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# SUPERFUND PROGRAM REPRESENTATIVE SAMPLING GUIDANCE

**VOLUME 1: SOIL** 

**Interim Final** 

Environmental Response Team

Office of Emergency and Remedial Response Office of Solid Waste and Emergency Response U.S. Environmental Protection Agency Washington, DC 20460

#### **Notice**

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For more information on Soil Sampling and Surface Geophysics procedures, refer to the *Compendium of ERT Soil Sampling and Surface Geophysics Procedures*, OSWER directive 9360.4-02, EPA/540/P-91/006. Topics covered in this compendium include Sampling Equipment Decontamination, Soil Sampling, Soil Gas Sampling, and General Surface Geophysics. The compendium describes procedures for collecting representative soil samples and provides a quick means of waste site evaluation. It also addresses the general procedures used to acquire surface geophysical data.

Questions, comments, and recommendations are welcomed regarding the Superfund Program Representative Sampling Guidance, Volume 1 -- Soil. Send remarks to:

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#### 1.0 INTRODUCTION

#### 1.1 OBJECTIVE AND SCOPE

This is the first 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. This document specifically addresses representative sampling for soil. The information presented here is valid throughout the Superfund program, but focuses on the objectives of early action activities and emergency responses. Topics covered in the document include: assessing available information; selecting an appropriate sampling approach; selecting and geophysical, analytical screening, and sampling equipment; utilizing proper sample preparation techniques; incorporating suitable types and numbers of Quality Assurance/Quality Control (QA/QC) samples; and interpreting and presenting the analytical and geophysical data.

In the Superfund program, representative sample data collected during emergency responses or early actions may form the basis of remedial response. Longer, more complex responses require a variety of sampling objectives, including identifying threat, delineating sources and extent of contamination, and confirming the achievement of clean-up standards. important and potentially costly decisions are based on the sampling data, making it very important that OSCs and field personnel understand how accurately the sampling data characterize the actual site conditions. In keeping with this strategy, this document emphasizes analytical screening and geophysical techniques as cost effective approaches to characterize the site and to select sampling locations.

# 1.2 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); drum dump; agricultural activities

Potential Exposure Pathway (Soil):

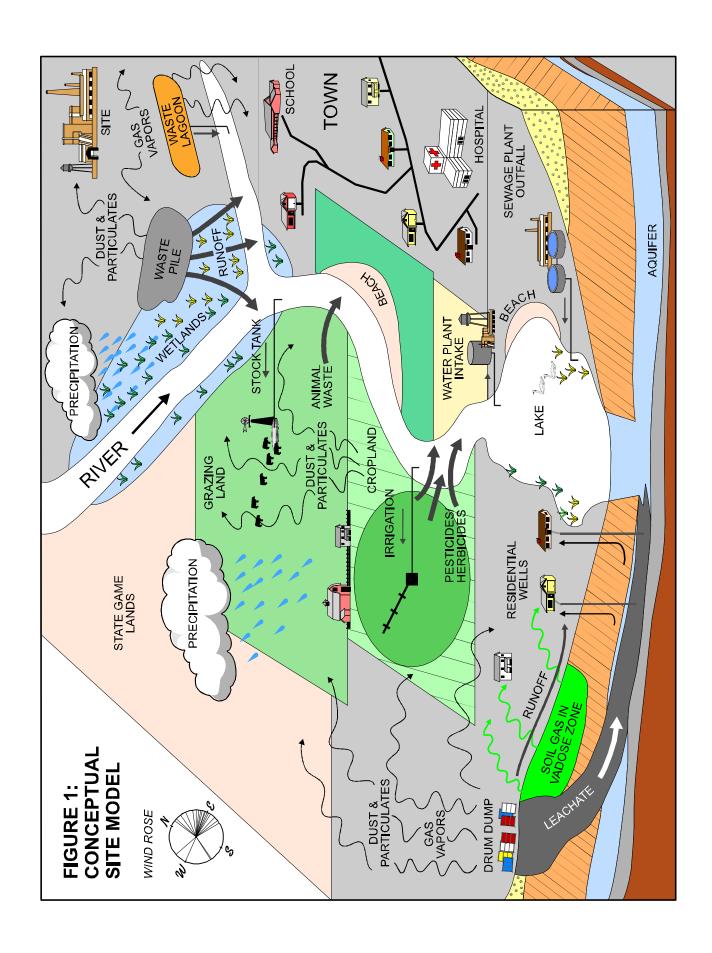
Leachate from the waste pile or drum dump; contaminated soil from direct contact with the waste pile or drum dump; agricultural activities such as pesticide application onto cropland

NOTE: Soil is described as an *exposure* pathway rather than a *migration* pathway because, unlike other media (e.g., air), contact between contaminated soil and a receptor is initiated by the receptor.

Potential Exposure Routes:

Ingestion -- Soil particles from the waste pile, drum dump or area of agricultural activity

Absorption/direct contact -- Soil near the waste pile, drum dump or area of agricultural activity



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

#### **Human Population**

#### Residents/Trespassers:

Leachate into soil from the drum dump; direct contact with soil contaminated by pesticides or other agricultural activities in the cropland

#### Workers/Trespassers:

Leachate into soil from the waste pile; contaminated soil associated with the waste pile or agricultural activities in the cropland

#### Biota

Endangered/threatened species or human food chain organisms, if suspected to be in contact with an area of potentially contaminated soil

Preliminary site information may provide the identification of the contaminant(s) of concern and the level(s) 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 offsite sampling locations.

# 1.3 REPRESENTATIVE SAMPLING OBJECTIVES

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

- 1. Establishing threat to public health or welfare or to the environment;
- 2. Locating and identifying potential sources of contamination;
- 3. Defining the extent of contamination;
- 4. Determining treatment and disposal options; and
- 5. Documenting the attainment of clean-up goals.

These objectives are discussed in detail in Section 2.5.

#### 1.4 REPRESENTATIVE SAMPLING

Representative soil sampling ensures that a sample or group of samples accurately reflects the concentration of the contaminant(s) of concern at a given time and location. Analytical results from representative samples reflect the variation in pollutant presence and concentration throughout a site.

This document concentrates on the variables that are introduced in the field -- namely, those that relate to the site-specific conditions, the sampling design approach, and the techniques for collection and preparation of samples. The following variables affect the representativeness of samples and subsequent measurements:

- Geological variability -- Regional and local variability in the mineralogy of rocks and soils, the buffering capacity of soils, lithologic permeability, and in the sorptive capacity of the vadose zone.
- Contaminant concentration variability --Variations in the contaminant concentrations throughout the site.
- Collection and preparation variability -Deviations in analytical results attributable to
  bias introduced during sample collection,
  preparation, and transportation (for analysis).
- Analytical variability -- Deviations in analytical results attributable to the manner in which the sample was stored, prepared, and analyzed by the on-site or off-site laboratory. Although analytical variability cannot be corrected through representative sampling, it can falsely lead to the conclusion that error is due to sample collection and handling procedures.

## 1.5 EXAMPLE SITE

An example site, presented at the end of each chapter, illustrates the development of a representative soil sampling plan that meets Superfund



Program objectives for early actions or emergency responses.

#### 2.0 SAMPLING DESIGN

#### 2.1 INTRODUCTION

The following procedures are recommended for developing a sound sampling design. Many steps can be performed simultaneously, and the sequence is not rigid.

- Review existing historical site information;
- Perform a site reconnaissance;
- Evaluate potential migration pathways and receptors;
- Determine the sampling objectives;
- Establish the data quality objectives;
- Utilize screening techniques;
- Select parameters for which to be analyzed;
- · Select an appropriate sampling approach; and
- Determine the locations to be sampled.

Real-time analytical screening techniques can be used throughout the removal action. The results can be used to modify the site sampling plan as the extent of contamination becomes known.

#### 2.2 HISTORICAL DATA REVIEW

Unless the site is considered a classic emergency, every effort should be made to first thoroughly review relevant site information. An historical data review examines past and present site operations and disposal practices, providing an overview of known and potential site contamination and other site hazards. Sources of information include federal, state and local officials and files (e.g., site inspection reports and legal actions), deed or title records, current and former facility employees, potentially responsible parties, local residents, and facility records or files. For any previous sampling efforts, obtain information regarding sample locations (on maps, if possible), matrices, methods of collection and analysis, and relevant contaminant concentrations. Assess the reliability and usefulness of existing analytical data. Even data which are not substantiated by documentation or QA/QC controls may still be useful. Collect information that describes any specific chemical processes used on site, as well as descriptions of raw materials used, products and wastes, and waste storage and disposal practices. Whenever possible, obtain site maps, facility blueprints, and historical aerial photographs, detailing past and present storage, process, and waste disposal locations. The local Agricultural Extension Agent, a Soil Conservation Service (SCS) representative, has information on soil types and drainage patterns. County property and tax records, and United States Geological Survey (USGS) topographic maps are also useful sources of site and regional information.

#### 2.3 SITE RECONNAISSANCE

A site reconnaissance, conducted either prior to or in conjunction with sampling, is invaluable to assess site conditions, to evaluate areas of potential contamination, to evaluate potential hazards associated with sampling, and to develop a sampling plan. During the reconnaissance, fill data gaps left from the historical review by:

- Interviewing local residents, and present or past employees about site-related activities;
- Researching facility files or records (where records are made accessible by owner/operator);
- Performing a site entry, utilizing appropriate personal protective equipment and instrumentation. Observe and photo-document the site; note site access routes; map process and waste disposal areas such as landfills, lagoons, and effluent pipes; inventory site wastes; and map potential transport routes such as ponds, streams, and irrigation ditches. Note topographic and structural features, dead animals and dead or stressed vegetation, potential safety hazards, and visible label information from drums, tanks, or other containers found on the site.

# 2.4 MIGRATION PATHWAYS AND RECEPTORS

The historical review and site visit are the initial steps in defining the source areas of contamination which could pose a threat to human health and the environment. This section addresses how to delineate the spread of contamination away from the source areas. Included are pollutant migration pathways and

the routes by which persons or the environment may be exposed to the on-site chemical wastes.

2.4.1 Migration Pathways and Transport Mechanisms

Migration pathways are routes by which contaminants have moved or may be moved away from a contamination source. Pollutant migration pathways may include man-made pathways, surface drainage, vadose zone transport, and wind dispersion. Human activity (such as foot or vehicular traffic) also transports contaminants away from a source area. These five transport mechanisms are described below.

- Man-made pathways -- A site located in an urban setting has the following man-made pathways which can aid contaminant migration: storm and sanitary sewers, drainage culverts, sumps and sedimentation basins, French drain systems, and underground utility lines.
- Surface drainage -- Contaminants can be adsorbed onto sediments, suspended independently in the water column, or dissolved in surface water runoff and be rapidly carried into drainage ditches, streams, rivers, ponds, lakes, and wetlands. Consider prior surface drainage routes; historical aerial photographs can be invaluable for delineation of past surface drainage patterns. An historical aerial photograph search can be requested through the EPA Regional Remote Sensing Coordinator.
- Vadose zone transport -- Vadose zone transport is
  the vertical or horizontal movement of water and
  of soluble and insoluble contaminants within the
  unsaturated zone of the soil profile.
  Contaminants from a surface source or a leaking
  underground storage tank can percolate through
  the vadose zone and be adsorbed onto subsurface
  soil or reach groundwater.
- Wind dispersion -- Contaminants deposited over or adsorbed onto soil may migrate from a waste site as airborne particulates. Depending on the particle-size distribution and associated settling rates, these particulates may be deposited downwind or remain suspended, resulting in contamination of surface soils and/or exposure of nearby populations.
- Human and animal activity -- Foot and vehicular traffic of facility workers, response personnel, and trespassers can move contaminants away from a source. Animal burrowing, grazing, and

migration can also contribute to contaminant migration.

### 2.4.2 Receptors

Once the migration pathways have been determined, identify all receptors (i.e., potentially affected human and environmental populations) along these pathways. Human receptors include on-site and nearby residents and workers. Note the attractiveness and accessibility of site wastes (including contaminated soil) to children and other nearby residents. Environmental receptors include Federal- or state-designated endangered or threatened species, habitats for these species, wetlands, and other Federal- and state-designated wilderness, critical, and natural areas.

### 2.5 SOIL REPRESENTATIVE SAMPLING OBJECTIVES

Collect samples if any of the following sampling objectives in the scope of the project are not fulfilled by existing data.

- 1. Establishing Threat to Public Health or Welfare or to the Environment -- The Comprehensive Environmental Response, Compensation and Liability Act of 1980 (CERCLA) and the National Contingency Plan (NCP) establish the funding mechanism and authority which allow the OSC to activate a Federal removal action. The OSC must establish (often with sampling) that the site poses a threat to public health or welfare or to the environment.
- Locating and Identifying Potential Sources of Contamination -- Sample to identify the locations and sources of contamination. Use the results to formulate removal priorities, containment and clean-up strategies, and cost projections.
- 3. Defining the Extent of Contamination -- Where appropriate, sample to assess horizontal and vertical extent of contaminant concentrations. Use the results to determine the site boundaries (i.e., extent of contamination), define clean areas, estimate volume of contaminated soil, establish a clearly defined removal approach, and assess removal costs and timeframe.
- 4. Determining Treatment and Disposal Options -- Sample to characterize soil for in situ or other onsite treatment, or excavation and off-site treatment or disposal.

Documenting the Attainment of Clean-up Goals

 During or following a site cleanup, sample to
 determine whether the goals were achieved, and
 to delineate areas requiring further treatment or
 excavation when appropriate.

#### 2.6 DATA QUALITY OBJECTIVES

Data quality objectives (DQOs) state the level of uncertainty that is acceptable from data collection activities. DQOs also define the data quality necessary to make a certain decision. Consider the following when establishing DQOs for a particular project:

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

In addition to these considerations, the quality assurance components of precision, accuracy (bias), completeness, representativeness, and comparability should also be considered. 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 component.
- Completeness -- percentage of sampling measurements which are judged to be valid.
- Representativeness -- degree to which sample data accurately and precisely represent the characteristics of the site contaminants and their concentrations.

• Comparability -- evaluation of the similarity of conditions (e.g., sample depth, sample homogeneity) under which separate sets of data are produced.

Quality assurance/quality control (QA/QC) objectives are discussed further in Chapter 5.

# 2.7 ANALYTICAL SCREENING AND GEOPHYSICAL TECHNIQUES

There are two primary types of analytical data which can be generated during sampling: laboratory analytical data and analytical screening data. Analytical screening techniques (e.g., using a photoionization detector (PID), portable X-ray fluorescence (XRF) unit, and hazard categorization kits) provide real-time or direct reading capabilities. These screening methods can narrow the possible groups or classes of chemicals for laboratory analysis and are effective and economical for gathering large amounts of site data. Once an area is identified using screening techniques, a subset of samples can be sent for laboratory analysis to substantiate the screening results. Under a limited sampling budget, analytical screening (with laboratory confirmation) will generally result in more analytical data from a site than will sampling for off-site laboratory analysis alone. To minimize the potential for false negatives (not detecting on-site contamination), use only those analytical screening methods which provide detection limits below applicable action levels. It should be noted, that some analytical screening methods which do not achieve detection limits below site action levels can still detect grossly contaminated areas, and can be useful for some sampling events.

Geophysical techniques may also be utilized during a removal action to help depict locations of any potential buried drums or tanks, buried waste, and disturbed areas. Geophysical techniques include ground penetrating radar (GPR), magnetometry, electromagnetic conductivity (EM) and resistivity surveys.

#### 2.8 PARAMETERS FOR ANALYSIS

If the historical data review yields little information about the types of waste on site, use applicable screening methods to narrow the parameters for analysis by ruling out the presence of high concentrations of certain contaminants. If the screening results are inconclusive, send a subset of samples from the areas of concern for a full chemical

characterization by an off-site laboratory. It is advised that samples from known or suspected source areas be sent to the laboratory for a full chemical characterization so that all contaminants of concern can be identified (even at low detection levels), and future sampling and analysis can then focus on those substances.

Away from source areas, select a limited number of indicator parameters (e.g., lead, PAHs) for analysis based on the suspected contaminants of concern. This will result in significant cost savings over a full chemical characterization of each sample. Utilize EPA-approved methodologies and sample preparation, where possible, for all requested off-site laboratory analyses.

#### 2.9 SAMPLING APPROACHES

Selecting sampling locations for screening or laboratory analysis entails choosing the most appropriate sampling approach. Representative sampling approaches include judgmental, random, stratified random, systematic grid, systematic random, search, and transect sampling. A representative sampling plan may combine two or more of these approaches. Each approach is defined below.

#### 2.9.1 Judgmental Sampling

Judgmental sampling is the subjective selection of sampling locations at a site, based on historical information, visual inspection, and on best professional judgment of the sampling team. Use judgmental sampling to identify the contaminants present at areas having the highest concentrations (i.e., worst-case conditions). Judgmental sampling has no randomization associated with the sampling strategy, precluding any statistical interpretation of the sampling results.

### 2.9.2 Random Sampling

Random sampling is the arbitrary collection of samples within defined boundaries of the area of concern. Choose random sample locations using a

random selection procedure (e.g., using a random number table). Refer to U.S. EPA, 1984a, for a random number table. The arbitrary selection of sampling points requires each sampling point to be selected independent of the location of all other points, and results in all locations within the area of concern having an equal chance of being selected. Randomization is necessary in order to make probability or confidence statements about the sampling results. The key to interpreting these probability statements is the assumption that the site 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 at the site. Because hazardous waste sites are very rarely homogeneous, other statistical sampling approaches (discussed below) provide ways to subdivide the site into more homogeneous areas. These sampling approaches may be more appropriate for removal activities than random sampling. Refer to U.S. EPA, February 1989, pages 5-3 to 5-5 for guidelines on selecting sample coordinates for random sampling. Figure 2 illustrates a random sampling approach.

### 2.9.3 Stratified Random Sampling

Stratified random sampling often relies on historical information and prior analytical results (or screening data) to divide the sampling area into smaller areas called strata. Each strata is more homogeneous than the site is as a whole. Strata can be defined based on various factors, including: sampling depth, contaminant concentration levels, and contaminant source areas. Place sample locations within each of these strata using random selection procedures. Stratified random sampling imparts some control upon the sampling scheme but still allows for random sampling within each stratum. Different sampling approaches may also be selected to address the different strata at the site. Stratified random sampling is a useful and flexible design for estimating the pollutant concentration within each depth interval or area of concern. Figure 3 illustrates a stratified random sampling approach where strata are defined based on depth. In this example, soil coring devices are used to collect samples from given depths at randomly selected locations within the strata.

Figure 2: Random Sampling

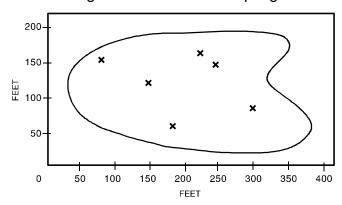


Figure 3: Stratified Random Sampling

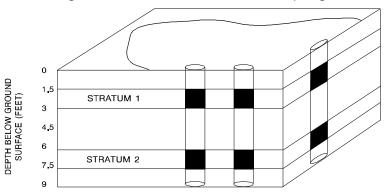
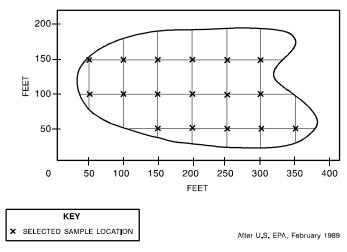


Figure 4: Systematic Grid Sampling



### 2.9.4 Systematic Grid Sampling

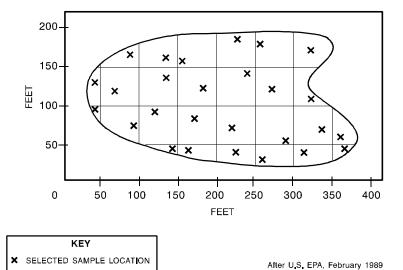
Systematic grid sampling involves subdividing the area of concern by using a square or triangular grid and collecting samples from the nodes (intersections of the grid lines). Select the origin and direction for placement of the grid using an initial random point. From that point, construct a coordinate axis and grid over the whole site. The distance between sampling locations in the systematic grid is determined by the size of the area to be sampled and the number of samples to be collected.

Systematic grid sampling is often used to delineate the extent of contamination and to define contaminant concentration gradients. Refer to U.S. EPA February 1989, pages 5-5 to 5-12, for guidelines on selection of sample coordinates for systematic grid sampling. Figure 4 illustrates a systematic grid sampling approach.

### 2.9.5 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 described in Section 2.9.4) 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. Figure 5 illustrates a systematic random sampling approach.

Figure 5: Systematic Random Sampling



#### 2.9.6 Search Sampling

Search sampling utilizes either a systematic grid or systematic random sampling approach to search for areas where contaminants exceed applicable clean-up standards (hot spots). The number of samples and the grid spacing are determined on the basis of the acceptable level of error (i.e., the chance of missing a hot spot). Search sampling requires that assumptions be made about the size, shape, and depth of the hot spots. As illustrated in Figure 6, the smaller and/or narrower the hot spots are, the smaller the grid spacing must be in order to locate them. Also, the smaller the acceptable error of missing hot spots is, the smaller the grid spacing must be. This, in effect, means collecting more samples.

Once grid spacing has been selected, the probability of locating a hot spot can be determined. Using a systematic grid approach, Table 1 lists approximate probabilities of missing an elliptical hot spot based on the grid method chosen as well as the dimensions of the hot spot. The lengths of the long and short axes (L and S) are represented as a percentage of the grid The triangular grid method spacing chosen. consistently shows lower probabilities of missing a hot spot in comparison to the block grid method. Table 1 can be used in two ways. If the acceptable probability of missing a hot spot is known, then the size of the hot spot which can be located at that probability level can be determined. Conversely, if the approximate size of the hot spot is known, the probability of locating it can be determined.

For example, suppose the block grid method is chosen with a grid spacing of 25 feet. The OSC is willing to accept a 10% chance of missing an elliptical hot spot. Using Table 1, there would be a 90% probability of locating an elliptical hot spot with L equal to 90% of the grid spacing chosen and S equal to 40% of the grid spacing chosen. Therefore the smallest elliptical hot spot which can be located would have a long axis  $L=0.90\times25 {\rm ft.}=22.5~{\rm ft.}$  and a short axis  $S=0.40\times25 {\rm ft.}=10~{\rm ft.}$ 

Similarly, if the approximate size of the hot spot being searched for is known, then the probability of missing that hot spot can be determined. For example, if a triangular grid method was chosen with a 25 foot grid spacing and the approximate shape of the hot spot is known, and L is approximately 15 feet or 60% of the grid spacing, and S is approximately 10 feet or 40% of the grid spacing, then there is approximately a 15% chance of missing a hot spot of this size and shape.

### 2.9.7 Transect Sampling

Transect sampling involves establishing one or more transect lines across the surface of a site. 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 sim-

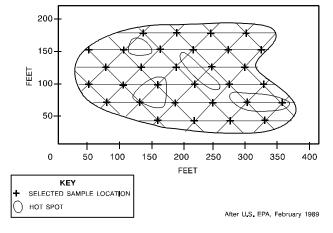
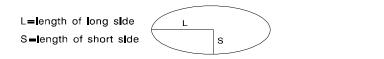


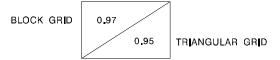
Figure 6: Search Sampling

# Table 1: Probability of Missing an Elliptical Hot Spot LENGTH OF SHORT AXIS AS A PERCENTAGE OF GRID SPACING

5 <u>N</u>		10%	20%	30%	40%	50%	60%	70%	80%	90%	100%
LENGTH OF LONG AXIS AS A PERCENTAGE OF GRID SPACING	10%	0.97									
	20%	0.95	0.88								
	30%	0.92	0.83	0.72							
	40%	0.88	0.75	0.65	0.50						
	50%	0.85	0.69	0.54	0.38	0.21					
	60%	0.80	0.62	0.45	0.27	0.12	0.06				
	70%	0.77	0.56	0.38	0.18	0.07	0.03	0.0			
	80%	0.75	0.54	0.32	0.12	0.05	0.0	0.0	0.0		
	90%	0.72	0.51	0.30	0.10	0.03	0.0	0.0	0.0	0.0	
	100%	0.70	0.45	0.24 0.18	0.08	0.01	0.0	0.0	0.0	0.0	0.0

From tables in Gilbert, 1987





ilar to systematic grid sampling. A primary benefit of transect sampling over systematic grid sampling is the ease of establishing and relocating individual transect lines versus an entire grid. Transect sampling is often used to delineate the extent of contamination and to define contaminant concentration gradients. It is also used, to a lesser extent, in compositing sampling schemes. For example, a transect sampling approach might be used to characterize a linear feature such as a drainage ditch. A transect line is run down the center of the ditch, along its full length. Sample aliquots are collected at regular intervals along the transect line and are then composited. Figure 7 illustrates transect sampling.

Table 2 summarizes the various representative sampling approaches and ranks the approaches from most to least suitable, based on the sampling objective. Table 2 is intended to provide general guidelines, but it cannot cover all site-specific conditions encountered.

#### 2.10 SAMPLING LOCATIONS

Once a sampling approach has been selected, the next step is to select sampling locations. For statistical (non-judgmental) sampling, careful placement of each sampling point is important to achieve representativeness.

Factors such as the difficulty in collecting a sample at a given point, the presence of vegetation, or discoloration of the soil could bias a statistical sampling plan.

Sampling points may be located with a variety of methods. A relatively simple method for locating

random points consists of using either a compass and a measuring tape, or pacing, to locate samplingpoints with respect to a permanent landmark, such as a survey marker. Then plot sampling coordinates on a map and mark the actual sampling points for future reference. Where the sampling design demands a greater degree of precision, locate each sample point by means of a survey. After sample collection, mark each sample point with a permanent stake so that the survey team can identify all the locations.

#### 2.11 EXAMPLE SITE

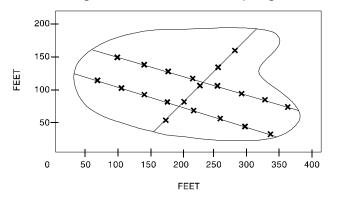
# 2.11.1 Background Information



The ABC Plating Site is located in Carroll County, Pennsylvania,

approximately 1.5 miles north of the town of Jonesville (Figure 8). The site covers approximately 4 acres, and operated as an electroplating facility from 1947 to 1982. During its years of operation, the company plated automobile and airplane parts with chromium, nickel, and copper. Cyanide solutions were used in the plating process. ABC Plating deposited electroplating wastes into two shallow surface settling lagoons in the northwest sector of the site. The county environmental health department was attempting to enforce cleanup by the site owner, when, in early 1982, a fire on site destroyed most of the process building. The owner then abandoned the facility and could not be located by enforcement and legal authorities. The county contacted EPA for an assessment of the site for a possible response.





KEY

★ SELECTED SAMPLE LOCATION

After U.S. EPA, February 1989

Table 2: Representative Sampling Approach Comparison

#### SAMPLING APPROACH

SAMPLING OBJECTIVE	JUDGEMENTAL	RANDOM	STRATIFIED RANDOM	SYSTEMATIC GRID	SYSTEMATIC RANDOM	SEARCH	TRANSECT
ESTABL <b>I</b> SH THREAT	1	4	3	2 ª	3	3	2
IDENTIFY SOURCES	1	4	2	2 ª	3	2	3
DELINEATE EXTENT OF CONTAMINATION	4	3	3	1 <sup>b</sup>	1	1	1
EVALUATE TREATMENT & DISPOSAL OPTIONS	3	3	1	2	2	4	2
CONFIRM CLEANUP	4	1 <sup>C</sup>	3	<b>1</b> b	1	1	<b>1</b> d

- 1 PREFERRED APPROACH
- 2 ACCEPTABLE APPROACH
- 3 MODERATELY ACCEPTABLE APPROACH
- 4 LEAST ACCEPTABLE APPROACH
- a SHOULD BE USED WITH FIELD ANALYTICAL SCREENING
- b PREFERRED ONLY WHERE KNOWN TRENDS ARE PRESENT
- c ALLOWS FOR STATISTICAL SUPPOFT OF CLEANUP VERIFICATION IF SAMPLING OVER ENTIRE SITE
- d MAY BE EFFECTIVE WITH COMPOSTING TECHNIQUE IF SITE IS PRESUMED TO BE CLEAN

# 2.11.2 Historical Data Review and Site Reconnaissance

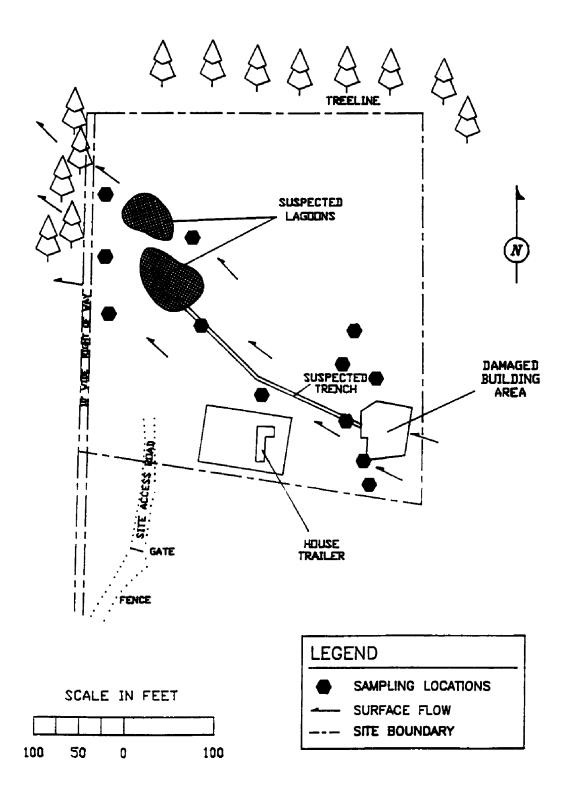
The EPA On-Scene Coordinator (OSC) reviewed the county site file, finding that in 1974, the owner was cited for violating the Clean Streams Act and for storing and treating industrial waste without a permit. The owner was ordered to file a site closure plan and to remediate the storage lagoons. The owner, however, continued operations and was then ordered to begin remediation in 90 days or be issued a cease and desist order. Soon after, a follow-up inspection revealed that the lagoons had been backfilled without removing the waste.

The OSC and response contractor arrived on site to interview local officials, fire department officers, neighboring residents (including a past facility employee), and county representatives, regarding site

operating practices and other site details. A past employee sketched facility process features on a map which was obtained from the county (Figure 8). The features included two settling lagoons and a feeder trench which transported plating wastes from the process building to the lagoons. The OSC obtained copies of aerial photographs of the site area from the district office of the U.S. Soil Conservation Service. The county also provided the OSC with copies of all historical site and violation reports.

The OSC and response contractor made a site entry utilizing appropriate personal protective equipment and instrumentation. They observed 12 vats, likely containing plating solutions, on a concrete pad where the original facility building once stood.

Figure 8: Site Sketch and Phase I Soil Sampling Locations ABC Plating Site



Measurements of pH ranged from 1 to 11. In addition, 50 drums and numerous smaller containers (some on the concrete pad, others sitting directly on the ground) were leaking and bulging, due to the fire. The response contractor noted many areas of stained soil, which indicated container leakage, poor waste handling practices, and possible illegal dumping of wastes.

# 2.11.3 Identification of Migration Pathways, Transport Mechanisms and Receptors

During the site entry, the OSC noted that several areas were devoid of vegetation, threatening wind erosion which could transport heavy metal- and cyanide-contaminated soil particulates off site. These particulates could be deposited on residential property downwind or be inhaled by nearby residents.

Erosion gullies located on site indicated soil erosion and fluvial transport due to storms. Surface drainage sloped towards the northwest. The response contractor observed stressed and discolored vegetation immediately off site, along the surface drainage route. Surface drainage of heavy metals and cyanide was a direct contact hazard to local residents. Further downgradient, runoff enters an intermittent tributary of Little Creek. Little Creek in turn feeds Barker Reservoir, the primary water supply for the City of Jonesville and neighboring communities, which are located 2.5 miles downgradient of the site. The site entry team observed that the site was not secure and there were signs of trespass (confirming a neighbor's claim that children play at the facility). These activities could lead to direct contact with cvanide and heavy metal contaminants, in addition to the potential for chemical burns from direct contact with strong acids and bases.

Information obtained from the historical data review and site reconnaissance was used to create a site-specific conceptual model. Sources (e.g., vats, drums), pathways (e.g., gullies) and potential receptors (e.g., local residents) were detailed on a map to assist the selection of sampling approaches, objectives, and locations.

### 2.11.4 Sampling Objectives

The OSC selected three specific sampling objectives, as follows:

- Phase 1 -- Determine whether a threat to public health, welfare, and the environment exists.
   Identify sources of contamination to support an immediate CERCLA-funded activation for containment of contaminants and security fencing.
- Phase 2 -- Define the extent of contamination at the site and adjacent residential properties. Estimate the volume of contaminated soil and the associated removal costs.
- Phase 3 -- After excavation (or treatment), document the attainment of clean-up goals. Assess that cleanup was completed to the selected level.

# 2.11.5 Selection of Sampling Approaches

The OSC selected a judgmental sampling approach for Phase 1. Judgmental sampling supports the Action Memorandum process by best defining on site contaminants in the worst-case scenario in order to evaluate the threat to human health, welfare, and the environment. Threat is typically established using a relatively small number of samples (less than 20) collected from source areas, or suspected contaminated areas based on the historical data review and site reconnaissance. For this site, containerized wastes were screened to categorize the contents and to establish a worst- case waste volume, while soil samples were collected to demonstrate whether a release had already occurred.

For Phase 2, a stratified systematic grid design was selected to define the extent of contamination. The grid can accommodate analytical screening and geophysical surveys and allow for contaminated soil excavation on a cell-by-cell basis. Based on search sampling conducted at similar sites, the hot spots being searched for were assumed to be elliptical in shape and 45 feet by 20 feet in size. Under these assumptions, a block grid, with a 50 foot grid spacing, was selected. This grid size ensured a no more than 10% probability of missing a hot spot (see Table 1). The grid was extended to adjacent residential properties when contaminated soil was identified at grid points near the boundary of the site.

Phase 3 utilized a systematic grid sampling approach to confirm the attainment of clean-up goals. Following cleanup, analytical screening was conducted on excavated soil areas using a transportable X-ray fluorescence (XRF) unit mounted in a trailer (mobile laboratory instrument). Based on the results, each area was documented as clean, or was excavated to additional depth, as necessary.

# 2.11.6 Analytical Screening, Geophysical Techniques, and Sampling Locations

During Phase 1 operations, containerized wastes were screened using hazard categorization techniques to identify the presence of acids, bases, oxidizers, and flammable substances. Following this procedure, photoionization detector (PID) and flame ionization detector (FID) instruments, a radiation meter, and a cyanide monitor were used to detect the presence of volatile organic compounds, radioactive substances, and cyanide, respectively, in the containerized wastes. Phase 1 screening indicated the presence of strong acids and bases and the absence of volatile organic compounds. The response contractor collected a total of 12 surface soil samples (0-3 inches) during this phase and sent them to a laboratory for analysis. The soil sampling locations included stained soil areas, erosion channels and soil adjacent to leaking containers. Background samples were not collected during Phase 1 because they were unnecessary for activating funding. Phase 1 sampling locations are shown in Figure 8. Based on Phase 1 analytical results, consultation with a Regional EPA toxicologist and with the Agency for Toxic Substances and Disease Registry (ATSDR), an action level of 100 ppm for chromium was selected for cleanup.

During Phase 2 sampling activities, the OSC used a transportable XRF unit installed in an on-site trailer to screen samples for total chromium in order to limit the number of samples to be sent for off-site laboratory analysis. The transportable XRF (rather than a portable unit) was selected for analytical screening to accommodate the 100 ppm action level for chromium. Sampling was performed at all grid nodes at the surface (0-4 inches) and subsurface (36-40 inches) (Figure 9). The 36-40 inch depth was selected based on information obtained from county reports and local interviews which indicated the lagoon wastes were approximately 3 feet below ground surface. The samples were homogenized and sieved (discussed in Chapter 4), then screened for chromium using the XRF. The surface and subsurface samples from areas downgradient of the original facility (21 grid nodes) and three upgradient (background) locations were sent for off-site laboratory analysis following XRF

screening. The analytical results from these samples allowed for site-specific calibration of the XRF unit. Once grid nodes with a contamination level greater than the selected action level were located, composite samples were collected from each adjoining cell. Surface aliquots were collected and then composited, sieved, thoroughly homogenized, and screened using the XRF to pinpoint contaminated cells. Additionally, four subsurface aliquots were collected at the same locations as the surface aliquots. They were also composited, sieved, thoroughly homogenized, and screened using the XRF. Figure 10 illustrates a Phase 2 sampling grid cell diagram. Based on the XRF data, each adjoining cell was either identified as clean (below action level), or designated for excavation (at or above action level).

For Phase 3 sampling, cleanup was confirmed by collecting and compositing four aliquots from the surface of each grid cell excavated during Phase 2. The surface composites were then screened (as in Phase 2), using the transportable XRF. Ten percent of the screened samples were also sent to an off-site laboratory for confirmatory sampling. Based on the Phase 3 screening and sampling results, each cell was documented as clean, or, excavated to additional depth, as necessary.

During Phase 2, the OSC conducted ground penetrating radar (GPR) and electromagnetic conductivity (EM) geophysical surveys to help delineate the buried trench and lagoon areas along with any other waste burial areas. The GPR survey was run along the north-south grid axis across the suspected locations of the trench and lagoons. Several structural discontinuities, defining possible disturbed areas, were detected. One anomaly corresponded with the suspected location and orientation of the feeder trench. Several discontinuities were identified in the suspected lagoon areas; however, the data did not conclusively pinpoint precise locations. This could be due to a disturbance of that area during the backfilling process by the PRP. The GPR survey is illustrated in Figure 11.

Figure 9: Soil Sampling and SRF Screening Locations ABC Plating Site

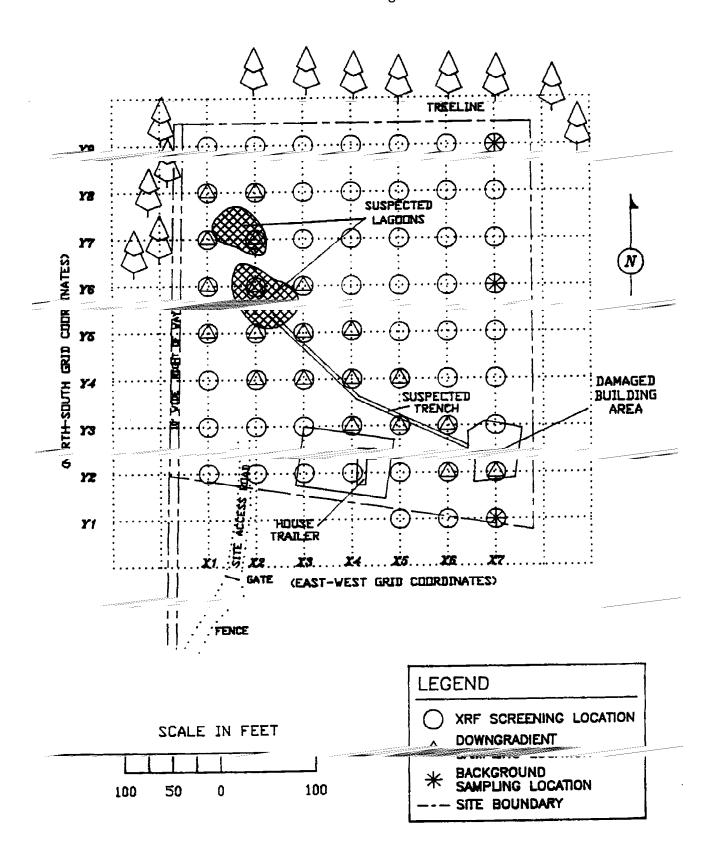
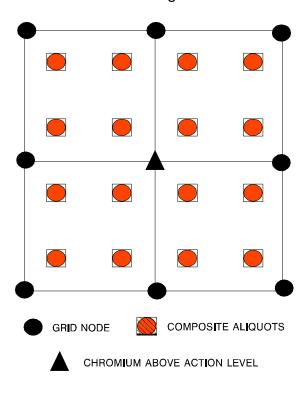


Figure 10: Phase 2 Sampling Grid Cell Diagram\*



\* Surface samples should be taken over a minimum area of one square foot. Sampling areas for depth sampling are limited by the diameter of the sampling equipment (e.g., auger, split spoon, or coring devices).

For the comprehensive EM survey, the original 50 foot grid spacing was decreased to 25 feet along the northsouth grid axis. The EM survey was run along the northsouth axes and readings were obtained at the established grid nodes. The EM survey was utilized throughout the site to detect the presence of buried metal objects (e.g., buried pipe leading to the lagoons), and potential subsurface contaminant plumes. The EM survey identified several high conductivity anomalies: the suspected feeder trench location, part of the lagoon area, and a small area west of the process building (Figure 12), which could have been an illegal waste dumping area. Several areas of interference were encountered due to the presence of large metal objects at the surface (a dumpster, surface vats and a junk car).

# 2.11.7 Parameters for Analysis

During Phase 1 sampling activities, full priority pollutant metals and total cyanide analyses were conducted on all samples. Since Phase 1 samples were collected from the areas of highest suspected contaminant concentration (i.e., sources and drainage pathways), Phase 2 samples were run for total chromium and cyanide, the only analytes detected during the Phase 1 analyses. During Phase 3, the samples sent to the laboratory for definitive analysis were analyzed for total chromium and cyanide. Throughout the removal, it was not possible to screen soils on site for cyanide, therefore the OSC requested laboratory cyanide analysis on the 10% confirmatory samples.

Figure 11: GPR Survey Results ABC Plating Site

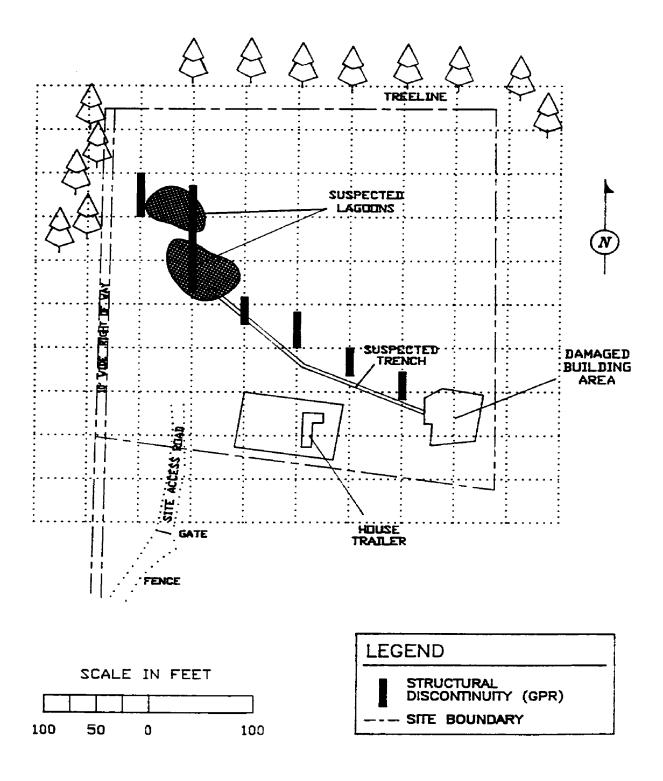
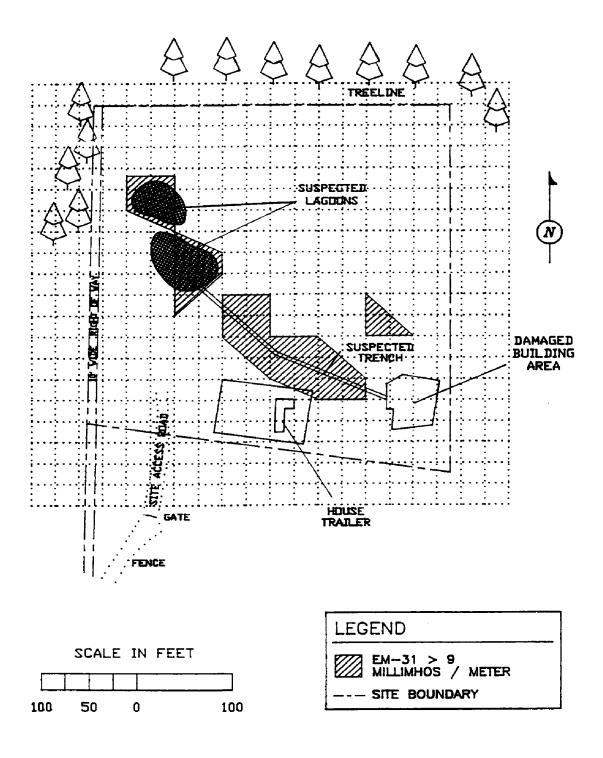


Figure 12: EM-31 Survey Results ABC Plating Site



#### 3.0 EQUIPMENT

#### 3.1 INTRODUCTION

Sample collection requires an understanding of the capabilities of the sampling equipment, since using inappropriate equipment may result in biased samples. This chapter provides information for selecting sampling and screening equipment.

# 3.2 ANALYTICAL SCREENING EQUIPMENT

Analytical screening methods provide on-site measurements of contaminants of concern, limiting the number of samples which need to be sent to an off-site laboratory for time-consuming and often costly analysis. Screening techniques can also evaluate soil samples for indications that soil contamination exists (e.g., X-ray fluorescence (XRF) for target metals or soil gas survey for identification of buried wastes or other subsurface contamination). All screening equipment and methods described in this section are **portable** (the equipment is hand-held, and generally no external power is necessary). Examples are photoionization detectors (PID), flame ionization detectors (FID), and some XRF devices.

Screening generally provides analytical data of suitable quality for site characterization, monitoring during response activities, and on-site health and safety decisions. The methods presented here can provide rapid, cost-effective, real-time data; however, results are often not compound-specific and not quantitative.

When selecting one screening method over another, consider relative cost, sample analysis time, potential interferences or instrument limitations, detection limit, QA/QC requirements, level of training required for operation, equipment availability, and data bias. Also consider which elements, compounds, or classes of compounds the screening instrument is designed to analyze. As discussed in Section 2.7, the screening method selected should be sensitive enough to minimize the potential for false negatives. When collecting samples for on-site analysis (e.g., XRF), evaluate the detection limits and bias of the screening method by sending a minimum of 10% of the samples to an off-site laboratory for confirmation. Table 3 summarizes the advantages and disadvantages of selected portable screening equipment.

#### 3.3 GEOPHYSICAL EQUIPMENT

Geophysical techniques can be used in conjunction with analytical screening to help delineate areas of subsurface contamination, including buried drums and tanks. Geophysical data can be obtained relatively rapidly, often without disturbing the site. Geophysical techniques suitable for emergency or removal activities include: ground penetrating radar (GPR), magnetometry, electromagnetic conductivity (EM) and resistivity. Specific advantages and disadvantages associated with geophysical equipment are summarized in Table 4. See also EPA ERT Standard Operating Procedure (SOP) #2159, General Surface Geophysics (U.S. EPA, January 1991).

# 3.4 SELECTING SAMPLING EQUIPMENT

The mechanical method by which a sampling tool collects the sample may impact representativeness. For example, if the sampling objective is to determine the concentrations of contaminants at each soil horizon interface, using a hand auger would be inappropriate: the augering technique would disrupt and mix soil horizons, making the precise horizon interface difficult to determine. Depth of sampling is another factor to consider in the proper selection of sampling equipment. A trowel, for example, is suitable for unconsolidated surface soils, but may be a poor choice for sampling at 12 inches, due to changes in soil consistency with depth.

All sampling devices should be of sufficient quality not to contribute contamination to samples (e.g., painted surfaces which could chip off into the sample). In addition, the sampling equipment should be either easily decontaminated, or cost-effective if considered to be expendable. Consider ease of use when selecting sampling equipment.

Table 3: Portable Field Analytical Screening Equipment

<u>Equipment</u>	Application to Sampling Design	Advantages and Disadvantages
X-ray fluorescence (portable)	Detects heavy metals in soils	Rapid sample analysis; may be used in situ; requires trained operator; potential matrix interferences; may be used with a generic or site-specific calibration model; detection limit may exceed action level; detects to ppm level; detection limit should be calculated on a site-specific basis.
Flame ionization detector (FID)	Semi-quantitatively detects VOCs in soils	Immediate results; can be used in GC mode to identity specific organic compounds; detects VOCs only; detects to ppm level.
Photoionization detector (PID)	Detects total concentration of VOCs and some non- volativle organics and inorganics in soils	Immediate results; easy to use; non-compound specific; results affected by high ambient humidity and electrical sources such as radios; does not respond to methane; detects to ppm level.
Field test kits	Detects specific elements, compounds, or compound classes in soils	Rapid results; easy to use; low cost; limited number of kit types available; kits may be customized to user needs; semi-quantitative; interferences by other analytes is common; colorimetric interpretation is needed; detection level dependent upon type of kit used; can be prone to error.
Radiation detector	Detects the presence of selected forms of radiation in soils or other waste materials	Easy to use; low cost; probes for one or a combination of alpha, beta or gamma forms of radiation; unit and detection limits vary greatly; detailed site surveys are time intensive and require experienced personnel to interpret results.

Sources: U.S. EPA, September 1988a; U.S. EPA, December 1987; U.S. EPA 1987.

Table 4: Geophysical Equipment

<u>Equipment</u>	Application to Sampling Design	Advantages and Disadvantages
Ground penetrating radar (GPR)	Detects reflection anomalies caused by lithology changes buried objects; varying depths of investigation, 15 to 30 feet, are possible.	Capable of high resolution; generates continuous measurement profile; can survey large area quickly; site specific; best results are achieved in dry, sandy soils; clay-rich and water saturated soils produce poor reflections and limit depth of penetration; data interpretation requires a trained geophysicist.
Magnetometer	Detects presence and areal extent of ferromagnetic material in subsurface soils, including buried metal containers. Single 55-gallon drums can be identified at depths up to 10 feet and large massed of drums up to 30 feet or more.	Quick and easy to operate; good initial survey instrument; readings are often affected by nearby man-made steel structures (including above-ground fences, buildings, and vehicles); data interpretation may require geophysicist.
Electromagnetic conductivity meter (EM)	Detects electrical conductivity changes in subsurface geologic lithology, pore fluids, and buried objects. Depth of investigation varies from 9 feet to 180 feet depending on instrument used, coil spacing, and coil configuration.	Rapid data collection; can delineate inorganic and large-scale organic contamination in subsurface fluids; sensitive to man-made structures (including buried cables, above-ground steel structures and electrical power lines); survey planning and data interpretation may require geophysicist.
Wadi	Detects electrical conductivity changes in surface and sub-surface materials utilizing existing very low frequency (VLF) radio waves.	Utilizes existing long-distance communication VLF radio waves (10-30 Khz range); no need to induce electrical field; directional problems can be overcome with portable transmitters.
Resistivity meter	Detects electrical resistivity variations in subsurface materials (e.g., lithology, pore fluids, buried pipelines and drums). Vertical resolution to depths of 100 feet are possible.	Detects lateral and vertical variations; instrument requires direct ground contact, making it relatively labor intensive; sensitive to outside interference; data interpretation requires a trained geophysicist.

Sources: Benson, et. al. 1988; NJDEP, 1988.

Complicated sampling procedures usually require increased training and introduce a greater likelihood of procedural errors. Standard operating procedures help to avoid such errors. Sample volume is another selection concern. Specific advantages and disadvantages of soil sampling equipment are given in Table 5. Refer also to EPA ERT SOP #2012, Soil Sampling (in U.S. EPA, January 1991) for guidance on using various types of soil sampling equipment.

#### 3.5 EXAMPLE SITE

# 3.5.1 Selection of Sampling Equipment



Dedicated plastic scoops were used for Phase 1 soil sampling. For Phase 2, the OSC used bucket augers for both surface and subsurface soil sampling because of their ease of use, good vertical depth range, and uniform surface sampling volume. Standard operating procedures were followed to promote proper sample collection, handling, and decontamination. From the bucket auger, each sample was placed into a dedicated plastic pan and mixed using a dedicated plastic scoop. Samples were further prepared for XRF screening and laboratory analysis (Section 4.8).

# 3.5.2 Selection of Analytical Screening Equipment

Phase 1 sampling identified the sources and types of on-site contaminants in order to establish a threat. Hazard categorization techniques, organic vapor detecting instruments, and radiation and cyanide monitors were utilized to tentatively identify containerized liquid wastestreams in order to select initial judgmental soil sampling locations. During Phase 2 sampling, a portable XRF unit was used to determine the extent of contamination and to identify additional hot spots. Samples to be sent for laboratory analysis were then placed into sampling jars (as discussed in Section 4.8). Samples collected from upgradient grid nodes for XRF screening only were stored on site for later treatment/disposal. For Phase 3, the XRF was used to confirm whether contaminated areas identified during Phase 2 were sufficiently excavated.

# 3.5.3 Selection of Geophysical Equipment

The GPR instrument delineated buried trench and lagoon boundaries. The EM meter detected subsurface conductivity changes due to buried metal containers and contaminants. The EM-31 (a shallower-surveying instrument than the EM-34) was selected because expected contaminant depth was less than 10 feet and because of the instrument's maneuverability and ease of use.

Table 5: Soil Sampling Equipment

<u>Equipment</u>	Applicability	Advantages and Disadvantages
Trier	Soft surface soil	Inexpensive; easy to use and decontaminate; difficult to use in stony, dry, or sandy soil.
Scoop or trowel	Soft surface soil	Inexpensive, easy to use and decontaminate; trowels with painted surfaces should be avoided.
Tulip bulb planter	Soft soil, 0-6 in.	Easy to use and decontaminate; uniform diameter and sample volume; preserves soil core (suitable for VOA and undisturbed sample collection); limited depth capability; not useful for hard soils.
Soil coring device	Soft soil, 0-24 in.	Relatively easy to use; preserves soil core (suitable for VOA and undisturbed sample collection); limited depth capability; can be difficult to decontaminate.
Thin-wall tube sampler	Soft soil, 0-10 ft.	Easy to use; preserves soil core (suitable for VOA and undisturbed sample collection); may be used in conjunction with bucket auger; acetate sleeve may be used to help maintain integrity of VOA samples, easy to decontaminate; can be difficult to remove cores from sampler.
Split spoon sampler	Soil, 0 inbedrock	Excellent depth range; preserves soil core (suitable for VOA and undisturbed sample collection); acetate sleeve may be used to help maintain integrity of VOA samples; useful for hard soils; often used in conjunction with drill rig for obtaining deep cores.
Shelby tube sampler	Soft soil, 0 inbedrock	Excellent depth range; preserves soil core (suitable for VOA and undisturbed sample collection); tube may be used to ship sample to lab undisturbed; may be used in conjunction with drill rig for obtaining deep cores and for permeability testing; not durable in rocky soils.
Bucket auger	Soft soil, 3 in10 ft.	Easy to use; good depth range; uniform diameter and sample volume; acetate sleeve may be used to help maintain integrity of VOA samples; may disrupt and mix soil horizons greater than 6 inches in thickness.
Hand-operated power auger	Soil, 6 in15 ft.	Good depth range; generally used in conjunction with bucket auger for sample collection; destroys soil core (unsuitable for VOA and undisturbed sample collection); requires 2 or more equipment operators; can be difficult to decontaminate; requires gasoline-powered engine (potential for cross-contamination).

Sources: NJDEP, 1988; U.S. EPA, January 1991.

#### 4.0 SAMPLE COLLECTION AND PREPARATION

#### 4.1 INTRODUCTION

In addition to sampling equipment, sample collection includes sample quantity and sample volume. Sample preparation refers to all aspects of sample handling after collection, until the sample is received by the laboratory. Sample preparation for soils may include, but is not limited to:

- removing extraneous material;
- sieving samples;
- homogenizing samples;
- splitting samples;
- compositing samples; and
- final preparation.

Sample preparation depends on the sampling objectives and analyses to be performed. Proper sample preparation and handling help to maintain sample integrity. Improper handling can result in a sample becoming unsuitable for the type of analysis required. For example, homogenizing, sieving, and compositing samples all result in a loss of volatile constituents and are therefore inappropriate when volatile contaminants are the concern.

#### 4.2 SAMPLE COLLECTION

How a sample is collected can affect its representativeness. The greater the number of samples collected from a site and the larger the volume of each sample, the more representative the analytical results will be. However, sampling activities are often limited by sampling budgets and project schedules. The following sections provide guidelines on appropriate sample numbers and volumes.

#### 4.2.1 Sample Number

The number of samples needed will vary according to the particular sampling approach that is being used. For example, in grid sampling, one sample is generally collected at each grid node, regardless of grid size. As discussed in Section 2.11.6, once contaminated grid node samples are located, adjoining grid cells can be sampled more thoroughly to define areas of contamination. Four aliquots from each grid cell, situated equidistant from the sides of each cell and each other (as illustrated in Figure 10), are recommended for grid cells measuring up to 100 x 100 feet. One additional aliquot may be collected from the

center of each cell, making a total of five aliquots per cell. For grid sizes greater than 100 feet x 100 feet, nine aliquots, situated equidistant from the sides of each cell and each other (as illustrated in Figure 13), are recommended. Depending on budget and other considerations, grid cell aliquots can be analyzed as separate samples or composited into one or more samples per cell.

#### 4.2.2 Sample Volume

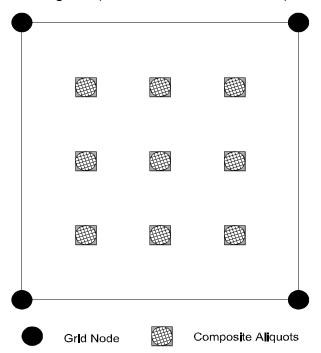
Both sample depth and area are considerations in determining appropriate sample volume. Depending on the analytes being investigated, samples are collected at the surface (0-3 in.), extended surface (0-6 in.), and/or at one-foot depth intervals. Nonwater soluble contaminants such as dioxin and PCBs are often encountered within the first six inches of soil. Water-soluble contaminants such as metals, acids, ketones, and alcohols will be encountered at deeper depths in most soils except clays. Contaminants in solution, such as PCPs in diesel fuel and pesticides in solvents, can penetrate to great depths (e.g., down to bedrock), depending on soil type.

For surface samples, collect soil over a surface area of one square foot per sample. A square cardboard template measuring 12 in. x 12 in., or a round template with a 12 in. diameter can be used to mark sampling areas. For subsurface samples, one of several coring devices may be used (see Table 5). Using a coring device results in a smaller diameter sampling area than a surface template, and therefore somewhat lessens the representativeness of the sample.

# 4.3 REMOVING EXTRANEOUS MATERIAL

Identify and discard materials in a sample which are not relevant or vital for characterizing the sample or the site, since their presence may introduce an error in the sampling or analytical procedures. Examples of extraneous material in soil samples include pieces glass, twigs or leaves. However, not all non-soil material is extraneous. For example, when sampling at a junkyard, lead-contaminated battery casing pieces should not be removed from a sample if the casing composes more than 10% of the sample composition. For a sample to be representative, it must also incorporate the lead from the casing. Collect samples

Figure 13: Phase 2 Sampling Grid Cell Diagram (Grid Sizes > 100 x 100 ft.)



of any material thought to be a potential source of contamination for a laboratory extraction procedure. Discuss any special analytical requirements for extraneous materials with project management, geologists, and chemists and notify the laboratory of any special sample handling requirements.

#### 4.4 SIEVING SAMPLES

Sieving is the process of physically sorting a sample to obtain uniform particle sizes, using sieve screens of predetermined size. For example, the sampler may wish to sieve a certain number of samples to determine if particle size is related to contaminant distribution. Sieving is generally only conducted when preparing soil samples for XRF screening. For this purpose, a 20-mesh screen size is recommended.

Be aware of the intent of the sampling episode, when deciding whether to sieve a sample prior to analysis. Prior to sieving, samples may need to be oven-dried. Discarding non-soil or non-sieved materials, as well as the sieving process itself, can result in physical and chemical losses. Sieving is not recommended where volatile compounds are of concern. Analyze the discarded materials, or a fraction thereof, to determine their contribution to the contamination of the site being investigated.

#### 4.5 HOMOGENIZING SAMPLES

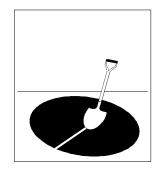
Homogenization is the mixing or blending of a soil sample in an attempt to provide uniform distribution of contaminants. (Do not homogenize samples for volatile compound analysis). Ideally, proper homogenization ensures that portions of the containerized samples are equal or identical in composition and are representative of the total soil sample collected. Incomplete homogenization will increase sampling error. All samples to be composited or split should be homogenized after all aliquots have been combined. Manually homogenize samples using a stainless steel spoon or scoop and a stainless steel bucket, or use a disposable scoop and pan. Quarter and split the sample as illustrated in Figure 14, repeating each step a minimum of 5 times until the sample is visually homogenized. Samples can also be homogenized using a mechanicallyoperated stirring device as depicted in ASTM standard D422-63.

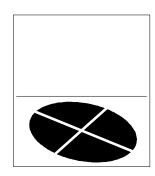
#### 4.6 SPLITTING SAMPLES

Splitting samples after collection and field preparation into two or more equivalent parts is performed 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 (PRP). Split samples also provide a measure of the sample variability, and a measure of the analytical and extraction errors. Before splitting, follow homogenization techniques outlined above. Fill two sample collection jars simultaneously with alternate spoonfuls (or scoopfuls) of homogenized sample. To simultaneously homogenize and split a sample, quarter (as illustrated in Figure 14) or **mechanically** split the sample using a riffle sample splitter. The latter two techniques are described in detail in ASTM Standard C702-87.

Figure 14: Quartering to Homogenize and Split Samples







#### Step 1:

- Cone sample on hard, clean surface
- Mix by forming new cone

#### Step 2:

- Flatten cone
- Divide sample into quarters

Step 3: (not shown)

- Remix opposite quarters
- · Reform cone
- Repeat a minimum of 5 times

#### 4.7 COMPOSITING SAMPLES

Compositing is the process of physically combining and homogenizing several individual soil aliquots. Compositing samples provides an average concentration of contaminants over a certain number of sampling points, which reduces both the number of required lab analyses and the sample variability. Compositing can be a useful technique, but must always be implemented with caution. Compositing is not recommended where volatile compounds are of concern.

Specify the method of selecting the aliquots that are composited and the compositing factor in the sampling plan. The compositing factor is the number of aliquots to be composited into one sample (e.g., 3 to 1; 10 to 1). Determine this factor by evaluating detection limits for parameters of interest and comparing them with the selected action level for that parameter. Compositing also requires that each discrete aliquot be the same in terms of volume or

weight, and that the aliquots be thoroughly homogenized. Since compositing dilutes high concentration aliquots, the applicable detection limits should be reduced 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 (e.g., if the action level for a particular substance is 50 ppb, an action level of 10 ppb should be used when analyzing a 5-aliquot composite). The detection level need not be reduced if the composite area is assumed to be homogeneous in concentration (for example, stack emission plume deposits of particulate contamination across an area, or roadside spraying of waste oils).

### 4.8 FINAL PREPARATION

Select sample containers on the basis of compatibility with the material being sampled, resistance to breakage, and volume. For soil sampling, use widemouth glass containers with Teflon-lined lids. Appropriate sample volumes and containers will vary according to the parameter being analyzed. Keep low and medium concentration soil samples to be analyzed for organic constituents at 4EC. Actual sample volumes, appropriate containers, and holding times are specified in the QA/QC Guidance for Removal Activities (U.S. EPA, April 1990), in 40 CFR 136, and in the Compendium of ERT Soil Sampling and Surface Geophysics (U.S. EPA, January 1991). Package all samples in compliance with Department of Transportation (DOT) or International Air Transport Association (IATA) requirements.

It is sometimes possible to ship samples to the laboratory directly in the sampling equipment. For example, the ends of a Shelby tube can be sealed with caps, taped, and sent to the laboratory for analysis. To help maintain the integrity of VOA samples, collect soil cores using acetate sleeves and send the sleeves to the laboratory. To ensure the integrity of the sample after delivery to the laboratory, make laboratory sample preparation procedures part of all laboratory bid contracts.

### 4.9 EXAMPLE SITE

After placing each sample in a dedicated pan and mixing (as discussed in Section 3.5.1), plant matter, stones, and broken glass were removed. Soil samples were oven-dried (at 104E C) and sieved using a 20-mesh screen in preparation for XRF analysis.



Samples were then homogenized and split using the quartering technique. Opposite quarters were remixed and quartering was repeated five times to ensure thorough homogenization. A portion of each sample was placed into XRF analysis cups for screening. The remainder of each sample was placed into 8-ounce, wide-mouth glass jars with Teflon-lined lids and sent to a laboratory for inorganic analysis. The samples were packaged in compliance with IATA requirements. Chain-of-custody paperwork was prepared for the samples. Laboratory paperwork was completed as appropriate and the samples were shipped to the predesignated laboratories for analysis.

### 5.0 QUALITY ASSURANCE/QUALITY CONTROL EVALUATION

### 5.1 INTRODUCTION

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

QA/QC samples evaluate the degree of site variation, whether samples were cross-contaminated during sampling and sample handling procedures, or if a discrepancy in sample results is due to laboratory handling and analysis procedures. The QA/QC sample results are used to assess the quality of the analytical results of waste and environmental samples collected from a site.

### 5.2 DATA CATEGORIES

EPA has established a process of 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

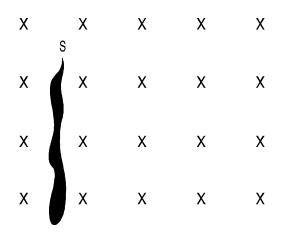
Identifying and quantifying the error or variation in sampling and laboratory analysis can be difficult. However, it is important to limit their effect(s) on the data. Four potential sources of error are:

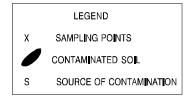
- sampling design;
- sampling methodology;
- sample heterogeneity; and
- analytical procedures.

### 5.3.1 Sampling Design

Site variation includes the variation both in the types and in the concentration levels of contaminants throughout a site. Representative sampling should accurately identify and define this variation. However, error can be introduced by the selection of a sampling design which "misses" site variation. For example, a sampling grid with relatively large distances between sampling points or a biased sampling approach (i.e., judgmental sampling) may allow significant contaminant trends to go unidentified, as illustrated in Figure 15.

Figure 15: Sampling Error Due to Sampling Design





### 5.3.2 Sampling Methodology

Error can be introduced by the sampling methodology and sample handling procedures, as in crosscontamination from inappropriate use of sample collection equipment, unclean sample containers, improper sampling equipment decontamination and shipment procedures, and other factors. Standardized procedures for collecting, handling, and shipping samples allow for easier identification of the source(s) of error, and can limit error associated with sampling methodology. The use of standard operating procedures ensures that all sampling tasks for a given matrix and analyte will be performed in the same manner, regardless of the individual sampling team, date, or location of sampling activity. Trip blanks, field blanks, replicate samples, and rinsate blanks are used to identify error due to sampling methodology and sample handling procedures.

### 5.3.3 Sample Heterogeneity

Sample heterogeneity is a potential source of error. Unlike water, soil is rarely a homogeneous medium and it exhibits variable properties with lateral distance and with depth. This heterogeneity may also be present in the sample container unless the sample was homogenized in the field or in the laboratory. The laboratory uses only a small aliquot of the sample for analysis; if the sample is not properly homogenized, the analysis may not be truly representative of the sample and of the corresponding site. Thoroughly homogenizing samples, therefore, can limit error associated with sample heterogeneity.

### 5.3.4 Analytical Procedures

Error which may originate in analytical procedures includes cross-contamination, inefficient extraction, and inappropriate methodology. Matrix spike samples, replicate samples, performance evaluation samples, and associated quality assurance evaluation of recovery, precision, and bias, can be used to distinguish analytical error from error introduced during sampling activities.

### 5.4 QA/QC SAMPLES

This section briefly describes the types and uses of QA/QC samples that are collected in the field, or prepared for or by the laboratory. QA/QC samples are analyzed in addition to field samples and provide information on the variability and usability of environmental sample results. They assist in identifying the origin of analytical discrepancies to help determine how the analytical results should be used. They are used mostly to validate analytical results. Field replicate, collocated, background, and rinsate blank samples are the most commonly collected field QA/QC samples. Performance evaluation, matrix spike, and matrix spike duplicate samples, either prepared for or by the laboratory, provide additional measures of control for the data generated. 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. Refer to data validation procedures in U.S. EPA, April 1990, for guidelines on utilizing OA/OC analytical results. The following paragraphs briefly describe each type of QA/QC sample.

### 5.4.1 Field Replicates

Field replicates are field samples obtained from one location, homogenized, divided into separate containers and treated as separate samples throughout the remaining sample handling and analytical processes. These samples are used to assess error associated with sample heterogeneity, sample methodology and analytical procedures. Use field replicates when determining total error for critical samples with contamination concentrations near the action level. For statistical analysis to be valid in such a case, a minimum of eight replicate samples would be required.

### 5.4.2 Collocated Samples

Collocated samples are collected adjacent to the routine field sample to determine local variability of the soil and contamination at the site. Typically, collocated samples are collected about one-half to three feet away from the selected sample location. Analytical results from collocated samples can be used to assess site variation, but only in the immediate sampling area. Due to the non-homogeneous nature of soil at sites, collocated samples should not be used to assess variability across a site and are not recommended for assessing error. Determine the applicability of collocated samples on a site-by-site basis. Collecting many samples (more than 50 samples/acre), is sufficient to demonstrate site variation.

### 5.4.3 Background Samples

Background samples are collected upgradient of the area(s) of contamination (either on or off site) where there is little or no chance of migration of the contaminants of concern. Background samples determine the natural composition of the soil with important in areas (especially concentrations of naturally-occurring metals) and are considered "clean" samples. They provide a basis for comparison of contaminant concentration levels with samples collected on site. At least one background soil sample should be collected; however, more are warranted when site-specific factors such as natural variability of local soil, multiple on-site contaminant source areas, and presence of off-site facilities potentially contributing to soil contamination exist. Background samples may be collected for all QA objectives, in order to evaluate potential error associated with sampling design, sampling methodology, and analytical procedures.

### 5.4.4 Rinsate Blanks

Rinsate blanks are samples obtained by running analyte-free water over decontaminated sampling equipment to test for residual contamination. The blank is placed in sample containers for handling, shipment, and analysis identical to the samples collected that day. A rinsate blank is used to assess cross-contamination brought about by improper decontamination procedures. Where dedicated sampling equipment is not utilized, collect one rinsate blank, per type of sampling device, per day.

### 5.4.5 Performance Evaluation Samples

Performance evaluation (PE) samples evaluate the overall bias of the analytical laboratory and detect any error in the analytical method used. These samples are usually prepared by a third party, using a quantity of analyte(s) which is known to the preparer but unknown to the laboratory, and always undergo certification analysis. The analyte(s) used to prepare the PE sample is the same as the analyte(s) of concern. Laboratory procedural error is evaluated by the percentage of analyte identified in the PE sample (percent recovery). Even though they are not available for every single analyte, analysis of PE samples is required to obtain definitive data.

### 5.4.6 Matrix Spike Samples

Matrix spike and matrix spike duplicate samples (MS/MSDs) are environmental samples that are spiked in the laboratory with a known concentration of a target analyte(s) to verify percent recoveries. MS/MSDs are primarily used to check sample matrix interferences. They can also be used to monitor laboratory performance. However, a dataset of at least three or more results is necessary to distinguish between laboratory performance and matrix interference.

MS/MSDs can also monitor method performance. Again, a dataset is helpful to assess whether a method is performing properly. Generally, interference and poor method performance go together.

MS/MSDs can also evaluate error due to laboratory bias and precision (when four or more pairs are analyzed). Analyze one MS/MSD pair to assess bias for every 20 soil samples. Use the average percent recovery for the pair. To assess precision, analyze at least 8 matrix spike replicates from the same sample, determine the standard deviation and the coefficient of variation. See pages 9 - 10 of the *QA/QC Guidance* 

for Removal Activities (U.S. EPA, April 1990) for procedures on calculating analytical error. MS/MSDs are optional when the goal is to obtain screening data and required to obtain definitive data as one of several methods to determine analytical error.

### 5.4.7 Field Blanks

Field blanks are samples prepared in the field using certified clean sand or soil and are then submitted to the laboratory for analysis. A field blank is used to evaluate contamination error associated with sampling methodology and laboratory procedures. If available, submit field blanks at a rate of one per day.

### 5.4.8 Trip Blanks

Trip blanks are samples prepared prior to going into the field. Trip blanks consist of certified clean sand or soil and are handled, transported, and analyzed in the same manner as the other volatile organic samples acquired that day. Trip blanks are used to evaluate error associated with sampling methodology and analytical procedures by determining if any contamination was introduced into samples during sampling, sample handling and shipment, and/or during laboratory handling and analysis. If available, utilize trip blanks for volatile organic analyses.

### 5.5 EVALUATION OF ANALYTICAL ERROR

The percentage and types of QA/QC samples needed to help identify the error and confidence in the data is based on the sampling objectives and the corresponding QA/QC objectives. The acceptable level of error is determined by the intended use of the data and the sampling objectives, including such factors as: the degree of threat to public health, welfare, or the environment; selected action levels; litigation concerns; and budgetary constraints.

The use of replicate samples is one method to evaluate error. To evaluate the total error of samples with contaminant concentrations near the selected action level, prepare and analyze a minimum of eight replicates of the same sample. Analytical data from replicate samples can also be used for a quick check on errors associated with sample heterogeneity, sample methodology and analytical procedures. Differing analytical results from two or more replicate samples could indicate improper sample preparation (e.g., incomplete homogenization), or that contamination was introduced during sample collection, preparation, handling, shipment, or

analysis.

It may be desirable to try to quantify confidence; however, quantification or analytical data correction is not always possible. A 95% confidence level (i.e., 5% acceptable error) should be adequate for most sampling activities. Experience will provide the best determination of whether to use a higher (e.g., 99%) or lower (e.g., 90%) level of confidence. It must be recognized that the use of confidence levels is based on the assumption that a sample is homogeneous. See also Section 6.8 for information on total error.

## 5.6 CORRELATION BETWEEN SCREENING RESULTS AND DEFINITIVE RESULTS

One cost-effective approach for delineating the extent of site contamination is to correlate inexpensive screening data and other field measurements (e.g., XRF, soil-gas measurements) with laboratory results. The relationship between the two methods can then be described by a regression analysis and used to predict laboratory results based on screening measurements. In this manner, cost-effective screening results may be used in addition to, or in lieu of, off-site laboratory sample analysis.

Statistical regression involves developing a model (equation) that relates two or more variables at an acceptable level of correlation. When screening techniques, such as XRF, are used along with laboratory methods (e.g., atomic absorption (AA)), a regression equation can be used to predict a laboratory value based on the results of the screening device. The model can also be used to place confidence limits around predictions. Additional discussion of correlation and regression can be found in most introductory statistics textbooks. A simple regression equation (e.g., linear) can be developed on many calculators or computer databases; however, a statistician should be consulted to check the accuracy of more complex models.

Evaluation of the accuracy of a model in part relies on statistical correlation. Statistical correlation 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, or simply  $\mathbb{R}^2$ , is an estimate of the proportion of variance

in one variable (the dependent variable) that can be accounted for by the independent variables. The R<sup>2</sup> value that is acceptable depends on the sampling objectives and intended data uses. As a rule of thumb, statistical relationships should have an R<sup>2</sup> value of at least 0.6 to determine a reliable model; however, for health or risk assessment purposes, the acceptable R<sup>2</sup> value may be made more stringent (e.g., 0.8). Analytical calibration regressions have an R<sup>2</sup> value of 0.98 or better.

Once a reliable regression equation has been derived, the 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 Chapter 6). These maps can be examined to evaluate the estimated extent of contamination and the adequacy of the sampling program.

### 5.7 EXAMPLE SITE

The screening of containerized liquid wastes was performed to quickly obtain data indicating general chemical class. Definitive analysis was run on



10% of the samples in order to verify screening results. The definitive analyses provided were analyte and concentration specific. Recoveries of matrix spike and matrix spike duplicate samples indicated no matrix interferences. Dedicated equipment was used during Phase 1 sampling, making rinsate blanks unnecessary. Phase 2 screening was performed using XRF. During Phase 2, samples were collected at 30% of the nodes screened with the XRF. These samples were sent for laboratory AA analysis. A correlation was established by plotting the Phase 2 AA and XRF data. This allowed the XRF data from the other 70% of the nodes to be used to evaluate the chromium levels across the site.

For Phase 2 and 3 sampling, 10% of the data were confirmed by running replicate analyses to obtain an estimate of precision. The results indicated good correlation. Matrix spikes and matrix spike duplicate samples indicated no matrix interferences. During Phase 2, the OSC included performance evaluation (PE) samples for metals to evaluate the overall laboratory bias. The laboratory achieved 92% recovery, which was within the acceptable control limits.

During Phases 2 and 3, a rinsate blank was collected each day. Following the decontamination of the bucket augers, analyte-free water was poured over the augers and the rinsate was placed into 1-liter polyethylene bottles and preserved. The rinsate blanks were analyzed for total metals and cyanide to determine the effectiveness of the decontamination procedures and the potential for cross-contamination. All rinsate blank samples were "clean", indicating sufficient decontamination procedures.

The correlation analysis run on Phase 2 laboratory (AA) data and corresponding XRF values resulted in r values of 0.97 for both surface and subsurface data, which indicated a strong relationship between the AA and XRF data. Following the correlation analyses, regression analyses were run and equations to predict laboratory values based on the XRF data were developed. The resulting equation for the surface data was: AA = 0.87 (XRF) + 10.16. The resulting regression equation for the subsurface data was: AA = 0.94 (XRF) + 0.30.

### 6.0 DATA PRESENTATION AND ANALYSIS

#### 6.1 INTRODUCTION

Data presentation and analysis techniques are performed with analytical, geophysical, or screening results. The techniques discussed below can be used to compare analytical values, to evaluate numerical distribution of data, to determine and illustrate the location of hot spots and the extent of contamination across a site, and to assess the need for removal of contaminated soil with concentrations at or near the action level. The appropriate methods to present and analyze sample data depend on the sampling objectives, the number of samples collected, the sampling approaches used, and a variety of other considerations.

### 6.2 DATA POSTING

Data posting involves placement of sample values on a site basemap. Data posting is useful for displaying the spatial distribution of sample values to visually depict extent of contamination and to locate hot spots. Data posting requires each sample to have a specific location (e.g., X and Y coordinates). Ideally, the sample coordinates would be surveyed values to facilitate placement on a scaled map.

### 6.3 GEOLOGIC GRAPHICS

Geologic graphics include cross-sections and fence diagrams, which are two- and three-dimensional depictions, respectively, of soils and strata to a given depth beneath the site. These types of graphics are useful for posting subsurface analytical data as well as for interpreting subsurface geology and contaminant migration.

### 6.4 CONTOUR MAPPING

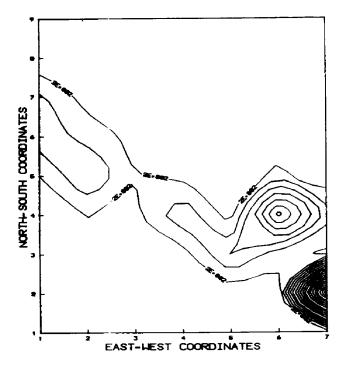
Contour maps are useful for depicting contaminant concentration values throughout a site. Contour mapping requires an accurate, to-scale basemap of the site. After data posting sample values on the basemap, insert contour lines (or isopleths) at a specified contour interval, interpolating values

between sample points. Contour lines can be drawn manually or be generated by computer using contouring software. Although the software makes the contouring process easier, computer programs have a limitation: they may interpolate between all data points, attempting to fit a contour interval to the full range of data values. This can result in a contour map that does not accurately represent general site contaminant trends. Typical emergency or early action sites have low concentration/non-detect areas and hot spots. Computer contouring programs may represent these features as in Figure 16 which illustrates a site that has a 4000 mg/kg hot spot. Because there is a large difference in concentration between the hot spot and the surrounding area, the computer contouring program used a contour interval that eliminated most of the subtle site features and general trends. However, if that same hot spot concentration value is posted at a reduced value, then the contouring program can select a more appropriate contour interval to better illustrate the general site trends. Figure 17 depicts the same site as in Figure 16, but the hot spot concentration value has been arbitrarily posted at 1400 mg/kg. The map was recontoured and the contouring program selected a contour interval that resulted in a map which enhanced the subtle detail and general site contaminant trends.

### 6.5 STATISTICAL GRAPHICS

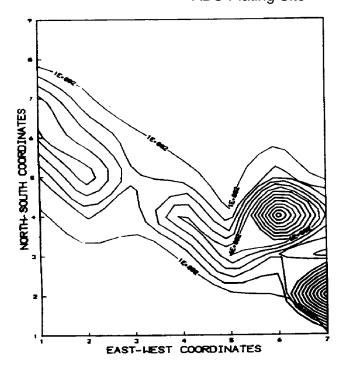
The distribution or spread of the data set is important in determining which statistical techniques to use. Common statistical analyses such as the t-test relies on normally distributed data. The histogram is a statistical bar graph which displays the distribution of a data set. A normally distributed 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. Use a histogram or probability plot to see trends and anomalies in the data prior to conducting more rigorous forms of statistical analysis.

Figure 16: Computer-Generated Contour Map (4000 mg/kg Hot Spot)
ABC Plating Site



Total Chromium Concentration
Units = mg/kg
Contour Interval = 100 mg/kg
Includes 4000 mg/kg Hot Spot

Figure 17: Computer-Generated Contour Map (1400 mg/kg Hot Spot) ABC Plating Site



Total Chromium Concentration Units = mg/kg Contour Interval = 100 mg/kg

includes 1400 mg/kg Hot Spot\*

1400 mg/kg hot spot is substituted for 4000 mg/kg hot spot

### 6.6 GEOSTATISTICS

Geostatistical methods are useful for data analysis and presentation. The characteristic feature of geostatistics is the use of variograms to quantify and model the spatial relationship between values at different sampling locations and for interpolating (e.g., kriging) estimated values across a site. The geostatistical analysis can be broken down into two phases. First, a model is developed that describes the spatial relationship between sample locations on the basis of a plot of spatial variance versus the distance between pairs of samples. This plot is called a variogram. Second, the spatial relationship modeled by the variogram is used to compute a weightedaverage interpolation of the data. The result of geostatistical mapping by data interpolation is a contour map that represents estimates of values across a site, and maps depicting potential error in the estimates. The error maps are useful for deciding if additional samples are needed and for calculating best or worst-case scenarios for site cleanup. More information on geostatistics can be found in U.S. EPA, September 1988b and U.S. EPA, 1990. Geo-EAS and GEOPACK, geostatistical environmental assessment software packages developed by U.S. EPA, can greatly assist with geostatistical analysis methods.

### 6.7 RECOMMENDED DATA INTERPRETATION METHODS

The data interpretation method chosen depends on project-specific considerations, such as the number of sampling locations and their associated range in values. A site depicting extremely low data values (e.g., non-detects) with significantly higher values (e.g., 5,000 ppm) from neighboring hot spots, with little or no concentration gradient in-between, does not lend itself to contouring and geostatistics, specifically the development of variograms. However, data posting would be useful at such a site to illustrate hot spot and clean areas. Conversely, geostatistics and contour mapping, as well as data posting, can be applied to site data with a wide distribution of values (i.e., depicting a "bell shaped" curve) with beneficial results.

#### 6.8 UTILIZATION OF DATA

When conducting search sampling to determine the locations of hot spots (as discussed in Section 2.9), analyze the data using one of the methods discussed in this chapter. For each node that is determined to be close to or above the action level, the following procedure is recommended.

Investigate all neighboring grid cells to determine which areas must be excavated and/or treated. From each grid cell, take a composite sample consisting of four or more aliquots, using the procedure described in Section 2.11.6. Grid cells with contaminant concentrations significantly above the action level (e.g., 20%) should be marked for removal. Grid cells with contaminant concentrations significantly less than the action level should be designated as clean. For grid cells with contaminant concentrations close to the action level, it is recommended that additional sampling be done within that grid cell to determine whether it is truly a hot spot, or whether the analytical result is due to sampling and/or analytical procedural error. If additional sampling is to be performed, one of the following methods should be considered:

- Collect a minimum of four grab samples within the grid cell in question. Use these samples to develop a 95% confidence interval around the mean concentration. If the action level falls within or below this confidence interval, then consider removal/treatment of the soil within that grid cell. More information on confidence intervals and standard deviation can be found in Gilbert, 1987.
- Collect additional composite samples from the grid cells in question using the technique discussed in Section 2.11.6. From these additional samples, determine the need for removal/treatment.

These two practical approaches help to determine the total error associated with collecting a sample from a non-homogeneous site. Total error includes design error, sampling error, non-homogeneous sampling error, and analytical error.

If additional sampling is being considered, weigh the cost-effectiveness of collecting the additional samples versus removing the soil from the areas in question. This decision must be made on a site-by-site basis.

After removal/treatment of the contaminated soil, reinvestigate the grid cells to verify cleanup below the action level. Each grid cell that had soil removed must either be composite sampled again, or have multiple grab samples collected with a 95% confidence interval set up again. Again, this decision must be made on a site-by-site basis. The methodology should be repeated until all grid cells are determined to have soil concentrations below the action level.

### 6.9 EXAMPLE SITE

The Phase 2 XRF/atomic absorption (AA) data were examined to determine the appropriate data interpretation method to use. A histogram



was generated to illustrate the distribution of the data as depicted in Figure 18. The histogram showed an uneven distribution of the data with most values less than 50 (approximately 4 on the LN scale of the histogram). Also, the presence of a single data point of 4000 (8 on the LN scale) was shown on the histogram. The data were initially posted as illustrated in Figures 19 and 20. Data posting was performed manually to give the OSC a quick depiction of the general site contamination trends. A contour mapping program was used to generate contours based on the posted data. Figure 16 illustrates the results of contouring with the 4000 mg/kg hot spot included. This contour map exaggerated the hot spot while eliminating the subtle site features and contaminant trends. Figure 17 depicts the same site data with the hot spot arbitrarily reduced to 1400 mg/kg. The resulting contour map enhanced more of the subtle site features and trends while reducing the effects of the hot spot.

AA concentrations predicted by the regression equations were kriged and contoured using Geo-EAS (Figures 21 and 22). Both the kriged contours and the data posting showed the same general site contaminant trends. However, data posting gave a more representative depiction of actual levels of contamination and the OSC used data posting for decision-making.

For each node with chromium concentrations close to or above the 100 ppm action level, the adjacent grid cells were further investigated. Composite samples consisting of four aliquots of soil were taken from within each grid cell in question and analyzed. If the soil concentration level was significantly below 100 ppm of chromium, the cell was designated as clean. Each cell that had a soil concentration level well above the action level was marked removal/treatment. Any cells having concentrations close to the action level were sampled further using the compositing method to better quantify the actual contaminant concentration. Since the surrounding area is residential, on-site landfilling was not considered a viable treatment option. To expedite treatment/disposal, all excavated soil from contaminated cells was stockpiled on site until treatment/disposal could be accomplished under a fixed-price contract. The stockpile, placed in the area of the most highly contaminated grid cells (where the lagoons were located), was covered until treatment/disposal could be arranged. Cleanup was verified with composite sampling in the excavated cells. Results of the composite sampling were compared with the action level to verify cleanup. All action levels were met. The excavation pits were filled with stone and clean soil, covered with topsoil, graded and seeded.

Figure 18: Histogram of Surface Chromium Concentrations ABC Plating Site

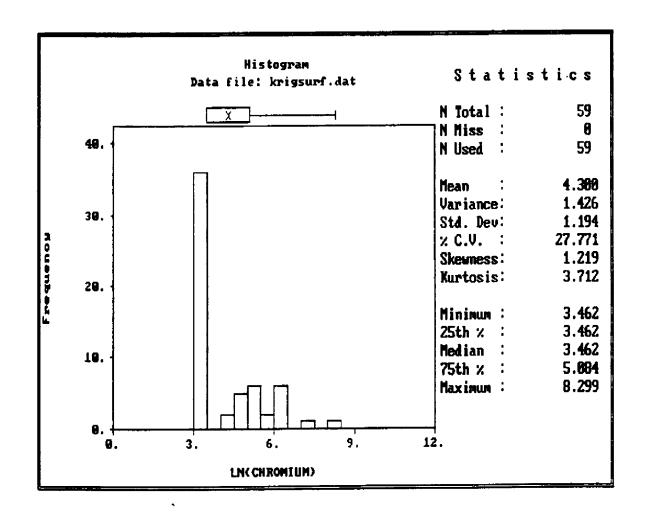


Figure 19: Phase 2 Surface Data Posting for Chromium ABC Plating Site

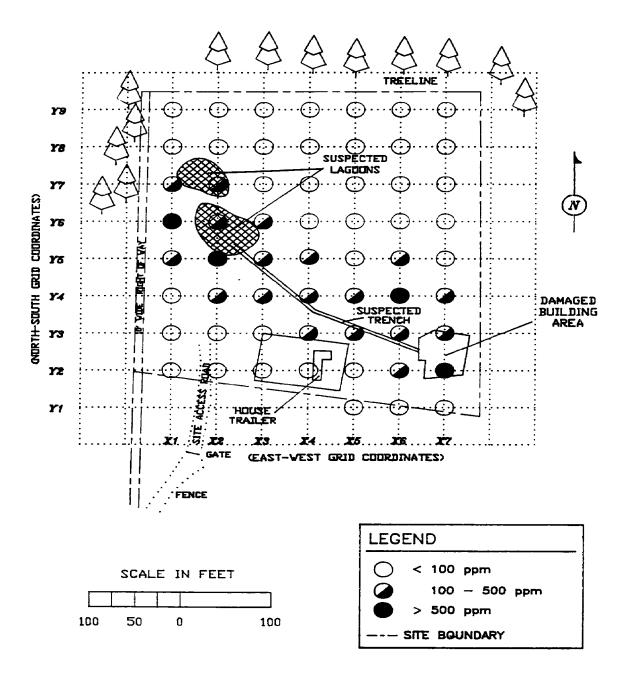


Figure 20: Phase 2 Subsurface Data Posting for Chromium ABC Plating Site

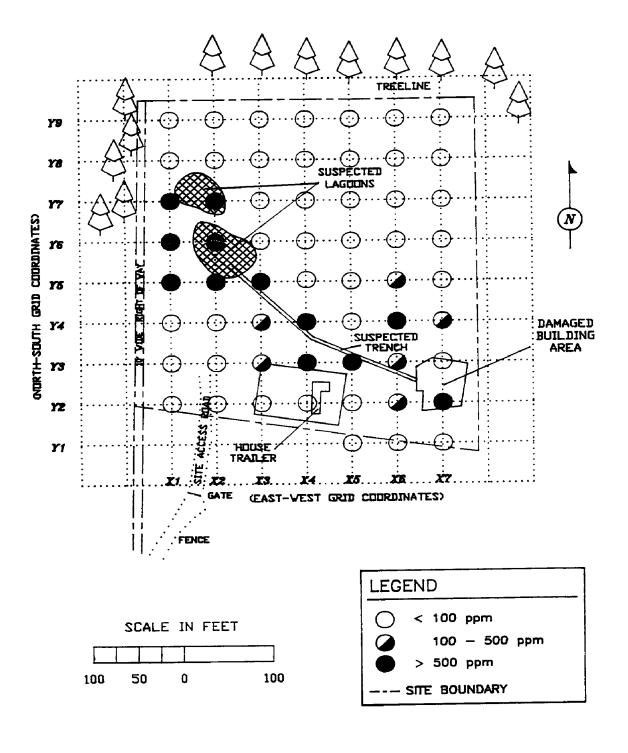


Figure 21: Contour Map of Surface Chromium Data (ppm) ABC Plating Site

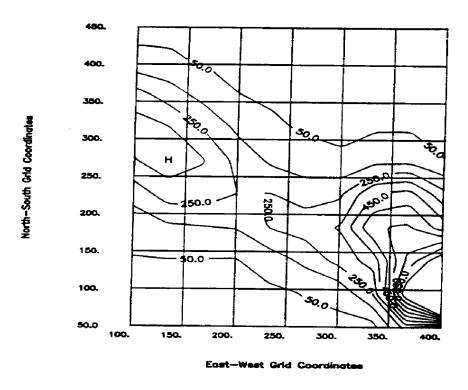
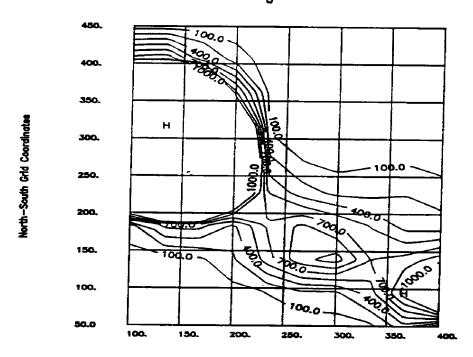


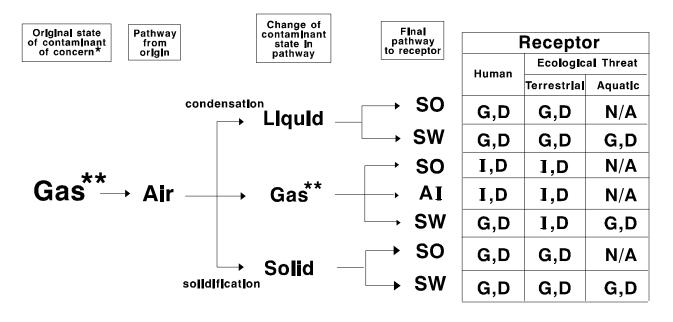
Figure 22: Contour Map of Subsurface Chromium Data (ppm)
ABC Plating Site



### 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

Beceptor Key

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

Pathway Key

AI = AI'
SO = Soil
SW = Surface Water
(Including sediments)
GW = Ground Water

Figure A-2

## Migration Routes of a Liquid Contaminant from Origin to Receptor

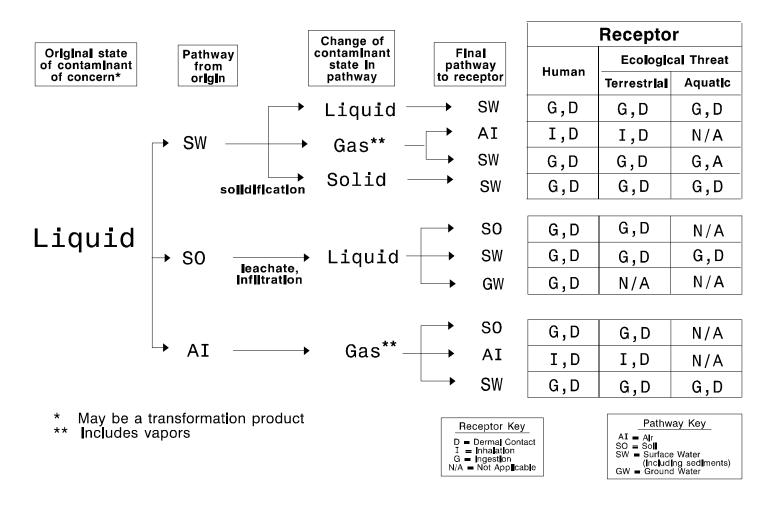
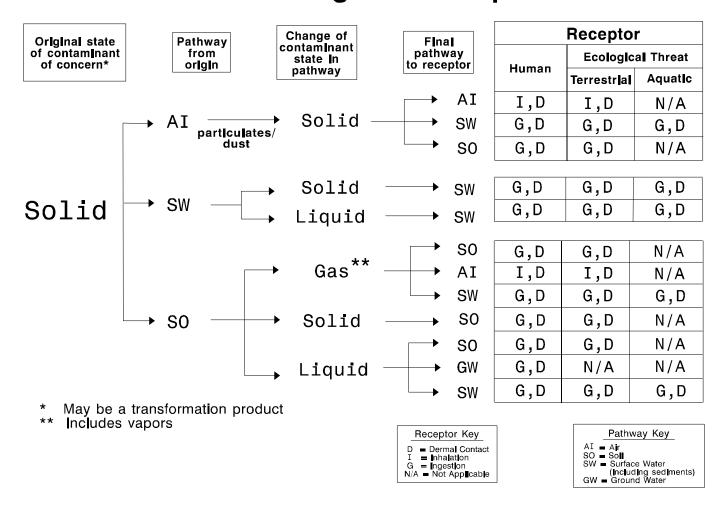


Figure A-3

## Migration Routes of a Solid Contaminant from Origin to Receptor



#### References

- American Society for Testing and Materials. 1987. Standard Practice for Reducing Field Samples of Aggregate to Testing Size. ASTM C702-87.
- American Society for Testing and Materials. 1972. Standard Method for Particle-Size Analysis of Soils. ASTM D422-63.
- Barth, Delbert. 1987. Statistical Considerations to Include the Importance of a Statistics Design for Soil Monitoring Programs. Proc. Third Annual Solid Waste Testing and Quality Assurance Symposium, Washington D.C., July 13-17, 1987.
- Benson, R., Robert A. Glaccum, and Michael R. Noel. 1988. *Geophysical Techniques for Sensing Buried Wastes and Waste Migration*. National Water Well Association: Ohio.
- Brady, Nyle C. 1974. The Nature and Properties of Soils. 8th ed. McMillan: New York.
- Clark, I. 1979. Practical Geostatistics. Elsevier Science: New York.
- Flatman, George T. 1987. QA/QC Considerations to Include Types and Numbers of Samples: Proc. Third Annual Waste Testing and Quality Assurance Symposium, Washington, D.C., July 13-17, 1987.
- Gilbert, Richard O. 1987. Statistical Methods for the Environmental Pollution Monitoring. Pacific Northwest Laboratory. Van Nostrand Reinhold: New York.
- Gy, P.M. 1982. Sampling of Particulate Material. Elsevier: Amsterdam.
- Jernigan, R.W. 1986. A Primer on Kriging. Washington, D.C.: U.S. EPA Statistical Policy Branch. 83 pp.
- Journal, A.G. 1989. *Geostatistics for the Environmental Sciences*. (Draft). Las Vegas: U.S. EPA Environmental Monitoring Systems Laboratory. 135 pp.
- New Jersey Department of Environmental Protection. February 1988. Field Sampling Procedures Manual.
- Taylor, J.K. 1987. Quality Assurance of Chemical Measurements.
- U.S. Environmental Protection Agency. Office of Emergency and Remedial Response. January 1991. Compendium of ERT Soil Sampling and Surface Geophysics Procedures. Interim Final. OSWER Directive 9360.4-02.
- U.S. Environmental Protection Agency. 1990. Geostatistics for Waste Management: A User's Manual for the GEOPACK (Version 1.0) Geostatistical Software System. Robert S. Kerr Environmental Research Laboratory, Ada, Oklahoma. EPA/600/8-90/004.
- U.S. Environmental Protection Agency. February 1989. *Methods for Evaluating the Attainment of Cleanup Standards*. Volume 1, *Soils and Solid Media*. EPA/230/02-89/042.
- U.S. Environmental Protection Agency. 1989a. *Soil Sampling Quality Assurance User's Guide*. 2nd ed. (Draft). EPA/600/8-89/046. Environmental Monitoring Systems Laboratory, Las Vegas, Nevada.
- U.S. Environmental Protection Agency. 1989b. Data Quality Objectives Workshop. (Briefing Notes).

- U.S. Environmental Protection Agency. December 1988. *User's Guide to the Contract Laboratory Program*. EPA/540/8-89/012.
- U.S. Environmental Protection Agency. September 1988a. Field Screening Methods Catalog User's Guide. EPA/540/2-88/005.
- U.S. Environmental Protection Agency. September 1988b. Geo-EAS (Geostatistical Environmental Assessment Software) User's Guide.
- U.S. Environmental Protection Agency. December 1987. *A Compendium of Superfund Field Operations Methods*. EPA/540/P-87/001. OSWER Directive 9355.0-14.
- U.S. Environmental Protection Agency. 1987. *Data Quality Objectives for Remedial Response Activities*. EPA/540/G-87/004. OSWER Directive 93550.7B.
- U.S. Environmental Protection Agency, Region IV. April 1986. Engineering Support Branch Standard Operating Procedures and Quality Assurance Manual. Environmental Services Division, Athens, Georgia.
- U.S. Environmental Protection Agency. 1986. Test Methods for Evaluating Solid Waste. Volume II, Field Manual Physical/Chemical Methods.
- U.S. Environmental Protection Agency. 1984a. Characterization of Hazardous Waste Sites A Methods Manual. Volume I, Site Investigations. Section 7: Environmental Monitoring Systems Laboratory, Las Vegas, Nevada. EPA/600/4-84/075.
- U.S. Environmental Protection Agency. 1984b. *Characterization of Hazardous Waste Sites A Methods Manual*. Volume II, *Available Sampling Methods, Second Edition*. Environmental Monitoring Systems Laboratory, Las Vegas, Nevada. EPA/600/4-84/076.
- U.S. Environmental Protection Agency. 1983. *Preparation of Soil Sampling Protocol: Techniques and Strategies*. EPA/600/4-83/020.
- van Ee, J.J., L.J. Blume, and T.H. Starks. 1989. A Rationale for the Assessment of Errors in the Sampling of Soils. U.S. EPA Environmental Monitoring Systems Laboratory, Las Vegas, Nevada.