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SUPERFUND PROGRAM
REPRESENTATIVE SAMPLING GUIDANCE

VOLUME 4: WASTE

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Environmental Response Team
Office of Emergency and Remedial Response
Office of Solid Waste and Emergency Response

U.S. Environmental Protection Agency
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Notice

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For more information on Waste Sampling procedures, refer to the *Compendium of ERT Waste Sampling Procedures*, OSWER Directive 9360.4-07, EPA/540/P-91/008. Topics covered in this compendium include: sampling equipment decontamination; drum sampling; tank sampling; chip, wipe, and sweep sampling; and waste pile sampling.

Please note that the procedures in this document should only be used by individuals properly trained and certified under a 40-hour hazardous waste site training course that meets the requirements set forth in 29 CFR 1910.120(e)(3). It should not be used to replace or supersede any information obtained in a 40-hour hazardous waste site training course.

Questions, comments, and recommendations are welcomed regarding the *Superfund Program Representative Sampling Guidance, Volume 4 -- Waste*. Send remarks to:

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Contents

Notice	ii
Disclaimer	iii
List of Figures	vii
List of Tables	vii
1.0 INTRODUCTION	1
1.1 OBJECTIVE AND SCOPE	1
1.2 CHARACTERISTICS OF WASTE	1
1.3 REPRESENTATIVE SAMPLING OBJECTIVES	1
1.4 CONCEPTUAL SITE MODEL	3
1.5 EXAMPLE SITE	5
2.0 SAMPLING DESIGN	6
2.1 INTRODUCTION	6
2.2 SAMPLING PLAN	6
2.2.1 Historical Data Review	7
2.2.2 Site Reconnaissance	7
2.2.3 Physiographic and Other Factors	7
2.3 WASTE SAMPLE TYPES	7
2.3.1 Grab Sample	7
2.3.2 Composite Sample	8
2.4 WASTE TYPES	8
2.5 WASTE CHARACTERISTICS	9
2.5.1 Homogeneity	9
2.5.2 Physical State	9
2.5.3 Chemical Stability	9
2.5.4 Particle Size (solids)	9
2.5.5 Viscosity (liquids)	10
2.6 WASTE SOURCES	10
2.6.1 Containerized Waste	10
2.6.2 Uncontainerized Waste	11
2.6.3 Surfaces and Debris	11
2.7 QUALITY ASSURANCE CONSIDERATIONS	11
2.8 DATA QUALITY OBJECTIVES	12
2.9 FIELD ANALYTICAL SCREENING AND GEOPHYSICAL TECHNIQUES	12
2.10 ANALYTICAL PARAMETERS AND METHODS	12
2.11 REPRESENTATIVE SAMPLING APPROACHES	13
2.11.1 Judgmental Sampling	13
2.11.2 Random Sampling	13
2.11.3 Systematic Grid Sampling	14
2.11.4 Systematic Random Sampling	14
2.11.5 Transect Sampling	14
2.12 SAMPLING LOCATIONS AND NUMBER	15
2.13 EXAMPLE SITE	16
2.13.1 Background	16
2.13.2 Site Entry	16
2.13.3 Site Inventory	17

2.13.4	Selecting Analytical Parameters	18
3.0	FIELD ANALYTICAL SCREENING AND SAMPLING EQUIPMENT	20
3.1	INTRODUCTION	20
3.1.1	Combustible Gas Indicator	20
3.1.2	Radiation Screening Instruments	20
3.1.3	Flame Ionization Detector	20
3.1.4	Photoionization Detector	21
3.1.5	Colorimetric Tubes	21
3.1.6	Hazard Categorization	21
3.1.7	Immunoassay Tests	22
3.1.8	X-Ray Fluorescence	22
3.1.9	Gas Chromatograph	22
3.2	SAMPLING EQUIPMENT	23
3.2.1	Drum Openers	23
3.2.2	Liquid Samplers	23
3.2.3	Sludge Samplers	24
3.2.4	Solids Samplers	25
3.3	EXAMPLE SITE	26
3.3.1	Drum Screening and Sampling Equipment	26
3.3.2	Plating Vat Screening and Sampling Equipment	26
3.3.3	Waste Pile Screening and Sampling Equipment	26
3.3.4	Impoundment Screening and Sampling Equipment	27
3.3.5	Transformer Screening and Sampling Equipment	27
4.0	FIELD SAMPLE COLLECTION AND PREPARATION	29
4.1	INTRODUCTION	29
4.2	SAMPLE VOLUME	29
4.3	SOURCE SAMPLING	29
4.3.1	Drum Sampling	29
4.3.2	Bulk Storage Tank and Transformer Sampling	30
4.3.3	Lab Pack Sampling	30
4.3.4	Surface Impoundment Sampling	30
4.3.5	Waste Pile Sampling	30
4.3.6	Surface Sampling	31
4.3.7	Debris Sampling	32
4.3.8	Compressed Liquid/Gas Cylinders	32
4.4	SAMPLE PREPARATION	32
4.4.1	Removing Extraneous Material	33
4.4.2	Homogenizing	33
4.4.3	Splitting	33
4.4.4	Final Preparation	33
4.5	EXAMPLE SITE	34
4.5.1	Source Sampling	34
4.5.2	Sample Preparation	34
5.0	QUALITY ASSURANCE/QUALITY CONTROL	36
5.1	INTRODUCTION	36
5.2	DATA CATEGORIES	36
5.3	SOURCES OF ERROR	36
5.3.1	Sampling Design	36
5.3.2	Sampling Methodology	37
5.3.3	Sample Heterogeneity	37

5.3.4	Analytical Procedures	37
5.4	QA/QC SAMPLES	37
5.4.1	Field Replicate Samples	38
5.4.2	Collocated Samples	38
5.4.3	Background Samples	38
5.4.4	Performance Evaluation/Laboratory Control Samples	38
5.4.5	Matrix Spike/Matrix Spike Duplicate Samples	38
5.4.6	Rinsate Blank Samples	39
5.4.7	Field Blank Samples	39
5.4.8	Trip Blank Samples	39
5.4.9	Laboratory Duplicate Samples	39
5.5	EVALUATION OF ANALYTICAL ERROR	39
5.6	CORRELATION BETWEEN FIELD SCREENING RESULTS AND LABORATORY RESULTS	39
5.7	EXAMPLE SITE	40
5.7.1	QA Objectives	40
5.7.2	Sources of Error	40
5.7.3	Field QA/QC Samples	40
5.7.4	Laboratory QA/QC Samples	41
6.0	DATA PRESENTATION AND ANALYSIS	42
6.1	INTRODUCTION	42
6.2	DATA POSTING	42
6.3	CROSS-SECTION/FENCE DIAGRAMS	42
6.4	CONTOUR MAPPING	42
6.5	STATISTICAL GRAPHICS	42
6.6	RECOMMENDED DATA INTERPRETATION METHODS	43
6.7	EXAMPLE SITE	43
Appendix A	EXAMPLE OF FLOW DIAGRAM FOR CONCEPTUAL SITE MODEL	47
References	50

List of Figures

1	Conceptual Site Model	4
2	Random Sampling	13
3	Systematic Grid Sampling	14
4	Systematic Random Sampling	15
5	Transect Sampling	15
6	ABC Plating Site Sketch	17
7	Example of a Drum Inspection Log	19
8	Example of a Hazard Categorization Data Sheet	28
9	Posted Total Chromium Data for Impoundment No. 1	44
A-1	Migration Routes of a Gas Contaminant	47
A-2	Migration Routes of a Liquid Contaminant	48
A-3	Migration Routes of a Solid Contaminant	49

List of Tables

1	ABC Plating Sample Log	35
2	Haz-Cat Results -- ABC Plating Site	45

1.0 INTRODUCTION

1.1 OBJECTIVE AND SCOPE

This is the fourth volume in a series of guidance documents that assist Superfund Program Site Managers, On-Scene Coordinators (OSCs), Remedial Project Managers (RPMs), and other field staff in obtaining representative samples at Superfund sites. The objective of representative sampling is to ensure that a sample or a group of samples accurately characterizes site conditions. The representative sampling principles discussed in this document are applicable throughout the Superfund Program. The following chapters will help field personnel to assess available information, select an appropriate sampling approach, select and utilize field analytical screening methods and sampling equipment, incorporate suitable types and numbers of quality assurance/quality control (QA/QC) samples, and interpret and present the site analytical data.

As the Superfund Program has developed, the emphasis of this response action has expanded beyond addressing emergency response and short-term cleanups. Each planned response action must consider a variety of sampling objectives, including identifying threat, delineating sources of contamination, and confirming the achievement of clean-up standards. Because many important and potentially costly decisions are based on the sampling data, Site Managers and other field personnel must characterize site conditions accurately. To that end, this document emphasizes the use of cost-effective field analytical screening techniques to characterize the site and aid in the selection of sampling locations.

1.2 CHARACTERISTICS OF WASTE

Waste, in general terms, can include solid, liquid, and sludge material typically generated as a by-product of an industrial process. Assume that containerized wastes comprise high concentrations of hazardous substances, unless clearly indicated otherwise through previous sample analysis or other reliable documentation. Waste samples are often of high concentration and phased (e.g., light liquid, dense

liquid, and sludge), an important point to consider when developing a sampling strategy. This document specifically addresses the sampling of wastes typically found in drums, tanks, lab packs, transformers, impoundments, waste piles, and on surfaces.

The National Oil and Hazardous Substances Pollution Contingency Plan (NCP) definition for a hazardous substance includes "...any substance designated pursuant to section 311(b)(2)(A) of the Clean Water Act; any element, compound, mixture, solution, or substance designated pursuant to Section 3001 of the Solid Waste Disposal Act (but not including any waste the regulation of which under the Solid Waste Disposal Act has been suspended by an Act of Congress); any hazardous air pollutant listed under Section 112 of the Clean Air Act; and any imminently hazardous chemical substance or mixture with respect to which the EPA Administrator has taken action pursuant to Section 7 of the Toxic Substances Control Act. The term does not include petroleum including crude oil or any fraction thereof which is not otherwise specifically listed or designated as a hazardous substance..., and the term does not include natural gas, natural gas liquids, liquified natural gas, or synthetic gas usable for fuel (or mixtures of natural gas and such synthetic gas)." Pursuant to 40 CFR 261, Subpart C, a waste is considered hazardous if it exhibits any of the following characteristics: ignitability, corrosivity, reactivity, or toxicity; or if it is a listed hazardous waste under 40 CFR 261.30, Subpart D. Asbestos and "mixed" waste (having radioactive and hazardous waste components), while included in this definition, require specialized sampling methods and techniques and will not be addressed in this document.

1.3 REPRESENTATIVE SAMPLING OBJECTIVES

Representative sampling applies to all phases of a Superfund response action. Representative sampling objectives for waste include:

- Identify the waste, including composition and characteristics, and determine if it is hazardous.

- Determine if there is an imminent or substantial threat to public health or welfare or the environment.
- Determine the need for long-term action.
- Develop containment and control strategies.
- Evaluate appropriate disposal/treatment options.
- Verify treatment goals or clean-up levels.

Determine Hazard and Identify Waste

One of the first objectives during a response action at a site is to determine the presence, identity, and potential threat of any hazardous materials. Use field screening techniques (discussed in Chapter 3) for rapid detection of wastes. Upon confirming the presence of hazardous materials, sample and/or continue screening to identify their compositions and determine their concentrations.

In addition to characterizing the waste sufficiently, conduct compatibility tests to help classify waste by composition and other physical characteristics into compatible waste streams (e.g., acid, base, or oxidizer). This will ensure safer handling, staging, bulking, storage, and transportation of wastes both on and off site.

Establish Threat

Establishing threat to the public or environment is a primary objective during a response action. The data obtained from characterizing the waste will help the Site Manager to determine whether an imminent or substantial threat exists and whether a removal action or other response action is necessary. The type and degree of threat determines the rate at which a response action is taken.

Determine Need for Long-Term Action

Site conditions may establish a long-term threat that is not imminent or substantial. Characterization of the waste can assist the Site Manager in setting a priority for long-term remediation evaluation and response. Waste characterization data are required to evaluate the site under the Hazard Ranking System and to identify sites eligible for inclusion on the National Priorities List (NPL). The NPL is the ranking list of

those sites at highest national priority for long-term evaluation and remediation.

Develop Waste Containment and Control Strategies

Once the chemical constituents and threat have been established, many strategies for waste containment and control are available to the Site Manager. Analytical data indicating the presence of chemical hazards are not in themselves sufficient to select a containment or control strategy. Site reconnaissance and historical site research provide information on site conditions and the physical state of the waste sources; waste containment and control strategies are largely determined by this information. For example, site security measures (such as erecting a fence) may be sufficient to stabilize a site containing intact drums of solvents, and overpacking may be sufficient to contain a corroded drum of organophosphate pesticides. Unstable or explosive wastes, such as picric acid, may require immediate removal by demolition experts.

Identify Available Treatment/Disposal Options

The site contaminants should be identified, quantified, and compared to selected action levels. Where regulatory action levels do not exist, site-specific clean-up levels are determined by the EPA Region (often in consultation with the Agency for Toxic Substances and Disease Registry (ATSDR)). If action levels are exceeded, a series of chemical and physical tests may be required to evaluate possible treatment and/or disposal options. Each treatment or disposal method has a corresponding set of waste parameters that must be evaluated, e.g., ash content, British thermal unit (BTU) value, total metals concentration, total organic halides, cyanide, total chlorine and NO_x are minimum requirements for incineration. It is important to test for treatment/disposal parameters as early as possible during the site assessment and characterization procedure. Relatively inexpensive tests such as total organic carbon (TOC), BTU, and pH should be considered early in the response action in order to contribute to later treatability studies. The test results will ultimately help to determine the most appropriate treatment or disposal option for meeting regulatory requirements.

Verify Treatment Goals or Clean-up Levels

After treatment or disposal, representative sampling results should either confirm that the response actions

have met the site-specific treatment goals or clean-up levels, or indicate that further treatment or removal is necessary. Refer to the *Representative Sampling Guidance, Volume 1 -- Soil*, OSWER Directive 9360.4-10, for guidelines on soil sample collection and preparation for confirming cleanup.

Sampling to verify cleanup requires careful coordination with demobilization activities. After treatment of one area on a site, verification sampling can begin in that area by using field screening and on-site analysis. Meanwhile, other areas can be treated. Lab confirmation of the screening performed in the treated areas can help ensure accuracy of screening for subsequent areas to meet QA objectives (as discussed in Section 5.2).

1.4 CONCEPTUAL SITE MODEL

A conceptual site model is a useful tool for selecting sampling locations. It helps ensure that sources, pathways, and receptors throughout the site have been considered before sampling locations are chosen. The conceptual model assists the site manager in evaluating the interaction of different site features. Risk assessors use conceptual models to help plan for risk assessment activities. Frequently, a conceptual model is created as a site map (see Figure 1) or it may be developed as a flow diagram which describes potential migration of contaminants to site receptors (see Appendix A).

A conceptual model follows contaminants from their sources, to pathways (e.g., air, surface water), and eventually to the assessment endpoints. Consider the following when creating a conceptual model:

- The state(s) of each contaminant and its potential mobility
- Site topographical features
- Meteorological conditions (e.g., wind direction/speed, average precipitation, temperature, humidity)
- Human/wildlife activities on or near the site

The conceptual site model on the next page is an example created for this document. The model assists in identifying the following site characteristics:

Potential Sources:

Site (waste pile, lagoon); drum dump; sewage plant discharge

Potential Migration Pathways:

Soil -- Leachate from the waste pile or drum dump; soil in direct contact with solids in the waste pile or drum dump

Surface Water -- Liquid waste from the lagoon or sewage plant discharge (into the lake)

Sediments -- Liquid waste from the lagoon or sewage plant discharge (into the lake)

Air -- Release of vapors/particulates from the waste pile, drum dump or lagoon

Potential Exposure Routes:

Ingestion -- Particles from the waste pile or drum dump; liquid from the lagoon or lake (from sewage plant discharge)

Inhalation -- Vapors from the waste pile, drum dump, lagoon, or lake (sewage plant discharge)

Absorption/direct contact -- Contact with the waste pile, drum dump, lagoon, or lake (sewage plant discharge)

Potential Receptors of Concern (and associated potential exposure sources):

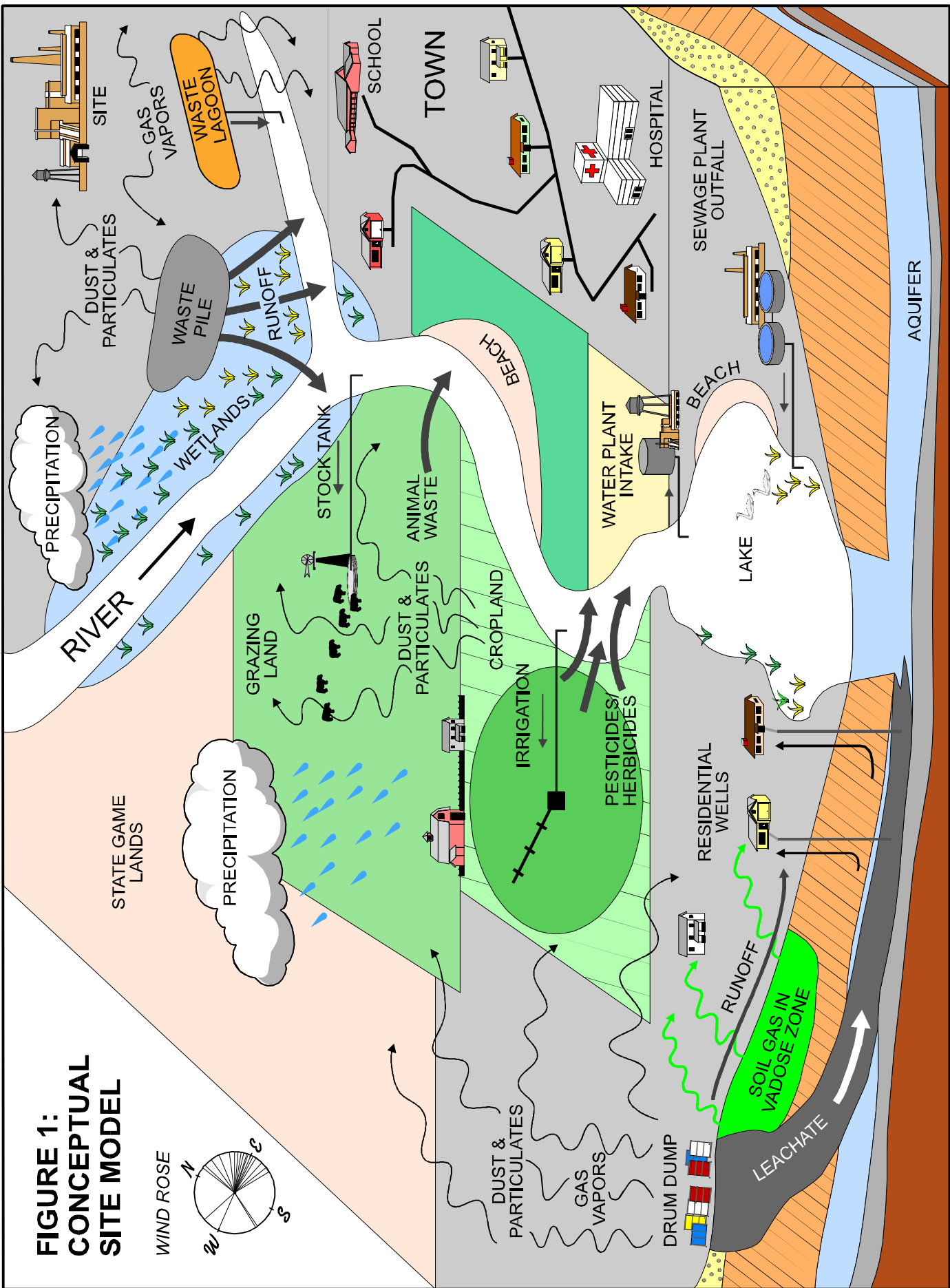
Human Population

Residents/Trespassers:

Soil -- Leachate from the drum dump; direct contact with soil from solids in the drum dump

Surface water -- Liquid waste from the lagoon into the river or sewage plant discharge into the lake

Air -- Vapors/particulates from the waste pile, drum dump, lagoon, or lake (sewage plant discharge)



**FIGURE 1:
CONCEPTUAL
SITE MODEL**

Workers/Trespassers:

Soil -- Leachate from the waste pile; direct contact with soil from solids in the waste pile

Surface water -- Liquid waste in the lagoon or associated with the sewage plant discharge

Air -- Vapors/particulates from the waste pile, drum dump, lagoon, or sewage plant discharge

Biota

Threatened or endangered species or human food chain organisms known to frequent areas near the waste pile, drum dump, lagoon, or lake (sewage plant discharge)

Preliminary site information may provide the identification of the contaminant of concern and the level of the contamination. A sampling plan should be developed based upon the selected receptors of concern and the suspected sources and pathways. The model may assist in the selection of on-site and off-site sampling locations.

1.5 EXAMPLE SITE

An example site presented at the end of each chapter illustrates the development of a representative waste sampling plan that meets Superfund Program objectives for an early action.

2.0 SAMPLING DESIGN

2.1 INTRODUCTION

There is no universal sampling method for characterizing wastes because site characteristics vary widely. The sampling methods and equipment must be suited to the specific sampling situation. A properly developed waste sampling design defines the sampling purpose, protects site worker health and safety, effectively utilizes resources, and minimizes errors. The sampling design will vary according to the type of waste sampled (including type of containers or sources), and the characteristics of the site. When developing a sampling design, consider: prior actions at the site (e.g., sampling practices, compliance inspections); properties and characteristics of the wastes sampled; site waste sources (e.g., impoundments, waste piles, drums); topographic, geologic, hydrologic, and meteorologic conditions of the site; and flora, fauna, and human populations in the area.

Waste material may be liquid, solid, or sludge, and may be contained in drums, tanks, waste piles, surface impoundments, on surfaces (e.g., building structures, floors, equipment), in lab packs, or other sources. Sampling each waste stream may require a variety of sampling techniques, equipment, sample packaging, and sample analyses.

2.2 SAMPLING PLAN

Many site-specific factors are important in the development of a good sampling plan, including: data use and quality assurance objectives; sampling equipment; sampling design; standard operating procedures (SOPs); field analytical screening; analytical method selection; decontamination; sample handling and shipment; and data validation.

The U.S. EPA *Quality Assurance Sampling Plan for Environmental Response (QASPER)* was designed to develop sampling plans for response actions. QASPER is menu-driven software which prompts the user to input background information and to select prescribed parameters for development of a site-specific sampling plan. It also gives the user access

to any previously developed site-specific sampling plans.

The following procedures are recommended for developing a thorough waste sampling plan. Many steps can be performed concurrently, and the sequence is flexible.

- Review the history of the site, including regulatory and reported spill history; note current and former locations of buildings, tanks, and process, storage, and disposal areas.
- Perform a site reconnaissance; categorize physical/chemical properties and hazardous characteristics of materials involved.
- Identify topographic, geologic and hydrologic characteristics of the site including surface water, groundwater, and soil characteristics, as well as potential migration pathways and receptors.
- Determine geographic and demographic information, including population size and its proximity to the site (e.g., public health threats, source of drinking water); identify threatened environments (e.g., potentially contaminated wetlands or other sensitive ecosystems).
- Select sampling strategies, considering field analytical screening and statistical applications, when appropriate.
- Determine data quality and quality assurance objectives for field analytical screening, sampling, and analysis. As the extent of contamination becomes quantified, the sampling plan can be modified to better assess sampling objectives throughout the action.

It is recognized that many of these steps (described in detail below) would not be applicable during a classic emergency response because of the lack of advance notice. Emergency response sampling nevertheless requires good documentation of sampling events.

2.2.1 Historical Data Review

The first step in developing a sampling plan is a review of historical site data, examining past and present site operations and disposal practices to provide clues on possible site contamination. Available sources of information include: federal, state and local agencies and officials; federal, state, and local agency files (e.g., site inspection reports and legal actions); deed or title records; current and former facility employees; potentially responsible parties (PRPs); local residents; and facility records or files.

A review of previous sampling information should include sampling locations, matrices, methods of collection and analysis, and relevant contaminant concentrations. Assess the reliability and usefulness of existing analytical data, including those which are not substantiated by documentation or QA/QC controls, but which may still illustrate general site trends.

Collect information that describes specific chemical processes, raw materials used, products and wastes, and waste storage and disposal practices. Review any available site maps, facility blueprints, and historical aerial photographs detailing past and present storage, process, and waste disposal locations. County property and tax records and U.S. Geological Survey (USGS) topographic maps are useful sources of information on the site and its surroundings.

2.2.2 Site Reconnaissance

A site reconnaissance can be conducted at an earlier date or immediately prior to sampling activities. It allows field personnel to assess site conditions, evaluate areas of potential contamination, evaluate potential hazards associated with sampling, and finalize a sampling plan. Site reconnaissance activities include: observing and photographing the site; noting site access routes and potential evacuation routes; noting potential safety hazards; recording label information from drums, tanks, or other containers; mapping process and waste disposal areas such as landfills, impoundments, and effluent pipes; making an inventory of the wastes on site; mapping potential contaminant migration routes such as drainage, streams, and irrigation ditches; noting the condition of animals and/or vegetation; and noting topographic and/or structural features. Field personnel should use

appropriate personal protective equipment (PPE) when engaged in any site activities.

2.2.3 Physiographic and Other Factors

Other procedures, such as determining data quality and QA/QC objectives, utilizing field analytical screening techniques, identifying topographic, geologic and hydrologic characteristics, and determining geographic and demographic information are important steps of an overall sampling plan. Field analytical screening techniques and equipment are discussed in Chapter 3; QA objectives are discussed in Chapter 5. Since this document specifically pertains to waste sampling, the remaining procedures listed above will not be addressed in detail here. (Please refer to the *Representative Sampling Guidance, Volume 1 -- Soil*, OSWER Directive 9360.4-10.) The U.S. EPA is currently developing an ecological sampling guidance document that will contain a detailed checklist for collecting ecological data.

2.3 WASTE SAMPLE TYPES

Design sampling procedures to match sampling objectives. The type of sample collected may depend on suspected waste types and characteristics; size and accessibility of waste containers, impoundments and other media; target analytes; and health and safety requirements.

The following section describes and gives examples of the two types of waste samples.

2.3.1 Grab Sample

A *grab* sample is a discrete aliquot collected from one specific sampling location at a specific point in time, and may be considered representative of a homogeneous and stable waste. When obtaining grab samples from containers or from an impoundment having stratified layers, sample each phase or stratum separately; the separate aliquots are representative of their respective stratum. When sampling stratified sources, determine as many properties of the wastes as possible through historical data and site reconnaissance prior to sampling, and use caution because the individual phase components may be more concentrated than the original waste material.

2.3.2 Composite Sample

A *composite* sample is a non-discrete sample composed of two or more equal aliquots collected at various sampling points or times. There are four types of composite samples: *areal*, *vertical*, *flow proportional*, and *time*. An *areal composite* is comprised of individual aliquots collected over a defined area (e.g., surface of a waste pile). It is made up of aliquots of equal volume, each collected in an identical manner at the same horizon (depth). A *vertical composite* is composed of individual aliquots collected at different depths but along the same vertical line (e.g., borehole). It is made up of aliquots of equal volume which are collected in an identical manner. A *flow proportional composite* is a sample collected proportional to the flow rate during the compositing period by either a time-varying/constant volume or time-constant/varying volume method. A *time composite* is composed of a varying number of discrete, equal-volume aliquots collected at equal time intervals during the compositing period. (Both flow and time composite samples are appropriate for sampling wastewater or streams.)

By design, composite samples reflect an "average" concentration within the composite area, flow, or interval. Compositing is appropriate when determining the general characteristics or the representativeness of certain sources (e.g., a waste pile or impoundment) when considering methods of treatment or disposal. When compositing samples from a waste stream, note that resulting concentrations are representative of the waste stream's average concentration, but not of discrete areas within the waste stream.

Composite sampling should be performed only on like waste streams. Do not composite dissimilar waste streams or waste sources (e.g., drums with unknown contents or dissimilar materials) because of health and safety risks associated with possible reactions; in addition, the resulting sample will not define or represent the origin of the mixed contaminants. Composite aliquots from tanks, drums, or other containers only after adequate hazardous characterization screening to prevent mixing of incompatible wastes.

A result of sample compositing is the dilution of high concentration aliquots. To compensate for dilution, reduce the applicable detection limits accordingly. If

the composite value is to be compared to a selected action level, then the action level must be divided by the number of aliquots that make up the composite in order to determine the appropriate detection limit. For example, if the action level for a particular substance is 40 ppb, a detection limit of 10 ppb should be used when analyzing a 4-aliquot composite.

When compositing waste, four aliquots per sample are recommended because two ounces of each aliquot can be added to an 8-ounce (or larger) jar. Individual aliquots in storage from any "hit" composites can be analyzed later to pinpoint contamination.

2.4 WASTE TYPES

The types of wastes encountered at a site greatly influence the development of the site sampling plan. The number of grab and composite samples, type of screening/sampling equipment used, and analytical methods all depend on the types of wastes present at the site. Waste solids can vary from granular or powdered materials to contaminated structural surfaces or demolition debris. Waste liquids can include solvents, acids, bases, process solutions, and lubricants, among others. Waste sludges have characteristics of both solids and liquids.

Each type of waste may be highly concentrated, consisting of virtually pure industrial products, raw or spent materials, chemicals, or process by-products. Methods for sampling and analyzing vary by waste type, and the sampling plan should specify appropriate sample collection and analysis methods.

Waste samples are often complex mixtures and may be difficult to analyze in the laboratory. Provide the analytical laboratory with as much information as possible to help minimize delays in analysis. The laboratory will find the following information helpful in expediting the analysis of waste samples:

- Whether the sample is pure waste or an environmental sample (e.g., oil as opposed to oily water).
- Viscosity, particle size, or an accurate description of the waste characteristics.
- Qualitative estimate of concentration (i.e., low, medium, high).

- Presence of extreme pH levels (i.e., less than 2 or greater than 12); some analytical methods will not yield successful results on such samples; it may be necessary to consult with a chemist to change the method.
- Presence of chlorinated dioxins, even if the samples are being analyzed for another parameter (e.g., metals). The Occupational Safety and Health Administration (OSHA) specifies special handling facilities for samples contaminated with dioxin; many laboratories are not set up to handle these samples.
- Presence of high concentrations of organic substances, particularly aromatics, in samples to be analyzed for metals (some methods for metals analysis are not compatible with high concentrations of organic materials).

Inform the laboratory in advance about important sample constituents of interest and QA/QC criteria. Waste samples typically must be diluted before analysis, which may prevent detection of these constituents. Also consult with the laboratory on how to prepare subaliquots of non-typical samples.

2.5 WASTE CHARACTERISTICS

Waste characteristics, including homogeneity, physical state, chemical stability, particle size (solids), and viscosity (liquids) are other factors that influence the number and types of samples collected.

2.5.1 Homogeneity

Wastes may be homogeneous or heterogeneous. The solubility, specific gravity, and mechanical mixing ability of the waste can affect its degree of homogeneity. A single grab sample per waste stream may be appropriate for a homogeneous material; however, heterogeneous and unclassified wastes often require more extensive sampling and analysis to ensure that the various phases and concentrations of the waste are represented in the samples. The sampling strategy should reflect the homogeneity, random heterogeneity, or stratification of the waste over space or time.

2.5.2 Physical State

The physical state of waste (i.e., solid, liquid, gas, or multiphase) will influence the selection of sampling devices and many other aspects of the sampling effort. Variances in each physical state can also affect sampling. For example, free-flowing liquid would require a different sampling approach than a viscous liquid.

Sample containers with wide mouths are best for solid samples, sludges, and liquids with substantial amounts of suspended matter. Bottles with air-tight closures are needed for gas samples or gases adsorbed onto solids or dissolved in liquids.

The sampling strategy will vary if the physical state of the waste is subject to stratification (for example, liquid wastes with differing densities or viscosities, or those with suspended solids), homogenization, or random heterogeneity.

2.5.3 Chemical Stability

Waste materials can differ considerably in their inherent chemical stability. Exposure to the elements (e.g., sunlight, air, rain) and leaching may cause chemical degradation or reaction, thereby creating new compounds. Heterogeneous materials may undergo physical separation, resulting in pockets or layers of different compounds. Sampling methods and shipping practices will vary according to the toxicity, ignitability, corrosivity, and reactivity of the waste.

2.5.4 Particle Size (solids)

Waste solids are often made up of materials with different particle sizes. This variation can influence analytical results by introducing either a negative or positive bias. For example, if large pieces of waste material (e.g., slag) are not collected and included with a sample, a negative bias of contaminants may result (analytical results may be lower than what is actually representative). Small particle size can also bias a sample. Some pollutants adsorb more readily onto small particles, so a small-fraction sample may result in a positive bias (analytical results may be higher than what is representative). If it is necessary to sample material that has unusual particle size characteristics, identify an approximate size distribution and consult the laboratory in advance to determine a method for representative analysis of the

irregular materials. Sieving of waste is not usually recommended. If grinding or pulverizing large pieces is desired, make special arrangements with the laboratory.

2.5.5 Viscosity (liquids)

Viscosity is the internal friction of a fluid that produces a resistance to flow. The viscosity of waste liquids often greatly affects the effort required for sample collection and may indirectly determine the volume of sample required. Because viscosity can affect the representativeness of the sample, and can itself be a physical limitation for sampling, a sampling technique suited to the viscosity of the material must be selected. Very viscous materials (greater than 100,000 centipoise (cps)) must be scooped, while low viscosity materials may be aspirated, encapsulated, or poured. To collect a representative sample for viscosity testing, it is important to limit handling and contact time. The sample must be allowed to return to equilibrium before measurement. Without a viscometer, viscosity may be roughly determined by comparison to water (low viscosity), syrup (medium viscosity), and mayonnaise or taffy (high viscosity), as well as to other materials of known viscosity.

Several sampling devices have been designed to sample waste liquids within a specific viscosity range. Weighted bottle samplers, PACS grab samplers, and composite liquid waste samplers (COLIWASAs) are suited for sampling less viscous liquids and become difficult to use in very viscous liquids. The glass thief and bacon bomb sampler are suitable for sampling moderately to highly viscous materials. See Section 3.2 for a discussion of sampling equipment.

2.6 WASTE SOURCES

There are a variety of potential waste sources commonly found at waste sites. The type of waste source affects many aspects of the sampling design, such as sampling approach (e.g., judgmental or random), sampling equipment, and types/numbers of samples (including QA/QC samples). The type of source will also affect many logistical considerations, such as cost, level of effort, and duration of a response action. This section introduces the three categories of waste sources: containerized waste, uncontainerized waste, and surfaces and debris.

2.6.1 Containerized Waste

Containerized waste consists of solids, liquids, or sludges that are found in drums, bulk storage tanks, transformers, and lab packs. Evaluate container label information before making sampling decisions. It may be possible to identify numerous containers of similar material. Wherever possible, use screening techniques to substantiate label information. Screening results should be confirmed with laboratory test results prior to making any treatment or disposal decisions (affirming that screening was effective). Specialized equipment (e.g., forklift, grapple, manlift) may be needed to access drums and tanks safely.

The sampling objective determines which and how many containers need to be sampled. For example, if the objective is to establish threat, it may be most important to sample a few containers having visible leaks or spills. If the objective is to estimate the disposal cost, it may be appropriate to sample each of the largest volume containers to identify the predominant waste streams.

Drums can be of different volumes (typically from 30 to 90 gallons), varied construction (e.g., top bung, side bung, removable top, lined), and be made of a variety of materials (e.g., steel, polyethylene, fiber, combinations). Drums located at a waste site often vary in condition, sometimes showing deterioration, bulging, and/or damage. These physical criteria can be useful in making assumptions about a drum's contents. For example, strong acids, caustics, or other corrosives are typically stored in 30- to 55-gallon polyethylene or polyethylene-lined steel drums with top bung holes. While this is not a fool-proof method of determining drum contents, it gives the investigator an indication prior to sampling of the general types of materials to be encountered.

Bulk storage tanks include tank cars/trucks, vats, storage vessels, and transformers. They range in size from less than 100 gallons to millions of gallons. Like drums, bulk storage tanks are constructed of many different materials and are designed in many configurations.

Lab packs consist of small, individually-labelled containers of laboratory waste or unused reagents. The chemical containers are usually not more than five gallons each, and are often packaged or transported together in a larger Department of

Transportation (DOT) shippable container (these are typically 30- to 55-gallon drums). Be sure to screen lab packs for radioactivity, since radioactive substances (alpha, beta, and gamma emitters) are commonly found in lab packs.

Another type of waste that can be found on site is biological waste, also known as "red bag waste." Note that contaminated biological waste (dressings, syringes, etc.) may not always be found in the required red bag. It should be handled only by personnel specifically trained and authorized to deal with biological waste. If "red bag waste" is encountered at a site, notify the ATSDR, or local health authorities.

2.6.2 Uncontainerized Waste

Uncontainerized waste consists of solids, liquids, and sludges that are found in waste piles and surface impoundments.

Waste piles may be composed of solid wastes such as tank bottom solids, contaminated soil, ash, solidified sludges, or a mixture of liquid and solid chemical wastes. The shape and size of waste piles can vary greatly, depending on the generating process or facility. The sampling plan should take into account the chemical and physical characteristics of the waste pile. For example, contaminants can leach out of the surface layers of a waste pile, resulting in deceptively low or nonrepresentative concentrations at the top. The sampling plan should account for leaching by taking composite samples from various horizons within the pile to determine an "average" concentration.

Surface impoundments include lined and unlined lagoons, ponds, and trenches that contain predominantly liquids and sludges from site processes or surface runoff. The liquids may be homogeneous or stratified, depending on the chemical and physical properties of the wastes. Reactions may occur within the impoundment to alter or degrade the original chemicals. Do not compromise any existing liners when sampling bottom sludges.

2.6.3 Surfaces and Debris

Surfaces and debris require specialized sampling techniques. During a response action, it may be necessary to sample object and structural surfaces for

contamination to determine the need for dismantling and eventual disposal. Virtually any surface on the site may have to be sampled, including walls and floors of buildings, process machinery, tanks, vats, air ducts, vehicles, and furniture. There are three methods for sampling surfaces: wipe sampling, chip sampling, and dust sampling. Each method is described further in Section 4.3.6.

Debris can be highly variable and includes demolition rubbish, construction and destruction materials, paint cans, empty 55-gallon drums, battery-casings, shredded automobiles, and other miscellaneous matrices such as process waste, tannery waste, and slag. Debris may be composed of plastic, metal, rubber, paper, concrete, wood, glass, masonry, and municipal waste. It can include contaminated waste sampling articles such as protective disposable clothing (e.g., Tyvek suits), sample collection jars, and disposable sampling equipment (e.g., plastic scoops).

2.7 QUALITY ASSURANCE CONSIDERATIONS

Quality assurance components are defined as follows:

- Precision -- measurement of variability in the data collection process
- Accuracy (bias) -- measurement of bias in the analytical process; the term "bias" throughout this document refers to the QA/QC accuracy measurement
- Completeness -- percentage of sampling measurements which are judged to be valid
- Representativeness -- degree to which sample data accurately and precisely represent the characteristics and concentrations of the waste contaminants
- Comparability -- evaluation of the similarity of conditions (e.g., sample depth, sample homogeneity) under which separate sets of data are produced

To ensure that the analytical samples are representative of site conditions, quality assurance measures must be associated with each sampling and

analysis event. The sampling plan must specify these measures. QA measures include, but are not limited to: laboratory SOPs, sample bottle preparation, equipment decontamination, field blanks, replicate samples, performance evaluation samples, sample preservation and handling, and chain-of-custody requirements (see Chapter 5, Quality Assurance/Quality Control).

2.8 DATA QUALITY OBJECTIVES

Data quality objectives (DQOs) state the level of uncertainty that is acceptable for data collection activities and define the certainty of the data necessary to make decisions. When establishing DQOs for a particular project, consider:

- Decision(s) to be made or question(s) to be answered
- Why analytical data are needed and how the results will be used
- Time and resource constraints on data collection
- Descriptions of the analytical data to be collected
- Applicable model or data interpretation method used to arrive at a conclusion
- Detection limits for analytes of concern
- Sampling and analytical error

2.9 FIELD ANALYTICAL SCREENING AND GEOPHYSICAL TECHNIQUES

There are two types of analytical data that can be generated during a response action: field analytical screening data and laboratory analytical data. Field analytical screening instruments and techniques provide real-time or direct (or colorimetric) readings. They include: flame ionization detectors (FIDs), photoionization detectors (PIDs), colorimetric tubes, portable X-ray fluorescence (XRF) units, portable gas chromatography (GC) units, immunoassay tests, and hazard categorization kits. These screening methods can assist with the selection of sample locations or samples to be sent for laboratory analysis by narrowing the possible groups or classes of chemicals. They are effective and economical for gathering large amounts of site data. After an area or group of containers has been characterized using field screening techniques, a subset of samples can be sent for laboratory analysis to substantiate the screening

results. Field analytical screening with laboratory confirmation usually generates more analytical data under a limited sampling budget than will sampling with off-site laboratory analysis alone. Whenever possible, use field analytical screening methods which provide detection limits below applicable action levels. If these methods are not available, field analytical screening can still be useful for waste sampling by detecting grossly contaminated areas as well as for on-site health and safety determination. Field analytical screening techniques to support waste sampling are discussed in more detail in Chapter 3.

Geophysical techniques may be utilized during a response action to locate potential buried drums or tanks, buried waste, and disturbed areas. Geophysical techniques include ground penetrating radar (GPR), magnetometry, electromagnetic conductivity (EM), and resistivity surveys. Refer to U.S. EPA *Representative Sampling Guidance, Volume 1 -- Soil*, OSWER Directive 9360.4-10, for a discussion of soil geophysical techniques that are also applicable for waste sampling.

2.10 ANALYTICAL PARAMETERS AND METHODS

Designing a representative waste sampling plan includes selecting analytical parameters and methods. Use data collected during the historical data review (e.g., past site processes, materials stored on site) to select appropriate analytical parameters and methods. If the historical data review reveals little information about the types of waste on site, select analytical parameters by initially characterizing the waste. Use applicable field screening methods and limited laboratory analysis to rule out the presence of high concentrations of certain contaminants, and to narrow the list of analytical parameters. Methods often used for characterization of waste include GC/MS (gas chromatography/mass spectroscopy) screening for tentatively identified compounds (TICs) in the volatile and semivolatile organic fractions, infrared spectroscopy (IR) for organic compounds, inductively coupled plasma (ICP) for inorganic substances, and product comparison. These methods are used to determine chemical percentages in waste samples. After characterization, future sampling and analysis efforts can focus on substances identified above the action level.

2.11 REPRESENTATIVE SAMPLING APPROACHES

Representative sampling approaches appropriate for waste sampling include judgmental, random, systematic grid, systematic random, and transect sampling. A representative sampling plan may use one or a combination of these approaches.

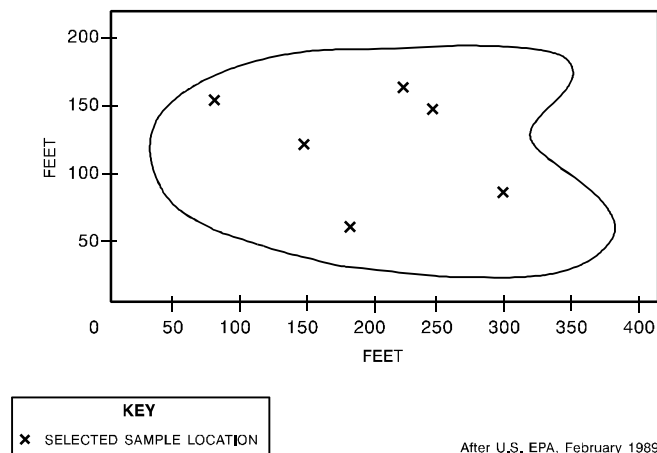
2.11.1 Judgmental Sampling

Judgmental sampling is the biased selection of sampling locations at a site, based on historical information, visual inspection, sampling objectives, and professional judgment. In waste sampling, three distinct situations prevail: 1) selecting locations within a large waste stream such as a waste pile or a stratum in an impoundment; 2) selecting a subset of containerized wastes when all containers cannot be sampled; and 3) sampling both containerized and non-containerized wastes in order to identify worst-case conditions and establish threat. If determining threat, the presence of certain site conditions such as leaking drums, spill areas, and large volume containers will indicate appropriate sampling locations if the source is known to be hazardous. Select drums to sample by existing labelling/markings or by container type, but not by random selection. When establishing threat, screen drums first to select a subset of drums containing hazardous materials or waste to be sent for analysis. This will avoid sampling drums of non-hazardous materials, which is not cost-effective. Judgmental sampling includes no randomization in the sampling strategy, precluding statistical interpretation of the sampling results.

2.11.2 Random Sampling

Random sampling is the arbitrary collection of samples having like contaminants within defined boundaries of the area of concern. Choose random sampling locations using a random selection procedure (e.g., a random number table). (Refer to Ford and Turina, July, 1984, for an example of a random number table.) The arbitrary selection of sampling points ensures that each sampling point is selected independently from all other points, so that all locations within the area of concern have an equal chance of being sampled. Randomization is necessary in order to make probability or confidence statements about the sampling results. The key to interpreting these statements is the assumption that the site or waste stream is homogeneous with respect to the parameters being monitored. The higher the degree of heterogeneity, the less the random sampling approach will adequately characterize true conditions. The use of random sampling on a subset of containers is not appropriate if different waste streams or concentrations might be present. Random sampling of waste piles and impoundments is often appropriate because of their large areal extent and relative homogeneity. Use random sampling to confirm the attainment of treatment levels of contaminated waste. (Refer to U.S. EPA, *Methods for Evaluating the Attainment of Cleanup Standards*, Volume 1 -- *Soils and Solid Media*, EPA/230/02-89/042, pages 5-3 to 5-5 for guidelines on selecting sample coordinates for random sampling.) Figure 2 illustrates a random sampling approach.

Figure 2: Random Sampling

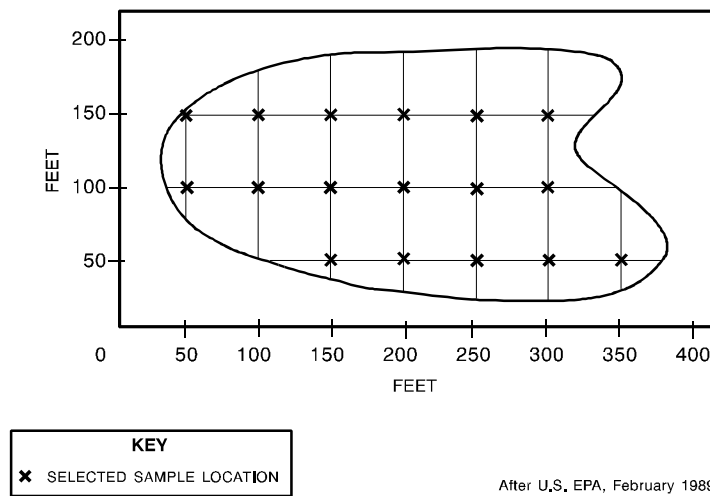


2.11.3 Systematic Grid Sampling

Systematic grid sampling involves subdividing the area of concern by using a square or triangular grid and collecting samples from the nodes (intersections of the grid lines). Select the origin and direction for placement of the grid using an initial random point. From that point, construct a coordinate axis and grid over the source. Generally, the more samples collected (and the smaller the grid spacing), the more reproducible and representative the results. Shorter distances between sampling locations also improve representativeness.

Systematic grid sampling can be used to characterize a waste pile, impoundments, or loose tank bottom solids. Systematic grid sampling is not applicable to sampling individual small containers or drums. (Refer to U.S. EPA, *Methods for Evaluating the Attainment of Cleanup Standards*, Volume 1 -- *Soils and Solid Media*, pages 5-5 to 5-12 for guidelines on selecting sample coordinates for systematic grid sampling.) Figure 3 illustrates a systematic grid sampling approach.

Figure 3: Systematic Grid Sampling



2.11.4 Systematic Random Sampling

Systematic random sampling is a useful and flexible design for estimating the average pollutant concentration within grid cells. Subdivide the area of concern using a square or triangular grid (as mentioned above) then collect samples from within each cell using random selection procedures. Systematic random sampling allows for the isolation of cells that may require additional sampling and analysis. Like systematic grid sampling, systematic random sampling can be used to characterize a waste pile, loose tank bottom solids, or impoundments, but not small containers or drums. Figure 4 illustrates a systematic random sampling approach.

2.11.5 Transect Sampling

Transect sampling involves establishing one or more transect lines across a surface. Collect samples at regular intervals along the transect lines at the surface and/or at one or more given depths. The length of the transect line and the number of samples to be collected determine the spacing between sampling points along the transect. Multiple transect lines may be parallel or non-parallel to one another. If the lines are parallel, the sampling objective is similar to systematic grid sampling. The primary benefit of transect sampling versus systematic grid sampling is the ease of establishing and relocating individual transect lines.

Transect sampling is applicable to waste piles or impoundments. Figure 5 illustrates a transect sampling approach.

Figure 4: Systematic Random Sampling

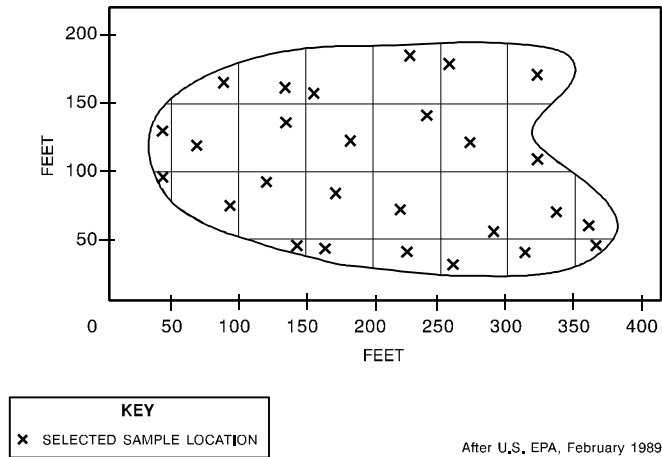
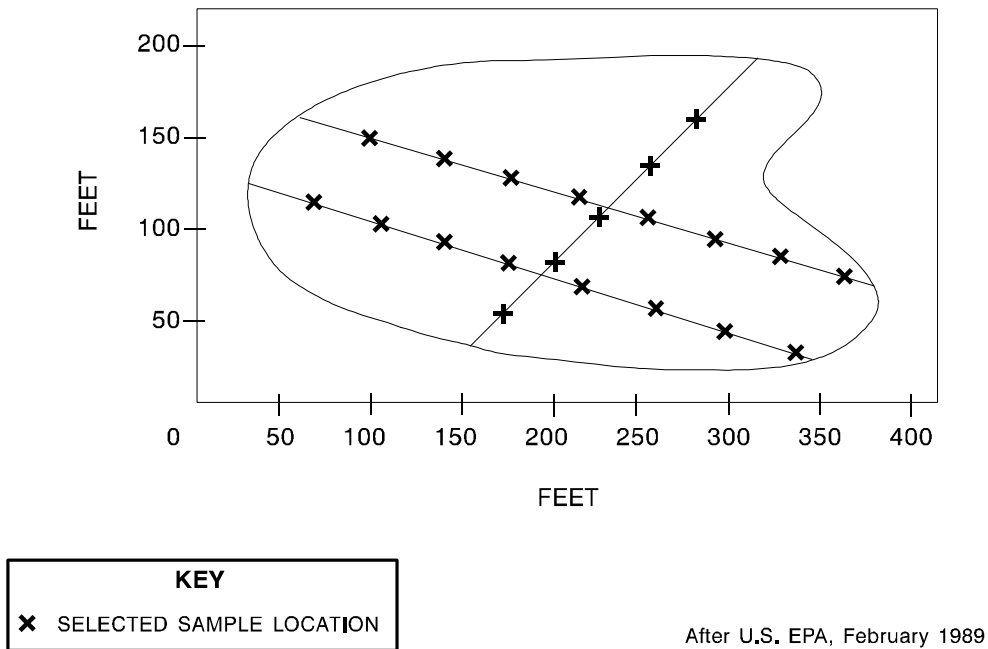


Figure 5: Transect Sampling



2.12 SAMPLING LOCATIONS AND NUMBER

The locations and number of samples to be collected must be carefully selected to obtain samples that are truly representative of the material being sampled, as well as of the general site area. The sampling objectives, waste type, container/source type,

sampling approach, and other factors determine where and how many samples are collected. For example, a judgmental sampling approach can establish threat or identify the presence of wastes with a few carefully selected samples. A larger number of samples are needed to characterize wastes, and sampling locations should be selected using random, systematic grid, and systematic random sampling techniques. Field screening techniques are valuable for selecting

sampling locations. In situations where there is a very large waste area or numerous containers of potentially different wastes, field screening techniques may identify similar waste streams. These wastes can be segregated into general chemical classes (e.g., strong acids, halogenated solvents) and then samples can be collected for confirmation by laboratory analysis.

Sampling locations which pose a severe chemical or physical hazard to sampling teams (e.g., cylinders of hydrofluoric acid, hydrogen cyanide, or nerve agents) should be avoided or sampled remotely. Sampling cylinders requires specially trained and authorized personnel.

2.13 EXAMPLE SITE

2.13.1 Background

ABC Plating, a multi-purpose specialty plating facility in northern Pennsylvania operating from 1947 to 1982, stored and treated its plating wastes by placing them in a series of unlined and unpermitted impoundments. State RCRA personnel cited the owner/operator for the operation of an unpermitted treatment system and ordered the owner to submit a remediation plan for state approval. Before the state could follow up on the order, the impoundments were partially backfilled with the wastes in place. The facility was later destroyed by a fire of suspicious origin. The owner abandoned the facility and could not be located by enforcement authorities. The state contacted U.S. EPA for an assessment of the site for a possible federally funded response action.

U.S. EPA initiated a removal assessment with the following primary sampling objectives:

- To establish the identities and volumes of hazardous materials present on the site to determine the potential threat to the surrounding population and the environment.
- To develop site stabilization strategies.

After federal funds were obtained and the site was stabilized, EPA addressed two additional objectives:

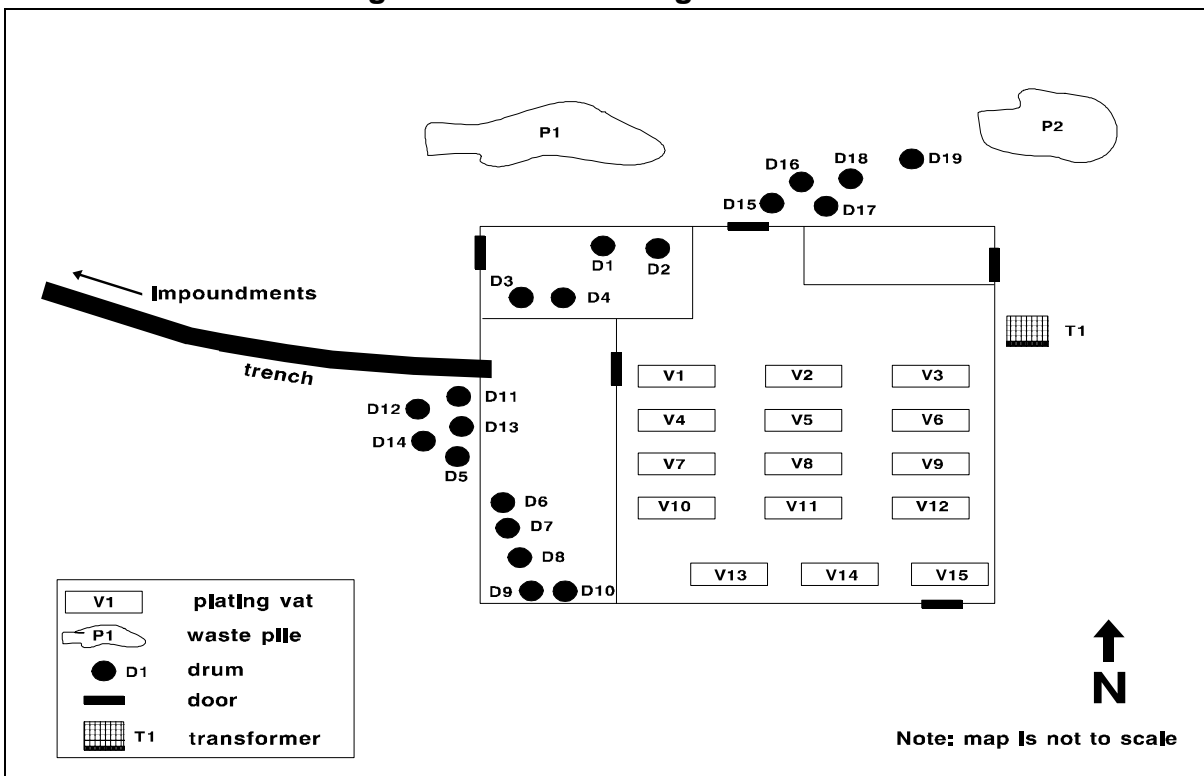
- To identify treatment and disposal options for the wastes on site.
- To verify that established clean-up levels are met.

2.13.2 Site Entry

Within four hours of the initial request for assistance from the state, an EPA On-Scene Coordinator (OSC) and other response personnel mobilized to the site with equipment to perform multi-media sampling. The next day, the OSC met with the township manager, representatives from the county health department and the Pennsylvania Department of Environmental Resources (PA DER), and the township Fire Chief. The OSC reviewed PA DER enforcement reports and aerial photographs which indicated the presence and locations of chromium, copper, and zinc plating process areas. The OSC interviewed local residents and performed a walk-through, donning Level B personal protective equipment (PPE), to survey the general site conditions. A site sketch was generated (Figure 6), indicating the locations and container types of the wastes. A total of nineteen 30- and 55-gallon fiber and metal drums, fifteen 250- to 500- gallon plating vats, two 10- to 15- cubic yard waste piles, a feeder trench leading to two 80 feet x 20 feet x 7 feet partially filled impoundments, and a transformer were located and noted in the site sketch. Some rooms of the building could not be entered because of unsafe structural conditions caused by the fire.

The OSC and PA DER reviewed all available information to formulate a sampling plan for the drums, vats, impoundments, and waste piles. The entry team used a judgmental sampling approach during the initial assessment, first collecting samples from containerized wastes for screening and possible analysis (suspecting that the containerized concentrated material posed the greatest potential hazard).

Figure 6: ABC Plating Site Sketch



2.13.3 Site Inventory

During this phase of the response, a field entry team in Level B personal protective equipment (PPE) inventoried drums, plating vats, waste piles, impoundments, and a transformer found at the site.

Drums

The entry team numbered each drum and noted drum type, size, condition, and label information on a drum inspection log (Figure 7). The chemical properties of constituents listed on labelled drums were researched. Typical hazardous materials used at plating facilities were determined from the references available; these substances include strong acids and bases, heavy metal solutions and solids, and cyanide-bearing compounds.

Vats

The plating vats were inspected, numbered, and noted on the site sketch. An estimate of the volume was documented for each of the vats. All vats were covered with non-reactive polyethylene sheeting to

prevent rain water from collecting in them and increasing the waste stream volume.

Waste Piles

The waste piles were inspected and noted on the site sketch. Transects were established along the longest horizontal axis of each pile. The transects were also noted on the site sketch. The samples will be collected and screening will be conducted along each transect.

Impoundments

The OSC determined that the contents of the impoundments posed a potential direct contact hazard to the surrounding population. In preparation for sampling, a transect was established from the entry point of the feeder trench across each impoundment. During site operation, wastes flowed from the feeder trench into the impoundments. Suspended solids were suspected to be present in a gradient decreasing with distance from the feeder trench.

Transformer

The OSC was concerned that the single transformer outside the plating building could contain PCB dielectric fluid. Inspection of the transformer determined that it was disconnected by first responders during the facility fire. The maximum transformer volume, the type of oil used, the date of manufacture, and the manufacturer's name were indicated on the metal plate on the side of the unit, and this information was noted. The unit was not leaking, so it was numbered and noted on the site sketch for future screening or sampling.

Information obtained from the site inventory and data review was used to create a site-specific conceptual model. Sources (e.g., vats, drums), pathways (e.g., vapors from the impoundments, soil under leaking drums), and potential receptors (e.g., local residents) were detailed to assist the selection of sampling approaches, objectives, and locations.

2.13.4 Selecting Analytical Parameters

Analytical parameters were selected based on research of plating chemistry and the initial site screening. Plating facilities generally use either an acid bath or basic cyanide bath to achieve the desired coating on their metal products. Based on the researched information and the measured pH of the liquid wastes on site, the following compounds were suspected to be present:

- Sodium and zinc cyanide salts and sodium hydroxide (highly basic, grey to green color) from zinc plating practices
- Chromic acid and sulfuric acid/sodium sulfate (acidic, yellow or dulled color) from chromium plating practices

- Copper sulfate and sulfuric acid (acidic, blue/green color) from copper plating practices.

During the assessment, liquid vat samples underwent field screening to assist in the selection of analytical parameters. Samples from all highly basic solutions were shipped to a laboratory for analysis of metals and cyanide. Acidic samples were sent for analysis of metals only. The composition of some of the drummed materials was initially unknown. In addition to the plating chemicals listed above, other possible drum contents included various cleaners, oils, fuels, and solvents. Many of the drums still had labels identifying their contents, and field screening was used to confirm content composition. Acids and bases were easily identified with pH paper, which was by far the most useful and inexpensive screening tool.

The waste piles were thought to include plating vat sludge waste. Samples collected from the piles were sent for laboratory metals and cyanide analyses.

Initial samples from impoundment liquids and solids (large-volume unsecured waste streams) were sent for full target analyte list (TAL), hexavalent chromium, pH, and total and amenable cyanide analyses. These analyses were conducted to fully characterize the liquid impoundment wastes for evaluation of the various on-site water treatment system needs. This information was later used to select optimum pH conditions and flocculent type for maximum settling efficiency. A local industrial wastewater treatment facility agreed to accept the liquid wastes if the heavy metal and cyanide levels were within their permit parameters. In addition to evaluating off-site treatment and disposal options, the impoundment bottom samples were characterized using target compound list (TCL), hexavalent chromium, total and amenable cyanide, pH, and total alkalinity analyses to allow evaluation of possible on-site stabilization, solidification and treatment techniques.

Figure 7: Example of a Drum Inspection Log

DRUM INSPECTION LOG	
Site: _____	
Location: _____	
Drum Number: _____	Date: _____
Project Code Number: _____	Time: _____
Type of Contents: ___ SOLID ___ LIQUID ___ SLUDGE ___ LAB PACK	
Color _____	PID _____
pH _____	CGI _____
FID _____	
Amount of Contents: ___ Full ___ 3/4 ___ 1/2 ___ 1/4 ___ Less than 1"	
Drum Size: ___ 55-gallon ___ 41-gallon ___ 30-gallon ___ 5-gallon	
Drum Markings:	

Hazard Class Label:	

Drum Type: ___ 17H ___ 17E ___ 37M ___ Fiber ___ Overpack ___ Other	
Drum Construction: ___ Metal ___ Poly ___ Fiber ___ Polylined ___ Other	
Drum Condition: ___ Deteriorated ___ Leaking ___ Dented ___ OK/DOT	
Sample Method: ___ Pipette ___ Trowel ___ Other	
Sample Number _____	Custody Sheet Number _____
Comments:	LAYER DESCRIPTION

Observations by: _____	

3.0 FIELD ANALYTICAL SCREENING AND SAMPLING EQUIPMENT

3.1 INTRODUCTION

Field analytical screening techniques and equipment may provide valuable information for developing sampling strategies. Field analytical screening can determine chemical classes of wastes and in some cases can identify particular substances of concern. Real-time or direct-reading capabilities narrow the possible groups or classes of substances which aids in selecting the appropriate laboratory analytical method. These screening techniques are useful and economical when gathering large amounts of site data. Some of the commonly used screening methods for waste analysis are presented in this chapter in the general order that they would initially be used at a waste site, although site-specific conditions may mandate a different sequence. This chapter focuses on site-screening methods, but the instruments described below have specific health and safety applications as well. Refer to the *Compendium of ERT Waste Sampling Procedures*, OSWER Directive 9360.4-07, for specific information about most of the following techniques or equipment. Refer to Standard Operating Safety Guides for each instrument, and the *Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities* (NIOSH Pub. 85-115) for site entry information.

3.1.1 Combustible Gas Indicator

The combustible gas indicator (CGI) measures the concentration of a flammable vapor or gas in the air, registering the results as a percentage of the lower explosive limit (LEL) of the calibration gas. The CGI is often combined with an oxygen meter; some contemporary models also have built-in compound-specific detectors (e.g., hydrogen sulfide, sulfur dioxide, carbon monoxide, and hydrogen cyanide). CGIs are particularly useful for entry into unknown and/or confined space atmospheres.

There are several factors that must be considered when using a CGI for waste site work. The accuracy of the reading is temperature dependent; the CGI must be calibrated at ambient temperatures. The sensitivity of the CGI is also a function of the physical and chemical properties of the calibration gas versus those of the unknown atmosphere. Oxygen concentrations

that are less than or greater than normal may cause erroneous readings. Leaded gasoline vapors, halogens, silicates, and sulfur compounds can decrease sensitivity. As a sample screening tool, the CGI is of limited value because it yields non-qualitative results for flammable vapors.

3.1.2 Radiation Screening Instruments

Screening for ionizing radiation is mandatory for all Superfund assessments, primarily for health and safety reasons. Since gamma rays and X-rays have high penetration capabilities even at extended distances, radiation screening instruments are generally used during the initial site entry. As containerized wastes are opened, alpha and beta radiation which was not detected during the initial walk-through screening may be encountered.

Most of the commonly used radiation screening instruments have gamma or beta/gamma detecting probes. Pure alpha detectors are not commonly used on site because the probes are too fragile and because pure alpha emitters are rare. However, an alpha/beta/gamma probe is suggested for screening wastes in lab packs, research facilities, laboratories, and on military installations where radioactive waste may be present (e.g., Department of Defense (DOD) and Department of Energy (DOE) installations).

3.1.3 Flame Ionization Detector

The flame ionization detector (FID) detects and measures the level of total organic compounds (including methane) in the ambient air or in a container headspace. The FID is used to evaluate existing conditions, identify potential sample locations and extent of contamination, and support health and safety decisions. The FID uses the principle of hydrogen flame ionization for detection and measurement. It is especially effective as an ethane/methane detector when used with an activated charcoal filter because most organic vapors are absorbed as the sample passes through the filter, leaving only ethane and methane to be measured.

The FID operates in one of two modes: the survey mode, or the gas chromatography (GC) mode. In the survey mode, the FID provides an approximate total concentration of all detectable organic vapors and gases measured relative to the calibration gas (usually methane). The GC mode identifies and measures specific components, some with detection limits as low as a few parts per million (ppm), using known standards run concurrently in the field. Since the GC mode requires standards to identify classes of compounds, before sampling it is necessary to have an idea of which compounds might be present on site. Advantages of the FID are that it is portable, relatively rugged, and provides real-time results.

The FID does not respond to inorganic substances. It has positive or negative response factors for each compound depending on the selected calibration gas standard. The FID does not recognize and may be damaged by acids; use pH paper to screen acids. Ambient air temperatures less than 40 degrees Fahrenheit will cause slower responses; relative humidity of greater than 95 percent can cause inaccurate and unstable responses. Low ambient oxygen levels can cause the flame to go out; use a CGI/oxygen meter in conjunction with an FID in confined space applications. Interpretation of readings (especially in the GC mode) requires training and experience with the instrument.

3.1.4 Photoionization Detector

Another portable air monitoring instrument frequently used for field screening is the photoionization detector (PID). Like the FID, the PID provides data for real-time total organic vapor measurements evaluating existing conditions, identifying potential sample locations and extent of contamination, and supporting health and safety decisions. The PID works on the principle of photoionization. Unlike the FID, the PID can be used to detect gross organic, and some inorganic vapors depending on the substance's ionization potential (IP) and the selected probe energy. It is portable and relatively easy to operate and maintain in the field.

The PID detects total concentrations and is not generally used to quantify specific substances. PIDs cannot detect methane; however, methane is an ultraviolet (UV) light absorber, and false negative instrument readings may register in methane-rich environments. The PID cannot detect substances with

IPs greater than that of the UV light source. Readings can be affected by high wind speeds, humidity, condensation, dust, power lines, and portable radios. Dust particles and water droplets (humidity) in the sample may collect on the light source and absorb or deflect UV energy, causing erratic responses in PIDs not equipped with dust and moisture filters.

3.1.5 Colorimetric Tubes

Colorimetric indicator tubes (e.g., Dräger, Sensidyne, MSA) provide real-time results in environments where a specific gas or vapor is suspected to be present. In waste sampling, they are useful for situations such as screening drums, where drum labels provide limited information on the contents of only some of the drums.

Colorimetric tubes consist of a glass tube filled with silica gel or a similar material impregnated with an indicator reagent which changes color in the presence of specific contaminants. The tube is attached to an intrinsically safe piston-syringe or bellows-type pump which slowly pulls a measured volume of air through the tube. The contaminant then reacts with the indicator chemical within the tube producing a color change proportional to the concentration of the chemical.

Although the indicator tubes are usually chemical or class specific, interferences can occur. Common interferences are noted in the directions for the specific tube. The tubes have a limited shelf life, and cannot be reused. Results can be misinterpreted due to cross-sensitivity, and there exists a potential for error in reading the end point of color change. Errors result if the limit of the tube has been exceeded (in very concentrated environments). High humidity may reduce tube sensitivity.

3.1.6 Hazard Categorization

Hazard categorization (haz-cattng) is performed as an initial screen for hazardous substances to provide identification of the classes/types of substances present in individual waste streams. Haz-cattng tests for general chemical characteristics or the presence of specific ions to determine chemical class; it is not compound-specific. The information from haz-cattng is useful for determining compatibility of unknown wastes.

Various indicators and wet chemistry tests characterize the wastes according to their chemical/physical properties (e.g., solubility, combustibility), and indicate the presence of sulfides, oxidizers, and cyanide. The haz-cating procedure requires numerous chemical reagents and interpretation of results. Common haz-cat tests include the *char* test for differentiating organic from inorganic substances; *chlorine hot wire* test to detect chlorine in organic solvents; *combustibility* test; *cyanide* test for cyanide salts; *flame* test for identifying cations and some anions; *iodine* crystal test for solvent classification; *oxidizer* test; *sulfide* test; *water solubility* test; and *pH* test.

3.1.7 Immunoassay Tests

Immunoassay tests can be used on site to screen for certain organic compounds such as pentachlorophenol (PCP), PCBs, and pesticides. Immunoassay tests are used for locating and mapping the extent of contamination, and for screening samples in the field prior to laboratory analysis.

Immunoassay tests utilize semi-quantitative, colorimetric methods. Some of the commonly used tests utilize tubes coated with a chemical that specifically binds to the contaminant. These types of tests utilize highly selective antibodies and sensitive enzyme reactions to yield qualitative, semi-quantitative, and quantitative results for a specific compound or for a closely related series of compounds (e.g., PCP, PCBs, and 2,4,D-pesticides). Antibodies can be either coated on the test tube, or attached to microparticulates or reaction well/ plates, depending on the brand. Other types of immunoassay tests utilize enzyme-linked, immunosorbent assays (ELISAs) and magnetic particles to bind to the contaminants.

The concentration range of a sample is determined by comparing the color change of the sample with that of duplicate standards of known concentrations. The color intensity in each tube decreases as the contaminant concentration increases. Photometers are available to "read" and digitally display, print, and store the color difference between the prepared sample and the standards. Since the results are compared to standards, the accuracy achievable is a contaminant range (e.g., greater than 100 ppm but less than 1,000 ppm). Laboratory confirmation is required when using these tests in the semi-quantitative and

qualitative modes. Performance evaluation spikes determine the efficiency of the test. Some training is needed to effectively run and interpret immunoassay tests.

3.1.8 X-Ray Fluorescence

Field analytical screening using X-ray fluorescence (XRF) is a cost-effective and time-saving method to detect and classify lead and other heavy metals in wastes. XRF screening provides immediate semi-quantitative results. The principle behind XRF is the detection and measurement of the X-rays released from an atom when it is ionized. The measure of energy released identifies the atom present.

Results of XRF analysis help determine the presence of metals and are often used to assess the extent of soil contamination at a site. For waste sampling, the XRF can be used for screening waste piles and for assessing metals in certain liquids such as paint. XRF use requires a trained operator and may require numerous site-specific calibration samples.

3.1.9 Gas Chromatograph

Although many FIDs are equipped with a GC mode, an independent, portable GC can also be used on site to provide a chromatographic profile of the occurrence and intensity of unknown volatile organic compounds (VOCs). The GC is useful as a screening tool to determine "hot spots," potential interferences, and semi-quantitation of VOCs and semi-volatile organic compounds (semi-VOCs).

Compounds with high response factors, such as benzene and toluene, produce large response peaks at low concentrations, and can mask the presence of compounds with lower response factors. However, recent improvements in GCs, such as pre-concentrator devices for lower concentrations, pre-column detection with back-flush capability for rapid analytical time, and the multi-detector (PID, FID, and electron capture detector (ECD)), all enable better detection of compounds. The GC is highly temperature-sensitive. It requires set-up time, many standards, and operation by trained personnel.

3.2 SAMPLING EQUIPMENT

Representative waste sampling requires an understanding of the capabilities of the sampling equipment, since the use of inappropriate equipment may result in biased samples. Select appropriate sampling equipment based on the sample type and matrix, physical location of the sample point, and other site-specific conditions. Consideration must be given to the compatibility of the waste with the design and composition of the sampling device. Follow SOPs for the proper use and decontamination of sampling equipment. This section provides descriptions of drum opening and sampling equipment descriptions, and other information to assist in selecting appropriate equipment. Refer to the *Compendium of ERT Waste Sampling Procedures*, OSWER Directive 9360.4-07, for expanded guidelines on the use of the equipment discussed below.

3.2.1 Drum Openers

Closed drums need to be opened for sampling. Tools suitable for opening drums include: universal bung wrench, drum deheader, backhoe spike, hydraulic drum opener, and pneumatic bung remover. Each of these devices has specific applications based on the drum type, composition, condition, location, and suspected contents.

Follow ERT SOP #2009 (Drum Sampling) in the *Compendium of ERT Waste Sampling Procedures*, OSWER Directive 9360.4-07, for guidelines on opening drums and the operating instructions for the particular equipment used. Always use non-sparking instruments to open drums and comply with proper health and safety protocols. Note that the use of a non-sparking tool does not completely eliminate the possibility of a spark being produced. Drums should be grounded to decrease the chance of static charges and sparks. Stage drums by suspected compatibility type prior to opening and sampling to decrease the risk of chemical reaction between incompatible substances.

3.2.2 Liquid Samplers

The following samplers are useful for collecting waste liquids from various sources: composite liquid waste sampler (COLIWASA), glass thief, bailer, and bacon bomb. Each has specific applications for use

depending on the type and nature of the waste and the type of source.

COLIWASA

The COLIWASA is a tool typically used for sampling stratified liquids in drums and other similar containers. It is a transparent or opaque glass, PVC, or Teflon tube approximately 60 inches in length and 1 inch in diameter. A neoprene stopper at the bottom of the tube can be opened and closed via a rod that passes through the length of the sampler.

The COLIWASA is difficult to decontaminate in the field but is versatile and simple to operate. Because of the relatively high cost of the COLIWASA, COLIWASA-type glass thieves have been developed which utilize neoprene or ground glass stopper mechanisms. Before conducting multiphased sampling, make sure that the physical and chemical properties of the container's contents and phases are understood.

Glass Thief

Another commonly used drum sampling device, the glass thief, is a hollow glass tube 40 to 48 inches in length and commonly 10 mm to 19 mm in diameter. The larger diameter tubes are used to collect more viscous materials. The glass thief is simple to operate, versatile, and disposable, eliminating the need for decontamination.

Conduct the sampling carefully in order to avoid sample spillage. Low viscosity liquids, thin-layered phases, and partially filled containers may be difficult to sample with a hollow glass thief. In those cases, use the COLIWASA-type glass thief, whose stopper mechanism prevents the sample from leaking out of the thief as it is removed from the container.

Bailer

A bailer is used to sample waste liquids in vessels (wells, tanks, or deep containers) where the liquid surface is far below the sampling entry access (i.e., too far for a glass thief or COLIWASA). The bailer consists of a hollow tube (constructed of relatively inert materials such as stainless steel, glass, or Teflon) with a bottom ball-check valve, usually suspended from a wire or rope for sampling. Bailers are good for

sampling VOCs because of the relatively low surface-to-volume ratio which reduces off-gassing.

Most bailers cannot obtain discrete depth or phased samples, so their use is primarily limited to homogeneous liquids or shallow (length of bailer) sampling. As with any sampling device, the construction material should not interfere with the wastes or the desired analytical parameters.

Bacon Bomb

The bacon bomb sampler is used to collect waste liquid samples from various levels within storage tanks or surface impoundments. Storage tank and impoundment wastes are often stratified, and the bacon bomb is useful when a discrete sample is needed from any level in the tank or impoundment. The bacon bomb consists of a cylindrical body with an internal tapered plunger for sampling. A separate line attached to the top of the plunger opens and closes the bottom valve at the desired depth or stratum. A removable top cover attaches the sample line and has a locking mechanism to keep the plunger closed after sampling. The bacon bomb is usually constructed of chrome-plated brass and bronze or stainless steel. A rubber O-ring acts as the plunger sealing surface.

Transfer of the sample to sample containers is sometimes difficult and tends to aerate the sample, resulting in loss of volatile constituents. The bacon bomb sampler can be more difficult to decontaminate than a bailer.

3.2.3 Sludge Samplers

The following devices are useful for sampling waste sludges: Ponar/Ekman dredge, sludge judge, and PACS grab sampler.

Ponar/Ekman Dredge

Ponar/Ekman dredges are clamshell-shaped scoops that are used to extract waste sludge samples from the bottom of impoundments, lakes, or other standing water bodies. The Ponar dredge's jaws are latched open and the unit is slowly lowered to the bottom of the area being sampled. When tension is eased on the lowering cable, the latch releases, and the lifting action of the cable on the lever system closes the dredge around a sample of sludge. The Ekman dredge

operates similarly but it closes by using a messenger or depressing a button on the upper end of the handle.

The substrate depth to which both dredges can sample usually does not exceed 4 to 6 inches, and they are not capable of collecting an undisturbed sample. As a result, material in the top inch of sludge cannot be separated from material at lower depths. The sampling action of the dredges causes agitation currents which may temporarily re-suspend some settled solids, especially the fine fraction.

Dredges are normally used from a boat or dock. Because the dredges are heavy, a boom is frequently used to ease the raising and lowering of them. Dredges are not usually effective in sampling hard or stony bottom material. Bottom vegetation will also limit dredge effectiveness.

Sludge Judge

The sludge judge is a long narrow tube with a check valve on the bottom, used primarily to obtain cores of waste sludge, or waste liquids mixed with sludge, from drums, tanks, or similar sources. Sludge judges are useful for determining the physical state of a tank's contents or its volume of settled sludge. The sludge judge is constructed of PVC which can limit its use because of potential interference with the contaminants of concern. The device is difficult to decontaminate and not recommended for use with very thick sludges.

PACS Grab Sampler

The PACS grab sampler is used to collect sludge samples at discrete depths from surface impoundments such as ponds and lagoons and also from certain types of containers. The PACS grab sampler consists of a 1000-ml wide-necked bottle with a control valve which screws on to the end of a 2-meter long handle. Large openings in the bottle facilitate sample collection. The control valve is operated from the top of the handle once the sampler is at the desired depth. Depth of sampling is limited by the length of the pole handle. The device is not useful in very viscous sludges, and it can be difficult to decontaminate.

3.2.4 Solids Samplers

The following devices are used to collect samples from waste piles and other sources of solid waste: scoop/trowel, bucket auger, sampling trier, waste pile sampler, cork screw auger, and split-barrel sampler.

Scoop/Trowel

Scoops or trowels are useful for collecting solid or sludge samples from waste piles. Collection is usually limited to near-surface and depends on the length of the scoop or trowel. These instruments are available in a variety of materials, including stainless steel and plastic. Stainless steel scoops and trowels are appropriate for VOC sample collection but require decontamination between sampling stations. Plastic scoops and trowels are suitable for metal analyses and are disposable, which eliminates the need to decontaminate between sampling stations. Do not use trowels with painted or chromium-plated surfaces, because the paint or plating can chip off into the sample.

Bucket Auger

Bucket augers are typically composed of stainless steel and are used to collect solid or sludge samples from waste piles or surface impoundments. The auger is effective for subsurface sampling, but tends to destroy horizons during sampling, making VOC collection difficult. The bucket auger is therefore not recommended for VOC collection; use a split-barrel sampler instead. Bucket augers can be used for sludge sampling in a surface impoundment, depending on accessibility.

Bucket augers provide uniform sampling diameter and good depth control, and are easy to decontaminate. Their effectiveness is reduced in rocky or hard solids, heavy clays, or very sandy solids.

Sampling Trier

A sampling trier is used to collect powdered or granular materials from bags, fiber or metal drums, sacks, or similar containers. A typical sampling trier is a long tube with a slot which extends almost its entire length. The tip and edges of the tube slot are sharpened so that when rotated after insertion into the material, the trier cuts out a sample core. Sampling triers range from 24 to 40 inches in length and from

1/2 to 1 inch in diameter, and are usually made of stainless steel (or a similar composition) with wooden handles. Triers are relatively easy to use and to decontaminate.

Waste Pile Sampler

The waste pile sampler is essentially a large sampling trier used for sampling large waste piles (with cross-sectional diameters greater than 1 meter). It can also be used for sampling granular or powdered wastes, and material in large bins or barges.

The waste pile sampler is commercially available, but one can be easily and inexpensively fabricated from sheet metal or plastic pipe. The sampler does not collect representative samples when the diameters of the solid particles are greater than one-half the diameter of the tube.

Cork Screw Auger

The cork screw auger is a hand-driven sampler used to sample bulk solid wastes such as waste piles. The auger tip resembles a large drill bit ranging from 3/4 to 1-1/2 inches in diameter. The auger is used for sampling at depth by adding rod extensions. It is effective in soft to hard materials, although saturated waste may be difficult to sample. Decontamination of the auger is relatively easy. The cork screw auger disturbs the waste profile and thus has limited utility for VOC sampling.

Split-Barrel Sampler

The split-barrel sampler (also called a split-spoon sampler) is used to collect waste samples from the bottom of boreholes. The split-barrel sampler consists of a hollow tube with a circular chisel or cutting shoe threaded onto one end and a driving head or collet threaded onto the other end. The sample tube is split lengthwise into two halves to facilitate sample removal and decontamination. A drilling rig is required to use the split-barrel sampler. The sampler is attached to the end of the drilling rod and is driven into the bottom of the borehole with a specially designed, 140-lb. drive hammer. The split-barrel sampler can be used to determine the relative density of the material that is being cored or drilled by counting the number of drive hammer blows it takes to drive the barrel 18 inches below the bottom of the borehole.

The split-barrel sampler is useful for collecting relatively undisturbed waste samples from great depths. Because split-barrel samplers do not disturb the sample, they are suitable for sampling VOCs. They can be used to sample deep into large waste piles, subsurface wastes, or dry lagoon beds, but they are not effective in rocky or very consolidated materials.

3.3 EXAMPLE SITE

After conducting proper site entry procedures (health and safety monitoring), the OSC utilized field screening techniques to the greatest extent possible. This allowed for the rapid collection of information to support the decision-making process and to limit the need for laboratory analysis. All waste streams were screened (as discussed below) to determine which laboratory analyses would be necessary, and to provide a logical basis for selecting a limited number of analytical parameters. All waste streams were also screened for radiation; none was detected.

3.3.1 Drum Screening and Sampling Equipment

Each of the closed drums appeared to be in relatively good condition, showing no signs of internal pressure or other instability, and were opened using a spark-proof bung wrench. Five drums were already open to the elements. After each drum was opened, samples were collected for screening using a glass thief. All drums were screened using haz-cattling procedures. All haz-cattling information was recorded in the field on a Hazard Categorization Data Sheet (illustrated in Figure 8). The first haz-cattling procedure conducted was for pH. Four drums were found to contain strong acids; five drums contained strong bases. Various colorimetric tubes were used in an attempt to identify specific acids; however, all the tubes exhibited positive reactions due to interferences from the presence of similar strong acids. Since cyanide-bearing solutions are typically basic, all basic solutions were screened for the presence of cyanide to prevent the potential generation of hydrogen cyanide gas (HCN) during handling. As a result of screening, three drums containing basic solutions were tentatively determined to contain cyanide. Additional haz-cat screening (PID, FID, solubility, chlorine, and peroxide) was conducted for the ten drums exhibiting relatively neutral pH. From this additional screening,

three drums of oil, one drum of halogenated solvents, and one drum of kerosene were tentatively identified. Two drums were suspected to contain rainwater. The screening results were inconclusive for three drums.

The drums containing strong acids and bases were, by definition, RCRA hazardous characteristic wastes. Because of the risks associated with strong acids and bases, there was no need for further data analysis to establish imminent threat. The entry team separated incompatible materials to reduce the risk of a chemical reaction/release.

3.3.2 Plating Vat Screening and Sampling Equipment

Some plating vats were already open; others had large, easy-to-open lids similar to that of a trash dumpster. The vat liquids and bottom solids were screened separately in the same manner as the drums, using a COLIWASA-style glass thief to collect the samples and haz-cattling to identify the wastes. The samplers were long enough to reach the bottom of the vats, providing a sample of the entire vertical column of liquid. A hollow glass thief was then used to collect a single grab sample from the bottom solids in each vat.

The vat contents were tentatively identified as strong acids, strong bases, and cyanide bases. Haz-cattling results were inconclusive for four vats. No volatile organic compounds were detected using PID and FID instruments.

3.3.3 Waste Pile Screening and Sampling Equipment

The piles contained blue and green solids that were assumed to be bottom solids cleaned from the plating vats. Screening samples from the waste piles were collected using a corkscrew auger and stainless steel trowels (because of the hardened texture of the piles). Waste pile samples were haz-catted in the same manner the samples from drums and vats. However, since haz-cat tests are better suited to liquid matrix waste streams, the two waste piles were difficult to classify into general hazard categories.

Screening results indicated that the wastes were not water reactive, flammable, combustible, or chlorine-bearing; organic compounds were not detected. Results of the cyanide test were positive. Sample

color and other visual signs of contamination were documented as the screening samples were collected.

3.3.4 Impoundment Screening and Sampling Equipment

Screening samples were collected from both surface impoundments. Waste liquids were sampled using a bacon bomb sampler, and waste bottom sludges were sampled using a Ponar dredge. Waste liquid samples were taken from the center of each impoundment at depths of 0 to 2 feet, and 2 feet to bottom. Five waste sludge samples were collected from the bottom of each impoundment at 20 foot intervals along a transect established across each impoundment. A small rowboat with stabilizing lines was moved along the transects to collect screening samples.

Impoundment liquids were screened by haz-catting. The results indicated that impoundment liquids contained water and were slightly acidic, possibly cyanide-bearing, non-flammable, and non-chlorinated. The PID and FID did not detect any organic vapors, suggesting a non-organic wastewater classification. A chemical test kit was used to identify low levels of

specific metals in the impoundment liquids. Screening of the bottom sludge had similar results, except for a higher metals content and the positive presence of cyanide.

3.3.5 Transformer Screening and Sampling Equipment

The transformer top was removed using a standard socket wrench. A transformer fluid screening sample was collected using a makeshift sampling device consisting of a clean, 4-oz sampling jar on a string. The transformer was screened for PCBs using a PCB screening kit. A grab sample was collected and the test was performed on site following the directions provided with the kit. The potential interferences listed in the directions were determined not to apply to this sampling event. The test indicated that the transformer contained less than the 50 ppm total PCBs action level (based on a colorimetric interpretation). The sample fluid was placed back into the transformer and the vessel was resealed.

Figure 8: Example of a Hazard Categorization Data Sheet

HAZARD CHARACTERIZATION DATA						Page ___ of ___
Site: _____					Date: _____	
Sampler(s): _____					Sample ID Number: _____	
Phase: All Top Bottom N/A					Sample Collected?: Yes No	
HAZARD CHARACTERIZATION RESULTS						
Soluble: Yes (dissolves/emulsifies in water)	Heavier (than water)					Lighter (than water)
pH: _____ (if using instrument, round to nearest whole number)						
Flammable: Yes No						
Chlorine: Yes No						
Oxidizer: Yes No						
Cyanide: Yes No						
Sulfide: Yes No						
Other Test A: _____						
Other Test B: _____						
Action Taken: Overpacked Staged (location _____) Bulked Other _____						
Sort Class: _____ (optional--specify 2 character alphanumeric designator to assign user sort class)						
SAMPLE DESCRIPTION						
Color(s): Colorless White Yellow Blue Red Green Purple Brown Black Other _____						
Clarity: Clear Cloudy Turbid (suspended solids) Opaque N/A (if solid)						
Viscosity: Water Light Oil Heavy Oil Sludge N/A (if solid)						
Impurities: _____						
Comments: _____ _____ _____						
Observations by: _____						

4.0 FIELD SAMPLE COLLECTION AND PREPARATION

4.1 INTRODUCTION

During a response action, proper field sample collection and preparation is as important as proper sampling equipment selection. Sample collection refers to the physical removal of a portion of waste material from its source for the purpose of either screening or laboratory analysis. Field sample preparation refers to all aspects of sample handling from collection to the time the sample is received by the laboratory. This chapter provides information on sample collection and preparation for various waste types and sources.

4.2 SAMPLE VOLUME

The volume of a sample should be sufficient to perform all required laboratory analyses with an additional amount remaining for analysis of QA/QC samples (including replicate analyses). However, because waste samples are generally of high concentration, sample volumes should be kept to a minimum (to minimize disposal costs). The EPA method description and the laboratory receiving the sample should be consulted for specific volume requirements for each parameter.

Make an initial estimate of the volume or area of waste represented by each sample. When obtaining representative samples from waste which appears to be relatively homogeneous, note the total waste volume in cubic yards or gallons.

4.3 SOURCE SAMPLING

The following sections provide general information on sampling several types of waste sources, including drums, bulk storage tanks, lab packs, surface impoundments, waste piles, surfaces, and debris. For specific sampling information on these waste types, refer to the *Compendium of ERT Waste Sampling Procedures*, OSWER Directive 9360.4-07.

4.3.1 Drum Sampling

Each drum can have different contents and concentrations, so each must be considered a unique waste source. A site-wide representative sampling approach is not appropriate. Screening techniques should be used on the contents of each drum to determine compatibility. This haz-cat information can help determine whether bulking of wastes is technically and economically feasible.

Drums may be sampled in place or staged in rows prior to sampling, depending on their condition and accessibility. If drums are stacked, a forklift or grapple may be needed to move them for sampling. (If drums cannot be safely moved, sample only accessible drums.) When moving drums, document on a site sketch their original locations. Number all drums and record their label information on a drum log sheet (see Chapter 2, Figure 6). Research all label information to determine health and safety precautions, including use of appropriate PPE. It does not necessarily follow that the labels affixed to the drums represent their actual contents. (Drums are often reused without regard to proper rinsing and relabelling procedures.) Further categorization is necessary to determine or confirm drum contents accurately. Be particularly cautious with drums that have crystalline deposits or a precipitate around the bung or lid. Some chemicals form a potentially shock-sensitive, explosive, or reactive phase as they degrade or react over time (e.g., picric acid forms shock-sensitive crystals). Do not move drums in this condition!

Bulging or misshapened drums and those with unknown contents should be opened remotely. Rough handling can trigger reactions which may cause them to rupture. Open unknown or unstable drums remotely. If drums contain a combination of solid, liquid, or sludge wastes, separate sampling of each phase may be necessary using chemically compatible sampling equipment (e.g., COLIWASA or glass thief). Where wastes are stratified, sample the top stratum first to avoid mixing strata. Since each drum may contain a different type of waste, it is usually not

possible to make composites from separate drums until the contents have been screened. As with most containers, drums should be sampled through upper bungs or openings whenever possible. Document contents, physical characteristics (e.g., color, viscosity) and field screening readings (e.g., FID, PID, CGI).

Refer to Section 2.11.1 for a discussion of judgmental sampling as it applies to drum sampling.

4.3.2 Bulk Storage Tank and Transformer Sampling

Bulk storage tank sampling involves many of the procedures and precautions noted for drum sampling. Number and document each tank, noting National Fire Protection Association (NFPA) 704 markings, if present. Document available information on vessel construction, tank location (e.g., in a tank farm), and the presence of any secondary containment. Estimate maximum tank volume using mathematical volume equations ($V=Br^2h$) or tank charts. Measure the content volume using exterior level indicators, if present.

Perform sampling through top hatches whenever possible; avoid using bottom valves because a spill is possible if the valve does not reseal. When there is more than one phase, identify the distinct phases and associated volumes. The objective is to identify volume in gallons (liquids) or cubic yards (solids) to determine the total waste volume that each sample represents. Sample each phase separately, including tank bottom sludges (use a bacon bomb sampler, PACS grab sampler, or a sludge judge). Obtain a sample from each compartment in multicompart-ment tanks.

When sampling specialized tanks or transformers, it may be necessary to use a manlift to gain access. Be certain that transformers are "off-line" and de-energized. Exercise spill control measures and ensure secondary containment is in place around a transformer before opening it. Access a transformer through the top and collect a stratified sample.

4.3.3 Lab Pack Sampling

Initial inspection of lab packs may uncover packing slips listing contents and associated volumes. The packing slips may be affixed to the outside of the

drum or under the drum lid. If the labels on individual containers are legible, inventory the containers and repackage them in inert cushioning and absorbent materials in accordance with 49 CFR 100-199. If the label is illegible or missing, the lab pack should be treated as an unknown. Unknowns are generally not manually opened because of the potential health and safety risks (exposure and reactivity). A safer approach is to use remote opening or crushing techniques and collecting the crushed containers and their contents in an absorptive medium, which is then sampled using a representative composite sampling method.

4.3.4 Surface Impoundment Sampling

When sampling a surface impoundment, consider its characteristics, which include size, depth, flow, liquid viscosity, bottom composition and whether a liner is present. The bottom sludges and liquid phases may be homogeneous or stratified.

Surface impoundments are often stratified by depth; each phase should be sampled separately. Transect sampling at various depths (including bottom sludge sampling) is generally recommended. Horizontal concentration gradients in the bottom sludges may be present from the point where liquids enter the impoundment. Vertical gradients may also be present in bottom sludges. The logistics and health and safety concerns of sampling large impoundments usually dictate the use of manlifts, boats, and safety lines. If a liner is present, take care to maintain its integrity.

4.3.5 Waste Pile Sampling

Waste pile sample collection techniques will depend upon the sampling objective. If the objective is to determine threat, grab sampling from the surface using a waste pile sampler or a scoop/trowel might be sufficient. If the objective is to obtain an average concentration value for the entire pile for treatment/disposal estimates, then the sampling should include grab samples or composite aliquots collected from the interior (using a waste pile sampler or an auger) and the surface of the pile. Composite aliquots collected at a given depth from several sides and the top of the pile can be used to obtain an estimate of the average pile concentration. The number of aliquots collected will depend on many site-specific factors,

including the size, composition, and accessibility of the waste pile, as well as on budget considerations.

The surface of a pile continually weathers chemically and physically. Depending on the size of the pile, it may be divided into sections for compositing at various depths. This will define an average concentration for each section of the pile. For large piles (e.g., large impoundment dredge or slag piles), a three-foot depth is generally adequate to reach the more representative materials. Extensive sampling of a pile for both chemical and physical characteristics is conducted during the evaluation of treatment and disposal options. If the pile has been stabilized (e.g., cover, liner), do not collect samples that might breach the integrity of the pile containment.

4.3.6 Surface Sampling

Special situations may present the need to sample surfaces such as floors, walls, or equipment. When sampling surfaces for contamination, choose sampling points based on site history, manufacturing processes, personnel practices, obvious contamination, and available surface area. Where possible, collect comparable media background samples from surfaces unlikely to have been contaminated. This is especially important when sampling for naturally occurring substances such as metals.

Surface sampling includes wipe, chip, and dust sampling. Analytical results for dust sampling are reported in weight/weight; wipe sampling results are reported in weight per unit area. Note that there are very few action levels or health standards reported in weight of contaminant per unit area to assist a Site Manager in decision-making.

The methods of sampling described below are appropriate for surfaces contaminated with non-volatile species of analytes (e.g., PCBs, PCDD, PCDF, metals, pesticides, cyanide). Detection limits are analyte-specific. Determine sample size based upon the detection limit desired, amount of sample requested by the analytical laboratory, and sampling locations and configuration.

Wipe Sampling

Wipe sampling is a method for collecting non-volatile species of analytes from relatively smooth, non-porous surfaces. It is appropriate for sampling walls,

floors, ventilation ducts and fans, empty transformers, process equipment, and vehicles. Wipe sampling can be used to confirm cleanup after steam cleaning or decontamination of smooth building walls.

To collect a wipe sample, use a piece of sterile medical gauze soaked in pesticide grade solvent (e.g., hexane, water, methanol, nitric acid). The type of solvent used depends on the target analytes. When requested in advance, analytical laboratories will often prepare the gauze and sample jars. Use caution and maintain proper safety protocols when handling hexane and other solvents.

Several wipe sampling techniques were developed for use in OSHA enforcement and industrial hygiene decision-making to evaluate potential sources of ingestion and direct contact exposures. Most of these techniques recommend a uniform wipe area of at least 100 cm², but larger areas may need to be wiped to collect enough sample for the analytical method detection limit. Disposable cardboard templates (or glass or stainless steel templates which can be decontaminated) are recommended to ensure a uniform surface area. Very few approved standards or action levels are available to compare with the wipe sampling results (this supports qualitative rather than quantitative conclusions). Wipe sampling is typically used to determine if decontamination has been effective or to select the type of disposal facility (e.g. hazardous vs. non-hazardous). A blank consisting of a solvent-soaked pad is required for each batch of samples.

Sampling locations are typically judgmental selections. They are chosen because they are areas of highest suspected contamination (for disposal decisions), or areas of suspected direct contact (for exposure and hygiene evaluations).

Chip Sampling

Chip sampling is a method for collecting non-volatile species of analytes from porous surfaces such as cement, brick, or wood. Sample points include floors near process areas, storage tanks, and loading dock areas. Chip sampling is usually performed with a hammer and chisel or with an electric hammer. It is important to ensure that the chipping device does not bias the integrity of the sample. Stainless steel tools allow for easy decontamination and preparation for reuse.

To collect the sample, chip the desired sampling area to a suitable depth (e.g., 1/8 inch). Gather the chips and place them in a sample container using forceps, a small scoop, or a dust pan. Make advance arrangements with the analytical laboratory prior to sampling to determine acceptable preparation and analysis procedures. The laboratory may require special grinding or extraction procedures.

Chip sampling is most often used to determine the necessity or effectiveness of decontamination, or the necessity for demolishing and disposing of a wall or building. As with wipe sampling, existing action levels may not be available for each application. Make appropriate decisions based on precedents and Regional guidelines.

Dust Sampling

Dust sampling is a method for collecting metal and semi-volatile contaminants in residue or dust found on porous or non-porous surfaces. Dust sampling techniques are used where a solvent cannot be used or where too much residue exists for a wipe sample to be easily collected. Dust sampling is used in industry to assess potential exposure of airborne contaminants to workers. For example, dust sampling would be effective in a bagging, processing, or grinding area where powdery contaminants and dust may have accumulated.

To collect a dust sample, select and sweep an appropriate area using a dedicated brush and dust pan. Transfer the sample to a sample container. Dust sampling can also be conducted using a cellulose fiber filter attached to a high-volume pump. Dust/residue is vacuumed onto the filter.

Dust sampling results are reported in mg/kg (weight/weight). The size of the area to be swept is dependent on the sample volume needed for the desired analysis and detection level. Dust sampling is often used to assess potential respiratory, direct contact, and ingestion hazards to workers and the public. It may also be used to determine the need and method for building decontamination.

4.3.7 Debris Sampling

The purpose of sampling waste debris is to select a disposal option. Since debris often consists of irregular pieces of material, it is a difficult matrix to

analyze in the laboratory. Provide the laboratory with instructions to guide it in preparing a representative subaliquot of debris samples for analysis.

Currently there are no standardized methods that reliably conserve VOCs during the grinding of large objects, nor are there good methods for extracting non-polar organic contaminants from plastic matrices without dissolving the plastic.

Use a judgmental sampling approach to sample debris, selecting sampling locations by matrix and physical properties. Use a chip sampling technique for porous materials and wipe sampling for non-porous materials. It is difficult to collect a representative sample of debris because of its heterogeneous composition. Compositing large objects will not result in meaningful data, and obtaining a sample of different components of debris is not always practical. Only if feasible, separate debris into components (e.g., metal, plastic, wood) and collect a representative surface sample of each.

4.3.8 Compressed Liquid/Gas Cylinders

Although dealing with compressed liquid/gas cylinders is outside the scope of this document, they are often found at waste sites. Compressed liquids and gases are stored in a variety of low- and high-pressure vessels or cylinders. Though the liquids or gases in the cylinders are rarely considered to be waste, the original cylinder may have been weakened by exposure to heat, pressure, or outside contamination. Cylinders represent a chemical, explosion/fire, and projectile hazard. Compressed liquids and gases, especially those in cylinders, should be sampled only by specialists.

4.4 SAMPLE PREPARATION

Sample preparation depends on the sampling objectives and analyses to be performed. Proper sample preparation and handling maintain sample integrity. Improper handling can render samples unsuitable for analysis. For example, homogenizing and compositing samples result in a loss of volatile constituents and are thus inappropriate when volatile contaminants are of concern. Sample preparation for waste may include, but is not limited to:

- Removing extraneous material
- Homogenizing
- Splitting
- Final preparation

Another field preparation technique is compositing of samples, which requires that each discrete aliquot be equal, and that the aliquots be thoroughly homogenized. Compositing waste samples is discussed in detail in Section 2.3.2.

4.4.1 Removing Extraneous Material

During sample collection, identify and discard materials from the sample which are not relevant or vital for characterizing the site, since their presence may introduce an error into the sampling or analytical procedures. Examples of extraneous material include pieces of glass, twigs, or leaves. However, not all external materials are extraneous. For example, when sampling at a junkyard, lead-contaminated battery casing pieces should not be removed from a sample if the casing comprises more than 10 percent of the sample volume. (For such a sample to be representative, it must incorporate the lead from the casing.) Collect samples of any material thought to be a potential source of contamination. Discuss any special analytical requirements for extraneous materials with the project team (project management, geologists, and chemists), and notify the laboratory of any special sample handling requirements or method changes.

4.4.2 Homogenizing

Homogenizing is the mixing or blending of a grab or composite sample to distribute contaminants uniformly within the sample. Ideally, proper homogenizing ensures that all portions of the sample are equal or identical in composition and are representative of the total sample collected. Incomplete homogenizing can introduce sampling error. Homogenizing requires additional handling of the waste and is not appropriate for all wastes. Unless layered, liquid wastes can be assumed to be homogeneous and do not require additional mixing. If they occur in phases, treat each phase as a unique homogeneous medium and sample each separately, as discussed in Section 2.3.1. Solid samples that will be composited should be homogenized after all aliquots have been combined. Manually homogenize solid and sludge samples using a stainless steel spoon or scoop

and a stainless steel bucket or pyrex bowl, or use a disposable plastic scoop and pan, depending on the analyses. Do not homogenize samples for VOC analysis.

4.4.3 Splitting

After collection and field preparation, samples are split into two or more equivalent parts when two or more portions of the same sample need to be analyzed separately. Split samples are most often collected in enforcement actions to compare sample results obtained by EPA with those obtained by the potentially responsible party. Split samples also provide measures of sample variability and analytical error. Before splitting, follow the homogenization techniques outlined above. Fill two sample collection jars at the same time, alternating spoonfuls (or scoopfuls) of homogenized sample between them. Samples for VOC analysis should not be homogenized; instead, collect two uniform samples concurrently from the same location (collocated).

4.4.4 Final Preparation

Select sample containers on the basis of compatibility with the material being sampled, resistance to breakage, and capacity. Appropriate sample volumes and containers will vary according to the parameters being analyzed. Actual sample volumes, appropriate containers, and holding times are specified in the U.S. EPA *Quality Assurance/Quality Control (QA/QC) Guidance for Removal Activities*, EPA/540/G-90/004, April 1990, in 40 CFR 136, and in the *Compendium of ERT Waste Sampling Procedures*, OSWER Directive 9360.4-07. Package all samples in compliance with current International Air Transport Association (IATA) or Department of Transportation (DOT) requirements, as applicable. Packaging should be performed by someone trained in current DOT shipping procedures.

Specific handling techniques may be required for physical parameters such as permeability or particle size distribution. Preservation of the original sample conditions will determine in part the representativeness of the analytical results. Permeability is affected by evaporation and by thermal variations; particle size is affected by handling. In general, cooling samples can help maintain original conditions; however, wastes are

often of such high concentration that cooling a sample is not necessary.

4.5 EXAMPLE SITE

Table 1 is a sample log for the ABC Plating site, illustrating the parameters used by the analytical laboratory to evaluate each waste source. The analytical results provided preliminary information on waste composition plus data necessary to begin clean-up strategy and treatment/disposal planning. The sampling objective of the initial assessment was to establish threat.

4.5.1 Source Sampling

The following is a detailed description of the sampling activities listed in Table 1.

Drum Sampling

Samples were collected from three drums tentatively identified from screening results as containing cyanide bases. They were sent for laboratory analysis of free and total cyanide, TCL organic compounds, and metals. Each sample was collected using a COLIWASA sampler which preserved phase layers that were present.

Vat Sampling

Samples were collected from two vats which were tentatively identified as containing cyanide bases. They were sent for laboratory analysis of free and total cyanide and metals. Vat samples were assumed to be relatively homogeneous and were collected using a COLIWASA-style glass thief.

Waste Pile Sampling

Two composite samples were collected (one from each waste pile) for laboratory analysis of free and total cyanide and metals. For each pile, four aliquots were collected from 12-inch depths at equally-spaced points located along the previously established transect. Sample aliquots were collected with a corkscrew auger and a hard plastic scoop. Aliquots from each pile were composited in separate disposable plastic trays.

Impoundment Sampling

Two waste liquid samples and five waste bottom sludge samples were collected from each impoundment. Waste liquids were analyzed in the laboratory for full TAL substances and the waste sludges were laboratory analyzed for full TCL substances. Sample locations and techniques were identical to those chosen for initial screening (Section 3.3.4). Waste liquid samples were collected using a bacon bomb sampler and waste sludge samples were collected with a Ponar dredge.

Surface Sampling

Non-porous walls and the concrete slab floor in the plating building were wipe and chip sampled, respectively. A one-square foot template was used to mark each area for wipe sampling. Sterile gauze pads soaked in hexane were used to collect four samples from non-porous walls. Four chip samples were collected from the floor using block hammers and chisels. Wipe and chip samples were analyzed for metals and cyanide to determine if the facility block walls and concrete floor needed to be sent to a secure or sanitary chemical landfill.

4.5.2 Sample Preparation

Removing Extraneous Material

Drum, vat, and impoundment liquid samples did not contain extraneous material. Stones and small pieces of stainless steel wire were removed from solid samples collected from the waste pile, but clumps of blue-green solid material were not removed. Based on screening data and knowledge of plating processes, the clumps were suspected to be plating solids containing high concentrations of metals and possibly cyanide. Sticks and other extraneous materials (e.g., plastic and metal objects) were discarded from dredged impoundment sludge samples. The presence of extraneous materials was documented for later consideration during treatment/disposal technology evaluation.

Homogenizing Samples

Homogeneity was assumed for most liquid samples, since plating processes require homogenous solutions to promote for even ion movement and uniformity of the coating. The liquid samples which appeared to be

uniform were not homogenized. Several drums containing liquid materials had distinct phases present which were visible in the glass thieves during sampling. Each phase was sampled separately using a COLIWASA. The solid waste piles and impoundment sludge samples were homogenized after screening results indicated a lack of volatile organic compounds. Since metals were a primary concern at the site, pyrex mixing bowls (instead of disposable aluminum pans) were used to homogenize samples.

Splitting Samples

At the request of the State, all initial containerized and impoundment waste samples were split during the removal assessment. The split samples were preserved and labelled, then chain of custody papers and samples were signed over to an on-site state representative.

Table 1: ABC Plating Sample Log

Sampling Locations	Number of Samples	Analytical Parameters
Plating Vats	2	metals, cyanide
Drums	3	metals, cyanide, full TCL substances
Waste Piles	2	metals, cyanide
Impoundment Liquids	4	full TAL substances
Impoundment Sludges	10	full TCL substances
Surfaces	8	metals, cyanide

5.0 QUALITY ASSURANCE/QUALITY CONTROL

5.1 INTRODUCTION

The goal of representative sampling is to obtain analytical results that accurately depict site conditions during a given time. The goal of quality assurance/quality control (QA/QC) is to implement correct methodologies which limit the introduction of error into the sampling and analytical procedures, and ultimately into the analytical data.

QA/QC samples evaluate three types of information: 1) the degree of site variation; 2) whether samples were cross-contaminated during sampling and sample handling procedures; and 3) whether a discrepancy in sample results is a result of laboratory handling and analysis procedures.

5.2 DATA CATEGORIES

EPA has established data quality objectives (DQOs) which ensure that the precision, accuracy, representativeness, and quality of environmental data are appropriate for their intended application. Superfund DQO guidance defines two broad categories of analytical data: *screening* and *definitive*.

Screening data are generated by rapid, less precise methods of analysis with less rigorous sample preparation. Sample preparation steps may be restricted to simple procedures such as dilution with a solvent, rather than elaborate extraction/digestion and cleanup. At least 10 percent of the screening data are confirmed using the analytical methods and QA/QC procedures and criteria associated with definitive data. Screening data without associated confirmation data are not considered to be data of known quality. To be acceptable, screening data must include the following: chain of custody, initial and continuing calibration, analyte identification, and analyte quantification. Streamlined QC requirements are the defining characteristic of screening data.

Definitive data are generated using rigorous analytical methods (e.g., approved EPA reference methods). These data are analyte-specific, with confirmation of analyte identity and concentration. Methods produce tangible raw data (e.g., chromatograms, spectra,

digital values) in the form of paper printouts or computer-generated electronic files. Data may be generated at the site or at an off-site location, as long as the QA/QC requirements are satisfied. For the data to be definitive, either analytical or total measurement error must be determined. QC measures for definitive data contain all of the elements associated with screening data, but also may include trip, method, and rinsate blanks; matrix spikes; performance evaluation samples; and replicate analyses for error determination.

For further information on these QA/QC objectives, please refer to EPA's *Quality Assurance/Quality Control Guidance for Removal Activities* or EPA's *Data Quality Objectives Process for Superfund*.

5.3 SOURCES OF ERROR

The four most common potential sources of data error in waste sampling are:

- Sampling design
- Sampling methodology
- Sample heterogeneity
- Analytical procedures

5.3.1 Sampling Design

Waste samples are often heterogeneous. Waste components separate into phases or layers by specific gravity and solubility. For example, an impoundment may have an oily layer on top and relatively contaminant-free water below. Failure to account for differences in composition of multiple phases can introduce sampling error. The sampling design must account for all phases and strata which may contain hazardous substances.

The sampling design should utilize approved SOPs and previously approved sampling designs to ensure uniformity and comparability between samples. The actual sample collection process should be determined prior to sampling. All samples should be collected using a uniform surface area and/or depth to ensure data comparability. Sampling equipment must be standardized for like sampling situations.

The sampling design should fulfill sampling and data quality objectives. The QA objectives selected should be built into the sampling design, including all necessary QA/QC samples.

5.3.2 Sampling Methodology

Sampling methodology and sample handling procedures have possible sources of error, including: cross-contamination from inappropriate use of sample collection equipment; unclean sample containers; improper sampling equipment decontamination; and improper shipment procedures. Procedures for collecting, handling, and shipping samples should be standardized to allow easier identification of any source(s) of error, and to minimize the potential for error. Use SOPs to ensure that all given sampling techniques are performed in the same manner, regardless of the individual sampling team, date, or location of sampling activity. Use field blanks, replicate samples, trip blanks, and rinsate blanks to identify errors due to improper sampling methodology and sample handling procedures.

Site screening and haz-cattng often employ kits or "cookbook" procedures requiring interpretations based on chemical reactions which produce a color change. The degree of subjectivity inherent in interpretation, and the complexity of some of the procedures, introduce a significant source of potential error.

5.3.3 Sample Heterogeneity

Wastes may become heterogeneous through vaporization, settling, solubility, migration, or addition of new wastes over time. Identify heterogeneity by obtaining several samples or composite aliquots from various depths.

Waste sources vary both in type and in concentration level. Incorporate representative sampling techniques into the sampling design to identify and define this variation accurately. Collect a grab sample of each phase or stratum suspected of containing contaminants of concern; the samples will be relatively homogeneous and representative of their respective phases. For example, if an impoundment has three liquid phases and sludge on the bottom, collect one sample of each liquid phase and a sample of the bottom sludge.

5.3.4 Analytical Procedures

Analytical procedures may introduce errors from: laboratory cross-contamination; inefficient extraction; and inappropriate methodology. High concentration waste samples tend to foul analytical equipment, which can lead to poor data reproducibility. Matrix spike, laboratory duplicate, performance evaluation, and laboratory control samples help to distinguish analytical error from sampling error.

5.4 QA/QC SAMPLES

QA/QC samples are collected at the site or prepared for or by the laboratory. Analysis of the QA/QC samples provides information on the variability and usability of waste sampling data, indicates possible field sampling or laboratory error, and provides a basis for future validation and usability of the analytical data. The most common field QA/QC samples are field replicate, background, and rinsate blank samples. The most common laboratory QA/QC samples are performance evaluation, matrix spike (MS), and matrix spike duplicate (MSD) samples. QA/QC results may suggest the need for modifying sample collection, preparation, handling, or analytical procedures if the resultant data do not meet site-specific quality assurance objectives.

Waste is typically characterized by high concentrations of contaminants, making precision and accuracy less important than for samples with lower concentrations (e.g., water, air). This eliminates the need for frequent field blanks. In addition, contaminant concentrations in waste samples are often several orders of magnitude higher than the concentrations of standard laboratory QA/QC mixes, which may render them useless in measuring laboratory error. The laboratory spikes are not detected because of masking caused by the high sample concentrations. Fouling of analytical equipment associated with high concentration samples may occur. Analytical error in waste sampling can be measured by performance evaluation samples and laboratory control samples, which are not subject to matrix interferences.

Refer to data validation procedures in U.S. EPA *Quality Assurance/Quality Control (QA/QC) Guidance for Removal Activities*, EPA/540/G-90/004, April 1990, for guidelines on utilizing QA/QC

analytical results. The following sections briefly describe the types of QA/QC samples appropriate for waste sampling.

5.4.1 Field Replicate Samples

Field replicates, also referred to as field duplicates and split samples, are field samples obtained from one sampling point, homogenized (where appropriate), divided into separate containers, and treated as separate samples throughout the remaining sample handling and analytical processes. Use replicate samples to assess error associated with sample heterogeneity, sample methodology, and analytical procedures. Field replicates can also be used when determining total error for critical samples with contamination concentrations near the action level. In such a case, a minimum of eight replicate samples is recommended for valid statistical analysis. Field replicates may be sent to two or more laboratories or to the same laboratory as unique samples. For total error determination, samples should be analyzed by the same laboratory.

5.4.2 Collocated Samples

A collocated sample is collected from an area adjoining a field sample to determine local variability of the waste. Collocated samples of solids, such as waste pile samples, are situated side by side. Collocated samples of liquids, such as vat samples, are collected from the same location and depth. Collocated samples are collected and analyzed as discrete samples; they are not composited. Because of the non-homogeneous nature of many waste sources, collocated samples should not be used to assess variability within a large source and are not recommended for assessing error. Determine the applicability of collocated samples on a site-by-site and source-by-source basis.

5.4.3 Background Samples

Waste sampling typically involves containerized or relatively immobile waste streams. Background sampling, which is appropriate when sampling soil, surface water, groundwater, and air, has less application to waste sampling. In some cases (e.g., uncontainerized waste) soil samples from uncontaminated areas can serve as background samples for waste sampling. Background samples are

appropriate for some surface sampling applications, as discussed in Section 4.3.6.

5.4.4 Performance Evaluation/ Laboratory Control Samples

A performance evaluation (PE) sample evaluates the overall error contributed by the analytical laboratory and detects any bias in the analytical method being used. PE samples contain known quantities of target analytes manufactured under strict quality control. They are usually prepared by a third party under an EPA certification program. The samples are usually submitted "blind" to analytical laboratories (the sampling team knows the contents of the samples, but the laboratory does not). Laboratory analytical error may be evaluated by the percent recoveries and correct identification of the components in the PE sample.

A blind PE sample may be included in a set of split samples provided to the PRP. The PE sample will measure PRP laboratory accuracy, which may be critical during enforcement litigation.

A laboratory control sample (LCS) also contains known quantities of target analytes in certified clean water. In this case, the laboratory knows the contents of the sample; the LCS is usually prepared by the laboratory. PE and LCS samples are not affected by waste matrix interference, and thus can provide a clear measure of laboratory error.

5.4.5 Matrix Spike/Matrix Spike Duplicate Samples

Matrix spike and matrix spike duplicate samples (MS/MSDs) are field samples that are spiked in the laboratory with a known concentration of a target analyte(s) in order to determine percent recoveries in sample extraction. The percent recovery from MS/MSDs indicates the degree to which matrix interferences will affect the identification of a substance. MS/MSDs can also be used to monitor laboratory performance. When four or more pairs of MS/MSDs are analyzed, the data obtained may also be used to evaluate error due to laboratory bias and precision. Analyze one MS/MSD pair to assess bias for every 20 samples, and use the average percent recovery for the pair. To assess precision, analyze at least 8 matrix spike replicates from the same sample, and determine the standard deviation and the

coefficient of variation. See pages 9-10 of the *U.S. EPA Quality Assurance/Quality Control (QA/QC) Guidance for Removal Activities*, April 1990, for directions on calculating analytical error. MS/MSDs are recommended for screening data and are required as one of several methods for determining analytical error for definitive data. Since the MS/MSDs are spiked field samples, provide sufficient volume for three separate analyses. Because the spiking solutions used in MS/MSDs are often obscured by high concentrations of contaminants or by matrix effects, the usefulness of MS/MSDs for high concentration samples may be limited.

5.4.6 Rinsate Blank Samples

A rinsate blank is used to assess cross-contamination from improper equipment decontamination procedures. Rinsate blanks are samples obtained by running analyte-free water over decontaminated sampling equipment. Any residual contamination should appear in the rinsate sample data. Analyze the rinsate blank for the same analytical parameters as the field samples collected that day. Handle and ship the rinsate like a low-concentration field sample. Where dedicated sampling equipment is not utilized, collect one rinsate blank per sampling batch per day.

5.4.7 Field Blank Samples

Field blanks are samples prepared in the field using certified clean water or sand which are then submitted to the laboratory for analysis. A field blank is used to evaluate contamination or error associated with sampling methodology, preservation, handling/shipping, and laboratory procedures. For high-concentration samples, the usefulness of field blanks is limited. Parts per billion (ppb) or low parts per million (ppm) error has little significance when identifying high concentration wastes or addressing action levels in the hundreds of ppm. If available and appropriate, submit one field blank per day.

5.4.8 Trip Blank Samples

Trip blanks are samples prepared prior to going into the field. They consist of certified clean water or sand, and are not opened until they reach the laboratory. If available, utilize trip blanks to meet QA objectives for volatile organic analyses only. Handle, transport, and analyze trip blanks in the same manner as the other volatile organic samples collected that

day. Trip blanks are used to evaluate error associated with sampling methodology, shipping and handling, and analytical procedures, since any volatile contamination of a trip blank would have to be introduced during one of those procedures. Since waste samples are often high concentration, trip blanks are not typically used during waste sampling.

5.4.9 Laboratory Duplicate Samples

A laboratory duplicate is a sample that undergoes preparation and analysis twice. The laboratory takes two aliquots of one sample and analyses them as separate samples. Comparison of data from the two analyses provides a measure of analytical reproducibility within a sample set. Discrepancies in duplicate analyses may indicate poor homogenization in the field or other sample preparation error, either in the field or in the laboratory. The benefit of laboratory duplicates in waste sampling may be limited. High concentration waste samples may foul analytical equipment and result in unavoidably poor reproducibility. Laboratory duplicates of high concentration waste samples should not be used to measure laboratory performance.

5.5 EVALUATION OF ANALYTICAL ERROR

Analytical error becomes significant in decision-making as sample results approach the action level. The acceptable level of error is determined by the intended use of the data and litigation concerns. Definitive data require quantitative measurement of analytical error with PE samples and replicates. The other QA samples identified in this section can indicate a variety of qualitative and quantitative sampling errors. As discussed earlier, error in the ppb or low ppm range may not be of concern when analyzing high concentration wastes.

5.6 CORRELATION BETWEEN FIELD SCREENING RESULTS AND LABORATORY RESULTS

A cost-effective approach for evaluating wastes and waste sources is to compare inexpensive field screening data and other field measurements (e.g., XRF) with laboratory results. This relies in part on

statistical correlation, which involves computing an index called the correlation coefficient (r) that indicates the degree and nature of the relationship between two or more sets of values. The correlation coefficient ranges from -1.0 (a perfect inverse or negative relationship), through 0 (no relationship), to +1.0 (a perfect direct or positive relationship). The square of the correlation coefficient, called the coefficient of determination, R^2 , is an estimate of the proportion of variance in one variable (the dependent variable) that can be accounted for by the independent variables. An acceptable R^2 value depends on the sampling objectives and intended data uses. As a rule, statistical relationships should have an R^2 value of at least 0.6 to determine a reliable model. For health or risk assessment purposes, the acceptable R^2 value may be more stringent (e.g., 0.6). Analytical calibration regressions have an R^2 value of 0.98 or greater. Once a reliable regression equation has been derived, the field screening data can be used to predict laboratory results. These predicted values can then be located on a base map and contoured (mapping methods are described in Section 6.4). The contour maps can illustrate the estimated extent of contamination (for certain waste sources) and the adequacy of the sampling program.

5.7 EXAMPLE SITE

5.7.1 QA Objectives

Screening data, which generate non-definitive, unconfirmed results (e.g., total hydrocarbons, total halogens, cyanide, PCBs) were used to select analytical parameters. Samples were sent to the analytical laboratory under protocols which provided definitive data. The rigorous laboratory analyses provided definitive identification and quantitation of contaminants (e.g., 50 ppm benzene, 110 ppm total chromium, 75 ppm total cyanide).

5.7.2 Sources of Error

All direct reading instruments were maintained and calibrated in accordance with their instruction manuals. Many of these instruments are class-specific (e.g. volatile organic vapors) with relative response rates that are dependent on the calibration gas selected. Instrument response to ambient vapor concentrations may differ by an order of magnitude from response to calibration standards. If compounds

of interest are known, site-specific standards may be prepared; they are most applicable for field gas chromatographs (GCs). These standards can be prepared on site in a gas bag or flask, but have limited holding times. Preparation of standards on site introduces its own potential error. For sites of long duration, specialty mixtures may be ordered from a specialty gas company or an analytical laboratory.

The number and location of initial field samples were based on observation and professional judgment (as outlined in Section 2.8). Liquid wastes in the vats, impoundments, and transformers were assumed to be homogeneous because there were no visible phases. (An erroneous observation could introduce significant error into the sampling design.)

Field standard operating procedures, documented in the site sampling plan, established consistent screening and sampling procedures among all samplers. This reduced the chances for variability and error during sampling. Site briefings were conducted prior to all sampling and screening events to review the use of proper screening and sampling techniques.

Other steps taken to limit error included proper sample preparation, adherence to sample holding times, and the use of proper shipment procedures. All off-site laboratory sample analyses were performed using EPA standard methods and protocols.

5.7.3 Field QA/QC Samples

Few field QA/QC samples were collected during waste sampling at the ABC Plating site. For the low-concentration impoundment liquids, a PE sample for metals was sent to the laboratory. (The PE sample is not affected by matrix interferences.) Field and trip blanks were not applicable since they are used to determine cross-contamination of low concentration samples. Cross-contamination that may occur during storage and shipping is minimal compared to the high ppm or percent level concentrations which are typically found in plating wastes. Nevertheless, suspected high concentration samples were shipped separately from the low level samples. One rinsate blank sample was collected from the impoundment sampling equipment (dredge and bacon bomb samplers) to check for cross-contamination during equipment decontamination.

5.7.4 Laboratory QA/QC Samples

Instructions on matrices, target compounds, and QA/QC criteria of particular interest were provided to the laboratory to help ensure that analytical results met the required objectives. The laboratory was instructed to run a duplicate of LCS samples for each batch of high concentration liquid vat samples to check reproducibility of the laboratory results. A matrix spike was not requested because the level of error measured by the standard low/medium spiking mixtures did not apply to the expected high concentrations in the samples. The laboratory analyzed the metals using the methods of inductively coupled plasma (ICP) spectrometry and atomic absorption (AA). The presence of cyanide was confirmed in the laboratory using total and amenable cyanide analyses (colorimetric manual method, SW-846 Method 9010).

PE sample results indicated low recoveries for some metals. The difference between LCS duplicate results was within the acceptable range, so these results were used as estimates with a low bias. The confirmation by a second method on 10 percent, or one per batch, of the high concentration samples indicated acceptable accuracy.

The waste pile was thought to be vat bottom materials of high contaminant concentrations, therefore a matrix spike was not requested. An LCS duplicate was used to evaluate the reproducibility of the results and to establish if the solid samples were homogeneous. Agreement between the LCS duplicates indicates good laboratory precision. When results of the LCS and LCS duplicate correlate, but the field replicates do not, two possible errors are indicated: either the matrix interfered with recovery, or there was poor sample homogenization in the field. The laboratory does not homogenize samples unless specified in the analytical method.

For the impoundment samples, matrix spike and LCS duplicate samples were used. Matrix spikes are applicable since the impoundment samples have a lower concentration than the vat samples. Matrix spike recoveries for certain metals were low. However, LCS results were within control limits, indicating good laboratory performance.

6.0 DATA PRESENTATION AND ANALYSIS

6.1 INTRODUCTION

Data presentation and analysis techniques are performed with analytical, field screening, or geophysical results. The techniques discussed below can be used to compare analytical values, to evaluate numerical distribution of data, and to reveal the location of "hot spots" and the extent of contamination at a site. The appropriate methods to present and analyze sample data depend on the sampling objectives, the number of samples collected, the sampling approaches used, and other considerations.

6.2 DATA POSTING

Data posting involves placement of sample values on a site base map or cross-section. Data posting is useful for displaying the distribution of sample values, visually depicting the location of wastes with associated assessment data. Data posting requires each sample to have a specific location (e.g., x , y , and sometimes z coordinates). Ideally, the sample coordinates are surveyed values or inventoried and numbered containers, facilitating placement on a scaled map. Data posting is useful for depicting concentration values of non-containerized wastes and surfaces, but has limited application to containerized wastes.

6.3 CROSS-SECTION/FENCE DIAGRAMS

Cross-section diagrams (two-dimensional) and fence diagrams (three-dimensional) depict layers or phases of wastes in sources such as tanks and impoundments. Two-dimensional cross-sections may be used to illustrate vertical profiles of waste concentrations in containerized wastes or impoundments. For solid wastes in waste piles, three-dimensional fence diagrams are often used to interpolate data between sampling locations. Solid wastes in waste piles do not usually form horizontal layers, so fence diagrams based on a few sampling points may not be representative. Both cross-sections and fence diagrams can provide useful visual interpretations of contaminant concentrations.

6.4 CONTOUR MAPPING

Contour maps are useful for depicting contaminant concentration values in waste piles or impoundments. Contour mapping requires an accurate, to-scale basemap of the site. After data posting sample values on the basemap, insert contour lines (or isopleths) at a specified contour interval, interpolating values between sample points. Contour lines can be drawn manually or can be generated by computer using contouring software. Although the software makes the contouring process easier, computer programs have a limitation: as they interpolate between data points, they attempt to "smooth" the values by fitting contour intervals to the full range of data values. This can result in a contour map that does not accurately represent general site contaminant trends. Typical waste sites have low concentration/non-detect areas and "hot spots." If there is a big difference in concentration between the waste "hot spot" and the surrounding area, the computer contouring program, using a contour interval that attempts to smooth the "hot spots," may eliminate most of the subtle site features and general trends. For waste sampling, contouring may apply only to large waste piles and impoundments.

6.5 STATISTICAL GRAPHICS

If using statistical interpretation, the distribution or spread of the data set is important in determining which statistical techniques to use. Common statistical analyses, such as the t-test, rely on normally distributed data. The histogram is a statistical bar graph which displays the distribution of a data set. A normally distributed data set takes the shape of a bell curve, with the mean and median close together about halfway between the maximum and minimum values. A probability plot depicts cumulative percent against the concentration of the contaminant of concern. A normally distributed data set, when plotted as a probability plot, would appear as a straight line. A histogram or probability plot can be used to see trends and anomalies in the data from a waste source (e.g., impoundment) prior to conducting more rigorous forms of statistical analysis. As with contour mapping, statistical data interpretation applications for waste are limited.

6.6 RECOMMENDED DATA INTERPRETATION METHODS

The data interpretation methods chosen depend on project-specific considerations, such as the number of sampling locations and their associated range in values. Data which are dissimilar in composition (e.g., drums with different chemicals or different waste media) should not be compared using statistical interpretation methods. Data posting, screening, and sampling data sheets, and cross-section/fence diagrams may be appropriate. A site feature depicting extremely low data values (e.g., non-detects), together with significantly higher values (e.g., 5000 ppm) from neighboring "hot spots" with little or no concentration gradient in between, does not lend itself to contouring.

6.7 EXAMPLE SITE

Figure 9 illustrates a transect of impoundment No.1 in a two-dimensional cross-section. The sampling intervals are indicated by the twenty foot markings along the transect of the cross-section. Analytical results were data posted on the cross-section to illustrate contaminant trends. Contaminant volume can be visualized by depicting both the sludge layer and impoundment bottom. The bottom sludges contained 300 to 427 ppm total chromium; other parameters exhibited a similar concentration gradient range.

Table 2 presents the haz-cattng results of all containerized waste and waste piles on site. This table was generated as the initial step in analyzing the data prior to posting on the base map and lists results from several different tests. These data were then posted on the base map.

Figure 9: Posted Total Chromium Data for Impoundment No. 1

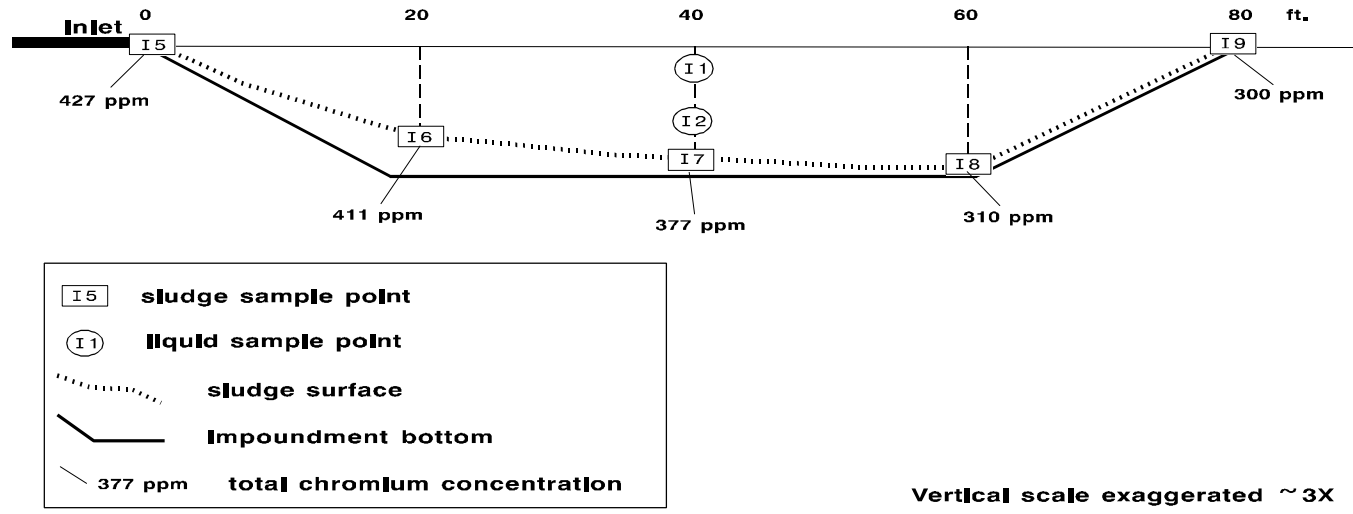


Table 2: Haz-Cat Results
 ABC Plating Site
 (page 1 of 2)

Container	Rad	pH	CN	PID	FID	Solubility in water	Chlorine Test	Peroxide Test	Phase	Cu ⁺	Comments
D1	ND	12	Y	N/A	N/A	Y	N	N	L	N	cyanide base
D2	ND	>12	Y	N/A	N/A	Y	N	N	L	N	cyanide base
D3	ND	>12	Y	N/A	N/A	Y	N	N	L	N	cyanide base
D4	ND	>12	N	N/A	N/A	Y	N	N	L	N	base
D5	ND	6	N/A	4	2	Y	N	N	L	N	inconclusive
D6	ND	7	N/A	18	35	N Floats	N	N	L	N	oil
D7	ND	7	N/A	26	70	N Floats	N	N	L	N	oil
D8	ND	7	N/A	14	39	N Floats	N	N	L	N	oil
D9	ND	7	N/A	45	128	N Sinks	N	N	L	N	halogenated solvent
D10	ND	N/A	N	18	26	Y	N	N	L	N	kerosene
D11	ND	5	N/A	6	11	Y	N	N	L	N	open; rain water?
D12	ND	5	N/A	8	6	Y	N	N	L	N	open; rain water?
D13	ND	5	N/A	ND	ND	Y	N	N	L	N	inconclusive
D14	ND	5	N/A	ND	ND	Y	N	N	L	N	inconclusive
D15	ND	<2	N/A	N/A	N/A	Reacts	N	N	L	N	strong acid
D16	ND	<2	N/A	N/A	N/A	Reacts	N	N	L	N	strong acid
D17	ND	<2	N/A	N/A	N/A	Reacts	N	N	L	N	strong acid
D18	ND	<2	N/A	N/A	N/A	Reacts	N	N	L	N	strong acid
D19	ND	>11	N	N/A	N/A	Y	N	N	S	N	caustic soda

Table 2: Haz-Cat Results (Cont'd)
 ABC Plating Site
 (page 2 of 2)

Container	Rad	pH	CN	PID	FID	Solubility in water	Chlorine Test	Peroxide Test	Phase	Cu ⁺	Comments
V1	ND	>12	Y	N/A	N/A	Y	N	N	L	N	cyanide base
V2	ND	>12	Y	N/A	N/A	Y	N	N	L	N	cyanide base
V3	ND	9	N	ND	ND	Y	N	N	L	N	inconclusive
V4	ND	2	N	N/A	N/A	Y	N	N	L	N	acid
V5	ND	2	N	N/A	N/A	Y	N	N	L	N	acid
V6	ND	8	N	ND	ND	Y	N	N	L	N	inconclusive
V7	ND	2	N	N/A	N/A	Y	N	N	L	N	acid
V8	ND	2	N	N/A	N/A	Y	N	N	L	N	acid
V9	ND	8	N	ND	ND	Y	N	N	L	N	inconclusive
V10	ND	<2	N	N/A	N/A	Reacts	N	N	L	Y	strong acid
V11	ND	<2	N	N/A	N/A	Reacts	N	N	L	Y	strong acid
V12	ND	8	N	ND	ND	Y	N	N	L	N	inconclusive
V13	ND	<2	N	N/A	N/A	Reacts	N	N	L	N	strong acid
V14	ND	<2	N	N/A	N/A	Reacts	N	N	L	N	strong acid
V15	ND	>12	N	N/A	N/A	Y	N	N	L	N	strong base
P1	ND	N/A	Y	ND	ND	N	N	N/A	S	N/A	inconclusive
P2	ND	N/A	Y	ND	ND	N	N	N/A	S	N/A	inconclusive

ND -- none detected
 NA -- not applicable

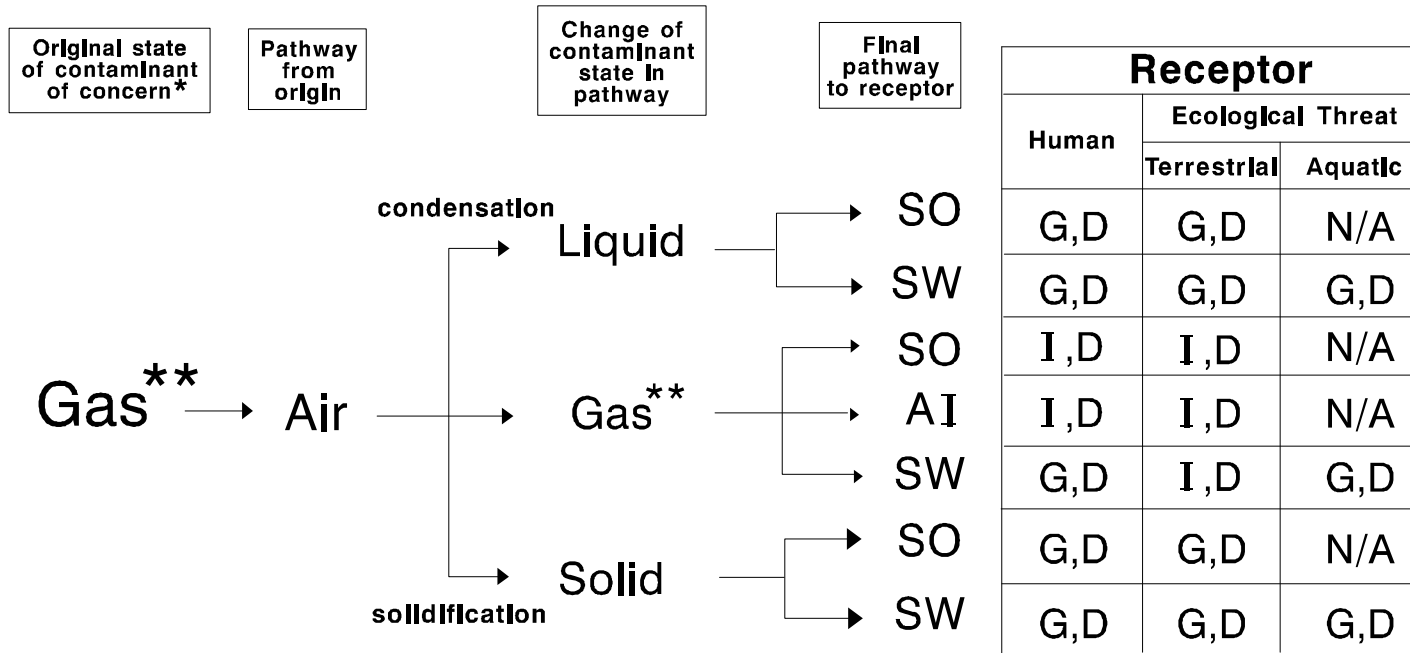
L -- liquid
 S -- solid

Y -- yes
 N -- no

APPENDIX A -- Example of Flow Diagram For Conceptual Site Model

Figure A-1

Migration Routes of a Gas Contaminant from Origin to Receptor



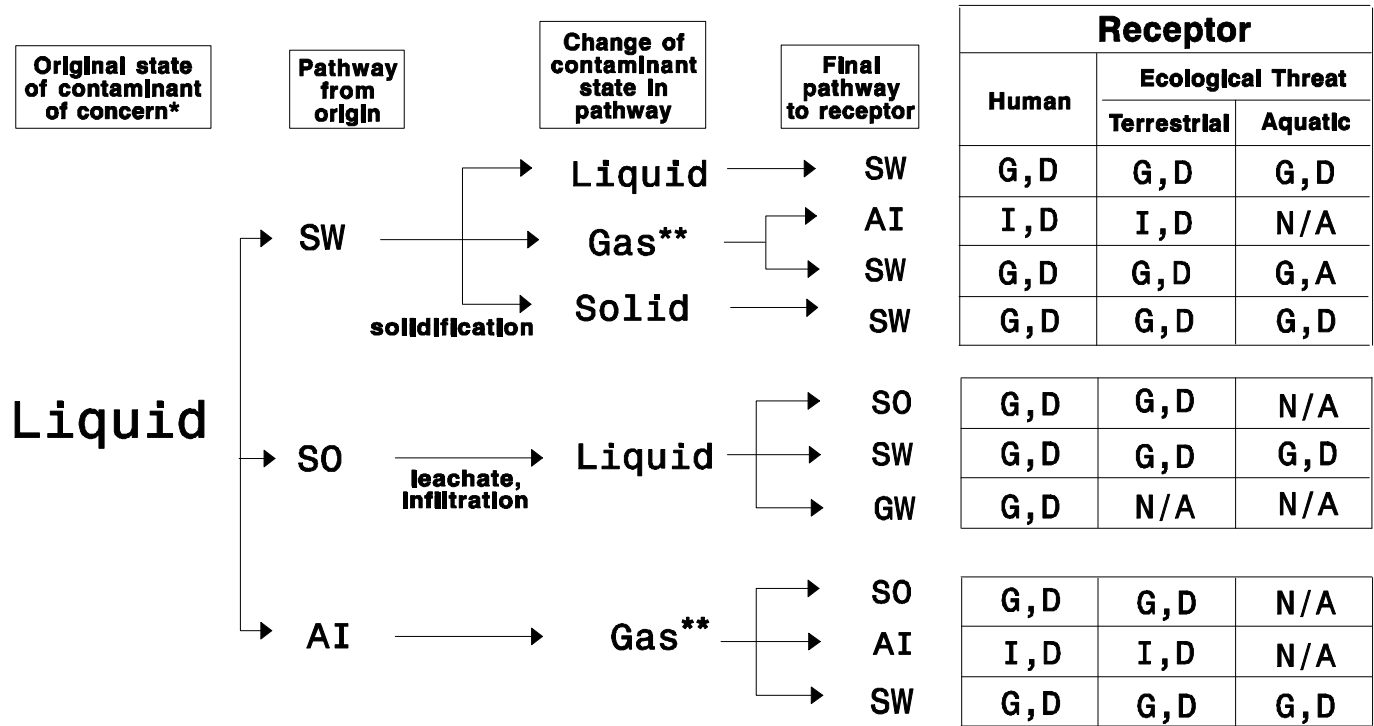
* May be a transformation product
 ** Includes vapors

Receptor Key
 D = Dermal Contact
 I = Inhalation
 G = Ingestion
 N/A = Not Applicable

Pathway Key
 AI = Air
 SO = Soil
 SW = Surface Water (Including sediments)
 GW = Ground Water

Figure A-2

Migration Routes of a Liquid Contaminant from Origin to Receptor



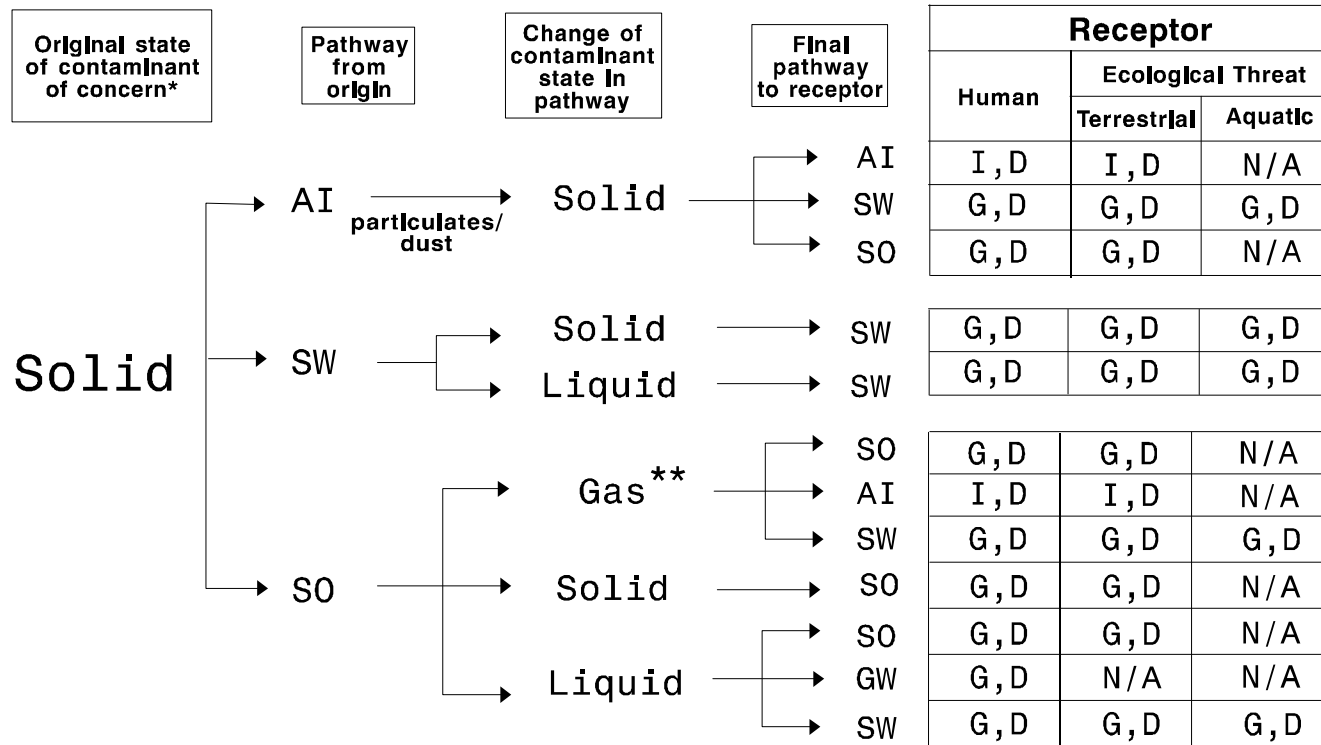
* May be a transformation product
 ** Includes vapors

Receptor Key
D = Dermal Contact
I = Inhalation
G = Ingestion
N/A = Not Applicable

Pathway Key
AI = Air
SO = Soil
SW = Surface Water (Including sediments)
GW = Ground Water

Figure A-3

Migration Routes of a Solid Contaminant from Origin to Receptor



* May be a transformation product

** Includes vapors

Receptor Key	
D	■ Dermal Contact
I	■ Inhalation
G	■ Ingestion
N/A	■ Not Applicable

Pathway Key	
AI	■ Air
SO	■ Soil
SW	■ Surface Water (Including sediments)
GW	■ Ground Water

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