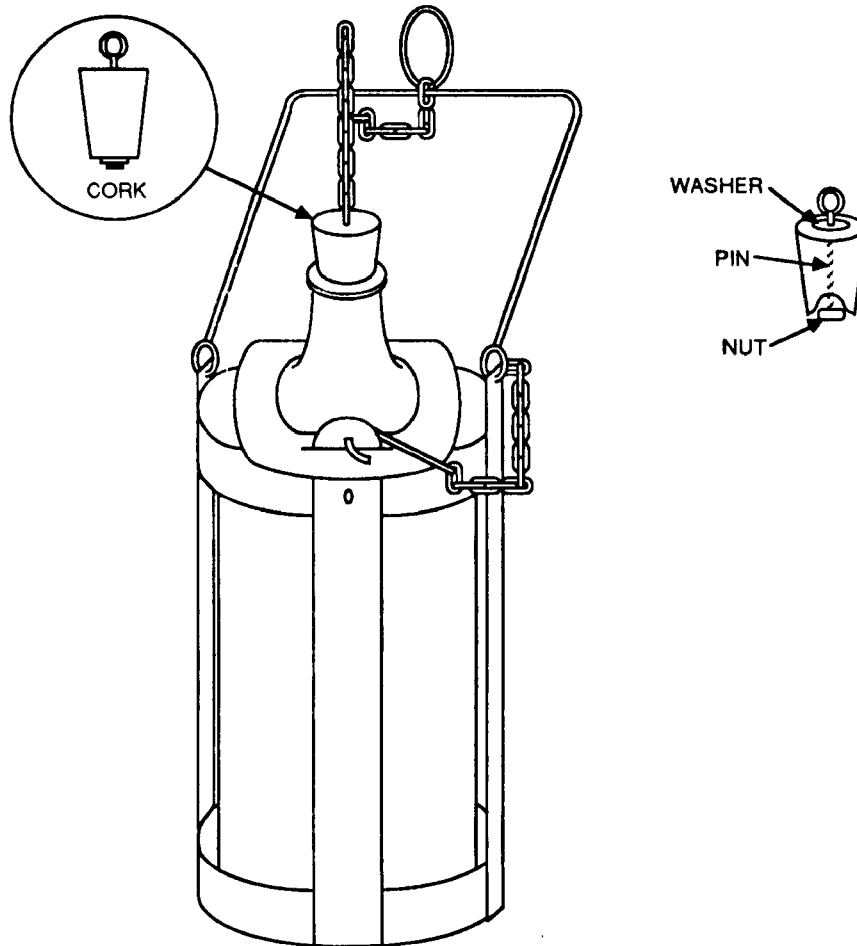


Figure 5-8. Weighted bottle sampler.

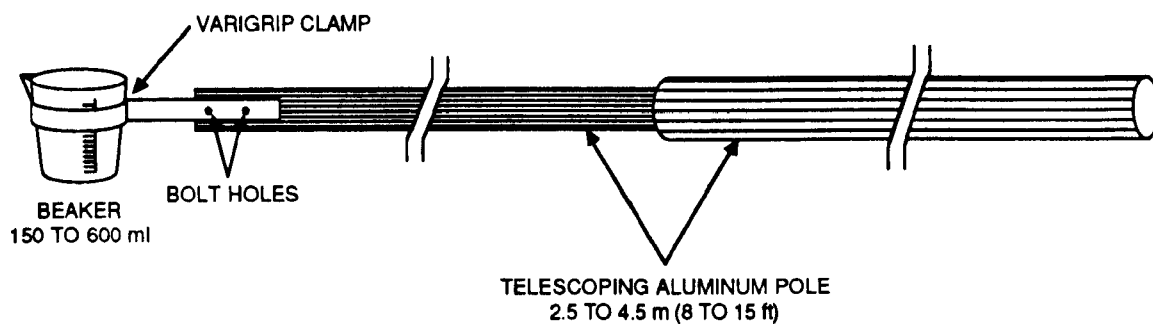


Figure 5-9. Dipper.

During sample collection, liquids and solids should be gently introduced into the vials to reduce any agitation that might drive off volatile compounds. Liquid samples should be poured into the vials without introducing air bubbles into the vial. Should bubbling result from overly aggressive pouring, the sample must be poured out and the vial refilled. Each VOA vial should be filled until the meniscus formed is higher than the lip of the vial. The screw-top lid with the septum (Teflon side toward the sample) should then be tightened onto the vial. After the lid is tightened, the vial should be inverted and tapped to check for air bubbles. If any air bubbles are present, the sample must be retaken. Two VOA vials should be filled at each sampling site.

The VOA vials for samples that have solid or semisolid (sludge) matrices should be filled to the extent possible. During the filling process, the vials should be lightly tapped to eliminate as much free air space as possible. Two vials should be filled at each sampling site.

When the VOA vials are filled, they should be immediately labeled at the point at which the sample is collected. They should not be filled near a running motor or any type of exhaust system because discharged fumes and vapors can contaminate the samples. The two vials from each sampling site should then be sealed in separate plastic bags to prevent cross-contamination between samples, particularly if the sample waste is suspected of containing high levels of volatile organics. (Activated carbon may be included in the bags to prevent cross-contamination from highly contaminated samples.) The VOA samples also can be contaminated by diffusion of volatile organics through the septum during shipment and storage. A trip blank prepared from distilled deionized water should be kept on hand throughout the sampling, storage, and shipping process to monitor for possible contamination.

Containers used to collect samples for determination of semivolatile organic compounds should be washed with soap and water and rinsed with methanol (or isopropanol). The sample containers should be glass or Teflon and have screw-top covers with Teflon liners. When Teflon is not available, solvent-rinsed aluminum foil may be used as a liner. Plastic containers or lids must not be used for sample storage because samples could become contaminated by the phthalate esters and other hydrocarbons within the plastic.

Four liters of aqueous liquids are required for semivolatile analysis, and 250-ml wide-mouth bottles are generally used for solids and sludges. Sample containers should be filled carefully to prevent any portion of the collected sample from coming in contact with the sampler's gloves and thereby causing contamination. Samples should not be collected or stored in the presence of exhaust fumes. If a sample comes in contact with the sampler (when an automatic sampler is used), reagent water should be run through the sampler and the sample should be used as a field blank.

Cleaning and Storage of Lab Glassware--

In the analysis of samples containing contaminants in the parts-per-billion (ppb) range, the use of scrupulously clean glassware is mandatory. Failure to use clean glassware can lead to a myriad of problems in the interpretation of the final chromatograms as a result of extraneous peaks caused by contamination. Particular care must be taken with glassware such as Soxhlet extractors, Kuderna-Danish evaporative concentrators, sampling-train components, or any other glassware that comes in contact with an extract that will be evaporated to a lesser volume. The process of concentrating the compounds of interest in this operation may similarly concentrate and thereby seriously distort the results.

The basic cleaning steps are as follows:

- 1) Removal of surface residuals immediately after use.
- 2) Hot soaking to loosen and flotate most particulate matter.
- 3) Hot-water rinse to flush away floating particulates.
- 4) Soaking with an oxidizing agent to destroy traces of organic compounds.
- 5) Hot-water rinse to flush away materials loosened by soaking in a deep penetrant.
- 6) Distilled-water rinse to remove metallic deposits from the tap water.
- 7) Methanol rinse to flush off any final traces of organic materials and to remove the water.
- 8) Flushing the item immediately before use with some of the same solvent that will be used in the analysis.

The preparation of sample containers should include Items 1 through 6 above. In addition to those steps, all glassware (except glass/metal containers) must be sterilized in an autoclave for 60 minutes at a temperature of 170°C. Glass/metal containers should be heated to 170°C in an autoclave for a minimum of 2 hours. Plastic sample containers may be sterilized in an autoclave at 121°C for 15 minutes, or ethylene oxide gas sterilization can be used.

Sample Preservation

Table 5-7 presents the required containers, preservation procedures, and holding times for aqueous samples. (Additional requirements can be found in Table 2-16 of SW-846, 3rd Edition.) Corresponding procedures for solids, nonaqueous liquids, slurries, particulates, and vapor samples may vary widely depending on the nature of the sample. Generally, solids, sludges, and non-aqueous liquids can be placed in glass containers with Teflon-lined caps and cooled to 4°C without jeopardizing the sample. Special samples, such as those collected in Tenax tubes, require special preparation and handling procedures, which are described in the methods. Separate samples must be obtained for analysis when the toxicity characteristic leaching procedure (TCLP) and zero-headspace extraction (ZHE) are of interest. No preservatives are added. The TCLP samples require 100 g; ZHE samples require 25 g. The ZHE sample is taken and bottled in the same way as are other samples for volatile organic analysis. Both TCLP and ZHE samples are stored and shipped at 4°C.

Sample Custody

An important part of the quality assurance program is assuring the integrity of the sample from collection to data reporting. This includes being able to trace the possession and handling of samples from the time of collection through analysis and final disposition. This documentation of the sample's history is referred to as "chain of custody." The components of the chain of custody (field-sampling protocol sample seals, sample labels with the sample number, a field logbook, chain-of-custody records, and sample analysis requests) and the procedures for their use are described in the following subsections.

TABLE 5-7. REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Measurement parameter	Container ^a	Preservation ^b	Maximum holding times ^c
Purgeable organics (EPA Method 8240)	G Teflon-lined septum	Cool to 4°C, protect from light ^d	14 days
Extractable organics (EPA Method 8250)	G Teflon-lined cap	Cool to 4°C, protect from light ^d	7 days until extraction, 40 days after extraction
Pesticides, PCB's (EPA Method 8080)	G Teflon-lined cap	Cool to 4°C pH 5 to 9	7 days until extraction, 40 days after collection
Metals (except mercury-chromium VI)	P,G	HNO ₃ to pH less than 2	6 months
Mercury (EPA Method 7470)	P,G	HNO ₃ to pH less than 2	28 days
Chromium VI	P,G	Cool, 4°C	24 hours
pH	P,G	None required	Analyze water immediately (on site); none specified for soil
Residue	P,G	Cool, 4°C	7 days
Organic carbon, total	P,G	Cool, 4°C, HCl or H ₂ SO ₄ to pH less than 2	28 days
Cyanide, total	P,G	Cool to 4°C; NaOH to pH greater than 12 ^e	14 days
Sulfide	P,G	Cool to 4°C; add zinc acetate plus NaOH to pH greater than 9	7 days

^a Polyethylene (P) or glass (G).

^b Sample should be preserved immediately upon sample collection.

^c Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. All data obtained beyond the maximum holding times will be flagged.

^d Add 0.08% Na₂S₂O₂ if residual chlorine may be present in aqueous samples.

^e 0.6 g of ascorbic acid in aqueous samples.

Field Sampling Protocol--

Chain-of-custody procedures begin with the preparation of reagents or supplies that eventually become a part of the sample (such as filters or absorbing reagents). The sample collector is responsible for initiating chain-of-custody procedures and documenting the sample source. Sample collection must be performed in accordance with standard sampling procedures. If these procedures are changed, a written justification for the deviation must be entered into the field survey log. After the sample is collected, it must be prepared in accordance with applicable sampling instructions (i.e., fixation, addition of preservative), labeled, sealed, and stored or shipped in a manner that will maintain the chain of custody.

Chain of Custody--

Chain of custody is the aspect of sample handling assurance that documents the custody chain that the sample follows. The chain-of-custody form documents where the sample originated, who authorized and who performed transmittals, how and when the sample was received, and who received it. Documentation must be clear.

After the chain-of-custody form is completed, each sample must be logged in on a sample receipt form and assigned a unique identification number. The sample receipt form identifies the condition of each sample and designates the analyses to be run. The sample description and project requirements must then be entered into the sample log, which also includes project number, date, and the analyses required. The sample is then properly stored according to prior transmitted instructions.

A sample checkout log form must be used for internal chain-of-custody tracking of samples placed in and taken out of sample refrigerators and secured storage areas. The person putting the sample in or taking it out of the designated location indicates this transaction with his/her initials, the date, and any pertinent comments. Examples of labels, chain-of-custody forms, sample receipt forms, and sample checkout log forms are shown in Figures 5-10 through 5-13, respectively.

Project Number: _____	Date: ____/____/____	Time: ____:____ am/pm
Site: _____	Sample Type: _____	C/G
Site Description: _____		
Analysis: _____	Preserv: _____	
Sampler: _____		

Figure 5-10. Sample bottle label.

When the sample analyses are completed and the sample is consumed, or discarded, the Sample Custodian logs and initials it accordingly. Only the Laboratory Director is authorized to transmit or discard samples, and then, only as authorized by the Laboratory Project Manager.

Sample Seals--

Sample seals are used to guard against and to detect unauthorized tampering of the samples between the time of collection and analysis. Items such as gummed paper seals and custody tape are used for this purpose. The seal is attached such that it must be broken to open the sample container. Seals should be affixed to containers before the samples leave the custody of the sampling personnel. Shipping containers should also contain seals to discourage and detect possible tampering.

Logbooks--

All information pertinent to a demonstration is recorded in ink in a bound, consecutively page-numbered logbook. Corrections should be lined out, not erased, and then be initialed and dated. Entries in a logbook should include the following, as applicable:

- 1) Location, description, and photographs (if applicable) of the sampling point. If the sample is obtained from a monitoring well, entry will include the well number.
- 2) Type of waste (e.g., ground water, soil, leachate, sludge, or wastewater).
- 3) Number and volume of sample taken.
- 4) Date and time of collection.
- 5) Collector's sample identification number(s).

CHAIN-OF-CUSTODY RECORD

						Samplers signature		Project No.			
Sample No.	Seq. No.	Date	Time	Sample location	Sample type	No. of conts.	Remarks				
					TOTAL NUMBER OF CONTAINERS						
Relinquished by (sign)		Date	Time	Received by (sign)		Relinquished by (sign)		Date	Time	Received by (sign)	
Relinquished by (sign)		Date	Time	Received by		Relinquished by		Date	Time	Received by (sign)	
Method of shipment				Shipped by (sign)		Received for laboratory (sign)			Time	Date	

Figure 5-11. Chain-of-custody record.

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Client: _____ Project No.: _____

Date Logged: _____

Logged by: _____

Shipper: _____

Condition of samples: _____

SAMPLES RECEIVED

Client ID	Sample no.	Analyses requested	Date of disposal	Type and volume of container

Figure 5-12. Sample receipt form.

Project: _____

- 6) Sample distribution and how transported; (e.g., name of the laboratory, the cartage agent (Federal Express, United Parcel, etc.).
- 7) References, material for the sampling site, such as maps or photographs.
- 8) Field observations.
- 9) Any field measurement made (e.g., pH, flammability, explosivity, and water depth).
- 10) The signature of the person responsible for and the date of the observation.

Summary--

Sampling situations vary widely. No general rules can be set to specify the extent of information that must be entered in a logbook. Nevertheless, records must contain sufficient information to allow someone to reconstruct the sampling activity without having to rely on the collector's memory. The logbooks should be kept in a safe place for protection.

The samples must be delivered to the laboratory for analysis within the specified allowable holding time and be accompanied by the chain-of-custody record. Samples must be packaged and shipped according to U.S. Department of Transportation and EPA regulations, and they must be delivered to the person authorized to receive samples at the laboratory.

Sampling Protocol for System Conditions

In addition to the collection of critical measurement data, monitoring the operation of system parameters is important. These parameters may vary greatly and they are normally monitored by process instrumentation. Instruments should be used to measure such variables as temperature, pressure, density, viscosity, specific heat, conductivity, pH, humidity, dew point, liquid level, flow rate, chemical composition, and moisture content. Process instruments, which are generally designed for broad application, may continuously record data on a strip chart (or some other medium). They also are used to control the operation of the process.

For example, process instrumentation might be used to measure pH in the biological degradation of phenol in soil. This hypothetical batch process might entail mixing microbial cultures into fixed volumes of contaminated soil. This process allows the microbes to break down phenol into various short-chain fatty acids (such as acetic acid) in a pH range of 6 to 9. Because the production of these fatty acids tends to lower the pH of the system, however, a buffer solution must be added to the mixing vessel. The use of pH monitoring equipment would permit the real-time measurement during processing, and the output from the pH equipment could be recorded on a strip chart for permanent storage. The pH measurements could also be used to control the addition of buffer solution. In this example, the measurement of process pH has provided the following:

- 1) The real-time measurement of the system's pH.
- 2) A permanent record of the system's pH on a strip chart.
- 3) The content of the addition of buffer solution to the process vessel.

For system conditions that are monitored on a routine basis (e.g., facility, design, or process controls and operating parameters), this section of the QAPP should include only the following items:

- ° Identification of all system condition data to be collected, descriptions of the collection methods, and a discussion of the reasons for collecting these data.
- ° Illustrations that accurately depict the monitoring locations at which the data will be collected.
- ° Specification of the frequencies of data collection.

5.5 ANALYTICAL PROCEDURES AND CALIBRATION

This section of the QAPP should include a description of or reference to an appropriate analytical method for each type of critical measurement to be made. The calibration procedures and frequency of calibration also should be discussed or referenced for each system, instrument, device, or technique used to obtain critical measurement data.

Officially approved EPA analytical methods and procedures should be selected for use if they are available and applicable. Previously validated methods should be used whenever possible. When standard EPA-approved methods are used, their descriptions can be referenced rather than included in the QAPP. (Approved methods include those in References 2 through 5.) The selected method must be appropriate for determination of the specific analytes of interest in the sample matrix involved.

When nonstandard methods of analysis are used, the QAPP must include detailed descriptions of the method, the standard operating procedure for implementing the method, and the pertinent validation data, either in this section or as an appendix.

Table 5-8 presents an example of the sample-preparation and analytical methods chosen for a project. All the methods listed are from U.S. EPA's "Test Methods for Evaluating Solid Waste", Third Edition, SW-846, November 1986, and "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020, March 1983. Each method includes a detailed discussion of the analytical procedures.

The correct calibration, operation, and maintenance of field equipment used to make measurements at the sites are extremely important throughout the demonstration. (The use of the term "calibration" is more generally applicable to chemical methodologies and field instrumentation.) Table 5-9 provides a sample list of field measurement equipment and corresponding Standard Operating Procedures (SOPs), field calibration procedures, and frequency of calibration.

Before onsite activities begin, specific personnel should be assigned responsibility for each piece of field measurement equipment. These persons should become familiar with the operation, calibration, and maintenance procedures for the items assigned to them. Calibration and maintenance activities are recorded in the daily logbooks.

Laboratory Calibration Procedures

Detailed calibration procedures for all sampling and analytical equipment required for a demonstration are provided in the respective analytical methods and should be included in the site-specific QAPP. This subsection provides general calibration procedures for the analytical instrumentation used in a wide variety of analytical methods.

TABLE 5-8. SAMPLE PREPARATION AND ANALYTICAL METHODS^a

Parameter class	Preparation method		Analytical method
	Liquid	Solid	
BDAT List			
Volatiles	NA	NA	8240
Semivolatiles	3520	3550	8270
Alcohols	NA	NA	Direct injection with a GC/FID
Metals			
Antimony	3005	3005	6010
Arsenic	3050	3050	7060
Barium	3005	3050	6010
Beryllium	3005	3050	6010
Cadmium	3005	3050	6010
Chromium(T)	3005	3050	6010
Chromium(VI)	NA	NA	7196
Copper	3005	3050	6010
Lead	3020	3050	7421
Mercury	7470	7471	NA
Nickel	3005	3050	6010
Osmium	3005	3050	7550
Selenium	3050	3050	7740
Silver	3020	3020	6010
Thallium	3020	3050	7841
Vanadium	3005	3050	6010
Zinc	3005	3050	6010
Inorganics (other than metals)			
Cyanide(T)	NA	NA	9010
Cyanide(A)	NA	NA	9010
Fluoride	NA	NA	340.2
Sulfide	NA	NA	9030
Organochlorine pesticides	3520	3550	8080
Phenoxyacetic acid herbicides	NA	NA	8150
Organophosphorus insecticides	3520	3550	8140
PCBs	3520	3550	8080
Dioxins and furans	NA	NA	8280

(continued)

TABLE 5-8 (continued)

Parameter class	Preparation method		Analytical method
	Liquid	Solid	
OAQPS List			
Volatiles	NA	NA	8240
Semivolatiles	3520	3550	8270
Ammonia	NA	NA	350.2
Alcohols	NA	NA	b
PCBs	3520	3550	8080
Dioxins and furans	NA	NA	8280
POC			
NPOC			
TCLP List			
Volatiles	NA	NA	8240
Semivolatiles	3520	NA	8270
Metals			
Arsenic	3050	NA	7060
Barium	3005	NA	6010
Cadmium	3005	NA	6010
Chromium	3005	NA	6010
Lead	3020	NA	7421
Mercury	7470	NA	7470
Selenium	3050	NA	7740
Silver	3020	NA	6010
Alcohols	NA	NA	8240
Organochlorine pesticides	3520	NA	8080
Phenoxyacetic acid herbicides	NA	NA	8150
Others			
pH	NA	9045	9040
Chloride	300.0	Water ex- traction	NA
Total residue	NA	NA	160.3
COD	NA	NA	410.3
TOC	NA	NA	415.1
Ethylenediamine	3520	3550	8270
Tartaric acid	3520	3550	8270
Ash (after ignition at 550°C)	-	-	160.4

^a Methods from "Test Methods for Evaluating Solid Waste", 3rd ed., SW-846, November 1986 and EPA 600/4-79-020, "Methods for Chemical Analysis of Water and Wastes".

TABLE 5-9. SAMPLE FIELD EQUIPMENT CALIBRATION CHECKLIST

Instrument	SOP	Calibration Procedure	Frequency
HNU Model P1-101 trace gas analyzer	See HNU instruction manual,	See HNU instruction manual, pp. 3-1 to 3-6 and 8-1 to 8-10 for calibration gas procedures	Daily
		Zero instrument	Daily or after 4 hours of use
pH meter	Per manufacturer's instructions	Immerse electrodes in buffer solution of known pH, adjust meter to proper reading. Remove and rinse electrodes. Immerse in second buffer solution of a known pH, adjust meter. Standard buffer solutions should bracket the sample pH. Repeat above until readings are within 0.05 units of buffer solution values. All solutions should be at ambient temperature	Daily or after 4 hours of use
Conductivity meter	Per manufacturer's instructions	Obtain correction factor by comparing observed reading with a standard salt solution and distilled or deionized water	Daily
		Zero instrument	Daily or after 4 hours of use
MSA Model 53 explosimeter	Per procedures stated on meter	Per procedures stated on meter	Daily
Foxboro/Century organic vapor analyzer, Model OVA-108	Per Foxboro/Century instruction and service manual Number MI-2R900AD	See Foxboro/Century instruction and service manual, pp. 7 to 9 ^a	Daily

Plasma Emission Spectrometer--

A plasma emission spectrometer, which operates under the same general principles as a UV light spectrometer, may be used to analyze for a variety of inorganic constituents. The general calibration procedure for the plasma emission spectrometer is as follows:

- 1) The instrument should be set up and programmed according to the *procedures contained in the instrument operating manual*.
- 2) Instrument linearity and detection limits are determined quarterly.
- 3) All calibration standards, the calibration blank, control samples, and regular samples are spiked with the internal standard.
- 4) All standards and samples are analyzed in duplicate, and a blank rinse is used between each sample and standard.
- 5) The first run of each analysis is a signal compensation blank, followed by a calibration blank and mixed calibration standards (5 ppm) for each element to be analyzed.
- 6) An independent control sample is run after the calibration standards, after every 10 to 15 samples, and after the last sample. If the results of this standard fail to agree within ± 10 percent of the accepted value, the previous 10 to 15 samples must be reanalyzed.
- 7) Each matrix is spiked with the elements being analyzed to determine any matrix interferences (acceptable recovery is 75 to 125 percent). At least 1 sample in 20 is spiked in duplicate.
- 8) Any sample exceeding the linear range for one or more elements is diluted and reanalyzed.

Other Inorganic Methods--

The following are general calibration procedures for other inorganic methods:

- 1) Analytical balances are checked with Class 5 weights each day they are used. If a trend toward inaccuracy is found and cannot be corrected by laboratory personnel, professional service must be obtained. The balances are professionally serviced and checked against NBS-certified weights each year.
- 2) Atomic absorption spectrophotometers are used in a number of EPA-approved methods, including 7060, 7080, 7130, and a variety of other metal analyses. Atomic absorption spectrophotometers are calibrated for each metal analyzed, and a record is kept of instrument response. A minimum of a blank plus four upscale points are

used for calibration. This full set of standards is analyzed initially and then periodically during a large set of samples and after the last sample. Should a lack of sensitivity or other malfunction be detected that cannot be corrected in-house, professional services must be obtained.

- 3) Ultraviolet/visible (UV) spectrophotometers may be used to analyze for a variety of ions, e.g., the permanganate ion. The UV spectrophotometers are checked with standard color cuvettes each day they are used and are checked for minor and grating alignment monthly. A full set of calibration standards (consisting of a blank and at least four upscale points) is analyzed to establish the calibration curve. A check sample is analyzed after every tenth sample and after the last sample. If the results of this check sample fail to agree within ± 10 percent of the accepted value, the instrument is recalibrated and the previous 10 samples are reanalyzed. Concentrations of the standards are as specified in the method. Service criteria are the same as those described for the other instruments.
- 4) The ion chromatograph is calibrated each day it is used. A minimum of a blank and four upscale points are used to establish the calibration curve. A check sample is analyzed after every tenth sample and after the last sample. If the results of this check sample fail to agree within ± 10 percent of the accepted value, the instrument is recalibrated and the previous 10 samples are reanalyzed.

Organic Methods--

The following are general calibration procedures for organic methods:

- 1) The high-pressure liquid chromatograph (HPLC) and the standard gas chromatographs (GCs) are calibrated each day analyses are performed. These instruments provide for the separation and analysis of numerous organic-based compounds, such as polychlorinated biphenyls (PCBs) (EPA Method 8080). At a minimum, a blank and five upscale points are used to establish the calibration curve. One of the calibration standards is reanalyzed after every tenth sample and after the last sample to establish the continuing validity of the initial curve. The criterion for continuing validity is specified in the individual methods. If the criterion is not met, the instrument is recalibrated and the previous 10 samples are reanalyzed.
- 2) The GC/mass spectrometers (GC/MS) provide for analysis of volatile and semivolatile organic compounds as outlined in EPA procedure 8240. The GC/MS's are checked for mass calibration with FC-43 each analysis day and are tuned to the criteria specified in the method with BFB or DFTPP (4-bromofluorobenzene or decafluorotriphenylphosphine) depending on whether samples are intended for volatile or semivolatile analysis. Calibration requires a minimum of a blank and three upscale points and the use of the internal standard method of determining response factors. Continuing calibration is based on satisfactory agreement of a daily check standard. The

requirements for satisfactory agreement are specified in the QA plan for the project or the specified method. If satisfactory agreement is not obtained, the instrument is recalibrated before proceeding with the analyses.

Preventive Maintenance

The preventive maintenance of analytical, laboratory, and operational process equipment is important to the success of the demonstration. The Contractor and Developer should assemble and regularly review a maintenance checklist to include the following:

- 1) A complete list of all operational process and analytical laboratory equipment and its location.
- 2) Frequency and description of each preventive maintenance activity, e.g.:

XYZ Sampling Pump:	Check seals	Weekly
	Repack bearing	Monthly
- 3) The individual designated to perform each preventive maintenance task.

Spare parts for process equipment, analytical equipment, and sample collection must be inventoried on a regular basis. Although it is not necessary to keep an onsite supply of those equipment parts that are stocked locally, a supply of hard-to-find parts should be kept on site for emergency repair. Availability of spare parts is essential to the implementation of a preventive maintenance program.

5.6 DATA REDUCTION, VALIDATION, AND REPORTING

For all laboratory data generated and processed to be scientifically valid, defensible, and comparable, the correct equations and procedures must be used to prepare those data. Each method presented in Reference 2 is accompanied by a set of calculations and appropriate forms for data presentation. Each analytical method presented in SW-846 provides detailed instructions for calculating concentrations of specific analytes. The following subsections also present general criteria concerning the processing of data.

Data Reduction

Analytical results should be reduced to concentration units specified in the analytical procedure, as cited in SW-846 (or a similar reference). If units are not specified in the analytical procedure, data from the analysis of water samples should be reported in milligrams/liter for inorganic parameters and micrograms/liter for organic parameters. Data from the analysis of solid samples should be converted to units of milligrams/kilogram for inorganic parameters by use of the following equation:

$$X_w = [X_v \times V \times \text{d.f.}] / w$$

where X_w = Reported value, mg/kg
 X_v = Reported sample value, mg/liter
 V = Sample volume, liters
d.f. = Dilution factor
 w = Sample weight, kg

This same basic equation is used for organic concentrations, except that X_w will be in units of micrograms/kilogram and X_v will be in units of micrograms/liter.

Data Validation

Data validation is the process of screening data and accepting or rejecting these data on the basis of sound criteria. The QA personnel should use validation methods and criteria that are appropriate for the type of data involved and purpose of the measurement. Validation procedures should include the following:

- 1) Ensuring adherence to the specified sampling, preparation, cleanup, and analysis procedures.
- 2) Examining precision, accuracy, and other QC data generated during the project.
- 3) Ensuring the use of properly calibrated and maintained sampling equipment and analytical instrumentation.

Outlier data may be thought of as QC data that fall outside of a specific QA objective window for accuracy or precision. Such data should be identified during the daily examination of QC data. When outlier data are identified, an investigation should be conducted to isolate the causes, and the results should be promptly reported to the Project Manager (or his representative). If possible, affected samples should be reanalyzed. If reanalysis corrects the problem, then only the reanalysis data should be reported. If both sets of analysis contain the outlier, both results should be reported, and the outlier should be identified in the final report. If reanalysis is not possible, the initial results should be reported, and the outlier should be identified in the final report. Records of all data should be maintained, even those judged to be "outlying" or of spurious value. The persons validating the data should be sufficiently knowledgeable with regard to the technical work to be able to identify questionable values.

Analytical data generated in this program are considered useful if the quality control data for a spiked sample achieve the precision and accuracy goals stated in the site-specific QA plans and the sample was analyzed within the maximum holding time. Spiked laboratory-pure water should be used in assessing data usefulness when the sample itself cannot be spiked because of its physical nature or the nature of the analysis parameters. If the precision and accuracy do not achieve the QA objectives, these data should be flagged, and the impact of not meeting the QA objectives should be delineated.

Problems of this type may be identified by the analysts or their supervisor, who may take the corrective action specified in the specific QA Plan. The QA Officer must be notified when this occurs. When corrective action is needed, sufficient reanalyses must be performed to establish whether the deviant QC result was caused by the sample matrix or by an out-of-control analysis. Out-of-control analyses must be repeated.

Data Reporting

Figure 5-14 presents a flow chart depicting the data-reporting scheme. Data will be reported in standard units, usually as micrograms/liter or milligrams/liter for trace analytes. Any necessary deviations from SW-846 procedures should be fully documented.

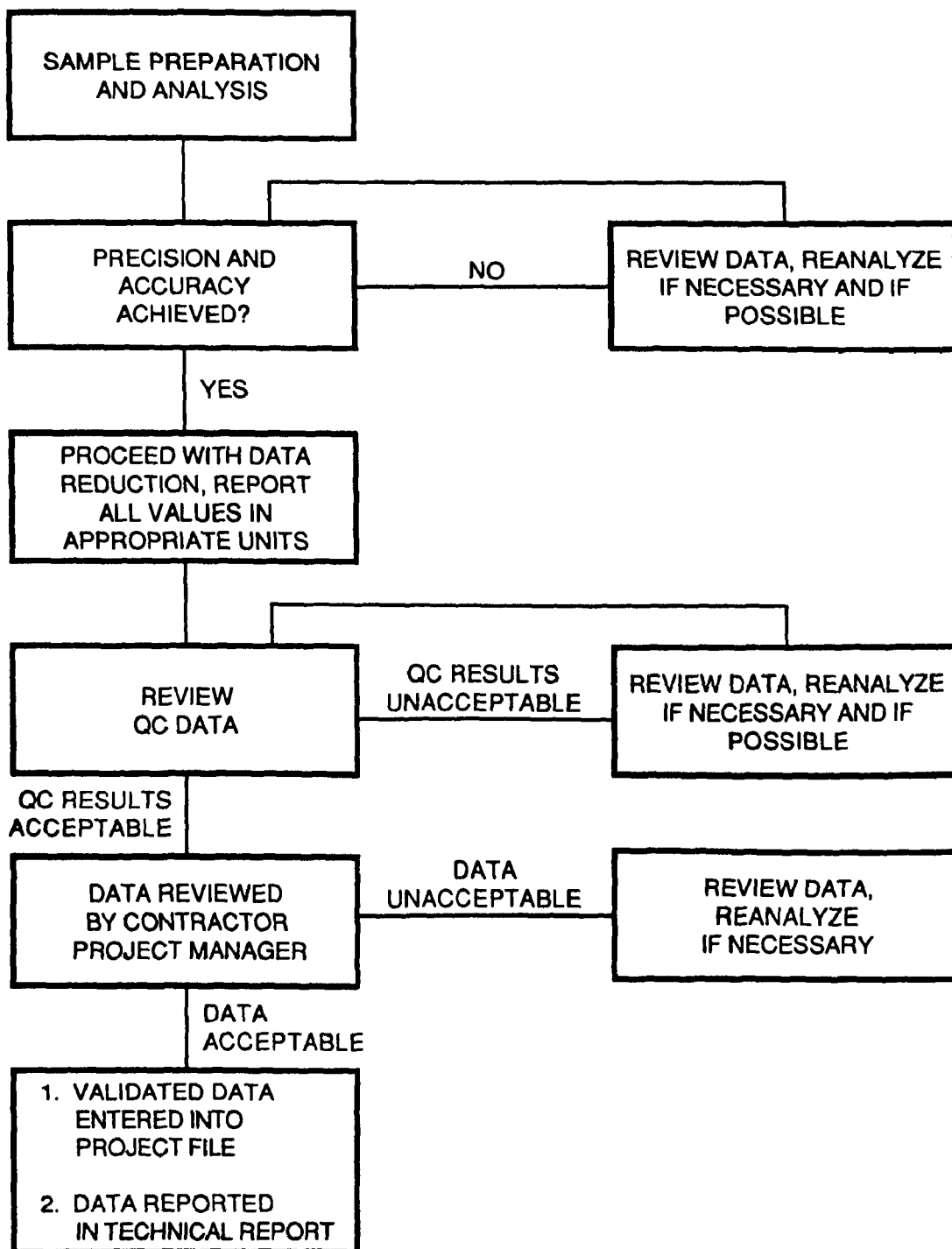


Figure 5-14. Data reporting scheme.

5.7 INTERNAL QUALITY CONTROL CHECKS

The QA Project Plan for each task should specify the internal quality control measures that will be used to ensure optimum valid data collection. The QA Plan should specify the appropriate QC checks, the control limits, and the planned corrective action to be used if control limits are exceeded. The quality control checks outlined in the following subsections are oriented toward chemical testing. Similar QC checks are not as specifically defined for biological tests. It is important that the Developer review all biological evaluations with appropriate laboratory personnel to ensure that appropriate measures are being taken during each analysis.

Internal Quality Control Measures

Internal quality control procedures for analytical programs are summarized in this subsection. The analytical QC program should make use of QC samples whose values are known, calibration check samples, method blanks, replicate aliquot analyses, surrogate spikes, and matrix spikes.

Known QC samples, called laboratory control standards (LCS) or standard reference solutions or laboratory-purewater spikes, are prepared by adding known quantities of EMSL-Cincinnati, NBS Standard Reference, or independently prepared stock materials to deionized water. The LCS are routinely used to establish that an instrument or procedure is in control before sample analysis begins. The analysts report the LCS result to the QC clerk, who plots it on the control chart.

A calibration check sample is one of the working calibration standards periodically used to check that the original calibration is still valid.

A method or reagent blank consists of deionized water carried through the entire preparation and analysis procedure. Analytical results should not be corrected for the method blank. Instead, both the uncorrected sample results and the blank results will be reported. Method blank samples for organic compositional analysis should be analyzed for volatile, base-neutral/acid, and PCB fractions. For inorganic analysis, separate method blanks for flame, furnace, and cold vapor digestates should be analyzed.

At least one sample, chosen at random, should undergo a complete duplicate analysis. Replicate aliquots of actual samples or QC samples are analyzed so that the precision of the analytical procedure can be estimated.

A surrogate spike compound is one that mimics the behavior of target analytes in terms of stability, preparation losses, measurement artifacts, etc., but does not interfere with the target analyte measurement. The surrogate spiking procedure should be used in the analysis of volatile and base-neutral fractions by GC/MS. Recovery data should be included in the QA/QC section of the final report.

A matrix spike sample is prepared by adding known amounts of the target analyte(s) to an aliquot of an actual sample. As indicated in SW-846 (3rd Edition), duplicate matrix spikes of a field sample are generally used to determine both precision and accuracy (percentage recovery). The frequency varies between 1 in 10 and 1 in 20 samples of a given matrix type, depending on the particular procedure.

Type and Frequency of Use of QC Measures

A minimum standard for the type and frequency of use of each QC measure should be presented. These requirements may be revised depending on the timing and scope of the demonstration. Any alterations to these measures require EPA approval. The following is a schedule of instrument QC checks and their frequency:

- 1) Calibrations (or calibration checks) should be performed at least daily. Laboratory personnel should consult standard operating procedures and manufacturers' literature to define specific intervals.
- 2) An LCS analysis value within control limits is performed daily before sample analysis is begun.
- 3) A calibration check or an LCS sample within the original value of the method limit is run after every 10 samples (inorganic analysis).
- 4) An appropriate calibration check or LCS samples may be run as often as every five samples for some organic analyses, whereas only a daily calibration check sample may be run for other organic analyses (such as GC/MS).
- 5) A method blank is used with each analysis batch (20 or less samples). The preparation and/or extraction dates should match those of the samples they represent.
- 6) Surrogate spikes are used in all organic GC/MS analyses.

- 7) Ten percent of all inorganic samples and five percent of all organic samples (or a minimum of one per sample batch of 20 or less, whichever is greater) should be split in the laboratory into three aliquots prior to the first sample preparation or extraction step in the procedure. Two aliquots should be spiked with the same amount of the desired constituents (duplicate matrix spikes). Duplicate matrix spikes are used to determine precision and accuracy (percentage recovery).
- 8) A trip blank should be used during each sampling episode. Trip blanks should be prepared beforehand in the laboratory, carried into the field, and then returned to the laboratory for analysis. A trip blank provides a check on sample contamination resulting from sample transport or shipping and from site conditions.

Special QC Considerations

Reagents used in the laboratory are normally of analytical reagent grade or higher purity; each lot of acid and solvent used is checked for acceptability prior to its use. All reagents should be labeled with the date received and date opened. Deionized water quality should be routinely checked. Subcontractor laboratories should make at least those QC checks outlined here and report the results of all QC samples with the data. No field spikes are planned because it is difficult to spike samples accurately in the field.

In the laboratory, preventive maintenance should include attention to glassware, water supply, reagents, and analytical balances, as well as the more complex instrumentation. Table 5-10 summarizes the preventive maintenance procedures for the instruments expected to be used during a demonstration.

5.8 PERFORMANCE AND SYSTEM AUDITS

The QA plan should describe the internal performance evaluation and technical system audits for monitoring the capacity and performance of each critical measurement system. Internal laboratory quality control checks, including analysis of duplicates, spikes, and blanks, are described in Subsection 5.9. Data generated as part of the internal quality control program should be reviewed by the QA Officer (or a subcontractor's QA Officer) and the Contractor Project Manager to assure the absence of systematic bias or trends and to ensure that appropriate corrective actions are taken as required. Quality problems identified and necessary corrective actions taken should be included in the SITE report.

TABLE 5-10. MAINTENANCE PROCEDURES AND SCHEDULE FOR MAJOR INSTRUMENTATION

Instrumentation	Maintenance procedure/schedule	Spare parts
GC/MS	<ol style="list-style-type: none"> 1. Replace pump oils annually. 2. Change septa daily. 3. Change gas line dryers quarterly. 4. Replace electron multiplier as needed. 	Syringes Septa Columns Liquid nitrogen
Inductively-coupled plasma spectrometer	<ol style="list-style-type: none"> 1. Clean optical surfaces monthly or as needed. 2. Clean torch assembly when discolored or after 8 h of running high-dissolved-solids samples. 	Autosampler tubing Liquid argon Injector tubes Spare windows RF coil
Perkin-Elmer model atomic absorption spectrophotometer graphite furnace	<ol style="list-style-type: none"> 1. Clean optical surfaces weekly. 2. Condition graphite tube before starting analysis. 3. Check condition of graphite contact rings weekly; replace if pitted or worn. 4. Clean atomizer windows weekly or as needed. 	Graphite tubes Graphite contact rings Autosampler tubing
GC	<ol style="list-style-type: none"> 1. Change septa daily. 2. Check syringe for burrs daily. 3. Change gas line dryers quarterly. 4. Leak check when installing a new analytical column. 5. Periodically check inlet system for residue buildup. 	Syringes Columns Septa Swagelok fittings FID jets Injection port liners

A technical systems audit consists of an evaluation of all components of a critical measurement system. It includes a careful evaluation of both field and laboratory quality control procedures. Normally, system audits are performed before or shortly after a system is operational. In addition, secondary audits should be made on a regularly scheduled basis during the lifetime of the project. An onsite systems audit also may be a requirement for many formal laboratory certification programs.

The field activities of a subcontractor should be audited internally at least once by the Contractor to assure that the required equipment and procedures for sample collection, preservation, shipping, handling, and documentation are being used. Audit results should be reported on a regular basis and also be included in the final SITE report.

5.9 CALCULATION OF DATA QUALITY INDICATORS: SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, COMPLETENESS, AND METHOD DETECTION LIMIT

The quality control activities undertaken during the demonstration should include:

- 1) Ongoing activities to assure that measurement systems are under control.
- 2) Activities specific to a given treatment technology evaluation experiment.

The ongoing quality control activities consist principally of evaluating data obtained (when possible) from the following sampling categories:

- | | |
|-------------------------|---------------------|
| ° Calibration standards | ° Laboratory blanks |
| ° Surrogate spikes | ° Control standards |
| ° Matrix spikes | ° Field samples |
| ° Duplicate analyses | |

Procedures used to evaluate these data should include calculations of arithmetic means, standard deviations, relative percent difference (relative range) if less than three data points, comparison of differences between duplicate analyses, and spike sample values expressed as percentage recovery. These values should be included as an appendix in each SITE report.

Project-specific data evaluation procedures depend on the demonstration and, in turn, on the types and numbers of field samples to be collected. In general, the overall objectives of the technology demonstration probably should include a comparison of concentrations of one or more measurement parameters in a waste before and after treatment and qualitative and quantitative determination of accompanying emissions, byproducts, etc. For the most part, the statistical procedures used for this work are expected to be simple and straightforward. For example, they should include calculation of limits of detection, limits of quantification, standard deviation, and relative percent difference (relative range), and an evaluation by least-squares linear regression. In all cases, these procedures should be taken from appropriate EPA documents and manuals for the media under investigation. Overall guidance should be obtained from the EPA document entitled "Calculation of Precision, Bias, and Method Retention Limit for Chemical and Physical Measurements," issued on March 30, 1984, as Chapter 5 in the EPA Quality Assurance Manual.

Precision

Precision should be estimated by the analysis of replicate samples. If three or more values are determined, they should be expressed as the standard deviation, s , which is determined by the following equation:

$$S = \frac{\sum_{i=1}^N X_i^2 - \frac{1}{N} \left(\sum_{i=1}^N X_i \right)^2}{N - 1}$$

where S = standard deviation
 X_i = individual measurement result
 N = number of measurements

Relative standard deviation may also be reported. If so, it should be calculated as follows:

$$RSD = 100 \cdot \frac{S}{\bar{X}}$$

where RSD = relative standard deviation, expressed in percent
 S = standard deviation
 \bar{X} = arithmetic mean of replicate measurement

If only two values are determined, precision should be estimated by calculating the relative percent difference (relative range) with the following equation:

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

where RPD = relative percent difference
 D_1 = the larger of the two observed values
 D_2 = the smaller of the two observed values

Accuracy

Accuracy should be estimated from the analysis of QC samples whose true values are known, surrogate spike recoveries, or matrix spike recoveries, and it should be expressed as percentage recovery. The formulas for calculating

these values are as follows:

- 1) For QC samples:

$$\text{Percentage recovery} = 100 \left(\frac{\text{measured value}}{\text{true value}} \right)$$

- 2) For surrogate spikes:

$$\text{Percentage recovery} = 100 \left(\frac{\text{measured value}}{\text{true value}} \right)$$

- 3) For matrix spikes:

$$\text{Percentage recovery} = 100 \left(\frac{C_i - C_o}{C_t} \right)$$

where C_i = value of spiked aliquot
 C_o = value of unspiked aliquot
 C_t = value for spike added

Completeness

Completeness should be reported as the percentage of all measurements made whose results are judged to be valid. The following formula should be used to estimate completeness:

$$C = 100 \left(\frac{V}{T} \right)$$

where C = percent completeness
 V = number of measurements judged valid
 T = total number of measurements

Method Detection Limit (MDL)

This approach for determining MDL and method quantitation limit (MQL) is taken from Chapter 1 of SW-846 (3rd Edition).

The detection and quantification limits of analytes are evaluated by determining the noise level for each analyte. If an analyte is present, the noise level adjacent in retention time to the analyte peak may be used. For wave length dispersive instrumentation, multiple determinations of digestates with no detectable analyte may be used to establish the noise level. The

method of standard additions should then be used to determine the calibration curve for the digestate or extracted sample in which the analyte was not detected. The slope of the calibration curve, m , should be used to calculate MDL and MQL by using the following:

m = slope of calibration line

S_B = standard deviation of the average noise level

$MDL (MQL) = K S_B / m$

For $K = 3$; MDL = method detection limit

For $K = 5$; MQL = method quantitation limit

The methods given in SW-846 (3rd edition) generally provide MDLs and practical quantitation limits (PQLs) for various matrices. Each laboratory must determine the MDLs for the methods and explain the procedure used to obtain them. These values cannot necessarily be obtained for all samples. The impact of varying matrices on MDLs should be addressed in the Developer's laboratory report.

5.10 CORRECTIVE ACTION

An analyst who obtains a value that fails to meet specifications for accuracy or precision should immediately notify the QA Officer, who will then take the following steps to correct the deficiency:

- 1) Review the calculations for calculating and transcribing errors.
- 2) Review the analysis with the analyst to determine whether any procedural errors were made.
- 3) Examine reagents and equipment to determine if they were functioning and were used properly.
- 4) Examine the instrumentation for calibration and signal response.

If calculation errors were found and corrected, no further action would be taken. If other causes were responsible, analyses would be rerun to obtain results within specifications or to document that the sample matrix is the cause of the problem.

When these procedures fail to reveal an apparent problem, the Quality Assurance Officer and the Project Manager must determine the appropriate action to be taken.

Because of its generic nature, this QA Plan cannot address every situation that may arise with the wide variety of sample types and analyses included in a particular demonstration. Therefore, the following cases are given as illustrations to assist in determining what corrective actions should be applied.

Examples of Out-of-Control Events and Corrective Actions

The following are general examples of out-of-control events and appropriate corrective actions:

- 1) Two testing laboratories do not agree in the determination of fecal coliform. The failure to dechlorinate samples or inaccurate temperature control may have caused the discrepancies. Laboratory personnel should review all procedures to ensure that the test is administered correctly.
- 2) The percent difference for response factors on GC/MS calibration check standards is greater than 20 percent. This is considered a warning limit. If the percent difference is greater than 25, corrective action must be taken. If the source of the problem cannot be determined after corrective action, a new initial calibration must be generated before sample analyses are continued.
- 3) The laboratory method blank exceeds the required criteria. The source of contamination must be investigated, and appropriate corrective measures must be taken and documented before sample analysis proceeds. Samples processed with an out-of-control method blank must be reanalyzed.
- 4) The analytical spike value for a graphite furnace metals analysis of a sample is not within the required 75 to 125 percent recovery window. The sample is diluted and another analytical spike is prepared and analyzed. If the spike value is still not within the required percent recovery window, the method of standard addition must be used for the sample analysis.

General

Because of the wide variety and total number of samples that must be analyzed for a demonstration within a very short time period, the QA objective is to apply currently accepted procedures and to obtain the best results possible with those procedures. The development of new procedures is not possible, but corrective action must be sufficient to show that the analytical procedures are in a state of statistical control and that QC results outside the QA objectives are due to problems in the sample matrix. Some of the

methods call for analyzing all samples by the method of standard addition if certain criteria are not met. Depending on the sample load and how critical the deadlines are, EPA QA personnel may waive this requirement. Sufficient spikes and/or reanalyses should be run to document these problems; however, it may be impossible to resolve them prior to the deadlines.

5.11 QUALITY CONTROL REPORTS

General

During the course of the project, a reporting mechanism must be in place to keep demonstration management and EPA informed of analytical progress, any problems, and the results of QA/QC assessments. These reports should address progress, data quality, analytical problems, and possible corrective action procedures and should include an up-to-date assessment of data quality in terms of precision, accuracy, completeness, and MDLs attained. Significant problems that come to light during the demonstration should be relayed to the EPA Project Manager. Also, any planned deviations to the approved QA Plan should first be cleared with the EPA Project Manager.

Final Reports

In the final demonstration report, a separate QA section should summarize the data quality information (including audit reports, if audits were conducted) obtained for each site. The QA section should contain the following:

- 1) A data quality statement for precision.
- 2) A data quality statement for accuracy.
- 3) A discussion of the QA objectives that were met and those not met.
- 4) If QA objectives were not met, a discussion of that impact on the project.

In addition to the final SITE report, EPA intends to develop an Application Analysis Report for each technology demonstration. This report will address the application of the technology to other waste/matrix scenarios based on the demonstration results.

5.12 REFERENCES FOR SECTION 5

1. Simes, G.F. Quality Assurance Procedures for HWERL. Hazardous Waste Engineering Research Library, Office of Research and Development, U.S. Environmental Protection Agency, August 26, 1985 (revised October 10, 1986). HWERL Document Control No. OAP-0006-GFS. 1986.
2. U.S. Environmental Protection Agency. Test Methods for Evaluating Solid Waste, Volumes 1A-1C: Laboratory Manual, Physical/Chemical Methods, SW-846, Third Edition. Office of Solid Waste. Document Control No. 955-001-00000-1. 1986.
3. American Water Works Association. Standard Methods for the Examination of Water and Wastewater. 16th Edition. 1985.
4. Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act. 40 CFR Part 136. Federal Register, Vol. 49 (209), Friday, October 26, 1984. Appendix A--Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater. 1984.
5. U.S. Environmental Protection Agency. Methods for the Chemical Analysis of Water and Wastes. EPA-600/4-79-020 (revised March 1983). Environmental Monitoring and Support Laboratory, Cincinnati. 1983, and subsequent EPA-600/4 Technical Additions thereto.

5.13 ANNOTATED GLOSSARY OF TERMS

Blank - A blank is an artificial sample designed to monitor the introduction of artifacts into the process. For aqueous samples, reagent water is used as a blank matrix; however, a universal blank matrix does not exist for solid samples; therefore, no matrix is used. The blank is taken through the appropriate steps of the process.

A reagent blank is an aliquot of analyte-free water or solvent analyzed with the analytical batch. Field blanks are aliquots of analyte-free water or solvents brought to the field in sealed containers and transported back to the laboratory with the sample containers. Trip blanks and equipment blanks are two specific types of field blanks. Trip blanks are not opened in the field. They serve as a check on sample contamination originating from sample transport, shipping and from site conditions. Equipment blanks are opened in the field and the contents are poured appropriately over or through the sample collection device, collected in a sample container, and returned to the laboratory as a sample. Equipment blanks serve as a check on sampling device cleanliness.

Calibration Standards and Devices (traceable) - Working standards or devices used for calibration purposes that are themselves calibrated or checked against a material or device with a known, true, reference value.

Collocated Samples - Air samples collected at the same time and location with adequate distance between them to preclude airflow interference. The difference in measured concentrations between the two collocated samplers is used to assess precision.

Control Charts - A quality control tool used to track the performance of field and laboratory instruments. After an instrument is calibrated, calibration limits are set (e.g., ± 7.5 percent) as a control measure. If the limits are exceeded, the control charts indicate the instrument must be recalibrated.

Independent Multilaboratory Analysis for Interlaboratory Comparison Studies - Used to determine precision between laboratories by using the same analytical methods and procedures and split samples or replicates. This also helps detect a laboratory bias (should any particular lab be significantly different), which could then be addressed.

Independent Multimethod Analysis - Used to determine the accuracy of a particular analytical method; may be used to study the appropriateness of a given method or procedure with a given matrix or background.

In-House Proficiency Testing Program - A program to determine and document the analysts' capabilities in the lab by incorporating practice runs of documented procedures and replicate or split samples, which are analyzed by different analysts to assess the individual analyst's accuracy and intralaboratory precision. This emphasizes to the analyst the importance of quality work and gives impetus to appropriate corrective action and further training as necessary.

Internal Standard - Known amount of a known compound (very similar in characteristics or behavior to a target compound) that is added to the sample immediately prior to analysis to provide a relative measure of instrument and analytical variability.

Laboratory Pure Water Spikes - Reagent, analyte-free, or laboratory pure water means distilled or deionized water or Type II reagent water that is free of contaminants that could interfere with the analytical test in question. Laboratory-pure-water spikes are made by adding a predetermined quantity of stock solution of certain analytes to the laboratory pure water prior to sample extraction/digestion and analysis.

Matrix/Spike Duplicate Analysis - In matrix/spike duplicate analysis, predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction/digestion and analysis. Samples are split into duplicates, spiked, and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative

percent difference between the samples is calculated and used to assess analytical precision. The concentration of the spike should be at the regulatory standard level or the estimated or actual method quantification limit. When the concentration of the analyte in the sample is greater than 0.1 percent, no spike of the analyte is necessary.

Reagent Checks - Reagents are analyzed prior to use to document impurity-free reagents (especially useful to document the absence to any target analyses). The reagents are certified to be of a known and acceptable quality.

Replicate Check Standard - Appropriate standard (may be laboratory pure water spike or independently prepared reference standard) that is run in replicate to generate precision data at a given concentration. This standard may be used to bracket field samples and blanks to demonstrate acceptable analytical precision throughout the run.

Replicate Sample - A replicate sample is a sample prepared by dividing a sample into two or more separate aliquots. Duplicate samples are considered to be two replicates.

Split Samples - Representative subsamples taken from the same sample. Split samples are normally provided when two or more parties are interested in analyzing the samples independently. Individual laboratories routinely split samples to perform matrix spikes and matrix spike duplicate analyses.

Surrogates - Surrogates are organic compounds that are similar to analytes of interest in chemical composition, extraction, and chromatography, but that are not normally found in environmental samples. These compounds are spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

Zero and Span Gases - At regular intervals between instrument calibrations, zero and span gases are used to check the calibration of an instrument. Zero-air, verified to be free of contaminants that would cause detectable responses in the instrument, is used to establish the baseline response for the contaminant of interest. Span gas, consisting of a relatively pure concentration of the contaminant of interest, is used to provide a single upscale recorder response in the range of 70 to 90 percent. The recorder response is used to determine the calibration relationship of the instrument.