SEVEN SISTERS
ENVIRONMENTAL

4609 Chokecherry Trail, no.4
Fort Collins Colorado 80526
970 631.8685
970 631.8082, fax

CDEPWMF #149002

June 16, 2009

Rodney Bailey, Water and Waste Group Lead
Chevron USA, Inc.
15 Smith Road
Midland, TX 79705

RE: Wilson Creek Landfarm Closure Plan

Dear Mr. Bailey:

This document has been prepared to provide guidance to Chevron for estimating future liabilities associated with the landfarm located at the Wilson Creek Facility. The landfarm is a centralized Exploration and Production (E&P) waste treatment facility primarily regulated by the 900 series rules of the Colorado Oil and Gas Conservation Commission (COGCC) under the provisions of §34-60-103(4.5) and §34-60-106 of the Colorado Revised Statutes (C.R.S.). The material treated on the landfarm are E&P wastes exempt from hazardous regulation under Subtitle C of the Resource and Recovery Act (RCRA) of 1976.

The Wilson Creek Facility is located in various sections of Townships 2 and 3 North, Range 94 West, of the 6th Principle Meridian in Rio Blanco County, Colorado. Figures 1 and 2 in Attachment II contain additional site location information. The town of Meeker, Colorado, which lies approximately 11 miles south of the facility, is the nearest population center.

The Wilson Creek Field, an onshore production facility owned and operated by Chevron, is located in rugged mountainous terrain consisting of steep canyon walls and deep gorges. Vehicular travel is restricted to roadways and foot travel off roadways is difficult.

The Landfarm Facility, less than 2 acres in extent, is sited in a remote area on the southwest quadrant of the basin at NE¼NW¼, Section 35, T3N, R94W (Attachment II, Figures 1 and 2 – General Area and Area Detail Maps). The closure plan consists of three phases:

1. Final Remediation
2. Equipment Decommissioning
3. Site Reclamation

Each of the phases is described in detail below. The attached spreadsheet (Attachment I) lists each phase and the associated detailed activities along with

estimated costs (2009 dollars). The primary assumptions in developing this Closure Plan are:

1. Chevron will obtain any needed variances to metals standards (e.g., arsenic) based on available data on background concentrations and other pertinent data.
2. Chevron will not obtain a variance on the current COGCC standard for Total Petroleum Hydrocarbons (TPH), which is 500 mg/kg.
3. There will be no groundwater impacts – currently there are no indications of groundwater impacts in the vicinity of the landfarm.

Final Remediation

After the final application of E&P waste on the landfarm, it is estimated that it will take five (5) years to remediate the Total Petroleum Hydrocarbons (TPH) to meet the COGCC Standard of 500 mg/kg. The current COGCC standards for soil are found in Attachment III.

During the five-year remediation span, quarterly soil sampling is recommended to provide the data necessary to optimize the operational parameters (irrigation, fertilization, soil tilling) to maximize the degradation rate. The sampling should be conducted according to the Annual Sampling Protocol contained in the Wilson Creek Landfarm Operating Manual. The cost of each sampling event (labor, equipment, and service fees) is estimated to be about $3,500. Normal operating costs would also be incurred during that 5-year period and would include, but not be limited to:

1. Irrigation
2. Fertilization
3. Soil manipulation
4. Storm water management
5. Routine patrol, security, and maintenance

A 30% premium should be added to these costs (over historical expenses) to account for the increased operating activity necessary to maximize degradation rates.

After the quarterly sampling results indicate the landfarm meets the COGCC standards for closure, one rigorous sampling event will be conducted to ensure the data are representative and unbiased. This final sampling event will be conducted in compliance with the ASTM Guidance Documents (Attachment IV):

1. ASTM D6009 – Standard Guide for Sampling Waste Piles, and
2. ASTM D6044 – Standard Guide for Representative Sampling for Management of Waste and Contaminated Media

Only after the results of the sampling event conducted according to the ASTM Standards confirm compliance with the COGCC Standards will Decommissioning

and Site Reclamation activities commence. The cost of this rigorous sampling event is estimated to be about $15,000.

Equipment Decommissioning

Certain equipment will need to be decommissioned and either moved to surplus equipment or appropriately disposed of. The on-site equipment includes:

1. Miscellaneous surface equipment
2. Fence

The costs of decommissioning this equipment are best estimated by Operations, however some placeholder values have been entered into the cost table (Attachment I) for reference purposes. The assumptions for the costs associated with this decommissioning include:

1. None of the miscellaneous surface equipment will be salvageable and will be disposed of in accordance will all applicable laws and regulations.
2. The fence material may be salvageable, but will have no appreciable salvage value. There is approximately 1200 linear feet of fence around the landfarm.

Site Reclamation

Final site reclamation will be similar to general construction project reclamation and will include:

1. Obtaining a Stormwater Construction Permit and subsequent stormwater management activities
2. Removal of excess soil for beneficial use
3. Final contouring of entire site consistent with natural slopes and drainages
4. Re-vegetation activities, including seeding (BLM approved seed mix) and erosion control structures
5. Final regulatory clearance activities with the COGCC, Rio Blanco County, and other applicable agencies.

Costs for Site Reclamation need to be modified by Facilities Engineering and/or Operations Personnel to reflect recent experience with this type of work at Wilson Creek.

Please don't hesitate if you have any question or concerns.

Sincerely,

Jack Matthews
Seven Sisters Environmental, Inc.

Attachments –
I – Closure Costs
II – Figures and Maps
III – COGCC Standards
IV – ASTM Guidance Documents

cc: Bill Savage – Chevron, Rangely
Dale Harber – Chevron, Rangely
Adam Berig – Olsson Associates, Golden

Attachment I

Closure Costs

Wilson Creek Facility
Landfarm Estimated Closure Costs

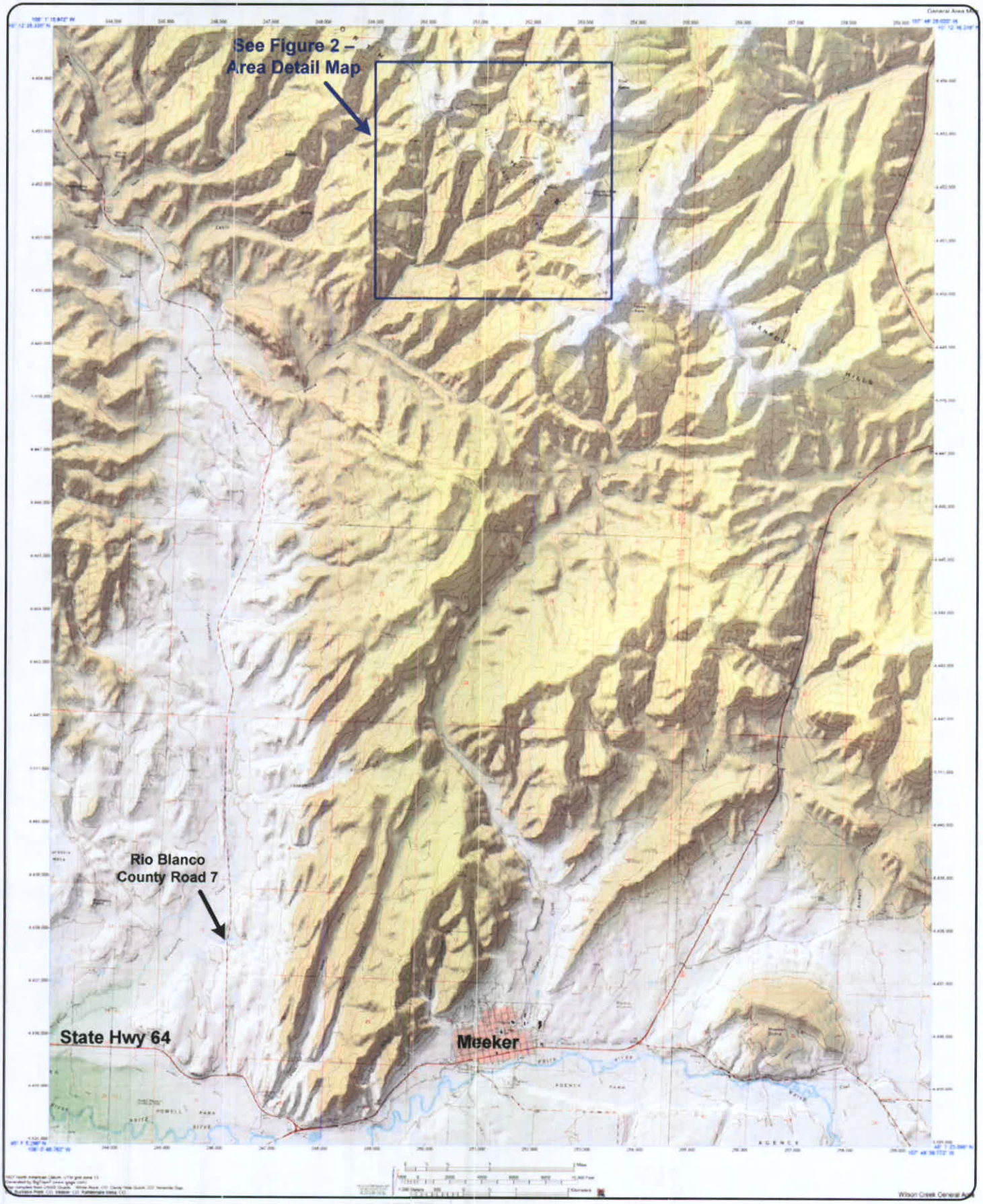
6/18/2009

- **Placeholder**, Actual costs to be input by Facilities Engineering or Operations Personnel

Phase / Activity					
Final Remediation	**Unit Cost**	**# Units/ year**	**Years**	**Premium**	**Total Cost**
Final Remediation Sampling and Analysis	$ 3,500	4	5	0%	$ 70,000
Annual Operating Costs	$ 2,000	1	5	30%	$ 13,000
ASTM Sampling and Analysis	$ 15,000	1	1	0%	$ 15,000
				Subtotal -	$ 98,000
Equipment Decommissioning	**Unit Cost**	**# Units**			**Total Cost**
Miscellaneous surface equipment	$ 5,000	1			$ 5,000
Fence Removal (per linear foot)	$ 2	1200			$ 2,400
				Subtotal -	$ 7,400
Site Reclamation	**Unit Cost**	**# Units/ year**	**Years**	**Premium**	**Total Cost**
Stormwater Permit and Management Activities, annual cost	$ 5,000	1	2	0%	$ 10,000
Excess Soil Removal, beneficial use	$ 2,000	1	1	0%	$ 2,000
Final contour, per acre	$ 1,600	2	1	0%	$ 3,200
Re-vegetation, per acre	$ 500	2	1	0%	$ 1,000
Final regulatory clearance by COGCC and RBC	$ 10,000	1	1	0%	$ 10,000
				Subtotal -	$ 26,200
				Total Cost - 2009 Dollars =	$ 131,600
				Contingency -	30%
				Total + Contingency -	**$ 171,080**

Attachment II

Figures and Maps

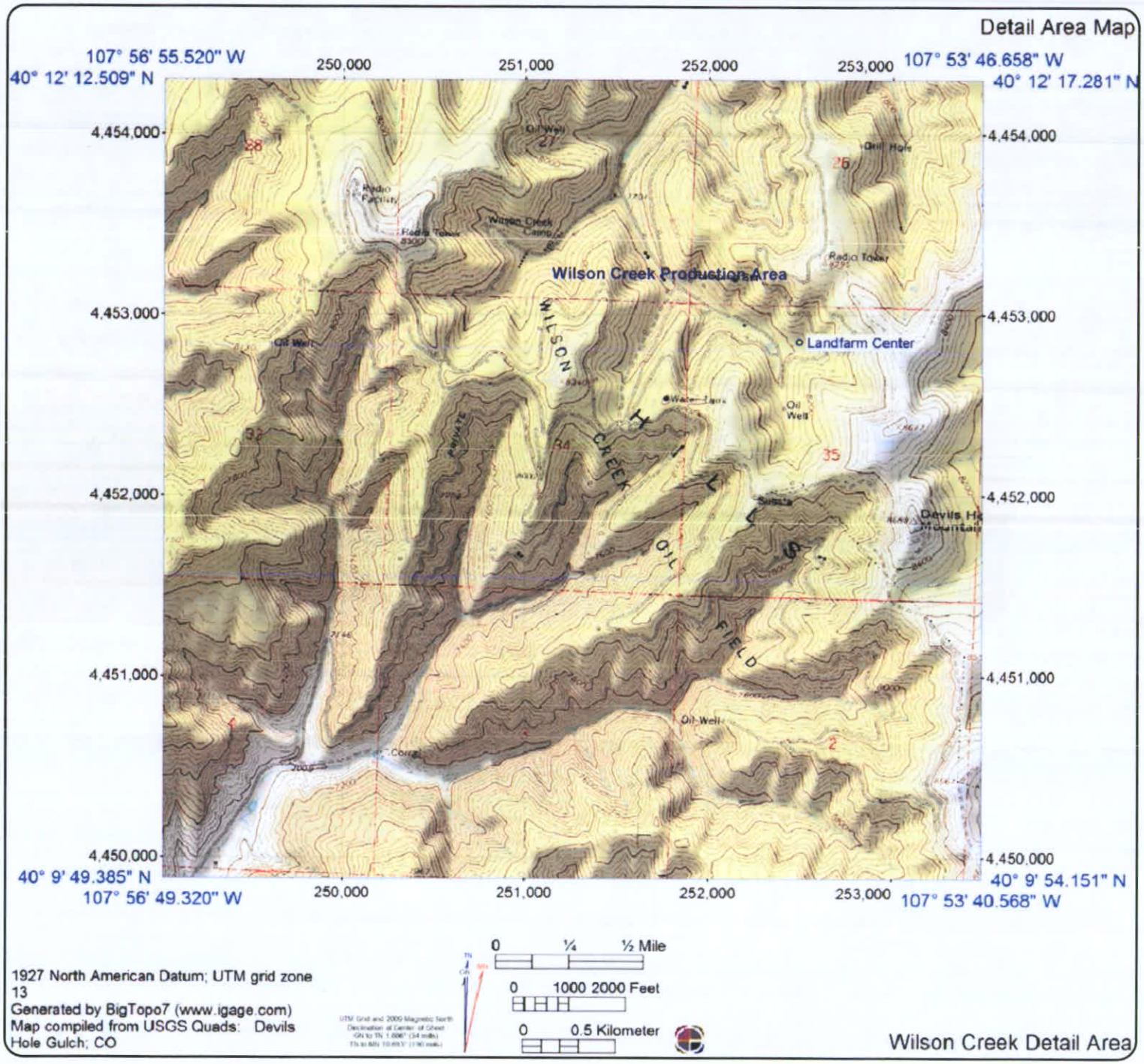


General Site Location Map Chevron USA, Inc. Wilson Creek Production Facility Rio Blanco County, Colorado	
Revision Date:	5/18/2009
Revision No.:	0
Revised By:	JYM
Approved By	JYM
Project No.:	OLS008-2084
Scale:	On map

SEVEN SISTERS ENVIRONMENTAL

Figure 1

Detail Area Map



0 ¼ ½ Mile

0 1000 2000 Feet

0 0.5 Kilometer

1927 North American Datum; UTM grid zone 13

Generated by BigTopo7 (www.igage.com)

Map compiled from USGS Quads: Devils Hole Gulch; CO

Wilson Creek Detail Area

RECEIVED
JUN 29 2009
COGCC

Figure 2

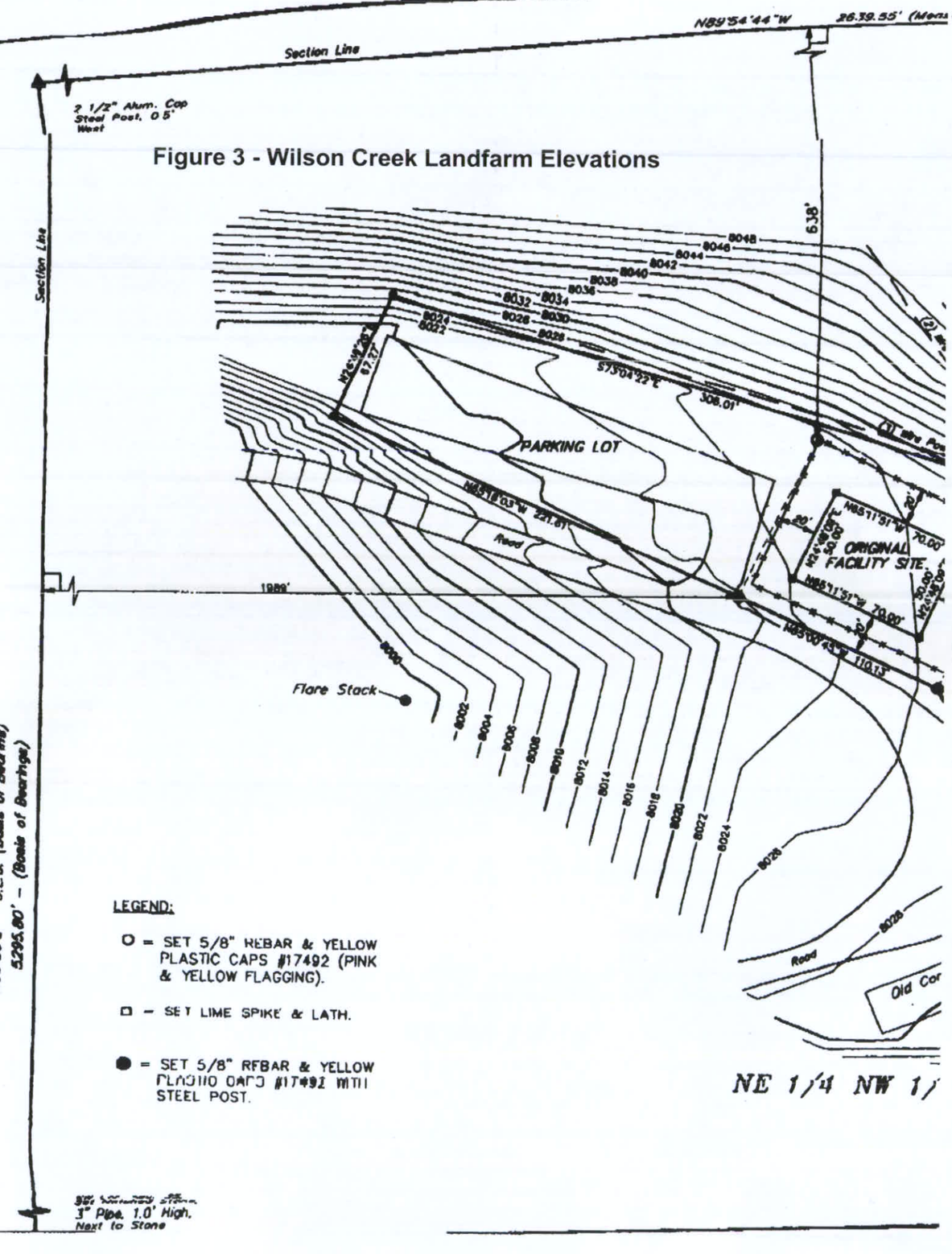
Area Detail Map
Chevron USA, Inc.
Wilson Creek Production Facility
Rio Blanco County, Colorado

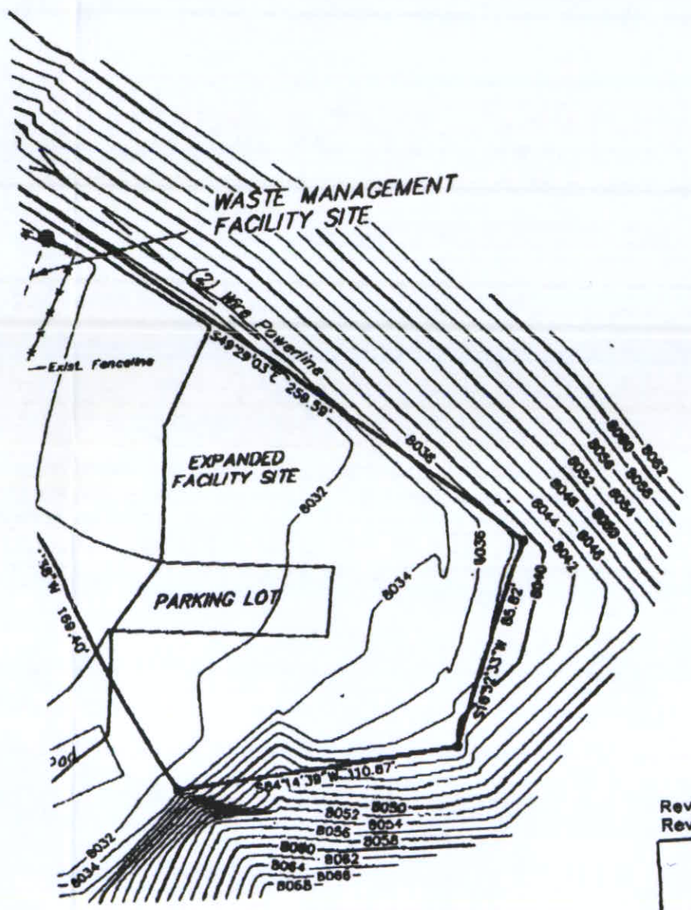
Revision Date:	6/15/2009
Revision No.:	0
Revised By:	JYM
Approved By	JYM
Project No.:	OLS008-2084
Scale:	On map

SEVEN SISTERS ENVIRONMENTAL

Figure 3 - Wilson Creek Landfarm Elevations

APPROXIMATE ACREAGE	
PROPERTY OWNER	ACREAGE
ORIGINAL FACILITY SITE	0.080
EXPANDED FACILITY SITE w/ ORIG. FACILITY SITE	1.360

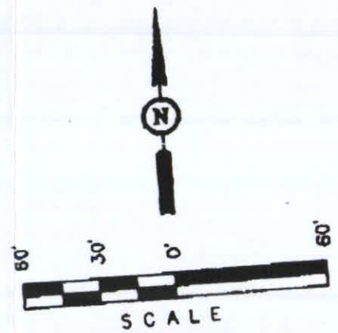




TEXACO EXPLR. & PROD., INC.

WASTE MANAGEMENT FACILITY SITE

LOCATED IN
SECTION 35, T3N, R94W, 6th P.M.
RIO BLANCO COUNTY, COLORADO.



CONTOUR INTERVAL = 2'

BASIS OF ELEVATION

SPOT ELEVATION AT A ROAD INTERSECTION IN THE NE 1/4 SW 1/4 OF SECTION 34, T3N, R94W, 6th P.M. TAKEN FROM THE DEVILS HOLE GULCH QUADRANGLE, COLORADO, 7.5 MINUTE QUAD. (TOPOGRAPHIC MAP) PUBLISHED BY THE UNITED STATES DEPARTMENT OF THE INTERIOR, GEOLOGICAL SURVEY. SAID ELEVATION IS MARKED AS BEING 8032 FEET.

CERTIFICATE

THIS IS TO CERTIFY THAT THE ABOVE PLAT WAS PREPARED FROM FIELD NOTES OF ACTUAL SURVEYS MADE BY ME OR UNDER MY SUPERVISION AND THAT THE SAME ARE TRUE AND CORRECT TO THE BEST OF MY KNOWLEDGE AND BELIEF.

REGISTERED LAND SURVEYOR
REGISTRATION NO. 17492
STATE OF COLORADO
STATE OF UTAH

Revised: 05-05-03 D.R.B.
Revised: 04-30-03 D.R.B.

UINTAH ENGINEERING & LAND SURVEYING
85 SOUTH - 200 EAST • (435) 789-1017
VERNAL, UTAH - 84078

SCALE	DATE
1" = 60'	07-20-00
PARTY	

Attachment III

COGCC Standards

TABLE 3: COGCC CONCENTRATION LEVELS

(SEE NOTES ON FOLLOWING PAGE)

Contaminant of Concern	Concentrations
Organic Compounds in Soil	
TPH (total volatile and extractable petroleum hydrocarbons)	500 mg/kg
Benzene	0.17 mg/kg[2]
Toluene	85 mg/kg2
Ethylbenzene	100 mg/kg2
Xylenes (total	175 mg/kg2
Acenaphthene	1,000 mg/kg2
Anthracene	1,000 mg/kg2
Benzo(A)anthracene	0.22 mg/kg2
Benzo(B)fluoranthene	0.22 mg/kg2
Benzo(K)fluoranthene	2.2 mg/kg2
Benzo(A)pyrene	0.022 mg/kg2
Chrysene	22 mg/kg2
Dibenzo(A,H)anthracene	0.022 mg/kg2
Fluoranthene	1,000 mg/kg2
Fluorene	1,000 mg/kg2
Indeno(1,2,3,C,D)pyrene	0.22 mg/kg2
Napthalene	23 mg/kg2
Pyrene	1,000 mg/kg2
Organic Compounds in Ground Water	
Benzene	5 µg/l[3]
Toluene	560 to 1,000 µg/l[3]
Ethylbenzene	700 µg/l[3]
Xylenes (Total)	1,400 to 10,000 µg/l[3,4]
Inorganics in Soils	
Electrical Conductivity (EC)	<4 mmhos/cm or 2x background
Sodium Adsorption Ratio (SAR)	<12[5]
pH	6-9
Inorganics in Ground Water	
Total Dissolved Solids (TDS)	<1.25 x background[3]
Chlorides	<1.25 x background[3]
Sulfates	<1.25 x background[3]
Metals in Soils	
Arsenic	0.39 mg/kg[2]
Barium (LDNR True Total Barium)	15,000 mg/kg[2]
Boron (Hot Water Soluble)	2 mg/l[3]
Cadmium	70 mg/kg[3,6]
Chromium (III)	120,000 mg/kg[2]
Chromium (VI)	23 mg/kg[2,6]
Copper	3,100 mg/kg[2]
Lead (inorganic)	400 mg/kg[2]
Mercury	23 mg/kg[2]
Nickel (soluble salts)	1,600 mg/kg[2,6]
Selenium	390 mg/kg[2,6]
Silver	390 mg/kg[2]
Zinc	23,000 mg/kg[2,6]
Liquid Hydrocarbons in Soils and Ground Water	
Liquid hydrocarbons including condensate and oil	Below detection level

Notes to Table 3

COGCC recommends that the latest version of EPA SW 846 analytical methods be used where possible and that analyses of samples be performed by laboratories that maintain state or national accreditation programs.

[1] Consideration shall be given to background levels in native soils and ground water.

[2] Concentrations taken from CDPHE-HMWMD Table 1 Colorado Soil Evaluation Values (December 2007).

[3] Concentrations taken from CDPHE-WQCC Regulation 41 - The Basic Standards for Ground Water.

[4] For this range of standards, the first number in the range is a strictly health-based value, based on the WQCC's established methodology for human health-based standards. The second number in the range is a maximum contaminant level (MCL), established under the Federal Safe Drinking Water Act which has been determined to be an acceptable level of this chemical in public water supplies, taking treatability and laboratory detection limits into account. The WQCC intends that control requirements for this chemical be implemented to attain a level of ambient water quality that is at least equal to the first number in the range except as follows: 1) where ground water quality exceeds the first number in the range due to a release of contaminants that occurred prior to September 14, 2004 (regardless of the date of discovery or subsequent migration of such contaminants) clean-up levels for the entire contaminant plume shall be no more restrictive than the second number in the range or the ground water quality resulting from such release, whichever is more protective, and 2) whenever the WQCC has adopted alternative, site-specific standards for the chemical, the site-specific standards shall apply instead of these statewide standards.

[5] Analysis by USDA Agricultural Handbook 60 method (20B) with soluble cations determined by method (2). Method (20B) = estimation of exchangeable sodium percentage and exchangeable potassium percentage from soluble cations. Method (2) = saturated paste method (note: each analysis requires a unique sample of at least 500 grams). If soils are saturated, USDA Agricultural Handbook 60 with soluble cations determined by method (3A) saturation extraction method.

[6] The table value for these inorganic constituents is taken from the CDPHE-HMWMD Table 1 Colorado Soil Evaluation Values (December 2007). However, because these values are high, it is possible that site-specific geochemical conditions may exist that could allow these constituents to migrate into ground water at levels exceeding ground water standards even though the concentrations are below the table values. Therefore, when these constituents are present as contaminants, a secondary evaluation of their leachability must be performed to ensure ground water protection.

Attachment IV

ASTM Guidance Documents

Designation: D 6009 – 96 (Reapproved 2001)

Standard Guide for Sampling Waste Piles[1]

This standard is issued under the fixed designation D 6009; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide provides guidance for obtaining representative samples from waste piles. Guidance is provided for site evaluation, sampling design, selection of equipment, and data interpretation.

1.2 Waste piles include areas used primarily for waste storage or disposal, including above-grade dry land disposal units. This guide can be applied to sampling municipal waste piles.

1.3 This guide addresses how the choice of sampling design and sampling methods depends on specific features of the pile.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1452 Practice for Soil Investigation and Sampling by Auger Borings[2]

D 1586 Test Method for Penetration Test and Split-Barrel Sampling of Soils[2]

D 1587 Practice for Thin-Walled Tube Geotechnical Sampling of Soils[2]

D 4547 Practice for Sampling Waste and Soils for Volatile Organics[3]

D 4687 Guide for General Planning of Waste Sampling[3]

D 4700 Guide for Soil Sampling from the Vadose Zone[2]

D 4823 Guide for Core-Sampling Submerged, Unconsolidated Sediments[4]

D 5088 Practice for Decontamination of Field Equipment Used at Nonradioactive Sites[5]

D 5314 Guide for Soil Gas Monitoring in the Vadose Zone[5]

D 5451 Practice for Sampling Using a Trier Sampler[3]

D 5518 Guide for Acquisition of File Aerial Photography and Imagery for Establishing Historic Site-Use and Surficial Conditions[5]

D 5730 Guide to Site Characterization for Environmental Purposes with Emphasis on Soil, Rock, the Vadose Zone and Ground Water[5]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *hot spots*—strata that contain high concentrations of the characteristic of interest and are relatively small in size when compared with the total size of the materials being sampled.

3.1.2 *representative sample*—a sample collected such that it reflects one or more characteristics of interest (as defined by the project objectives) of the population from which it was collected.

3.1.2.1 *Discussion*—A representative sample can be a single sample, a set of samples, or one or more composite samples.

3.1.3 *waste pile*—unconfined storage of solid materials in an area of distinct boundaries, above grade and usually uncovered. This includes the following:

3.1.3.1 *chemical manufacturing waste pile*—a pile consisting primarily of discarded chemical products (whether marketable or not), by-products, radioactive wastes, or used or unused feedstocks.

3.1.3.2 *scrap metal or junk pile*—a pile consisting primarily of scrap metal or discarded durable goods such as appliances, automobiles, auto parts, or batteries.

3.1.3.3 *trash pile*—a pile of waste materials from municipal sources, consisting primarily of paper, garbage, or discarded nondurable goods that contain or have contained hazardous substances. It does not include waste destined for recyclers.

4. Significance and Use

4.1 This guide is intended to provide guidance for sampling waste piles. It can be used to obtain samples for waste characterization related to use, treatment, or disposal; to monitor an active pile; to prepare for closure of the waste pile; or to investigate the contents of an abandoned pile.

4.2 Techniques used to sample include both in-place evaluations of the pile and physically removing a sample. In-place evaluations include techniques such as remote sensing, on-site gas analysis, and permeability.

4.3 Sampling strategy for waste piles is dependent on the following:

4.3.1 Project objectives including acceptable levels of error when making decisions;

---

[1] This guide is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.01.01 on Planning for Sampling.

Current edition approved Oct. 10, 1996. Published December 1996.

[2] *Annual Book of ASTM Standards*, Vol 04.08.

[3] *Annual Book of ASTM Standards*, Vol 11.04.

[4] *Annual Book of ASTM Standards*, Vol 11.02.

[5] *Annual Book of ASTM Standards*, Vol 04.09.

4.3.2 Physical characteristics of the pile, such as its size and configuration, access to all parts of it, and the stability of the pile;

4.3.3 Process that generated the waste and the waste characteristics, such as hazardous chemical or physical properties, whether the waste consists of sludges, dry powders or granules, and the heterogeneity of the wastes;

4.3.4 History of the pile, including dates of generation, methods of handling and transport, and current management methods;

4.3.5 Regulatory considerations, such as regulatory classification and characterization data;

4.3.6 Limits and bias of sampling methods, including bias that may be introduced by waste heterogeneity, sampling design, and sampling equipment.

4.4 It is recommended that this guide be used in conjunction with Guide D 4687, which addresses sampling design, quality assurance, general sampling considerations, preservation and containerization, cleaning equipment, packaging, and chain of custody.

4.5 A case history of the investigation of a waste pile is included in Appendix X1.

5. Site Evaluation

5.1 Site evaluations are performed to assist in designing the most appropriate sampling strategy. An evaluation may consist of on-site surveys and inspections, as well as a review of historical data. Nonintrusive geophysical and remote sensing methods are particularly useful at this stage of the investigation (see Guide D 5518). Table 1 summarizes the effects that various factors associated with the waste pile, such as the history of how the pile was generated, have upon the strategy and design of the sampling plan. The strategic and design considerations are discussed as well.

5.2 *Generation History*—The waste pile may have been created over an extended time period. A remote sensing method that is very useful in establishing historical management practices for waste piles is aerial imagery. Aerial photographs are widely available and may be used to determine the history of a waste pile, sources of waste, and the presence and distribution of different strata. Satellite imagery could be used for larger waste piles.

TABLE 1 Strategy Factors

Waste Pile Factors	Strategic Considerations	Design Considerations
Generation history	Date of generation	Analysis required
	Types of processes	Location of samples
	Characteristics by process	
	Delivery method	
	Current management	
	Regulatory considerations	
Physical characteristics of pile:	Physical variability of pile	Number of samples
– size	Access	Location of samples
– shape	Safety	Equipment selection
– stability		
Waste characteristics	Constituents present	Number of samples
	Constituent distribution	Analysis required
	Heterogeneity	Location of samples
	– physical variability	Representative samples
	– chemical variability	Equipment selection

5.2.1 The date of generation could be important with respect to the types of processes that generated the waste, the characteristics of the waste, the distribution of the constituents, and regulatory concerns.

5.2.2 The type of process that generated the waste will determine the types of constituents that may be present in the waste pile. Chemical variability will influence the number of samples that are required to characterize the waste pile unless a directed (biased) sampling approach is acceptable.

5.2.3 The delivery method of the material to the waste pile could influence the concentrations of the constituents, affect the overall shape of the pile, or create physical dissimilarity within the waste pile through sorting by particle size or density.

5.2.4 If the pile is under current management and use, the variability in constituent types and concentrations may be affected. Current management activities also may influence the regulatory status of the waste pile.

5.2.5 Regulatory considerations will typically focus on waste identification questions, in other words is the material a solid waste that should be regulated and managed as a hazardous waste **(1)**.[6] This may involve a limited, directed sampling approach, particularly if a regulatory agency is conducting the investigation. A more comprehensive sampling design may be required to determine if the waste classifies as hazardous. Remediation efforts and questions regarding permits may focus on characterizing the entire pile, possibly as the removal of material is occurring. It should be noted that concentrations of contaminants near regulatory levels may increase the number of samples required to meet the objectives of the investigation. These regulatory levels could be those established to determine if a waste is hazardous, or "cleanup" levels set for a removal or remediation.

5.3 *Physical Characteristics of Pile*— Several physical characteristics of the waste pile must be considered during the site evaluation. Variability in size, shape, and stability of the pile affects access to it to obtain samples as well as safety considerations. Physical variability will influence the number of samples that are required to characterize the waste pile unless a directed (biased) sampling approach is considered to be acceptable. Techniques that might be used include resistivity and seismic refraction (for determining the depth of very large piles).

5.3.1 The size of the waste pile will influence the sampling strategy in that increasing size is often accompanied by increased variability in the physical characteristics of the waste pile. The number of samples, however, that are needed to characterize a waste pile adequately will typically be a function of the study objectives as well as the inherent variability of the pile.

5.3.2 The shape of the waste pile can influence the sampling strategy by limiting access to certain locations within the pile, and if it is topologically complex it is difficult to lay out a sampling grid. Also, a waste pile may extend vertically both above and below grade, making decisions regarding the depth of sample collection difficult.

[6] The boldface numbers in parentheses refer to the list of references at the end of this standard.

5.3.3 The stability of the waste pile also can limit access to both the face and the interior of the pile. The use of certain types of heavier sampling equipment also could be limited by the ability of the pile to bear the weight of the equipment.

5.4 *Waste Characteristics*:

5.4.1 The constituents could include inorganics, volatile organic compounds (VOCs), and semivolatile organic compounds (including pesticides and polychlorinated biphenyls (PCBs)) (see Practice D 4547). Speciality analyses may be warranted, such as leaching tests or analyses for dioxin/furans or explosive compounds. Soil gas sampling is a minimally intrusive technique that may detect the presence and distribution of volatile organic compounds in soils and in porous, unconsolidated materials. Appropriate applications for soil gas monitoring are identified in Guide D 5314.

5.4.2 The distribution of constituents in the waste pile could be influenced by changes in the manufacturing process which resulted in changes in the composition of the waste; the length of time the material has remained in the pile (particularly for VOCs); the mode of delivery of the waste materials to the pile; and management practices, such as mixing together wastes from more than one process.

5.4.3 Physical and chemical variabilities would include variability in the chemical characteristics of the material within the pile, as well as variability in particle size, density, hardness, whether brittle or flexible, moisture content, consolidated, or unconsolidated. The variability may be random or found as strata of materials having different properties or containing different types or concentrations of constituents.

5.4.3.1 Geophysical survey methods may be used on piles to estimate physical homogeneity, which may or may not be related to chemical homogeneity, and to detect buried objects, both of which may need to be considered during the development of the sampling design and the safety plan for the investigation. The most suitable technique for detecting nonmetallic objects is electromagnetics. Ground-penetrating radar, a more sophisticated and complex technique, also may be considered. Electromagnetic techniques are suited particularly to large piles that contain leachate plumes (for example, mine tailings) or for the detection of large discontinuities in a pile (for example, different types of wastes or the transition from a disposal area to background soils). For metallic objects, metal detectors and magnetometers are useful and relatively easy to use in the field.

5.5 *Potential Investigation Errors*:

5.5.1 Equipment selection can bias sampling results even if the equipment is used properly. Bias can result from the incompatibility of the materials that the sampling equipment is made of with the materials being sampled. For example, the equipment could alter the characteristics of the sample. Some equipment will bias against the collection of certain particles sizes, and some equipment cannot penetrate the waste pile adequately.

5.5.2 Equipment, use, and operation can introduce error (bias) into the characterization of a waste pile. Sampling errors typically are caused when certain particle sizes are excluded, when a segment of the waste pile is not sampled, or when a location outside the pile is inadvertently sampled.

5.5.3 When stratification, layering, or solid phasing occurs it may be necessary to obtain and analyze samples of each of the distinct phases separately to minimize sampling bias. Care should be taken when sampling stratified layers to minimize cross contamination. Proper decontamination procedures should be used for all sampling equipment (see Practice D 5088).

5.5.4 Statistical bias includes situations where the data are not normally distributed or when the sampling strategy does not allow the potential for every portion of the pile to be sampled.

6. Sampling Strategy

6.1 Developing a strategy for sampling a waste pile requires a thorough examination of the site evaluation factors listed in Section 5. The location and frequency of sampling (number of samples) should be outlined clearly in the sampling plan, as well as provisions for the use of special sampling equipment, access of heavy equipment to all areas of the pile, if necessary, and so forth.

6.1.1 *Representative Sampling*—The collection of a representative set of samples from a waste pile typically will be complicated by the presence of a number of the site evaluation factors **(2,3)**.

6.1.2 *Heterogeneous Wastes*—Waste piles may be homogeneous, for applied purposes, or may be quite heterogeneous in particle size and contaminant distribution. If the particle sizes of the material in the waste pile and the distribution of contaminants are known, or can be estimated, then less sampling may be necessary to define the properties of interest in the waste pile. An estimate of the variability in contaminant distribution may be based on process knowledge or determined by preliminary sampling **(4)**. The more heterogeneous the waste pile is, the greater the planning and sampling requirements.

6.1.3 *Strata and Hot Spots*—A waste pile also could contain strata that have less internal variation in physical properties or concentrations of chemical constituents than the remainder of the waste pile **(2,5)**. For example, strata may be present in a waste pile due to changes in the process that generated the waste, or if different processes at a facility contribute waste to different parts of the waste pile. A stratified sampling strategy would consider this situation by conducting independent sampling of each stratum, which could reduce the number of samples required. These strata could be in specific areas of the waste pile **(4)**. Also, hot spots may be present in the waste pile that are unique in composition **(2,5)**.

6.2 *Specific Sampling Strategies*:

6.2.1 Although the most appropriate method for evaluating material in waste piles is to sample at or immediately following the point of generation (for example, conveyor belt), most sampling problems involve existing or in-place waste piles. Therefore, the following discussion will focus on in-place waste piles. Sampling strategies available for waste piles include directed or judgmental sampling, simple random sampling, stratified random sampling, systematic grid sampling, and systematic sampling over time **(2,6)**. General concerns about the collection of a representative sample, the existence of potential heterogeneity in the waste pile, the presence of strata

within the waste pile, and the existence of distinct hot spots within the waste pile may also influence the selection of an appropriate sampling strategy and development of the sampling plan **(5)**. The following paragraphs provide an introduction to determining the appropriate number of samples to collect and the sampling strategies available for determining sample locations.

6.2.2 *Determining the Frequency or Number of Samples*—The frequency of sampling or the number of samples to collect typically will be based on several factors including the study objectives, properties of wastes in the pile, degree of confidence required, access to sampling points, and budgetary constraints. Practical guidance for determining the number of samples is included in Guide D 4687 and Refs **(2, 3)**.

6.2.3 *Directed Sampling*—Directed sampling (Fig. 1) is based on the judgment of the investigator and will not result necessarily in a sample that reflects the characteristics of the entire waste pile. Directed sampling also is called judgmental sampling, authoritative sampling, or nonprobability sampling. The experience of the investigator often is the basis for sample collection, and, depending on the study objectives, bias should be recognized as a potential problem. For preliminary screening investigations of a waste pile and for certain regulatory investigations, however, directed sampling may be appropriate. A directed sampling strategy could call for the collection of a composite sample from the surface area or the collection of discrete grabs at the surface of the pile (see Fig. 1). Directed sampling would typically focus on worst case conditions in a waste pile, for example, the most visually contaminated area or most recently generated waste.

6.2.4 *Simple Random Sampling*—Simple random sampling (Fig. 2) ensures that each element in the waste pile has an equal chance of being included in the sample **(2)**. This may be the method of choice when, for purposes of the investigation, the waste pile is randomly heterogeneous **(5)**. If the waste pile contains trends or patterns of contamination, a stratified random sampling or systematic grid sampling strategy would be more appropriate **(2)** (see 6.2.5 and 6.2.6).

6.2.4.1 A simple random approach could use a grid with random grids selected for sample collection (see Fig. 2). Note that the grid size could be selected based on the number of samples that are required (some guidance suggests having at least ten times the number of grids as samples required). Once the grid is overlaid and the sampling locations are selected, the decision must be made to collect either a discrete grab sample (surface), a composite of surface samples taken from predesignated locations within the grid cell (based on compass points), a vertical composite to a specified depth, or discrete

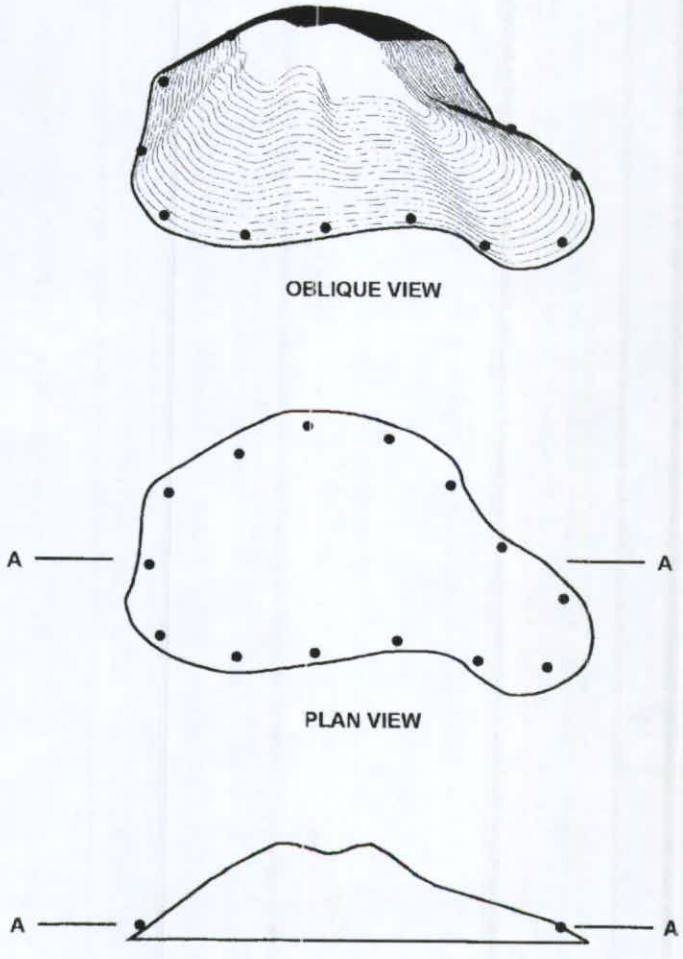


FIG. 1 Waste Pile Sampling Strategy—Directed Sampling

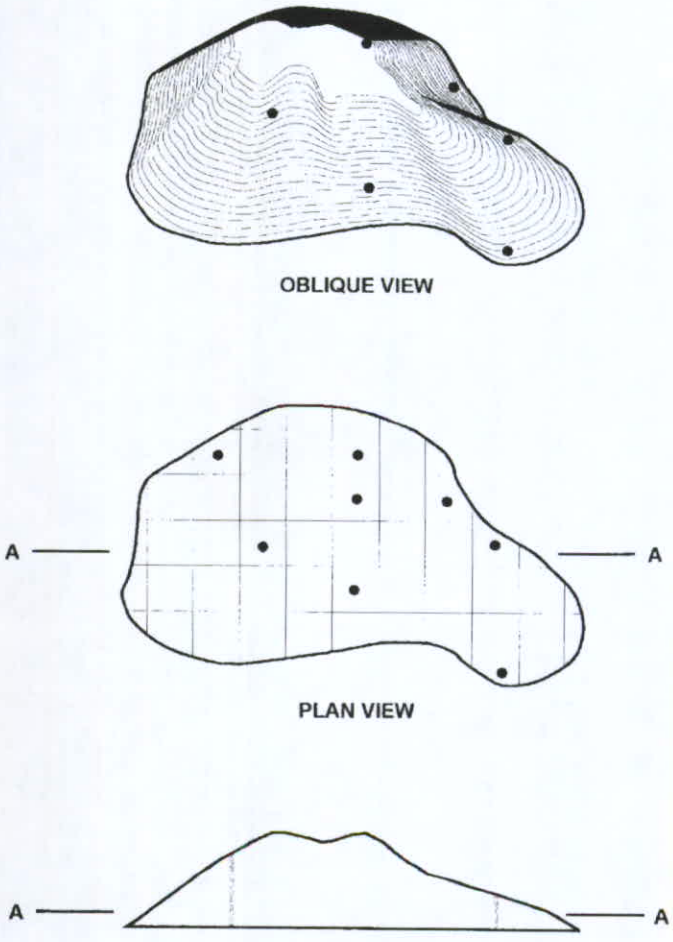


FIG. 2 Waste Pile Sampling Strategy—Simple Random Sampling

grab samples at specified depths. If discrete grab samples are desired at specified depths, they typically would be collected at the same location as the bore hole is advanced into the pile. Fig. 2 illustrates the collection of vertical composites at each of the randomly selected locations.

6.2.5 *Stratified Random Sampling*—Stratified random sampling (see Fig. 3) may be useful when distinct strata or homogeneous subgroups are identified within the waste pile **(2)**. The strata may be located in different areas of the pile or may be comprised of different layers (see Fig. 3). This approach is useful when the individual strata may be considered internally homogeneous or at least have less internal variation in what would otherwise be considered a heterogeneous waste pile **(2)**. Information on the waste pile usually is required to establish the location of individual strata unless process knowledge or changes in the composition of the material is obvious, such as with discoloration or with the type of waste. The grid may be utilized for sampling several horizontal layers if the strata are oriented horizontally **(4)**. A simple random sampling approach then is used within each stratum. The use of a stratified random sampling strategy may result in the collection of fewer samples. Fig. 3 illustrates a scenario where the number of samples collected in each stratum varies (plan view), and discrete grabs are collected in each boring at predesignated depths (side view).

6.2.6 *Systemic Grid Sampling*—Systematic grid sampling (see Fig. 4) involves the collection of samples at fixed intervals and is useful when the contamination is assumed to be distributed randomly **(2)**. This method also is commonly used with waste piles when estimating trends or patterns of contamination or when the objective is to locate hot spots. This approach may not be acceptable if the entire waste pile is not accessible or if the sampling grid locations become phased with variations in the distribution of contaminants within the waste pile **(6)**. It also may be useful for identifying the presence of strata within the pile. The grid and starting points should be laid out randomly over the waste pile, yet the method allows for rather easy location of exact sample locations by means of the grid (see Fig. 4). The same considerations discussed in 6.2.4 concerning the depth of each sample (surface, vertical composite, discrete grabs at depth) also should be considered. Fig. 4 illustrates the collection of vertical composites at each grid, which could be difficult and costly. Also note that the grid size typically would be adjusted according to the number of samples that are required.

6.2.7 *Systematic Sampling Over Time*—Systematic sampling over time at the point of generation is useful if the material is being sampled from a conveyor belt or being

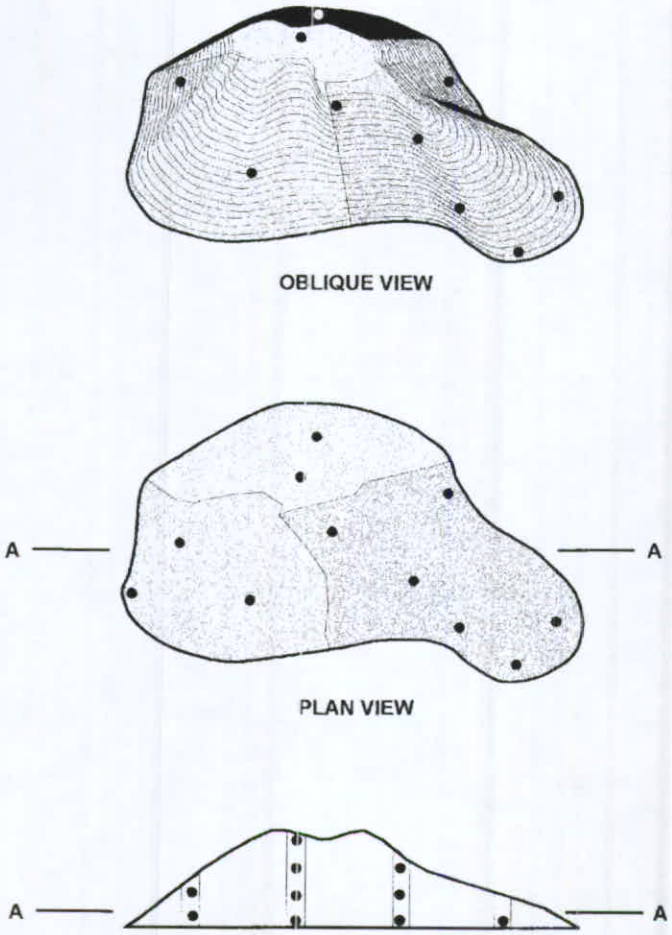


FIG. 3 Waste Pile Sampling Strategy—Stratified Random Sampling

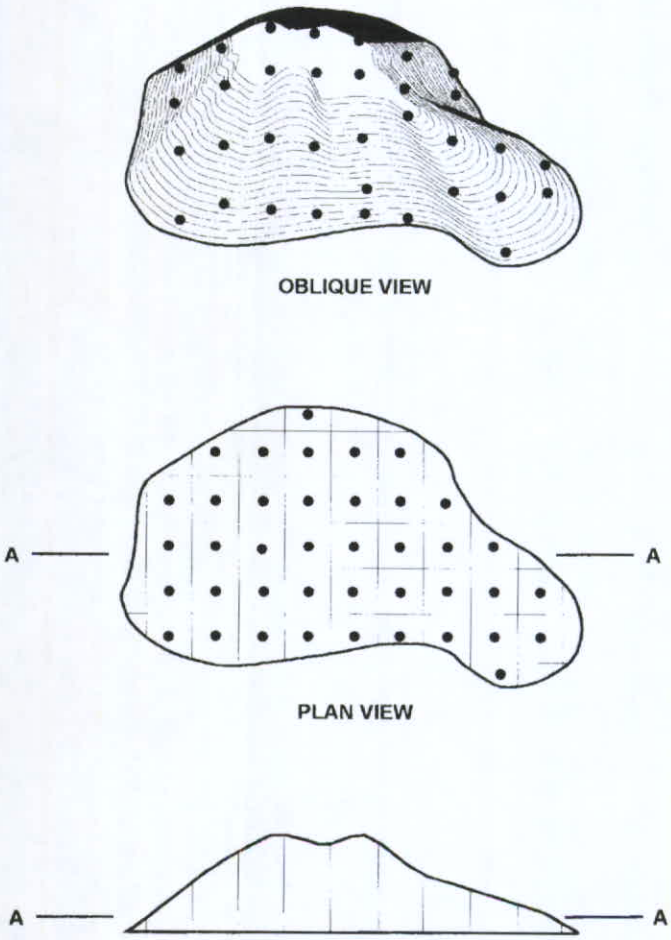


FIG. 4 Waste Pile Sampling Strategy—Systematic Grid Sampling

delivered by means of truck or pipeline to the waste pile. The sampling interval can be determined on a time basis, for example, every hour from a conveyor belt or pipeline discharge, or from every third truck load. The time between intervals is influenced by the factors addressed in 6.2.2.

6.2.8 *Alternative Approach*—In many cases, an objective of waste pile characterization is to determine the impact of the pile on the environment. At times this may be accomplished more easily by sampling the routes by which contaminants are dispersed from the pile than through direct sampling of the pile, especially for piles that are difficult to characterize. For example, ground water up-and-down gradient from the pile could be sampled to check for ground water contamination. The vadose zone below the pile also might be sampled to detect leachate (and potential ground water contamination) through soil sampling, vacuum lysimeters, or soil gas. Surface water and sediment in drainage channels down gradient from the pile also might be sampled. Surface soils, air samples, and contaminants deposited on vegetation can be used as indicators of atmospheric transport of contaminants from the pile, including both particulate and volatile materials. Such approaches will seldom replace pile sampling completely, but they may reduce the number of pile samples needed to make remedial action decisions (see Guide D 5730), also Refs **(7-9)**.

7. Selection of Sampling Equipment

7.1 Wastes in piles are often complex, multiphase mixtures of solids and semisolids. The wastes can range from powders to granules to large, heterogeneous solid fragments and can cover many acres in area. No single type of sampler can be used to collect representative samples of all types of waste from piles. Large, thick piles may require drill rigs to obtain samples from depth. The sampling of gases from within the pile requires other types of equipment. Table 2 lists typical waste types and the corresponding recommended samplers to use.

7.2 Sampling at depth from inside the pile may require heavy equipment designed for excavation or removal of soil or rock. Table 3 lists such equipment and its applications for sampling waste piles **(10)**.

7.3 Sampling equipment should be constructed of materials that are compatible with the waste to be sampled. Compatibility refers to the physical durability, lack of chemical reactivity with the waste, and lack of potential for contamination of the waste with analytes of concern. Typical materials of construction include stainless steel, plastic, and glass.

8. Data Use

8.1 The decisions that will be made based upon the data must be identified early in the planning process since these affect the approach to the problem and how the data will be evaluated. Decisions affecting waste classification, closure, and post-closure issues, are examples of the uses of the data. Methods to determine the volume of contaminated material in a pile or pile strata may be needed. Standard mathematical formulas for calculating the volume of a cone, cylinder, various prisms, and so forth, may be used.

8.2 *Statistical Considerations*:

8.2.1 Data quality assessment (DQA) methods are used to evaluate the data for any anomalies and to evaluate the assumptions for statistical evaluation. The statistician makes use of both subjective judgment (graphical analysis for identification of trends and anomalies) and statistical models and inference (for example, outlier detection, autocorrelation estimation) in the investigation of data for validity of the assumptions needed to make a statistical test. Classical statistical models assume that the samples collected from the population of interest are independent and have an identical probability distribution (that is, normal distribution with constant mean and variance). Random sampling is a method to ensure independence. The probability distributional assumptions are part of DQA that will determine if the classical statistical

TABLE 2 Sampling Devices Suitable for Waste Piles[A]

Location and Waste Type	Sampling Devices	ASTM Standard	Limitations
Subsurface			
Powdered, granular, or soil-like solids; sludges	split-barrel push coring device	D 1586 D 1587 D 4700 D 4823	Limited application for sampling moist and sticky solids, or particles with diameter 0.6 cm (0.25 in.) or more. Depth limitation of about 1 m.
	trier	D 5451	May not retain core sample of very dry granular materials. Not applicable to sampling solid wastes with particle diameter >½ the diameter of the sampling tube.
	auger	D 1452 D 4700	Does not collect undisturbed sample.
	thin-walled tube sampler	D 4823 D 4700	Collects relatively undisturbed core. Difficult to use on gravelly or rocky soils.
	drill rigs		Used for geoenvironmental exploration. To minimize sample contamination, avoid those using a water-based drilling fluid.
	soil gas samplers	D 5314	Used for volatile organic compounds.
Surface			
Powdered, granular, or soil-like solids; sludges	trowel or scoop	D 4700	Not applicable to sampling deeper than 8 cm (3 in.). Difficult to obtain reproducible mass of sample. May exclude certain particle sizes, especially large aggregates.
Slag	hammer/chisel Impact device		Changes particle size.

[A] This table is not all inclusive; other equipment may be used.

TABLE 3 Excavation and Removal Equipment for Waste Piles

Excavation and Removal Equipment	General Excavation	Ability to Excavate Hard and Compacted Material	Soil Hauling	Mixing of Solids, Soil	Spreading Cover	Site Maneuverability
Wheel or crawler Mounted backhoe	A[A]	A	B[B]/O[C]	A	A	A/B
Wheel or crawler Mounted front-end loader	A	A	A/B	A	A	A/B
Skid steer loader	A	B	B	A	B	A
Bulldozer	A	A	O	O	A	B

[A] A = Good choice. Equipment is fully capable of performing function listed.
[B] B = Secondary choice. Equipment is marginally capable of performing function listed.
[C] O = Not applicable or poor choice.

model is appropriate for the collected data. For directed sampling, the sampling is subjective and the sample results are typically judged on a qualitative basis.

8.2.2 Simple random sampling will provide an unbiased estimate of the average waste concentration, that is, an estimate of the mean. This unbiased estimate is independent of the geometry of the pile and of the distribution of the concentration of the contaminants, but it may not have the smallest variance. Other sampling designs, such as systematic grid sampling or stratified random sampling, may provide an average that has a smaller variance. If the waste pile has uneven topography, the calculation of the mean concentration of the pile should be a volume-weighted average, using core volume as the weighting factor to reduce the variance of the estimated mean.

8.2.2.1 For simple random sampling and systematic grid sampling designs, histogram and normal probability plots of the sample data can be used to judge if the data conform to normal distribution. If not, there are several alternatives. First, the classical statistical model may still be considered robust for the decision-making process. Second, a transformation of the data may approximate a normal distribution of the data. For example, logarithmic transformation will normalize data that are lognormal originally. If the data are lognormal, the question of whether to use the arithmetic mean or the geometric mean for decision-making purposes must be decided. Third, an alternative statistical model based on nonparametric methods, but which uses weaker assumptions, may be proposed to analyze the decision-making process. It may be advisable to consult a statistician.

8.2.2.2 For the stratified random sampling design, the test of normality is not straightforward. Generally, it requires a mathematical model to take out the strata effects first, then test for normality using the residuals. A statistician should be consulted.

8.2.2.3 In any of these cases, alternative consequences of the level of uncertainty can be calculated prior to collecting the data. These alternatives can be used by decision-makers to select the best strategy to minimize the environmental risks.

9. Keywords

9.1 piles; sampling; waste

APPENDIX

(Nonmandatory Information)

X1. WASTE PILE—A CASE HISTORY

X1.1 **Background**—The waste pile was generated by a facility that produces brass alloys from scrap metal. The byproduct from this operation was slag, which was generated in the recovery furnace. The slag was ground subsequently in a ball mill prior to being reintroduced into the recovery furnace. A large amount of the ground slag was disposed of in a waste pile which covered about one acre. No active management was occurring with the waste pile. No buried containers or extremely heterogenous material (unground slag) was suspected of being present in the waste pile based on facility records and interviews of personnel.

X1.1.1 Lead and cadmium were the constituents of concern based on process knowledge, and the possibility for the waste being hazardous was the regulatory consideration. The potential for off-site migration of contaminants was also an immediate concern, and this was considered in the development of the Phase 1 study design. Fig. X1.1 shows a site map of the facility and the slag pile. Fig. X1.2 shows a computer enhancement of the slag pile, and Fig. X1.3 shows a topographic view of the pile.

X1.2 Phase 1:

X1.2.1 *Objective*—The primary objective of the initial investigation was to determine if the slag in the waste pile classified as hazardous based on the concentration of lead and cadmium in a leach test. A secondary objective was to provide preliminary information on the potential migration and transport of contaminants from the waste pile off-site. The sampling plan for this initial investigation utilized a directed sampling strategy to provide a preliminary estimate of the lead concentration in the waste, the variability of contaminant concentrations in the pile, and the potential for leaching using the applicable leaching procedure mandated in regulations. Four composite samples were collected from the surface (0 to 15 cm or 0 to 6 in.) of the waste pile at locations within the four

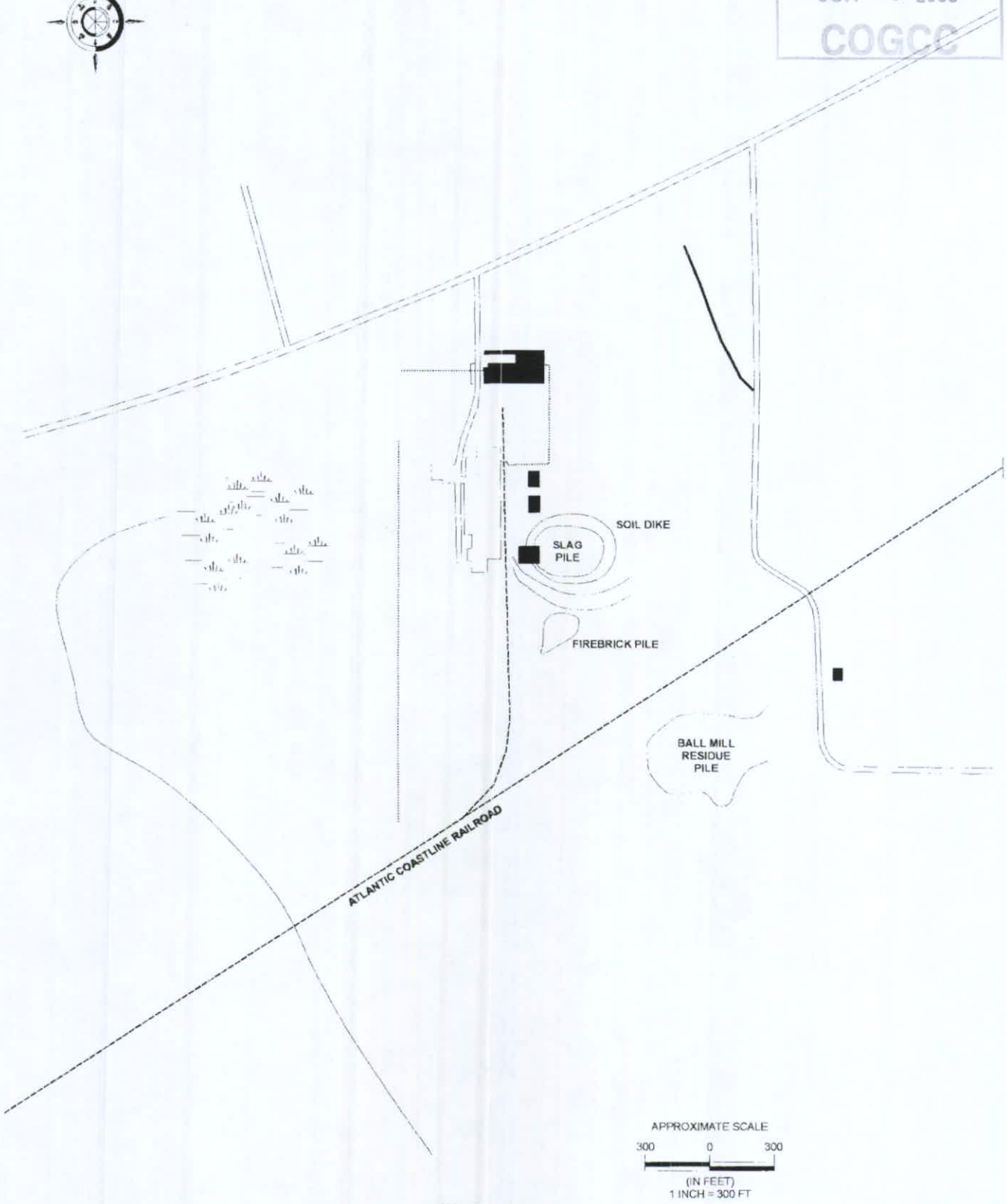


FIG. X1.1 Site Map

quadrants. The following environmental samples were also collected:

X1.2.1.1 Several soil samples in the vicinity of the waste pile,

X1.2.1.2 Sediment upstream and downstream in a stream which borders the facility,

X1.2.1.3 Sediment in a ditch which contained runoff from the pile, and

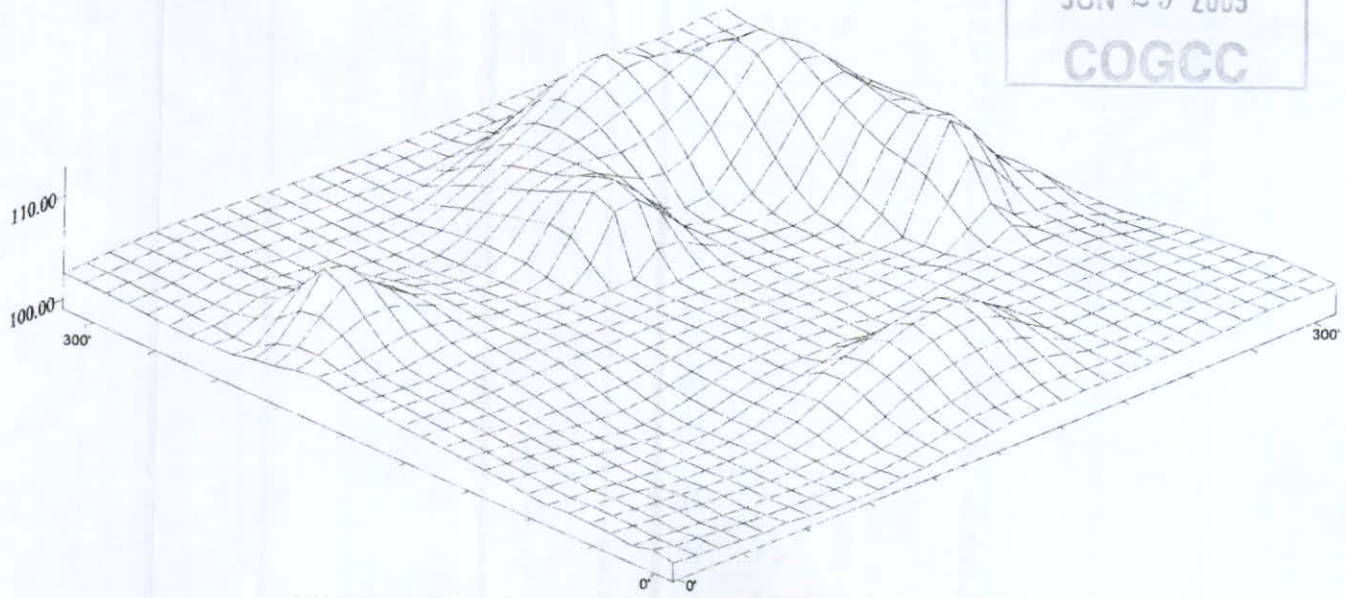


FIG. X1.2 Computer Enhancement of the Slag Pile (Front View) Scale 1:1:2

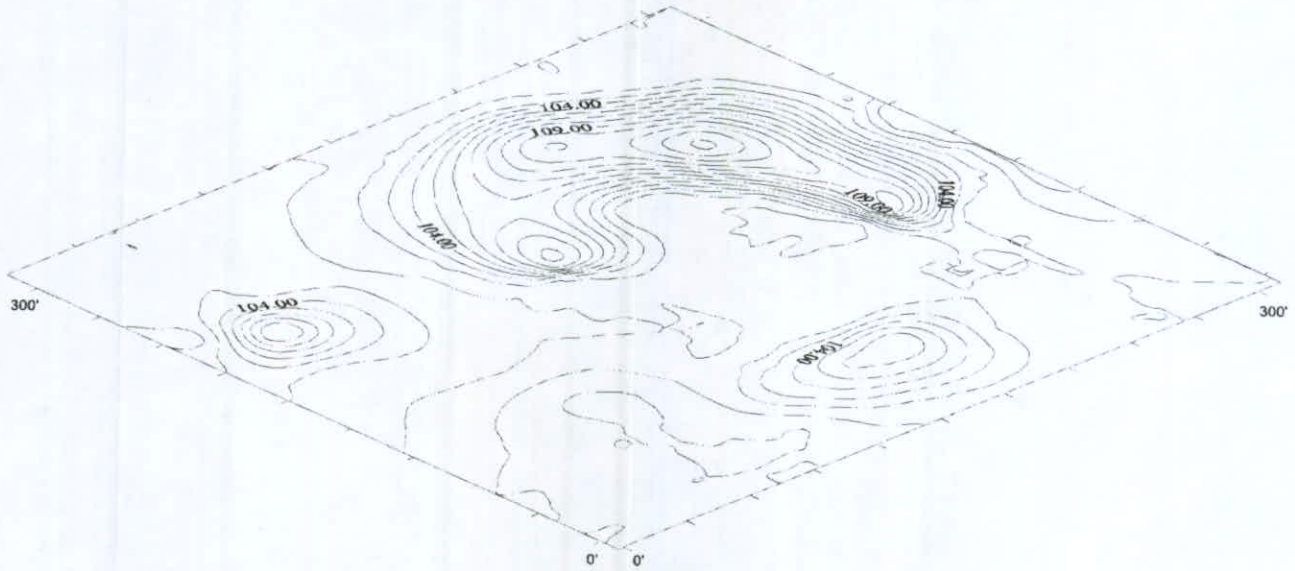


FIG. X1.3 Topographic View of the Slag Pile

X1.2.1.4 Two background soil samples.

X1.2.2 Fig. X1.4 shows the Phase 1 sampling locations within the slag pile, and Fig. X1.5 shows the same sampling locations on the topographic map of the pile.

X1.2.3 *Results*—Zinc, copper, cadmium, and lead were all elevated (compared to background) in the samples collected from the waste pile, and the concentrations did not appear to vary significantly between the samples. Since lead and cadmium are regulated constituents, a leach test was completed, and the lead results exceeded the regulatory level of 5 mg/L. Cadmium was just under the regulatory level of 1.0 mg/L. Lead and cadmium concentrations in the soil were 2 to 3 times above background, and the drainage ditch and downstream sediment sample also had elevated lead and cadmium levels.

X1.2.4 *Conclusion*— The waste pile contained slag that is hazardous for lead. The waste pile required further characterization to determine the variability in the pile. The presence of lead and cadmium in soils and the stream sediment downstream of the facility was confirmed and should be investigated further to determine the extent of contaminant transport.

X1.3 Phase 2:

X1.3.1 *Objective*—The objective is to characterize the waste pile further using a systematic grid sampling design. This design will delineate horizontal and vertical variability in lead and cadmium concentrations. The Phase 1 investigation also provided a good estimate of the anticipated variability in the waste pile. The number of samples required to characterize the waste pile adequately was calculated based on the average concentration, the anticipated variability, the regulatory level of concern, and the specified confidence interval. The grid size then was adjusted to accommodate the projection on the required number of samples. Composite samples were collected within each grid cell based on one center point and eight

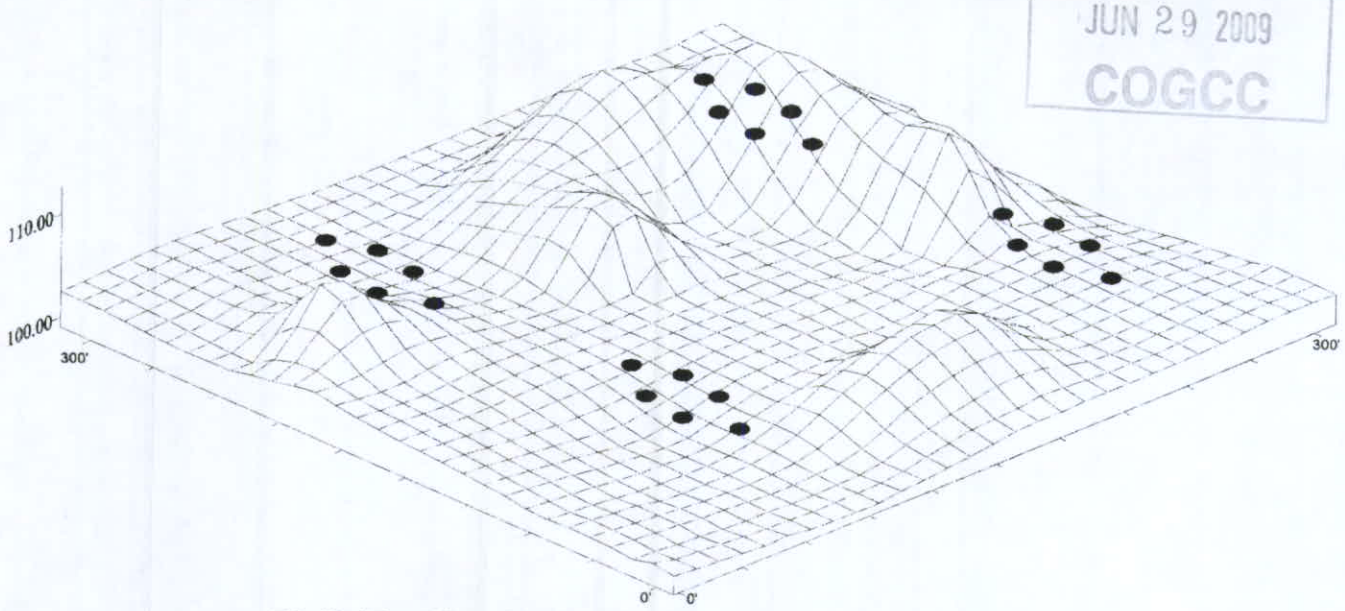


FIG. X1.4 Front View of the Slag Pile Showing Sampling Locations Scale 1:1:2

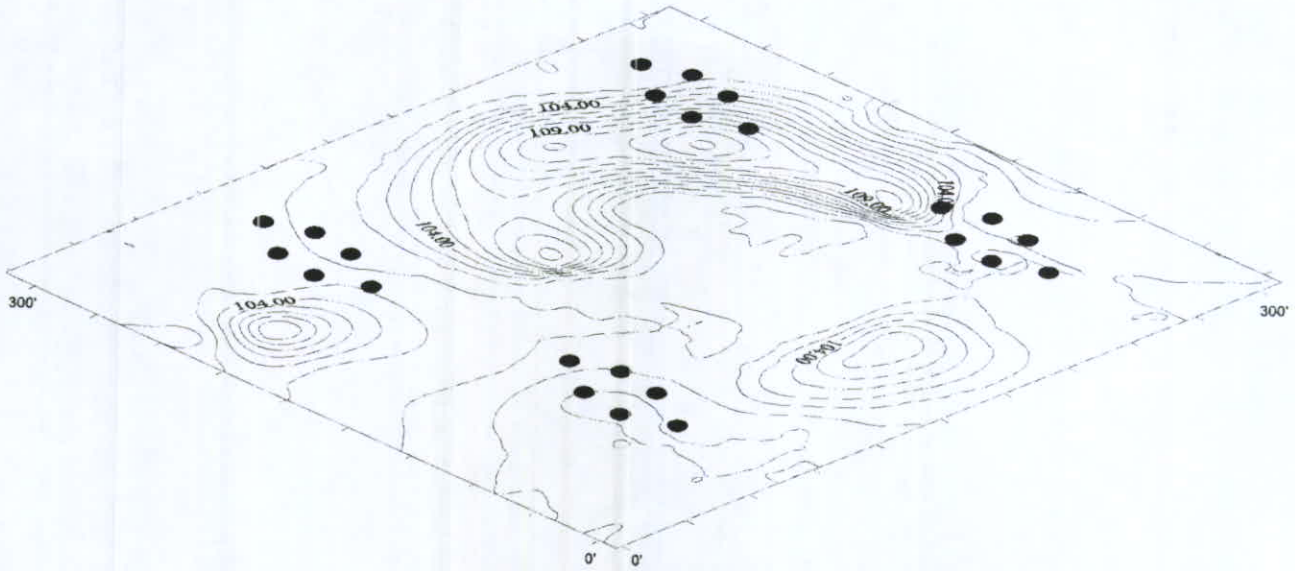


FIG. X1.5 Topographic View of the Slag Pile Showing Sampling Locations

points on the compass (45° intervals) equidistant from the center point. Ten percent of the grids were designated for vertical as well as surface (0 to 15 cm or 0 to 6 in.) sample collection. Additionally, 10 % of the grids were designated randomly for duplicate sampling (using a different aliquot pattern) to check the preliminary estimate on the variability. Additional environmental sampling was conducted but will not be covered in this discussion.

X1.3.2 *Results*—The results supported the initial Phase 1 investigation with lead consistently exceeding the regulatory level. Cadmium consistently was below the regulatory level.

X1.3.3 *Conclusion*— The waste pile was characteristic for lead and classified as hazardous according to the applicable regulations. There was no significant variability with depth, although several gradients were noticed across the grid based on lead concentration (scan) results.

X1.4 Phase 3:

X1.4.1 *Objective*—The objective is to determine thevolume of the waste pile in order to estimate both the disposal cost and the total amount of the civil penalty to be charged to the owner of the pile. The waste pile was surveyed using standard surveying techniques.

X1.4.2 *Results*—The results were used to calculate the volume using geometric principles. Also, a computer program was utilized which constructs contours based on the surveying information. The computer program was used as a check of the manual method, which produced a result that was 10 % higher in volume than the computer program.

X1.4.3 *Conclusion*— For penalty calculation purposes, the smaller estimate was utilized; however, the actual treatment and disposal costs could reflect the larger estimate.

REFERENCES

(1) U.S. Environmental Protection Agency (EPA). 1986. *Test Methods for Evaluating Solid Waste*; SW-846, 3rd Edition. EPA/530/SW-846 (NTIS PB88-239223); First update, 3rd edition. EPA/530/SW-846.3-1 (NTIS PB89-148076). Current edition and updates available on a subscription basis from U.S. Government Printing Office, Stock #955-001-00000-1.

(2) Gilbert, R. O., "Statistical Methods for Environmental Pollution Monitoring," Van Nostrand Reinhold Co., 1987.

(3) Ford, P. J., and Turina, P. J., *Characterization of Hazardous Waste Sites—A Methods Manual, Vol 1: Site Investigations*, EPA 600/4-84/075, (NTIS PB85-215960), 1985.

(4) U.S. Environmental Protection Agency (EPA), *Compendium of ERT Waste Sampling Procedures*, Section 5.0 Waste Pile Sampling, SOP No. 2017, EPA 540/P-91/008, OSWER Directive 9360.4-07, January 1991.

(5) Pitard, F., *Pierre Gy's Sampling Theory and Sampling Practice*, Vol 1: Heterogeneity and Sampling, Chemical and Rubber Company (CRC) Press, 1989.

(6) U.S. Environmental Protection Agency (EPA), *Characterizing Heterogeneous Wastes: Methods and Recommendations*, EPA 600/R-92/033, (NTIS PB92-216894) February 1992. [Also published as hardback Smoley Edition: Rupp and Joens (1993).]

(7) Keith, L., *Principles of Environmental Sampling*, Ed. ACS, 1988.

(8) U.S. Environmental Protection Agency (EPA). *Guidance Document on the Statistical Analysis of Ground Water Monitoring Data at RCRA Facilities*, Office of Solid Waste, 1993.

(9) McCoy and Associates, Inc., "Soil Sampling and Analysis—Practices and Pitfalls," Hazardous Waste Consultant, Vol 10, No. 6, Lakewood, CO, 1992.

(10) PEI Associates, 1991, *Survey of Materials-Handling Technologies Used at Hazardous Waste Sites*, EPA 540/2-91/010, June 1991. (NTIS PB91-186924), 225 pp.

Designation: D 6044 – 96

Standard Guide for Representative Sampling for Management of Waste and Contaminated Media[1]

This standard is issued under the fixed designation D 6044; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide covers the definition of representativeness in environmental sampling, identifies sources that can affect representativeness (especially bias), and describes the attributes that a representative sample or a representative set of samples should possess. For convenience, the term" representative sample" is used in this guide to denote both a representative sample and a representative set of samples, unless otherwise qualified in the text.

1.2 This guide outlines a process by which a representative sample may be obtained from a population. The purpose of the representative sample is to provide information about a statistical parameter(s) (such as mean) of the population regarding some characteristic(s) (such as concentration) of its constituent(s) (such as lead). This process includes the following stages: (*1*) minimization of sampling bias and optimization of precision while taking the physical samples, (*2*) minimization of measurement bias and optimization of precision when analyzing the physical samples to obtain data, and (*3*) minimization of statistical bias when making inference from the sample data to the population. While both bias and precision are covered in this guide, major emphasis is given to bias reduction.

1.3 This guide describes the attributes of a representative sample and presents a general methodology for obtaining representative samples. It does not, however, provide specific or comprehensive sampling procedures. It is the user's responsibility to ensure that proper and adequate procedures are used.

1.4 The assessment of the representativeness of a sample is not covered in this guide since it is not possible to ever know the true value of the population.

1.5 Since the purpose of each sampling event is unique, this guide does not attempt to give a step by step account of how to develop a sampling design that results in the collection of representative samples.

1.6 Appendix X1 contains two case studies, which discuss the factors for obtaining representative samples.

1.7 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 3370 Practices for Sampling Water from Closed Conduits[2]

D 4448 Guide for Sampling Groundwater Monitoring Wells[3]

D 4547 Practice for Sampling Waste and Soils for Volatile Organics[3]

D 4700 Guide for Soil Sampling from the Vadose Zone[4]

D 4823 Guide for Core-Sampling Submerged, Unconsolidated Sediments[5]

D 5088 Practice for Decontamination of Field Equipment Used at Nonradioactive Waste Sites[6]

D 5792 Practice for Generation of Environmental Data Related to Waste Management Activities: Development of Data Quality Objectives[3]

D 5956 Guide for Sampling Strategies for Heterogeneous Wastes[3]

D 6051 Guide for Composite Sampling and Field Subsampling for Environmental Waste Management Activities[3]

3. Terminology

3.1 *analytical unit, n*—the actual amount of the sample material analyzed in the laboratory.

3.2 *bias, n*—a systematic positive or negative deviation of the sample or estimated value from the true population value.

3.2.1 *Discussion*—This guide discusses three sources of bias—sampling bias, measurement bias, and statistical bias.

There is a sampling bias when the value inherent in the physical samples is systematically different from what is inherent in the population.

There is a measurement bias when the measurement process produces a sample value systematically different from that inherent in the sample itself, although the physical sample is

[1] This guide is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.01.01 on Planning for Sampling.

Current edition approved Nov. 10, 1996. Published January 1997.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 11.04.

[4] *Annual Book of ASTM Standards*, Vol 04.08.

[5] *Annual Book of ASTM Standards*, Vol 11.02.

[6] *Annual Book of ASTM Standards*, Vol 04.09.

itself unbiased. Measurement bias can also include any systematic difference between the original sample and the sample analyzed, when the analyzed sample may have been altered due to improper procedures such as improper sample preservation or preparation, or both.

There is a statistical bias when, in the absence of sampling bias and measurement bias, the statistical procedure produces a biased estimate of the population value.

Sampling bias is considered the most important factor affecting inference from the samples to the population.

3.3 *biased sampling, n*—the taking of a sample(s) with prior knowledge that the sampling result will be biased relative to the true value of the population.

3.3.1 *Discussion*—This is the taking of a sample(s) based on available information or knowledge, especially in terms of visible signs or knowledge of contamination. This kind of sampling is used to detect the presence of localized contamination or to identify the source of a contamination. The sampling results are not intended for generalization to the entire population. This is one form of authoritative sampling (see *judgment sampling*.)

3.4 *characteristic, n*—a property of items in a sample or population that can be measured, counted, or otherwise observed, such as viscosity, flash point, or concentration.

3.5 *composite sample, n*—a combination of two or more samples.

3.6 *constituent, n*— an element, component, or ingredient of the population.

3.6.1 *Discussion*—If a population contains several contaminants (such as acetone, lead, and chromium), these contaminants are called the constituents of the population.

3.7 *Data Quality Objectives, DQOs, n*—qualitative and quantitative statements derived from a DQO process describing the decision rules and the uncertainties of the decision(s) within the context of the problem(s) (see Practice D 5792).

3.8 *Data Quality Objective Process*—a quality management tool based on the Scientific Method and developed by the U.S. Environmental Protection Agency to facilitate the planning of environmental data collection activities. The DQO process enables planners to focus their planning efforts by specifying the use of data (the decision), the decision criteria (action level), and the decision maker's acceptable decision error rates. The products of the DQO process are the DQOs (see Practice D 5792).

3.9 *error, n*—the random or systematic deviation of the observed sample value from its true value (see *bias* and *sampling error*).

3.10 *heterogeneity, n*— the condition or degree of the population under which all items of the population are not identical with respect to the characteristic(s) of interest.

3.10.1 *Discussion*—Although the ultimate interest is in the statistical parameter such as the mean concentration of a constituent of the population, heterogeneity relates to the presence of differences in the characteristics (for example, concentration) of the units in the population. It is due to the presence of fundamental heterogeneity (or fundamental error)[7] in the population that sampling variance arises. Degree of sampling variance defines the degree of precision in estimating the population parameter using the sample data. The smaller the sampling variance is, the more precise the estimate is. See also *sampling error*.

3.11 *homogeneity, n*— the condition of the population under which all items of the population are identical with respect to the characteristic(s) of interest.

3.12 *judgment sampling, n*—taking of a sample(s) based on judgment that it will more or less represent the average condition of the population.

3.12.1 *Discussion*—The sampling location(s) is selected because it is judged to be representative of the average condition of the population. It can be effective when the population is relatively homogeneous or when the professional judgment is good. It may or may not introduce bias. It is a useful sampling approach when precision is not a concern. This is one form of authoritative sampling (see *biased sampling*.)

3.13 *population, n*— the totality of items or units of materials under consideration.

3.14 *representative sample, n*—a sample collected in such a manner that it reflects one or more characteristics of interest (as defined by the project objectives) of a population from which it is collected.

3.14.1 *Discussion*—A representative sample can be a single sample, a collection of samples, or one or more composite samples. A single sample can be representative only when the population is highly homogeneous.

3.15 *representative sampling, n*—the process of obtaining a representative sample or a representative set of samples.

3.16 *representative set of samples, n*—a set of samples that collectively reflect one or more characteristics of interest of a population from which they were collected. See *representative sample*.

3.17 *sample, n*—a portion of material that is taken for testing or for record purposes.

3.17.1 *Discussion*—Sample is a term with numerous meanings. The scientist collecting physical samples (for example, from a landfill, drum, or monitoring well) or analyzing samples considers a sample to be that unit of the population that was collected and placed in a container. A statistician considers a sample to be a subset of the population, and this subset may consist of one or more physical samples. To minimize confusion, the term *sample*, as used in this guide, is a reference to either a physical sample held in a sample container, or that portion of the population that is subjected to in situ measurements, or a set of physical samples. See *representative sample*.

3.17.1.1 The term *sample size* also means different things to the scientist and the statistician. To avoid confusion, terms such as sample mass/sample volume and number of samples are used instead of sample size.

3.18 *sampling error*— the systematic and random deviations of the sample value from that of the population. The

[7] Pitard, F. F., "*Pierre Gy's Sampling Theory and Sampling Practice: Heterogeneity, Sampling Correctness and Statistical Process Control*," 2nd ed., CRC Press Publishers, 1993.

systematic error is the *sampling bias*. The random error is the *sampling variance*.

3.18.1 *Discussion*—Before the physical samples are taken, potential sampling variance comes from the inherent population heterogeneity (sometimes called the "fundamental error," see *heterogeneity*). In the physical sampling stage, additional contributors to sampling variance include random errors in collecting the samples. After the samples are collected, another contributor is the random error in the measurement process. In each of these stages, systematic errors can occur as well, but they are the sources of bias, not sampling variance.

3.18.1.1 Sampling variance is often used to refer to the total variance from the various sources.

3.19 *stratum, n*—a subgroup of the population separated in space or time, or both, from the remainder of the population, being internally similar with respect to a target characteristic of interest, and different from adjacent strata of the population.

3.19.1 *Discussion*—A landfill may display spatially separated strata, such as old cells containing different wastes than new cells. A waste pipe may discharge into temporally separated strata of different constituents or concentrations, or both, if night-shift production varies from the day shift. In this guide, strata refer mostly to the stratification in the concentrations of the same constituent(s).

3.20 *subsample, n*— a portion of the original sample that is taken for testing or for record purposes.

4. Significance and Use

4.1 Representative samples are defined in the context of the study objectives.

4.2 This guide defines the meaning of a representative sample, as well as the attributes the sample(s) needs to have in order to provide a valid inference from the sample data to the population.

4.3 This guide also provides a process to identify the sources of error (both systematic and random) so that an effort can be made to control or minimize these errors. These sources include sampling error, measurement error, and statistical bias.

4.4 When the objective is limited to the taking of a representative (physical) sample or a representative set of (physical) samples, only potential sampling errors need to be considered. When the objective is to make an inference from the sample data to the population, additional measurement error and statistical bias need to be considered.

4.5 This guide does not apply to the cases where the taking of a nonrepresentative sample(s) is prescribed by the study objective. In that case, sampling approaches such as judgment sampling or biased sampling can be taken. These approaches are not within the scope of this guide.

4.6 Following this guide does not guarantee that representative samples will be obtained. But failure to follow this guide will likely result in obtaining sample data that are either biased or imprecise, or both. Following this guide should increase the level of confidence in making the inference from the sample data to the population.

4.7 This guide can be used in conjunction with the DQO process (see Practice D 5792).

4.8 This guide is intended for those who manage, design, and implement sampling and analytical plans for waste management and contaminated media.

5. Representative Samples

5.1 Samples are taken to infer about some statistical parameter(s) of the population regarding some characteristic(s) of its constituent(s) of interest. This is discussed in the following sections.

5.2 *Samples*—When a representative sample consists of a single physical sample, it is a sample that by itself reflects the characteristics of interest of the population. On the other hand, when a representative sample consists of a set of physical samples, the samples collectively reflect some characteristics of the population, though the samples individually may not be representative. In most cases, more than one physical sample is necessary to characterize the population, because the population in environmental sampling is usually heterogeneous.

5.3 *Constituents and Characteristics*—A population can possess many constituents, each with many characteristics. Usually it is only a subset of these constituents and characteristics that are of interest in the context of the stated problem. Therefore, samples need to be representative of the population only in terms of these constituent(s) and characteristic(s) of interest. A sampling plan needs to be designed accordingly.

5.4 *Parameters*—Similarly, samples need to be representative of the population only in the parameter(s) of interest. If the interest is only in estimating a parameter such as the population mean, then composite samples, when taken correctly, will not be biased and therefore constitute a representative sample (regarding bias) for that parameter. On the other hand, if the interest happens to be the estimation of the population variance (of individual sampling units), another parameter, then the variance of the composite samples is a biased estimate of the population variance and therefore is not representative. (It is to be noted that composite samples are often used to increase the precision in estimating the population mean and not to estimate the population variance of individual sampling units.)

5.5 *Population*—Since the samples are intended to be representative of a population, a population must be well defined, especially in its spatial or temporal boundaries, or both, according to the study objective.

5.6 *Representativeness*—The word "reflects" in this guide is used to mean a certain degree of low bias and high precision when comparing the sample value(s) to the population value(s). This is a broad definition of sample representativeness used in this guide. A narrower definition of representativeness is often used to mean simply the absence of bias.

5.6.1 *Bias*—Bias is sometimes mistakenly taken to be "a difference between the observed value of a physical sample and the true population value." The correct definition of bias is "a *systematic* (or consistent) difference between an observed (sample) value and the true population value." The word "systematic" here implies "on the average" over a set of physical samples, and not a single physical sample. Recall that sampling error consists of the random and systematic deviations of a sample (or estimated) value from that of the population. Although random deviations may occur on occasions due to imprecision in the sampling or measurement processes, or both, they balance out on the average and lead to no systematic difference between the sample (or estimated)

value and the population value. The random deviation corresponds to the observation of "a random difference between a single physical sample value and the true population value," which can be randomly positive or negative, and is not a bias. On the other hand, a persistent positive or negative difference is a systematic error and is a bias.

5.6.1.1 In order to assess bias, the true population value must be known. Since the true population value is rarely known, bias cannot be quantitatively assessed. However, this guide provides an approach to identifying the potential sources of bias and general considerations for controlling or minimizing these potential biases.

5.6.2 *Precision*—Precision has to do with the level of confidence in estimating the population value using the sample data. If the population is totally homogeneous and the measurement process is flawless, a single sample will provide a completely precise estimate of the population value. When the population is heterogeneous or the measurement process is not totally precise, or both, a larger number of samples will provide a more precise estimate than a smaller number of samples.

5.6.2.1 In the case of bias, the goal in environmental sampling is its absence. In the case of precision, the goal in sampling will depend on factors such as:

(1) The precision level needed to achieve the desired levels of decision errors, both false positive and false negative errors,

(2) If the true value is known or suspected to be well below the regulatory limit, high precision in the samples may not be needed, and

(3) The study budget.

5.6.2.2 Note that the second item applies similarly to bias as well.

5.6.2.3 Since bias, especially during sampling, can be very large when proper procedures are not followed, it is considered to be the first necessary condition for sample representativeness. On the other hand, precision can be more or less controlled, for example, by increasing the number of samples taken or by decreasing the sampling or measurement variabilities, or both.

5.6.2.4 The optimal number of samples to take to achieve a desired level of precision is typically an issue in optimization of a sampling plan. Therefore, the precision issue will be covered only briefly in this guide.

6. A Systematic Approach to Representative Sampling

6.1 A systematic approach is one that first defines the desired end result and then designs a process by which such a result can be obtained. In representative sampling, the desired end result is a sample or a set of samples that achieves desired levels of low bias and high precision.

6.2 A representative sampling process is described in Fig. 1. The key components in the process are described in this section.

6.3 *Study Objective*— A sampling plan is designed according to a defined problem or a stated study objective. The samples are then collected according to the sampling plan. Generally, the study objective dictates that representative samples be taken for the purpose of inference about the population. In that case, these samples will need to be collected according to this guide in order for the inference to be valid.

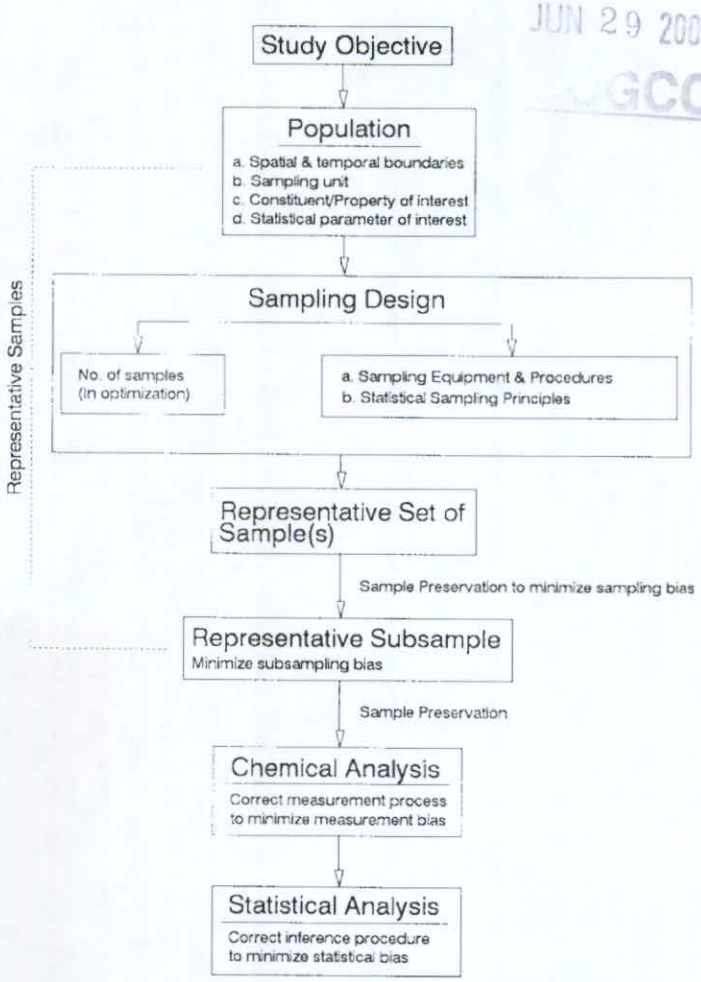


FIG. 1 A Systematic Approach to Representative Sampling

Occasionally, the objective is merely to detect the presence of a contaminant or to obtain a "worst case" sample. In that case, an authoritative sampling approach (biased sampling or judgment sampling) may be taken and this guide does not apply.

6.4 *Population*—A population consists of the totality of items or units of materials under consideration (Compilation of ASTM Standard Definitions, 1990). Its boundaries (spatial or temporal, or both) are defined according to the problem statement. This population is usually called the *target population*. In order to solve the stated problem, samples must be taken from the target population.

6.4.1 *Sampled Population*—Sometimes some parts of the target population may not be amenable to sampling due to factors such as accessibility. The boundaries of the target population actually sampled due to factors such as incomplete accessibility define the sampled population.

6.4.1.1 Although the samples taken from the sampled population may be representative of the sampled population, they may not be representative of the target population. In this case, potential exists that the samples taken from the sampled population may systematically deviate from the true value of the target population, thereby introducing bias when making inference from the samples to the target population.

6.4.1.2 When the boundaries of the target and sampled populations are not identical, some possible solutions are:

(1) The parties to the decision-making may agree that the

sampled population is a sufficient approximation to the target population. A sampling plan can then be designed to take representative samples from the" sampled population,"

(2) Qualifications on the sampling results are made based on the differences between the two populations. Some professional judgment may have to be exercised here, and

(3) Redefine the problem by considering what problem is solvable based on the observed differences between the two populations.

6.4.1.3 Occasionally, the sampled population is chosen on purpose to be different from the target population. For example, an investigator may be interested in the lead content in the sludge of a surface impoundment (the target population). He may decide to take samples from the sludge near the inlet (sampled population). Thus, the impoundment is the target population, while the inlet area is the sampled population. If the interest is in the target population, then this is an example of a biased sampling approach. On the other hand, the involved parties may decide to redefine the target population to include only the inlet area. Then the target population and the sampled population are identical. Again, the definition of a population depends on the problem statement.

6.4.1.4 In yet other circumstances, an investigator may take only a sample from the population. The following cases are possible:

(1) This one physical sample can be a sample from a biased sampling approach, for the purpose of detecting the presence of a contaminant or identifying the source of contamination. Therefore, it is not a representative sample due to its bias,

(2) This one physical sample can be a sample from judgment sampling, for the purpose of estimating the average condition of the population. Bias may or may not exist depending to some degree on the expertise of the sampler,

(3) This sample can be viewed as a population itself if the investigator is interested in the sample alone and a result from this sample is not to be used to infer to areas outside the sample. In this case, no bias exists, and

(4) If this sample is the composite of a few samples taken from the population, bias is likely to be minimal if the original samples are carefully taken.

6.4.2 *Decision Unit*— Often a population may be divided into several exposure units, cleanup units, or strata. If the environmental management decision is to be made for the entire population as a whole, representative samples can be obtained by designs such as a stratified random sampling design. Here the entire population is the decision unit. On the other hand, if the decision is to be made on each unit or stratum, then each unit or stratum is the decision unit. In this case, representative sample(s) need to be taken from each unit or stratum as if the unit or stratum is the population.

6.4.2.1 If the units or strata are relatively small in size or too numerous to take many samples per unit or stratum, composite sample(s) can be taken from each unit or stratum to increase precision without introducing bias. Alternatively, if precision is not a concern and there is sufficient professional expertise to avoid bias, a judgment sample(s) can be taken from each unit or stratum.

6.4.3 *Heterogeneity*— Heterogeneity is discussed in greater detail in Guide D 5956.

6.4.3.1 The degree and extent of population heterogeneity affect potential bias and precision in the samples. Population heterogeneity can be viewed at least in three different ways:

(1) When the population is heterogeneous in a random manner in only the distribution of the concentration, but not in the physical materials such as particle sizes, designs such as a simple random sampling design will generally produce samples with minimal bias. Its precision will then depend on the number of samples taken,

(2) When the population is randomly heterogeneous in concentrations due to large differences in the materials such as particle size, a simple random sampling design may still be effective if the sample volume/weight and sampling equipment are chosen to accommodate the largest particles and thereby prevent introduction of bias, and

(3) If the population is systematically heterogeneous, such as the presence of stratification in concentrations, then a simple random sampling design may not be biased, but will be less precise than an alternative design such as stratified random sampling.

6.4.3.2 Heterogeneity in the population affects the sampling variance. Sampling variance is a function of factors such as the population heterogeneity and the sample volume or weight. It is clear that the more heterogeneous the population is, the larger the inherent sampling variance is. It is also clear that samples of smaller volume or weight will have a higher sampling variance than those with greater volume or weight. However, the reduction in sampling variance due to increased volume or weight may eventually reach a limit. Determination of the optimal sample volume or weight is beyond the scope of this guide.[7]

6.4.3.3 The proper procedure is to first determine the right sample volume or weight, then to determine the number of samples needed for the chosen sample volume or weight.

6.4.3.4 Since stratification as a phenomenon of population heterogeneity is fairly common, it is discussed in greater details as follows.

6.4.4 *Stratification*— There are generally three types of stratification affecting sample representativeness. One is a stratification in the distribution of the contaminant concentration distribution alone. The second is a stratification in sampling materials or matrices alone. The third is a combination of both types. Stratification of any type is not a big problem regarding sample representativeness if each stratum is a decision unit. In that case, the units in a stratum are by definition relatively similar, apart from the random variations in concentrations. A simple random sampling design can be used to obtain representative samples (unbiased) for each stratum. The question of sample representativeness becomes more complicated when a decision is to be made over all the strata in the population.

6.4.4.1 *A Single Representative Sample in A Stratified Population*—When the objective is to obtain a single (physical) representative sample of all the strata, the sample must be a composite of individual samples from the strata (for example, at least one individual sample per stratum). Here the volumes or weights of the individual samples should be proportional to

the relative stratum sizes. The composite sample so obtained would be unbiased. However, since there is only one composite sample, precision of the composite sample cannot be estimated. If there are existing data on the precision of the individual samples in the strata, then the precision of the composite sample can be inferred from the precision of the individual samples by theoretical or empirical relationship. See Guide D 6051.

6.4.4.2 *A Representative Set of Samples*—When the population is stratified, a set of samples obtained by statistical designs such as stratified random sampling, where the number of samples to be taken from the strata are proportional to the relative sizes of the strata, is unbiased and more precise than a set of samples taken without considering the stratification.

6.4.5 *Parameter(s) of Interest*—This refers to the statistical parameter such as mean or variance of the population. It is often used with a characteristic such as concentration of a constituent(s) of the population. An example is the mean (parameter) concentration (characteristic) of lead (constituent). Another example is a population of mixture of silt-size calcium carbonate particles and large cobble-size particles of calcium carbonate. The interest here could be in the mean (parameter) particle size or chemical composition (characteristic) of calcium carbonate (constituent), depending on the study objective.

6.5 *Develop A Sampling Design*—The objectives of a sampling design are to minimize bias and achieve a desired level of precision. Precision and bias are an issue at various stages of the process of inferring from the samples to the population. The first stage is the act of obtaining the physical samples. The second stage is the act of analyzing the physical samples and translating them into data. The third stage is the use of statistical method to infer from the sample data to the population. At the first stage, the main concerns are sampling precision and bias. At the second stage, the concerns are measurement of precision and bias. At the third stage, the concern is statistical bias.

6.5.1 At the first stage of obtaining physical samples, the issues of precision and bias are sometimes grouped together as sampling design issues.

6.5.2 Bias at this stage is often called the sampling bias. Sampling bias is the systematic difference between the value inherent in the physical samples and the true population value. The word "inherent" is used because at this point the physical samples have not been translated into data.

6.5.3 The phrase "systematic difference" implies a persistent difference in long-term average or expectation, not the occasional random difference. Representative samples, apart from the issue of precision, are obtained when this long-term expected difference is zero or nearly so.

6.5.4 Since the true population value is typically not known, sampling bias cannot be assessed. However, efforts to minimize sampling bias can be attempted in at least two areas:

6.5.4.1 *Proper Statistical Sampling Design*—Statistical sampling design has to do with where and how samples are to be taken, where equal probability of selecting any of the units or items in the population is often a primary requirement. If the probability of selection is not equal, it is highly likely that bias will have been introduced into the physical samples so obtained. Depending on the layout of the population, designs such as simple random sampling or stratified random sampling can be used.

6.5.4.2 *Proper Sampling Procedures and Sampling Equipment*—This includes proper procedures for compositing, subsampling, sample preparation and preservation, and proper use of the chosen sampling equipment. This is a major source affecting precision and bias, especially bias.

6.5.5 In the case of precision, it can be controlled by things such as the number of samples taken, the use of composite samples, or more precise sampling techniques. Often, the number of samples to take is considered the key design issue. Some considerations regarding precision are:

6.5.5.1 If a population is relatively small compared to the sample mass/volume and the distribution of the characteristic of interest is random, it may be appropriate to collect a smaller number of samples by a random or systematic sampling approach, and

6.5.5.2 If a population is relatively large compared to sample mass/volume and the characteristic of interest is not randomly distributed (for example, stratified), a greater number of samples and a stratified sampling approach may be needed.

6.5.6 *Compositing*— Compositing is the combination of two or more individual physical samples into a single sample. It is often used to reduce the analytical costs, while maintaining or increasing precision relative to the individual samples (see Guide D 6051). Bias may or may not be introduced in compositing, depending on the study objective and the physical means of compositing. For example:

6.5.6.1 If the study calls for the estimation of the population variance (or standard deviation) of individual samples, then composite samples will surely underestimate the population variance, and

6.5.6.2 If the physical means of compositing changes the characteristics of the samples, then bias may have been introduced (unless such changes are part of the study design).

6.6 *Subsampling*— Sampling bias can be introduced in subsampling unless the same proper sampling protocol is followed as in taking samples from the original population.

6.6.1 *Discussion*—After the physical samples have been obtained and before they are measured, bias can be prevented by following proper sample preservation and preparation procedures. It is not important whether these procedures are viewed as part of the sampling process or as part of the measurement process. It is only important in following the proper procedures to prevent bias.

6.7 *Measurement of Precision and Bias*:

6.7.1 The measurement process, like the sampling process, also consists of a random error and a systematic error. The random errors define the degree of measurement precision, and the systematic error defines the degree of measurement bias.

6.7.2 Like sampling precision, measurement precision is controlled by things such as the number of replicate analyses performed per sample and refinements of the analytical method.

6.7.3 Measurement bias is a systematic difference between the sample value produced by the measurement process and the true population value, assuming that the physical samples are

unbiased before the analysis. The bias can come from contamination, loss or alteration of the sample materials, systematic errors in the measurement device, or from systematic human errors.

6.7.4 Often the measurement bias can be reasonably estimated in a laboratory testing setting when the true value is known. Laboratory samples spiked with known quantities of a chemical or certified reference standard can often be used to assess potential measurement bias. Minimization or adjustment for such estimable bias in the measurement process is essential in order to obtain data that are unbiased. When estimation of bias is not possible, care in measurement protocol and training is probably the only recourse.

6.7.4.1 *Discussion*—It is important to note that, when inferring from the sample data to the population, all the sources of imprecision, including sampling, subsampling, and measurement, need to be combined. The process of accumulating these sources of variation is sometimes called the "propagation of errors." The determination of the optimal numbers of samples, subsamples, and replicates are an issue of optimization and is not covered in this guide.

6.8 *Statistical Bias*— Statistical bias can result from an inappropriate sampling design or inappropriate estimation procedures, or both:

6.8.1 *Selection Bias from Sampling Design*—In the course of taking the sample, if the population units do not have the same probability of being selected, bias can be introduced. This bias can be prevented or minimized when a statistical sampling design is carefully selected, based on the study objective and the layout of the population. Some possible designs are the simple random sampling design and the stratified random sampling design.

6.8.2 *Estimation of Bias from Estimation Procedures*—This bias occurs when the expected value of the statistical estimator does not equal the true value.

6.8.2.1 Estimation bias can occur when the wrong statistical distribution of the data is used. For example, if the normal distribution assumption is used when the true data distribution is lognormal, the interval estimate of the mean concentration will be an biased estimate against the true interval. Thus, the expected value of the estimator will not be equal to the true value. To avoid this potential bias, it is wise to check the data distribution.

6.8.2.2 Estimation bias can also occur when a wrong statistical estimator is used. For example, if the sum of squares of deviations from the sample mean divided by the number of samples (that is, $\Sigma_{i=1,n}\ (x_i - \bar{x})^2/n$) is used to estimate the population variance, then this estimator is biased (its mathematical expected value is not equal to the population variance). If its denominator is modified to be $(n-1)$, then it is an unbiased estimator. For an unbiased statistical estimator, the reader is advised to check with a statistician.

7. Attributes of Representative Samples

7.1 The attributes of a representative (physical) sample or a representative set of (physical) samples can be described in the chronological order in which samples are taken. Note that these attributes apply only to how representative the physical samples are of the population. This corresponds to the upper half of Fig. 1.

7.2 *Design Considerations*:

7.2.1 A well-defined target population. The target population includes all the population units as determined from the stated problem.

7.2.2 The sampled population equals the target population in their spatial or temporal boundaries, or both. The sampled population consists of the population units directly available for measurement.[8]

7.2.2.1 When all the population units in the target population are accessible and directly available for measurement, then the sampled population is identical to the target population in its spatial or temporal boundaries, or both.

7.2.2.2 When not all the population units are directly available for measurement, then the inference from the sample is made to the sampled population, not the target population.

7.2.3 Size (weight or volume) of the sampling unit is well defined.

7.2.3.1 The population can be divided into various sizes (weight or volume) of population units. The size of the sampling unit is the size of the population unit most appropriate for the sampling purposes.

7.2.3.2 The appropriate size of the sample is determined by degree of heterogeneity of the materials to be sampled, such as particle size or shape.

7.3 *Sampling and Measurement Considerations*:

7.3.1 Correct sampling procedures are followed to minimize sampling bias.

7.3.1.1 Absence or minimization of bias is a key attribute of representative samples. Sampling bias can be minimized by following correct sampling procedures. Correct sampling procedures have two components.

(1) A sampling procedure that maximizes the potential of population units having equal probability of selection as sampled, and

(2) Correct sampling procedures. This includes the selection of appropriate equipment and proper use of that equipment.

7.3.2 Sample integrity is maintained during sampling and before chemical analysis.

7.3.3 If subsampling is performed, correct sampling procedures are followed to minimize sampling bias.

7.3.4 Sample preparation errors such as contamination and loss or alteration of constituents are prevented or minimized.

7.3.5 The samples, in the end, collectively reflect the target population within the context of the problem.

7.3.6 These attributes can be summarized into three broad categories:

7.3.6.1 A well-defined population,

7.3.6.2 Correct sampling procedures, and

7.3.6.3 Samples collected in the context of the stated problem.

[8] Gilbert, Richard O., *Statistical Methods for Environmental Pollution Monitoring*, Van Nostrand Reinholt Co., New York, NY 1987.

8. Practical Considerations

8.1 *Sampling Equipment*—The choice of appropriate sampling equipment can be crucial to the task of collecting a representative sample or a representative set of samples. Depending on the goals of the sampling activity, the sampling device used should minimize bias by having certain characteristics and capabilities, such as:

8.1.1 The ability to access and extract from every location in the target population,

8.1.2 The ability to collect a sample of proper shape,

8.1.3 The ability to collect a sufficient mass or volume of sample such that the distribution of particle sizes in the population are represented, and

8.1.4 The ability to collect a sample without the addition or loss of contaminants of interest.

8.2 *Equipment Design*— The improper design of sampling equipment may result in the collection of samples that are not representative of the population.

8.2.1 An example of equipment design influencing sampling results is samplers which exclude certain sized particles from a soil matrix or waste pile sample. The shape of some scoops may influence the distribution of particle sizes collected from a sample. Dredges used to collect river or estuarine sediments may also exclude certain sized particles, particularly the fines fraction which may contain a significant percentage of some contaminants such as polynuclear aromatic hydrocarbons (PAHs). Specific considerations in equipment design are outlined as follows.

8.2.1.1 *Sample Volume Capabilities*—Most sampling devices will provide adequate sample volume. However, the sampling equipment volumes should be compared to the volume necessary for all required analyses and the additional amount necessary for quality control (QC), split and repeat samples. Taking more than one aliquot to obtain an adequate sample volume can impact the representativeness of a sample.

8.2.1.2 *Compatibility*— It is important that sampling equipment, other equipment that may come in contact with samples (such as gloves, mixing pans, knives, spatulas, spoons, etc.) and sample containers be constructed of materials that are compatible with the matrices and analytes of interest. Incompatibility may result in the contamination of the sample and the degradation of the sampling equipment.

8.2.1.3 *Decontamination (see Practice D 5088) and Reuse*—Inadequate decontamination of sampling equipment can result in contamination of the sample and affects its representativeness. Due to design, some equipment is very difficult to adequately decontaminate. In some instances, it may even be desirable to either dispose of sampling equipment after use or to dedicate the equipment to a sampling point.

8.3 *Sampling Procedure*—Inappropriate use of sampling equipment is one of the largest sources of sampling bias. While it is beyond the scope of this guide to discuss it in depth, examples of how bias can be introduced during the sampling procedure are discussed in the following paragraphs. This guide does not provide comprehensive sampling procedures. It is the responsibility of the user to ensure that proper and adequate procedures are used.

8.3.1 *Ground Water*— For a more comprehensive discussion of sampling ground water refer to Guide D 4448.

8.3.1.1 Ground-water samples are usually collected through an in-place well, either temporarily or permanently installed. The following is a list of concerns that should be considered when collecting a ground-water sample.

(1) The well should be purged before collecting samples in order to clear the well of stagnant water which is not representative of aquifer conditions. Purging and sampling rates can cause chemical or physical changes in the water.

(2) Purging can be performed in such a way that the entire column of water is not removed. The best method for avoiding this situation is by lowering a pump or bailer into the top of the column of water.

(3) Bailing may stir up sediment in the well if conducted too vigorously. Increased turbidity can result in a higher metal content in the sample than in a non-turbid sample.

(4) Samples for volatile organic analysis should be collected in a fashion that minimizes agitation of the sample.

(5) Wells with in-place plumbing must also be purged. Samples should be collected immediately following purging. In order to collect a sample representative of ground water, samples should be collected before the water travels through any hoses or in-line treatment devices.

8.3.2 *Surface Water and Sediment*—For a more comprehensive discussion of sampling surface water and sediment, refer to Practice D 3370 and Guide D 4823. General and specific sampling concerns for collection of surface water and sediment samples are as follows:

8.3.2.1 *General Considerations*:

(1) Although bridges and piers may provide access for water and sediment sampling, these structures can also alter the nature of water flow and thus influence sediment deposition or scouring. Depending on the construction materials, these structures can contaminate samples collected in the immediate vicinity.

(2) Wading for water samples should be done with caution since bottom deposits are easily disturbed resulting in increased sediment in surface water samples and a removal of fines from the sediment sample.

8.3.2.2 *Rivers, Streams, and Creeks*:

(1) A good location to collect a vertically mixed surface water sample is immediately downstream of a riffle area. This location is also a likely area for deposition of sediment since the greatest deposition occurs where stream velocity slows down.

(2) Horizontal (cross-channel) mixing occurs in constrictions in the channel. However, this is a poor sediment sample collection area because of scouring.

(3) Surface water samples will be affected by point sources, such as tributaries and industrial and municipal effluents.

(4) Locations immediately upstream or downstream from the confluence of two streams or rivers may not immediately mix, and at times, due to possible back flow, can upset the normal flow patterns.

(5) Unless a stream is extremely turbulent, it is nearly impossible to measure the effect of a waste discharge or tributary immediately downstream of the source. Inflow frequently "hugs" the stream bank with very little cross-channel

mixing for some distance. Samples from quarter points across a stream may miss the wastes altogether and reflect only the quality of water upstream from the waste source. Samples collected within the portion of the cross section containing the wastes would indicate excessive effects of the wastes with respect to the river as a whole.

(6) When sampling tributaries, care should be exercised to avoid collecting water from the main stream that may flow into the mouth of the tributary on either the surface or bottom.

8.3.2.3 *Lakes, Ponds, and Impoundments*:

(1) Stratification of surface water is of greater concern in standing water. For example: A turbidity difference may occur vertically where a highly turbid river enters a lake, and each layer of the stratified water column may need to be considered. In addition, stratification may be caused by water temperature difference; cooler, heavier river water is beneath the warmer lake water.

(2) Dredges used to collect sediment samples can displace and miss lighter materials if allowed to drop freely.

(3) Core samplers used to sample vertical columns of sediment are useful when there is a need to know the history of sediment deposition. Coring devices also minimize the disturbance of fines at the sediment-water interface. However, coring devices can only sample a relatively small surface area. Depending on the core diameter, larger particles may be excluded and a single aliquot may not be sufficient for analytical needs.

8.3.3 *Soils*—For more detailed information, refer to Practice D 4547 and Guide D 4700. General areas of concern for sampling soils are as follows:

8.3.3.1 Soil samples for purgeable organic analyses should be collected with a minimum disturbance of the sample.

8.3.3.2 Samples for VOA analysis should not be mixed.

8.3.3.3 Two potential problems are associated with compositing soil samples. Low concentrations of contaminants present in individual aliquots may be diluted to the extent that the total composite concentration is below the minimum quantification limit. In addition, depending on the soil type, it can be very difficult to produce a homogeneous mixture.

8.3.4 *Waste*—Wastes referred to in this section include any liquid, solid, or sludge from pits, ponds, lagoons, waste piles, landfills, and open or closed containers such as drums, tank trucks, and storage tanks.

8.3.4.1 Any of these units may have multiple phases (floating solids, different density liquid phases, and sludge) and one or all of them may need to be sampled.

8.3.4.2 If sampling from access valves or ports on an open or closed container, care should be taken to be sure that the desired layer is sampled. For example, bottom sampling ports would allow only the heavier contents to be sampled while surface or top sampling would allow only sampling of the lighter layers.

8.4 *Subsampling (Field)*:

8.4.1 Different analyses require different types of bottles and preservation. For multiple analyses of the same waste stream, this may require subsampling in the field. Subsampling in the laboratory may require many of the same procedures; however, laboratory subsampling is beyond the scope of this guide.

8.4.1.1 Samples for organic analyses should always be taken from the first material collected. This minimizes loss of volatile organics during handling of the material.

8.4.1.2 If necessary, place the appropriate volume of material in a tray or other suitable container to composite. The volume is dependent on the needed analyses, and should be specified by the analytical laboratory.

8.4.1.3 Transfer the material into the required containers for analyses. If subsampling takes place, then the analytical sample is the final portion of the material subsampled from the original sampling unit and analyzed in the laboratory.

8.4.2 In subsampling, the original sampling unit can be considered as the population and the correct sampling procedures must be followed to ensure a representative subsample.

9. Keywords

9.1 bias; contaminated media; precision; representative; sample; waste; waste management

APPENDIX

(Nonmandatory Information)

X1. TWO CASE STUDIES OF REPRESENTATIVE SAMPLING

X1.1 Case Study One—Waste Pile Investigation

X1.1.1 *Background*— An industrial facility has managed recovery furnace slag and baghouse dust in a waste pile located on the site. No active management was occurring with the waste pile. No buried containers or extremely heterogenous material (debris) was suspected of being present in the waste pile based on facility records and interviews of personnel.

X1.1.1.1 Lead and cadmium were the constituents of concern based on process knowledge, and the possibility for the waste being hazardous by means of the Toxicity Characteristic (TC) Rule was the regulatory consideration. No preliminary information on the variability of lead and cadmium within the piles was available. The potential for off-site migration of contaminants by means of a drainage ditch that leads to a stream adjacent to the facility was an immediate concern.

X1.1.2 *Phase 1: Objective*—The primary objective of the initial investigation was to determine if the slag and baghouse dust in the waste piles were characteristic for lead via the Toxicity Characteristic Rule. A secondary objective was to provide preliminary information on potential migration and transport of contaminants from the waste piles off site.

X1.1.2.1 The sampling design for this initial investigation

utilized a judgmental sampling strategy to provide a preliminary estimate of the lead and cadmium concentrations in the waste pile, the variability of contaminant concentrations in the pile, and the potential for leaching using the TCLP. Four areal composite samples were collected from the surface (0 to 6 in.) at the four quadrants of the waste pile. Borings were completed at the center of each area that was sampled on the surface. Each four-foot interval was analyzed to assess vertical variability.

X1.1.2.2 The following environmental samples were also collected using a judgmental approach:

(1) Several soil samples in the vicinity of the waste pile,

(2) Sediment upstream and downstream in a stream that borders the facility,

(3) Sediment in a ditch which contained run-off from the pile, and

(4) Two background soil samples.

X1.1.2.3 *Results*—Zinc, copper, cadmium, and lead were all elevated (compared to background) in the samples collected from the waste piles. Since lead and cadmium are TC Rule constituents, the TCLP was completed, and the lead results exceeded the regulatory level of 5 mg/L. Cadmium was just under the regulatory level of 1.0 mg/L. Lead and cadmium concentrations in the soil near the waste piles were 2 to 3 times above background, and the drainage ditch and downstream sediment sample also had elevated lead and cadmium levels.

X1.1.2.4 *Conclusion*— The waste piles contain slag and baghouse dust that is hazardous for lead. The waste pile requires further characterization to determine the variability in the pile. The presence of lead and cadmium in soils and the stream sediment downstream of the facility was confirmed and should be further investigated to determine the extent of contaminant transport.

X1.1.3 *Phase 2: Objective*—The sampling design utilized a systematic grid approach. This design will delineate horizontal and vertical variability in lead and cadmium concentrations. The Phase 1 investigation also provided a good estimate of the anticipated variability in the waste pile.

X1.1.3.1 The number of samples required to adequately characterize the waste pile was calculated based on the anticipated variability, the regulatory level of concern, and the specified confidence interval. The grid sizes were then adjusted to accommodate the projection on the required number of samples. Composite samples were collected within each grid cell based on one center point and eight points on the compass (45 deg intervals) equidistant from the center point.

X1.1.3.2 Twenty percent of the grids were designated for vertical characterization (at the grid center) at four-foot intervals, as well as surface (0 to 6 in.) sample collection. Additionally, ten percent of the grids were randomly designated for duplicate sampling (using a different aliquot pattern within the cell) to check the preliminary estimate on the variability.

X1.1.3.3 Additional environmental sampling was conducted that included a systematic sampling design for the stream adjacent to the facility with sediment samples collected at 100-ft intervals. A systematic approach was also used for the drainage ditch (50-ft intervals), with judgmental samples being collected at any location where visible staining was observed.

X1.1.3.4 *Results*—The results supported the initial investigation with lead consistently exceeding the TC Rule regulatory level; cadium was consistently below the regulatory level. Vertical differences in the lead and cadmium concentrations were not significant. Lead and cadmium were detected at elevated concentrations (relative to background) in the adjacent stream at a point downstream of the confluence with the drainage ditch.

X1.1.3.5 *Conclusion*— The waste pile was characteristic for lead and subject to Subtitle C of RCRA. There was no significant variability with depth, although several gradients were noticed across the grid (horizontally) based on lead concentration (scan) results.

X1.2 Case Study Two—Drum Sampling

X1.2.1 *Background*— An industry has two areas where drums of waste have been stored. One area is a warehouse adjacent to an off-line plating process that contains less than 25 drums (55 gal). The drums have manufacturers' labels indicating they contain an acid solution, and all of the drums are similar in appearance. A second area is a covered shed that has an estimated 100 drums from a variety of processes, several of which are no longer in use at the facility. Information on the content of these drums is not available.

X1.2.2 *Objective*—The objective of the initial investigation was to survey both of the storage areas for safety purposes, assess and record information on the drums, and open drums that were candidates for screening. All drums that were opened were surveyed using an organic vapor analyzer (PID, FID), pH paper, halogen detector, cyanide detector, and radiation meter.

X1.2.2.1 A judgmental sampling design was utilized in the warehouse where the anticipated variability was low. Based on the site screening (pH measurement), six samples were collected for pH analysis from the warehouse.

X1.2.2.2 The drums in the shed were screened in a similar fashion. A variety of results were obtained which included elevated pH, high organic vapor readings, and so forth. A simple random sampling design was used which called for the collection of 15 samples, with five from each major group of drums based on the screening (five corrosives, five potential ignitables with no halogens, and five with elevated halogen readings).

X1.2.2.3 *Results*—The warehouse samples were all corrosive with pH values from 1 to 2 S.U. The shed samples resulted in the collection of five corrosive wastes, three that were both ignitable and characteristic for non-halogenated TC Rule constituents, and two that were ignitable and characteristic for halogenated constituents. In summary, of the 15 drums sampled, 10 contained hazardous waste.

X1.2.2.4 *Conclusions*— All of the drums in the warehouse are subject to Subtitle C of RCRA. The drums in the shed require further assessment due to the fact that several of those sampled did not contain hazardous waste.

Attachment III

COGCC Standards

TABLE 3: COGCC CONCENTRATION LEVELS

(SEE NOTES ON FOLLOWING PAGE)

Contaminant of Concern	Concentrations
Organic Compounds in Soil	
TPH (total volatile and extractable petroleum hydrocarbons)	500 mg/kg
Benzene	0.17 mg/kg[2]
Toluene	85 mg/kg2
Ethylbenzene	100 mg/kg2
Xylenes (total	175 mg/kg2
Acenaphthene	1,000 mg/kg2
Anthracene	1,000 mg/kg2
Benzo(A)anthracene	0.22 mg/kg2
Benzo(B)fluoranthene	0.22 mg/kg2
Benzo(K)fluoranthene	2.2 mg/kg2
Benzo(A)pyrene	0.022 mg/kg2
Chrysene	22 mg/kg2
Dibenzo(A,H)anthracene	0.022 mg/kg2
Fluoranthene	1,000 mg/kg2
Fluorene	1,000 mg/kg2
Indeno(1,2,3,C,D)pyrene	0.22 mg/kg2
Napthalene	23 mg/kg2
Pyrene	1,000 mg/kg2
Organic Compounds in Ground Water	
Benzene	5 µg/l[3]
Toluene	560 to 1,000 µg/l[3]
Ethylbenzene	700 µg/l[3]
Xylenes (Total)	1,400 to 10,000 µg/l[3,4]
Inorganics in Soils	
Electrical Conductivity (EC)	<4 mmhos/cm or 2x background
Sodium Adsorption Ratio (SAR)	<12[5]
pH	6-9
Inorganics in Ground Water	
Total Dissolved Solids (TDS)	<1.25 x background[3]
Chlorides	<1.25 x background[3]
Sulfates	<1.25 x background[3]
Metals in Soils	
Arsenic	0.39 mg/kg[2]
Barium (LDNR True Total Barium)	15,000 mg/kg[2]
Boron (Hot Water Soluble)	2 mg/l[3]
Cadmium	70 mg/kg[3,6]
Chromium (III)	120,000 mg/kg[2]
Chromium (VI)	23 mg/kg[2,6]
Copper	3,100 mg/kg[2]
Lead (inorganic)	400 mg/kg[2]
Mercury	23 mg/kg[2]
Nickel (soluble salts)	1,600 mg/kg[2,6]
Selenium	390 mg/kg[2,6]
Silver	390 mg/kg[2]
Zinc	23,000 mg/kg[2,6]
Liquid Hydrocarbons in Soils and Ground Water	
Liquid hydrocarbons including condensate and oil	Below detection level

Notes to Table 3

COGCC recommends that the latest version of EPA SW 846 analytical methods be used where possible and that analyses of samples be performed by laboratories that maintain state or national accreditation programs.

[1] Consideration shall be given to background levels in native soils and ground water.

[2] Concentrations taken from CDPHE-HMWMD Table 1 Colorado Soil Evaluation Values (December 2007).

[3] Concentrations taken from CDPHE-WQCC Regulation 41 - The Basic Standards for Ground Water.

[4] For this range of standards, the first number in the range is a strictly health-based value, based on the WQCC's established methodology for human health-based standards. The second number in the range is a maximum contaminant level (MCL), established under the Federal Safe Drinking Water Act which has been determined to be an acceptable level of this chemical in public water supplies, taking treatability and laboratory detection limits into account. The WQCC intends that control requirements for this chemical be implemented to attain a level of ambient water quality that is at least equal to the first number in the range except as follows: 1) where ground water quality exceeds the first number in the range due to a release of contaminants that occurred prior to September 14, 2004 (regardless of the date of discovery or subsequent migration of such contaminants) clean-up levels for the entire contaminant plume shall be no more restrictive than the second number in the range or the ground water quality resulting from such release, whichever is more protective, and 2) whenever the WQCC has adopted alternative, site-specific standards for the chemical, the site-specific standards shall apply instead of these statewide standards.

[5] Analysis by USDA Agricultural Handbook 60 method (20B) with soluble cations determined by method (2). Method (20B) = estimation of exchangeable sodium percentage and exchangeable potassium percentage from soluble cations. Method (2) = saturated paste method (note: each analysis requires a unique sample of at least 500 grams). If soils are saturated, USDA Agricultural Handbook 60 with soluble cations determined by method (3A) saturation extraction method.

[6] The table value for these inorganic constituents is taken from the CDPHE-HMWMD Table 1 Colorado Soil Evaluation Values (December 2007). However, because these values are high, it is possible that site-specific geochemical conditions may exist that could allow these constituents to migrate into ground water at levels exceeding ground water standards even though the concentrations are below the table values. Therefore, when these constituents are present as contaminants, a secondary evaluation of their leachability must be performed to ensure ground water protection.

Attachment IV

ASTM Guidance Documents

Designation: D 6009 – 96 (Reapproved 2001)

Standard Guide for Sampling Waste Piles[1]

This standard is issued under the fixed designation D 6009; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide provides guidance for obtaining representative samples from waste piles. Guidance is provided for site evaluation, sampling design, selection of equipment, and data interpretation.

1.2 Waste piles include areas used primarily for waste storage or disposal, including above-grade dry land disposal units. This guide can be applied to sampling municipal waste piles.

1.3 This guide addresses how the choice of sampling design and sampling methods depends on specific features of the pile.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1452 Practice for Soil Investigation and Sampling by Auger Borings[2]

D 1586 Test Method for Penetration Test and Split-Barrel Sampling of Soils[2]

D 1587 Practice for Thin-Walled Tube Geotechnical Sampling of Soils[2]

D 4547 Practice for Sampling Waste and Soils for Volatile Organics[3]

D 4687 Guide for General Planning of Waste Sampling[3]

D 4700 Guide for Soil Sampling from the Vadose Zone[2]

D 4823 Guide for Core-Sampling Submerged, Unconsolidated Sediments[4]

D 5088 Practice for Decontamination of Field Equipment Used at Nonradioactive Sites[5]

D 5314 Guide for Soil Gas Monitoring in the Vadose Zone[5]

D 5451 Practice for Sampling Using a Trier Sampler[3]

D 5518 Guide for Acquisition of File Aerial Photography and Imagery for Establishing Historic Site-Use and Surficial Conditions[5]

D 5730 Guide to Site Characterization for Environmental Purposes with Emphasis on Soil, Rock, the Vadose Zone and Ground Water[5]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *hot spots*—strata that contain high concentrations of the characteristic of interest and are relatively small in size when compared with the total size of the materials being sampled.

3.1.2 *representative sample*—a sample collected such that it reflects one or more characteristics of interest (as defined by the project objectives) of the population from which it was collected.

3.1.2.1 *Discussion*—A representative sample can be a single sample, a set of samples, or one or more composite samples.

3.1.3 *waste pile*—unconfined storage of solid materials in an area of distinct boundaries, above grade and usually uncovered. This includes the following:

3.1.3.1 *chemical manufacturing waste pile*—a pile consisting primarily of discarded chemical products (whether marketable or not), by-products, radioactive wastes, or used or unused feedstocks.

3.1.3.2 *scrap metal or junk pile*—a pile consisting primarily of scrap metal or discarded durable goods such as appliances, automobiles, auto parts, or batteries.

3.1.3.3 *trash pile*—a pile of waste materials from municipal sources, consisting primarily of paper, garbage, or discarded nondurable goods that contain or have contained hazardous substances. It does not include waste destined for recyclers.

4. Significance and Use

4.1 This guide is intended to provide guidance for sampling waste piles. It can be used to obtain samples for waste characterization related to use, treatment, or disposal; to monitor an active pile; to prepare for closure of the waste pile; or to investigate the contents of an abandoned pile.

4.2 Techniques used to sample include both in-place evaluations of the pile and physically removing a sample. In-place evaluations include techniques such as remote sensing, on-site gas analysis, and permeability.

4.3 Sampling strategy for waste piles is dependent on the following:

4.3.1 Project objectives including acceptable levels of error when making decisions;

---

[1] This guide is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.01.01 on Planning for Sampling.

Current edition approved Oct. 10, 1996. Published December 1996.

[2] *Annual Book of ASTM Standards*, Vol 04.08.

[3] *Annual Book of ASTM Standards*, Vol 11.04.

[4] *Annual Book of ASTM Standards*, Vol 11.02.

[5] *Annual Book of ASTM Standards*, Vol 04.09.

4.3.2 Physical characteristics of the pile, such as its size and configuration, access to all parts of it, and the stability of the pile;

4.3.3 Process that generated the waste and the waste characteristics, such as hazardous chemical or physical properties, whether the waste consists of sludges, dry powders or granules, and the heterogeneity of the wastes;

4.3.4 History of the pile, including dates of generation, methods of handling and transport, and current management methods;

4.3.5 Regulatory considerations, such as regulatory classification and characterization data;

4.3.6 Limits and bias of sampling methods, including bias that may be introduced by waste heterogeneity, sampling design, and sampling equipment.

4.4 It is recommended that this guide be used in conjunction with Guide D 4687, which addresses sampling design, quality assurance, general sampling considerations, preservation and containerization, cleaning equipment, packaging, and chain of custody.

4.5 A case history of the investigation of a waste pile is included in Appendix X1.

5. Site Evaluation

5.1 Site evaluations are performed to assist in designing the most appropriate sampling strategy. An evaluation may consist of on-site surveys and inspections, as well as a review of historical data. Nonintrusive geophysical and remote sensing methods are particularly useful at this stage of the investigation (see Guide D 5518). Table 1 summarizes the effects that various factors associated with the waste pile, such as the history of how the pile was generated, have upon the strategy and design of the sampling plan. The strategic and design considerations are discussed as well.

5.2 *Generation History*—The waste pile may have been created over an extended time period. A remote sensing method that is very useful in establishing historical management practices for waste piles is aerial imagery. Aerial photographs are widely available and may be used to determine the history of a waste pile, sources of waste, and the presence and distribution of different strata. Satellite imagery could be used for larger waste piles.

TABLE 1 Strategy Factors

Waste Pile Factors	Strategic Considerations	Design Considerations
Generation history	Date of generation	Analysis required
	Types of processes	Location of samples
	Characteristics by process	
	Delivery method	
	Current management	
	Regulatory considerations	
Physical characteristics of pile:	Physical variability of pile	Number of samples
– size	Access	Location of samples
– shape	Safety	Equipment selection
– stability		
Waste characteristics	Constituents present	Number of samples
	Constituent distribution	Analysis required
	Heterogeneity	Location of samples
	– physical variability	Representative samples
	– chemical variability	Equipment selection

5.2.1 The date of generation could be important with respect to the types of processes that generated the waste, the characteristics of the waste, the distribution of the constituents, and regulatory concerns.

5.2.2 The type of process that generated the waste will determine the types of constituents that may be present in the waste pile. Chemical variability will influence the number of samples that are required to characterize the waste pile unless a directed (biased) sampling approach is acceptable.

5.2.3 The delivery method of the material to the waste pile could influence the concentrations of the constituents, affect the overall shape of the pile, or create physical dissimilarity within the waste pile through sorting by particle size or density.

5.2.4 If the pile is under current management and use, the variability in constituent types and concentrations may be affected. Current management activities also may influence the regulatory status of the waste pile.

5.2.5 Regulatory considerations will typically focus on waste identification questions, in other words is the material a solid waste that should be regulated and managed as a hazardous waste **(1)**.[6] This may involve a limited, directed sampling approach, particularly if a regulatory agency is conducting the investigation. A more comprehensive sampling design may be required to determine if the waste classifies as hazardous. Remediation efforts and questions regarding permits may focus on characterizing the entire pile, possibly as the removal of material is occurring. It should be noted that concentrations of contaminants near regulatory levels may increase the number of samples required to meet the objectives of the investigation. These regulatory levels could be those established to determine if a waste is hazardous, or "cleanup" levels set for a removal or remediation.

5.3 *Physical Characteristics of Pile*— Several physical characteristics of the waste pile must be considered during the site evaluation. Variability in size, shape, and stability of the pile affects access to it to obtain samples as well as safety considerations. Physical variability will influence the number of samples that are required to characterize the waste pile unless a directed (biased) sampling approach is considered to be acceptable. Techniques that might be used include resistivity and seismic refraction (for determining the depth of very large piles).

5.3.1 The size of the waste pile will influence the sampling strategy in that increasing size is often accompanied by increased variability in the physical characteristics of the waste pile. The number of samples, however, that are needed to characterize a waste pile adequately will typically be a function of the study objectives as well as the inherent variability of the pile.

5.3.2 The shape of the waste pile can influence the sampling strategy by limiting access to certain locations within the pile, and if it is topologically complex it is difficult to lay out a sampling grid. Also, a waste pile may extend vertically both above and below grade, making decisions regarding the depth of sample collection difficult.

[6] The boldface numbers in parentheses refer to the list of references at the end of this standard.

5.3.3 The stability of the waste pile also can limit access to both the face and the interior of the pile. The use of certain types of heavier sampling equipment also could be limited by the ability of the pile to bear the weight of the equipment.

5.4 *Waste Characteristics*:

5.4.1 The constituents could include inorganics, volatile organic compounds (VOCs), and semivolatile organic compounds (including pesticides and polychlorinated biphenyls (PCBs)) (see Practice D 4547). Speciality analyses may be warranted, such as leaching tests or analyses for dioxin/furans or explosive compounds. Soil gas sampling is a minimally intrusive technique that may detect the presence and distribution of volatile organic compounds in soils and in porous, unconsolidated materials. Appropriate applications for soil gas monitoring are identified in Guide D 5314.

5.4.2 The distribution of constituents in the waste pile could be influenced by changes in the manufacturing process which resulted in changes in the composition of the waste; the length of time the material has remained in the pile (particularly for VOCs); the mode of delivery of the waste materials to the pile; and management practices, such as mixing together wastes from more than one process.

5.4.3 Physical and chemical variabilities would include variability in the chemical characteristics of the material within the pile, as well as variability in particle size, density, hardness, whether brittle or flexible, moisture content, consolidated, or unconsolidated. The variability may be random or found as strata of materials having different properties or containing different types or concentrations of constituents.

5.4.3.1 Geophysical survey methods may be used on piles to estimate physical homogeneity, which may or may not be related to chemical homogeneity, and to detect buried objects, both of which may need to be considered during the development of the sampling design and the safety plan for the investigation. The most suitable technique for detecting nonmetallic objects is electromagnetics. Ground-penetrating radar, a more sophisticated and complex technique, also may be considered. Electromagnetic techniques are suited particularly to large piles that contain leachate plumes (for example, mine tailings) or for the detection of large discontinuities in a pile (for example, different types of wastes or the transition from a disposal area to background soils). For metallic objects, metal detectors and magnetometers are useful and relatively easy to use in the field.

5.5 *Potential Investigation Errors*:

5.5.1 Equipment selection can bias sampling results even if the equipment is used properly. Bias can result from the incompatibility of the materials that the sampling equipment is made of with the materials being sampled. For example, the equipment could alter the characteristics of the sample. Some equipment will bias against the collection of certain particles sizes, and some equipment cannot penetrate the waste pile adequately.

5.5.2 Equipment, use, and operation can introduce error (bias) into the characterization of a waste pile. Sampling errors typically are caused when certain particle sizes are excluded, when a segment of the waste pile is not sampled, or when a location outside the pile is inadvertently sampled.

5.5.3 When stratification, layering, or solid phasing occurs it may be necessary to obtain and analyze samples of each of the distinct phases separately to minimize sampling bias. Care should be taken when sampling stratified layers to minimize cross contamination. Proper decontamination procedures should be used for all sampling equipment (see Practice D 5088).

5.5.4 Statistical bias includes situations where the data are not normally distributed or when the sampling strategy does not allow the potential for every portion of the pile to be sampled.

6. Sampling Strategy

6.1 Developing a strategy for sampling a waste pile requires a thorough examination of the site evaluation factors listed in Section 5. The location and frequency of sampling (number of samples) should be outlined clearly in the sampling plan, as well as provisions for the use of special sampling equipment, access of heavy equipment to all areas of the pile, if necessary, and so forth.

6.1.1 *Representative Sampling*—The collection of a representative set of samples from a waste pile typically will be complicated by the presence of a number of the site evaluation factors **(2,3)**.

6.1.2 *Heterogeneous Wastes*—Waste piles may be homogeneous, for applied purposes, or may be quite heterogeneous in particle size and contaminant distribution. If the particle sizes of the material in the waste pile and the distribution of contaminants are known, or can be estimated, then less sampling may be necessary to define the properties of interest in the waste pile. An estimate of the variability in contaminant distribution may be based on process knowledge or determined by preliminary sampling **(4)**. The more heterogeneous the waste pile is, the greater the planning and sampling requirements.

6.1.3 *Strata and Hot Spots*—A waste pile also could contain strata that have less internal variation in physical properties or concentrations of chemical constituents than the remainder of the waste pile **(2,5)**. For example, strata may be present in a waste pile due to changes in the process that generated the waste, or if different processes at a facility contribute waste to different parts of the waste pile. A stratified sampling strategy would consider this situation by conducting independent sampling of each stratum, which could reduce the number of samples required. These strata could be in specific areas of the waste pile **(4)**. Also, hot spots may be present in the waste pile that are unique in composition **(2,5)**.

6.2 *Specific Sampling Strategies*:

6.2.1 Although the most appropriate method for evaluating material in waste piles is to sample at or immediately following the point of generation (for example, conveyor belt), most sampling problems involve existing or in-place waste piles. Therefore, the following discussion will focus on in-place waste piles. Sampling strategies available for waste piles include directed or judgmental sampling, simple random sampling, stratified random sampling, systematic grid sampling, and systematic sampling over time **(2,6)**. General concerns about the collection of a representative sample, the existence of potential heterogeneity in the waste pile, the presence of strata

within the waste pile, and the existence of distinct hot spots within the waste pile may also influence the selection of an appropriate sampling strategy and development of the sampling plan **(5)**. The following paragraphs provide an introduction to determining the appropriate number of samples to collect and the sampling strategies available for determining sample locations.

6.2.2 *Determining the Frequency or Number of Samples*—The frequency of sampling or the number of samples to collect typically will be based on several factors including the study objectives, properties of wastes in the pile, degree of confidence required, access to sampling points, and budgetary constraints. Practical guidance for determining the number of samples is included in Guide D 4687 and Refs **(2, 3)**.

6.2.3 *Directed Sampling*—Directed sampling (Fig. 1) is based on the judgment of the investigator and will not result necessarily in a sample that reflects the characteristics of the entire waste pile. Directed sampling also is called judgmental sampling, authoritative sampling, or nonprobability sampling. The experience of the investigator often is the basis for sample collection, and, depending on the study objectives, bias should be recognized as a potential problem. For preliminary screening investigations of a waste pile and for certain regulatory investigations, however, directed sampling may be appropriate. A directed sampling strategy could call for the collection of a composite sample from the surface area or the collection of discrete grabs at the surface of the pile (see Fig. 1). Directed sampling would typically focus on worst case conditions in a waste pile, for example, the most visually contaminated area or most recently generated waste.

6.2.4 *Simple Random Sampling*—Simple random sampling (Fig. 2) ensures that each element in the waste pile has an equal chance of being included in the sample **(2)**. This may be the method of choice when, for purposes of the investigation, the waste pile is randomly heterogeneous **(5)**. If the waste pile contains trends or patterns of contamination, a stratified random sampling or systematic grid sampling strategy would be more appropriate **(2)** (see 6.2.5 and 6.2.6).

6.2.4.1 A simple random approach could use a grid with random grids selected for sample collection (see Fig. 2). Note that the grid size could be selected based on the number of samples that are required (some guidance suggests having at least ten times the number of grids as samples required). Once the grid is overlaid and the sampling locations are selected, the decision must be made to collect either a discrete grab sample (surface), a composite of surface samples taken from predesignated locations within the grid cell (based on compass points), a vertical composite to a specified depth, or discrete

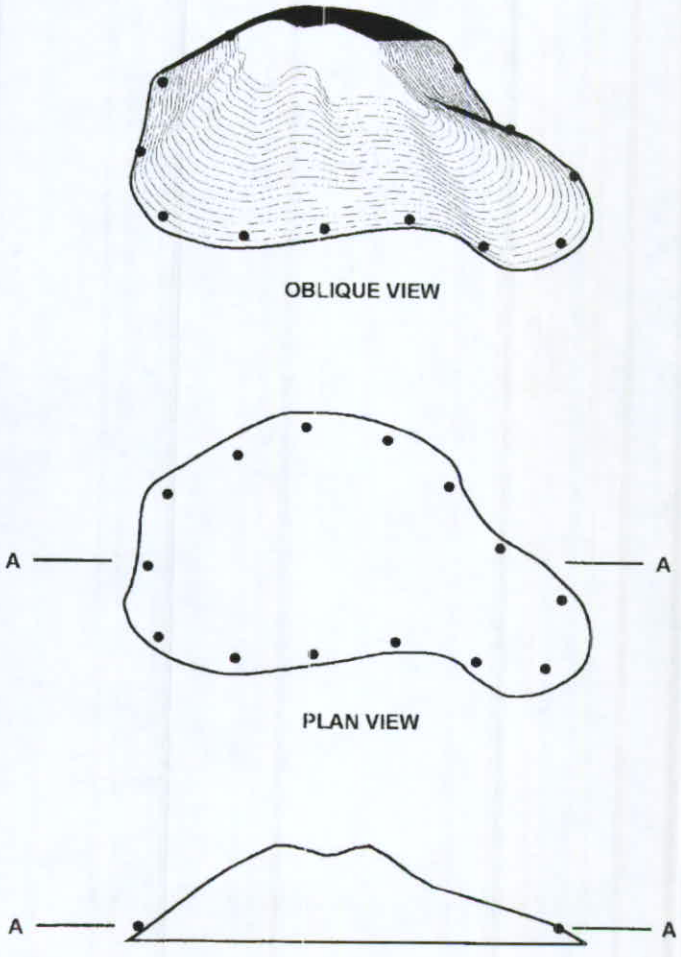


FIG. 1 Waste Pile Sampling Strategy—Directed Sampling

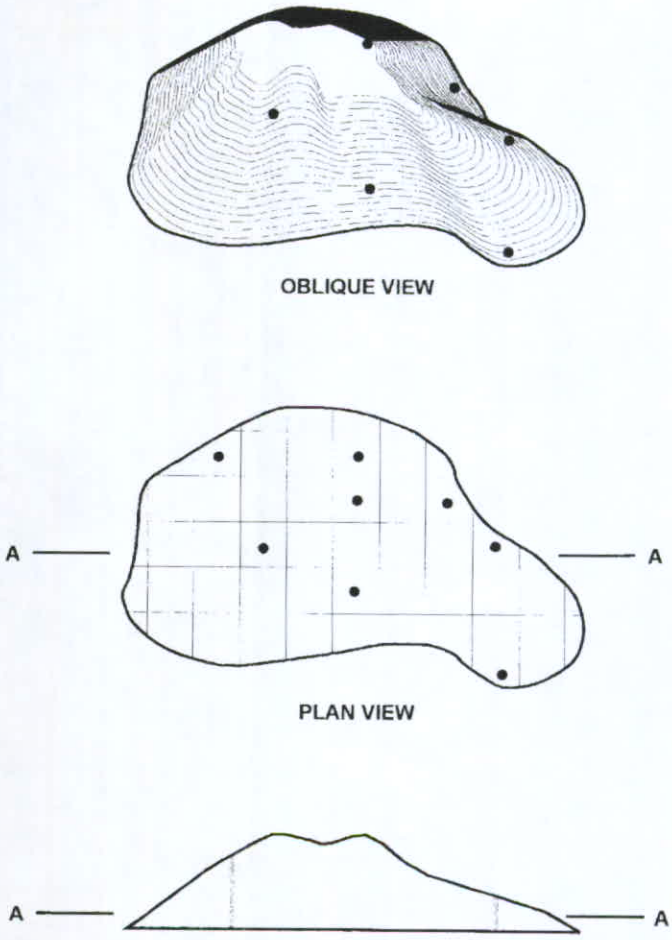


FIG. 2 Waste Pile Sampling Strategy—Simple Random Sampling

grab samples at specified depths. If discrete grab samples are desired at specified depths, they typically would be collected at the same location as the bore hole is advanced into the pile. Fig. 2 illustrates the collection of vertical composites at each of the randomly selected locations.

6.2.5 *Stratified Random Sampling*—Stratified random sampling (see Fig. 3) may be useful when distinct strata or homogeneous subgroups are identified within the waste pile **(2)**. The strata may be located in different areas of the pile or may be comprised of different layers (see Fig. 3). This approach is useful when the individual strata may be considered internally homogeneous or at least have less internal variation in what would otherwise be considered a heterogeneous waste pile **(2)**. Information on the waste pile usually is required to establish the location of individual strata unless process knowledge or changes in the composition of the material is obvious, such as with discoloration or with the type of waste. The grid may be utilized for sampling several horizontal layers if the strata are oriented horizontally **(4)**. A simple random sampling approach then is used within each stratum. The use of a stratified random sampling strategy may result in the collection of fewer samples. Fig. 3 illustrates a scenario where the number of samples collected in each stratum varies (plan view), and discrete grabs are collected in each boring at predesignated depths (side view).

6.2.6 *Systemic Grid Sampling*—Systematic grid sampling (see Fig. 4) involves the collection of samples at fixed intervals and is useful when the contamination is assumed to be distributed randomly **(2)**. This method also is commonly used with waste piles when estimating trends or patterns of contamination or when the objective is to locate hot spots. This approach may not be acceptable if the entire waste pile is not accessible or if the sampling grid locations become phased with variations in the distribution of contaminants within the waste pile **(6)**. It also may be useful for identifying the presence of strata within the pile. The grid and starting points should be laid out randomly over the waste pile, yet the method allows for rather easy location of exact sample locations by means of the grid (see Fig. 4). The same considerations discussed in 6.2.4 concerning the depth of each sample (surface, vertical composite, discrete grabs at depth) also should be considered. Fig. 4 illustrates the collection of vertical composites at each grid, which could be difficult and costly. Also note that the grid size typically would be adjusted according to the number of samples that are required.

6.2.7 *Systematic Sampling Over Time*—Systematic sampling over time at the point of generation is useful if the material is being sampled from a conveyor belt or being

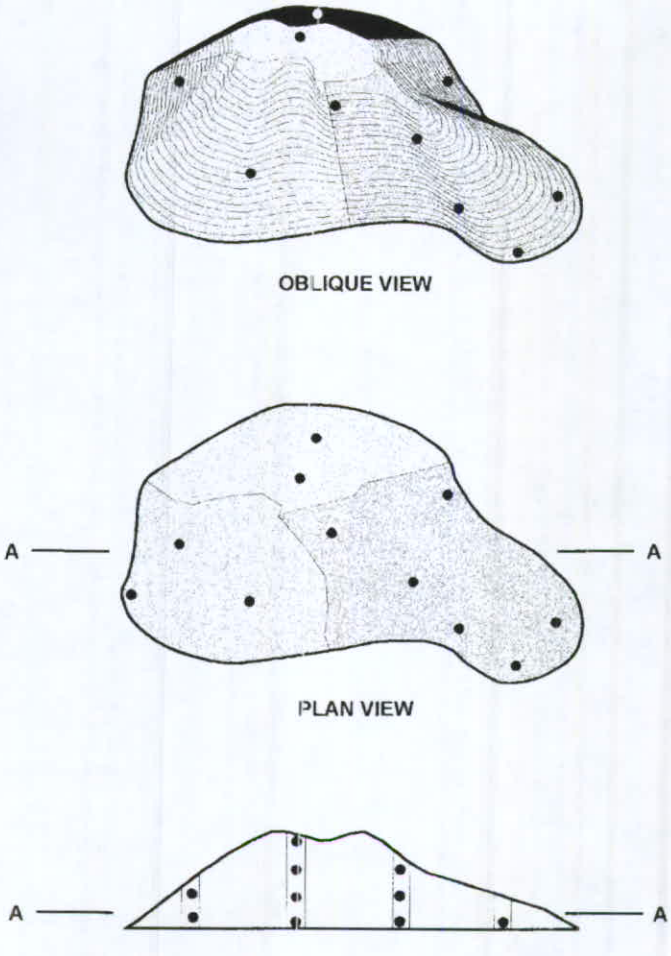


FIG. 3 Waste Pile Sampling Strategy—Stratified Random Sampling

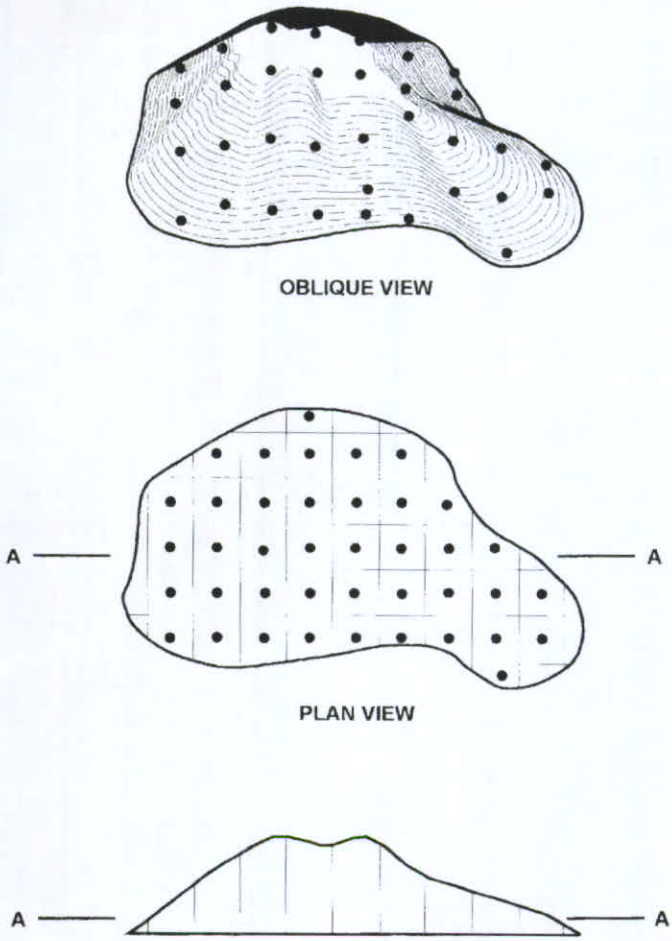


FIG. 4 Waste Pile Sampling Strategy—Systematic Grid Sampling

delivered by means of truck or pipeline to the waste pile. The sampling interval can be determined on a time basis, for example, every hour from a conveyor belt or pipeline discharge, or from every third truck load. The time between intervals is influenced by the factors addressed in 6.2.2.

6.2.8 *Alternative Approach*—In many cases, an objective of waste pile characterization is to determine the impact of the pile on the environment. At times this may be accomplished more easily by sampling the routes by which contaminants are dispersed from the pile than through direct sampling of the pile, especially for piles that are difficult to characterize. For example, ground water up-and-down gradient from the pile could be sampled to check for ground water contamination. The vadose zone below the pile also might be sampled to detect leachate (and potential ground water contamination) through soil sampling, vacuum lysimeters, or soil gas. Surface water and sediment in drainage channels down gradient from the pile also might be sampled. Surface soils, air samples, and contaminants deposited on vegetation can be used as indicators of atmospheric transport of contaminants from the pile, including both particulate and volatile materials. Such approaches will seldom replace pile sampling completely, but they may reduce the number of pile samples needed to make remedial action decisions (see Guide D 5730), also Refs **(7-9)**.

7. Selection of Sampling Equipment

7.1 Wastes in piles are often complex, multiphase mixtures of solids and semisolids. The wastes can range from powders to granules to large, heterogeneous solid fragments and can cover many acres in area. No single type of sampler can be used to collect representative samples of all types of waste from piles. Large, thick piles may require drill rigs to obtain samples from depth. The sampling of gases from within the pile requires other types of equipment. Table 2 lists typical waste types and the corresponding recommended samplers to use.

7.2 Sampling at depth from inside the pile may require heavy equipment designed for excavation or removal of soil or rock. Table 3 lists such equipment and its applications for sampling waste piles **(10)**.

7.3 Sampling equipment should be constructed of materials that are compatible with the waste to be sampled. Compatibility refers to the physical durability, lack of chemical reactivity with the waste, and lack of potential for contamination of the waste with analytes of concern. Typical materials of construction include stainless steel, plastic, and glass.

8. Data Use

8.1 The decisions that will be made based upon the data must be identified early in the planning process since these affect the approach to the problem and how the data will be evaluated. Decisions affecting waste classification, closure, and post-closure issues, are examples of the uses of the data. Methods to determine the volume of contaminated material in a pile or pile strata may be needed. Standard mathematical formulas for calculating the volume of a cone, cylinder, various prisms, and so forth, may be used.

8.2 *Statistical Considerations*:

8.2.1 Data quality assessment (DQA) methods are used to evaluate the data for any anomalies and to evaluate the assumptions for statistical evaluation. The statistician makes use of both subjective judgment (graphical analysis for identification of trends and anomalies) and statistical models and inference (for example, outlier detection, autocorrelation estimation) in the investigation of data for validity of the assumptions needed to make a statistical test. Classical statistical models assume that the samples collected from the population of interest are independent and have an identical probability distribution (that is, normal distribution with constant mean and variance). Random sampling is a method to ensure independence. The probability distributional assumptions are part of DQA that will determine if the classical statistical

TABLE 2 Sampling Devices Suitable for Waste Piles[A]

Location and Waste Type	Sampling Devices	ASTM Standard	Limitations
Subsurface			
Powdered, granular, or soil-like solids; sludges	split-barrel push coring device	D 1586 D 1587 D 4700 D 4823	Limited application for sampling moist and sticky solids, or particles with diameter 0.6 cm (0.25 in.) or more. Depth limitation of about 1 m.
	trier	D 5451	May not retain core sample of very dry granular materials. Not applicable to sampling solid wastes with particle diameter >½ the diameter of the sampling tube.
	auger	D 1452 D 4700	Does not collect undisturbed sample.
	thin-walled tube sampler	D 4823 D 4700	Collects relatively undisturbed core. Difficult to use on gravelly or rocky soils.
	drill rigs		Used for geoenvironmental exploration. To minimize sample contamination, avoid those using a water-based drilling fluid.
	soil gas samplers	D 5314	Used for volatile organic compounds.
Surface			
Powdered, granular, or soil-like solids; sludges	trowel or scoop	D 4700	Not applicable to sampling deeper than 8 cm (3 in.). Difficult to obtain reproducible mass of sample. May exclude certain particle sizes, especially large aggregates.
Slag	hammer/chisel Impact device		Changes particle size.

[A] This table is not all inclusive; other equipment may be used.

TABLE 3 Excavation and Removal Equipment for Waste Piles

Excavation and Removal Equipment	General Excavation	Ability to Excavate Hard and Compacted Material	Soil Hauling	Mixing of Solids, Soil	Spreading Cover	Site Maneuverability
Wheel or crawler Mounted backhoe	A[A]	A	B[B]/O[C]	A	A	A/B
Wheel or crawler Mounted front-end loader	A	A	A/B	A	A	A/B
Skid steer loader	A	B	B	A	B	A
Bulldozer	A	A	O	O	A	B

[A] A = Good choice. Equipment is fully capable of performing function listed.
[B] B = Secondary choice. Equipment is marginally capable of performing function listed.
[C] O = Not applicable or poor choice.

model is appropriate for the collected data. For directed sampling, the sampling is subjective and the sample results are typically judged on a qualitative basis.

8.2.2 Simple random sampling will provide an unbiased estimate of the average waste concentration, that is, an estimate of the mean. This unbiased estimate is independent of the geometry of the pile and of the distribution of the concentration of the contaminants, but it may not have the smallest variance. Other sampling designs, such as systematic grid sampling or stratified random sampling, may provide an average that has a smaller variance. If the waste pile has uneven topography, the calculation of the mean concentration of the pile should be a volume-weighted average, using core volume as the weighting factor to reduce the variance of the estimated mean.

8.2.2.1 For simple random sampling and systematic grid sampling designs, histogram and normal probability plots of the sample data can be used to judge if the data conform to normal distribution. If not, there are several alternatives. First, the classical statistical model may still be considered robust for the decision-making process. Second, a transformation of the data may approximate a normal distribution of the data. For example, logarithmic transformation will normalize data that are lognormal originally. If the data are lognormal, the question of whether to use the arithmetic mean or the geometric mean for decision-making purposes must be decided. Third, an alternative statistical model based on nonparametric methods, but which uses weaker assumptions, may be proposed to analyze the decision-making process. It may be advisable to consult a statistician.

8.2.2.2 For the stratified random sampling design, the test of normality is not straightforward. Generally, it requires a mathematical model to take out the strata effects first, then test for normality using the residuals. A statistician should be consulted.

8.2.2.3 In any of these cases, alternative consequences of the level of uncertainty can be calculated prior to collecting the data. These alternatives can be used by decision-makers to select the best strategy to minimize the environmental risks.

9. Keywords

9.1 piles; sampling; waste

APPENDIX

(Nonmandatory Information)

X1. WASTE PILE—A CASE HISTORY

X1.1 **Background**—The waste pile was generated by a facility that produces brass alloys from scrap metal. The byproduct from this operation was slag, which was generated in the recovery furnace. The slag was ground subsequently in a ball mill prior to being reintroduced into the recovery furnace. A large amount of the ground slag was disposed of in a waste pile which covered about one acre. No active management was occurring with the waste pile. No buried containers or extremely heterogenous material (unground slag) was suspected of being present in the waste pile based on facility records and interviews of personnel.

X1.1.1 Lead and cadmium were the constituents of concern based on process knowledge, and the possibility for the waste being hazardous was the regulatory consideration. The potential for off-site migration of contaminants was also an immediate concern, and this was considered in the development of the Phase 1 study design. Fig. X1.1 shows a site map of the facility and the slag pile. Fig. X1.2 shows a computer enhancement of the slag pile, and Fig. X1.3 shows a topographic view of the pile.

X1.2 Phase 1:

X1.2.1 *Objective*—The primary objective of the initial investigation was to determine if the slag in the waste pile classified as hazardous based on the concentration of lead and cadmium in a leach test. A secondary objective was to provide preliminary information on the potential migration and transport of contaminants from the waste pile off-site. The sampling plan for this initial investigation utilized a directed sampling strategy to provide a preliminary estimate of the lead concentration in the waste, the variability of contaminant concentrations in the pile, and the potential for leaching using the applicable leaching procedure mandated in regulations. Four composite samples were collected from the surface (0 to 15 cm or 0 to 6 in.) of the waste pile at locations within the four

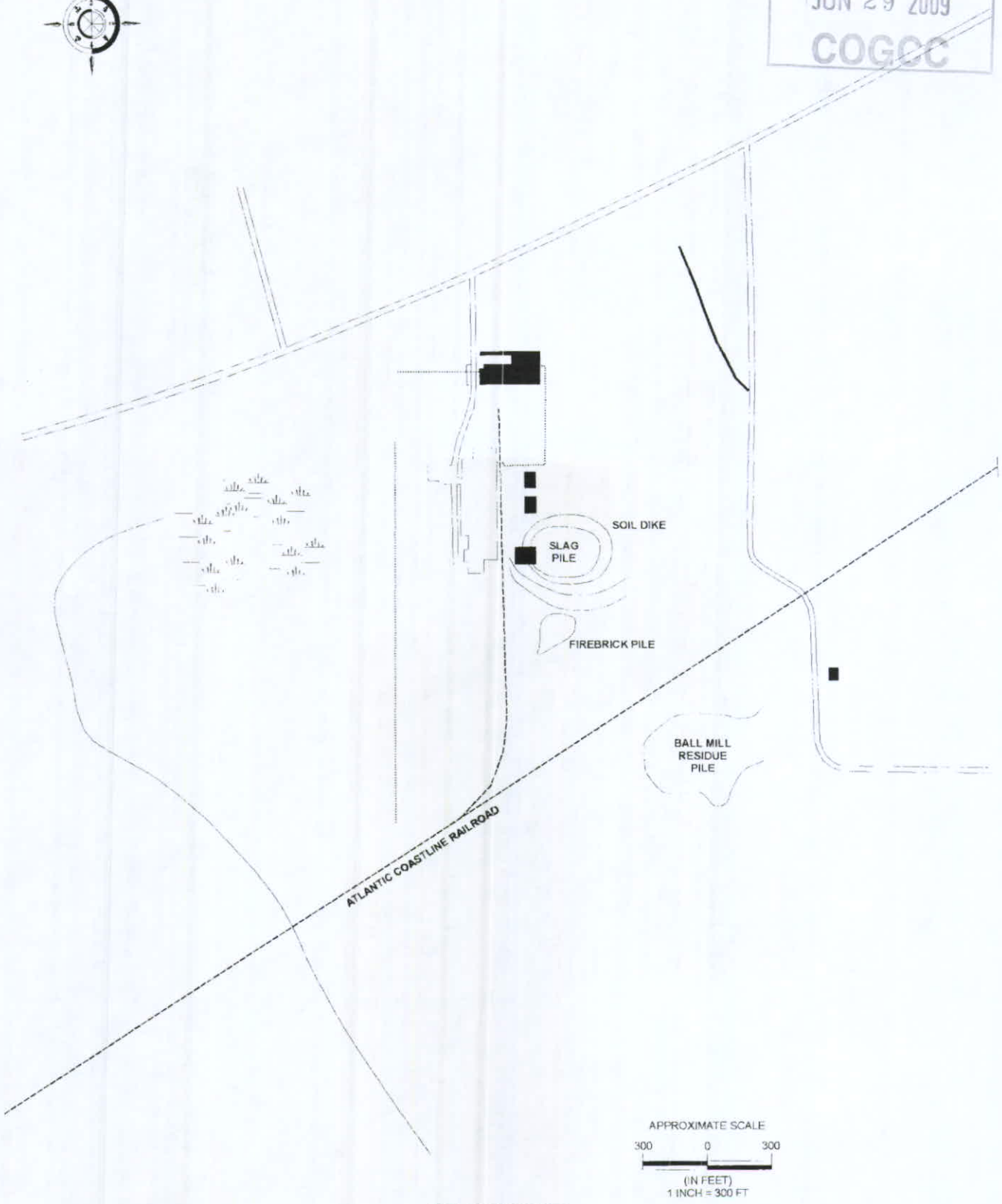


FIG. X1.1 Site Map

quadrants. The following environmental samples were also collected:

X1.2.1.1 Several soil samples in the vicinity of the waste pile,

X1.2.1.2 Sediment upstream and downstream in a stream which borders the facility,

X1.2.1.3 Sediment in a ditch which contained runoff from the pile, and

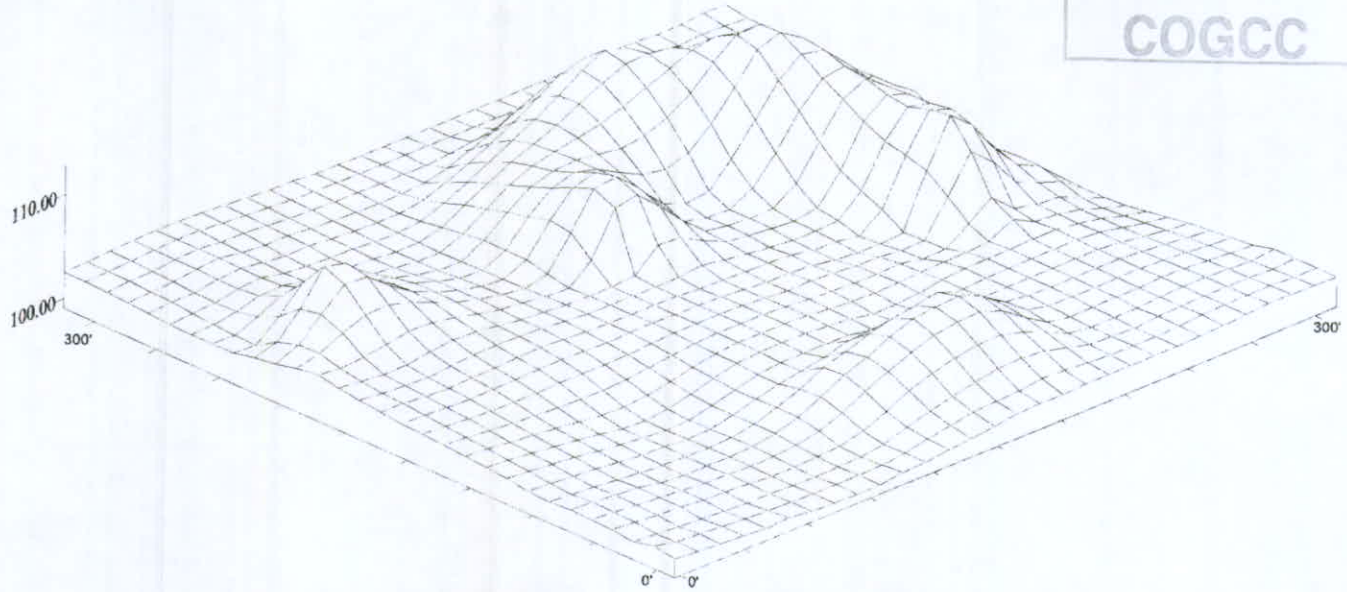


FIG. X1.2 Computer Enhancement of the Slag Pile (Front View) Scale 1:1:2

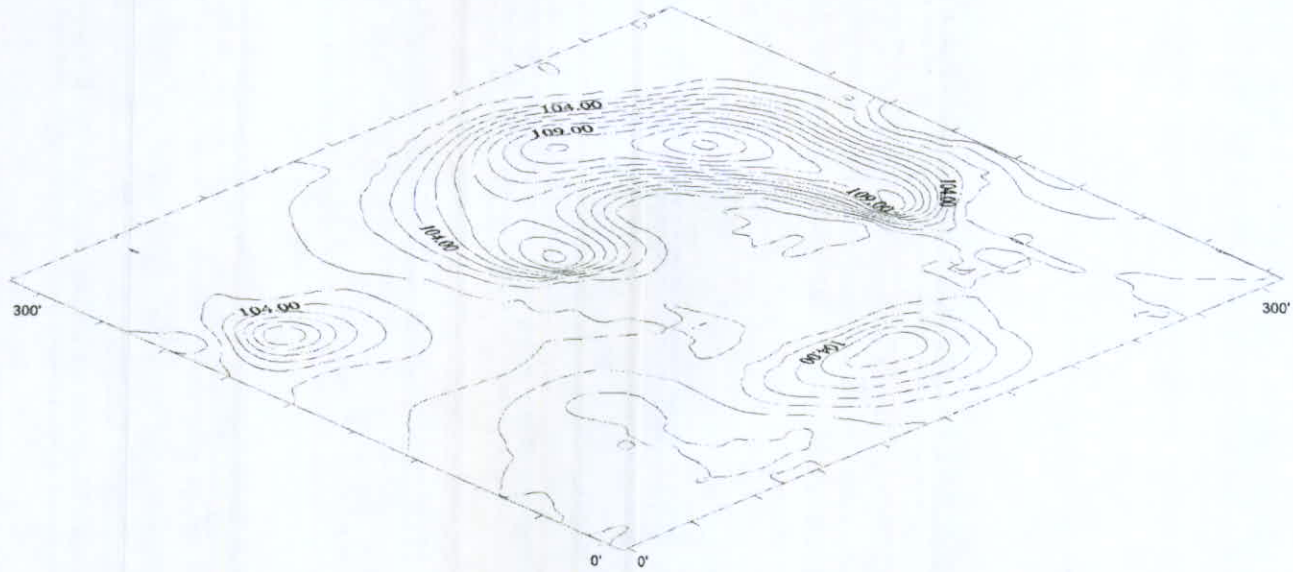


FIG. X1.3 Topographic View of the Slag Pile

X1.2.1.4 Two background soil samples.

X1.2.2 Fig. X1.4 shows the Phase 1 sampling locations within the slag pile, and Fig. X1.5 shows the same sampling locations on the topographic map of the pile.

X1.2.3 *Results*—Zinc, copper, cadmium, and lead were all elevated (compared to background) in the samples collected from the waste pile, and the concentrations did not appear to vary significantly between the samples. Since lead and cadmium are regulated constituents, a leach test was completed, and the lead results exceeded the regulatory level of 5 mg/L. Cadmium was just under the regulatory level of 1.0 mg/L. Lead and cadmium concentrations in the soil were 2 to 3 times above background, and the drainage ditch and downstream sediment sample also had elevated lead and cadmium levels.

X1.2.4 *Conclusion*— The waste pile contained slag that is hazardous for lead. The waste pile required further characterization to determine the variability in the pile. The presence of lead and cadmium in soils and the stream sediment downstream of the facility was confirmed and should be investigated further to determine the extent of contaminant transport.

X1.3 Phase 2:

X1.3.1 *Objective*—The objective is to characterize the waste pile further using a systematic grid sampling design. This design will delineate horizontal and vertical variability in lead and cadmium concentrations. The Phase 1 investigation also provided a good estimate of the anticipated variability in the waste pile. The number of samples required to characterize the waste pile adequately was calculated based on the average concentration, the anticipated variability, the regulatory level of concern, and the specified confidence interval. The grid size then was adjusted to accommodate the projection on the required number of samples. Composite samples were collected within each grid cell based on one center point and eight

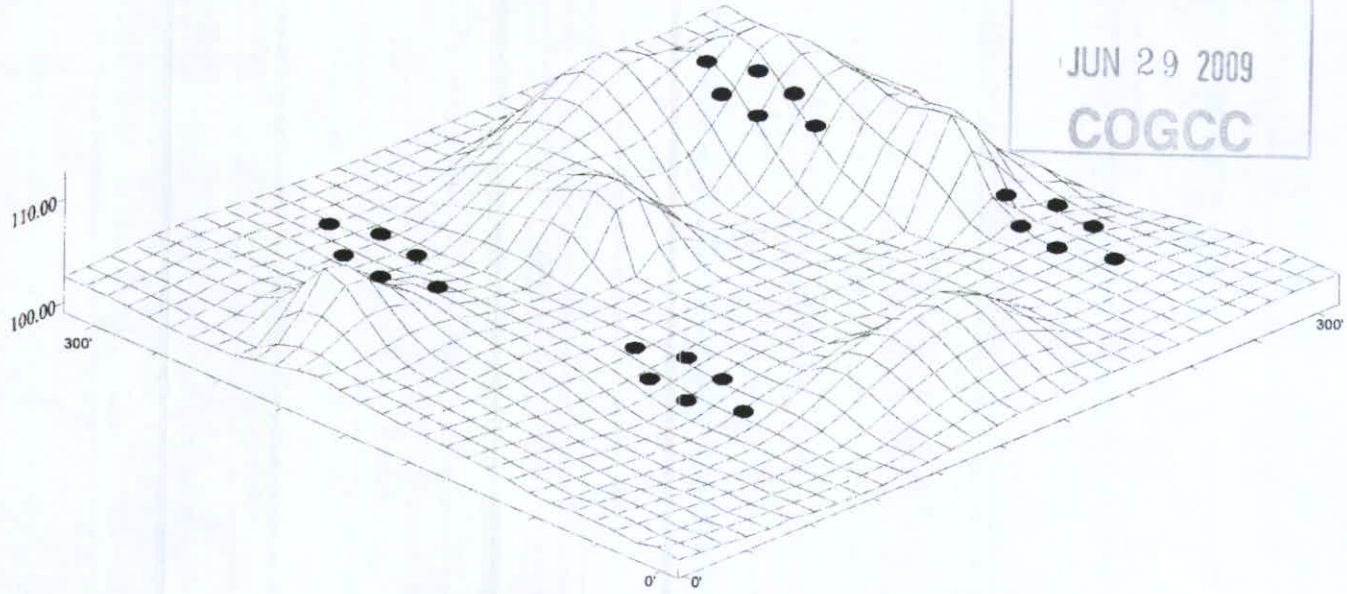


FIG. X1.4 Front View of the Slag Pile Showing Sampling Locations Scale 1:1:2

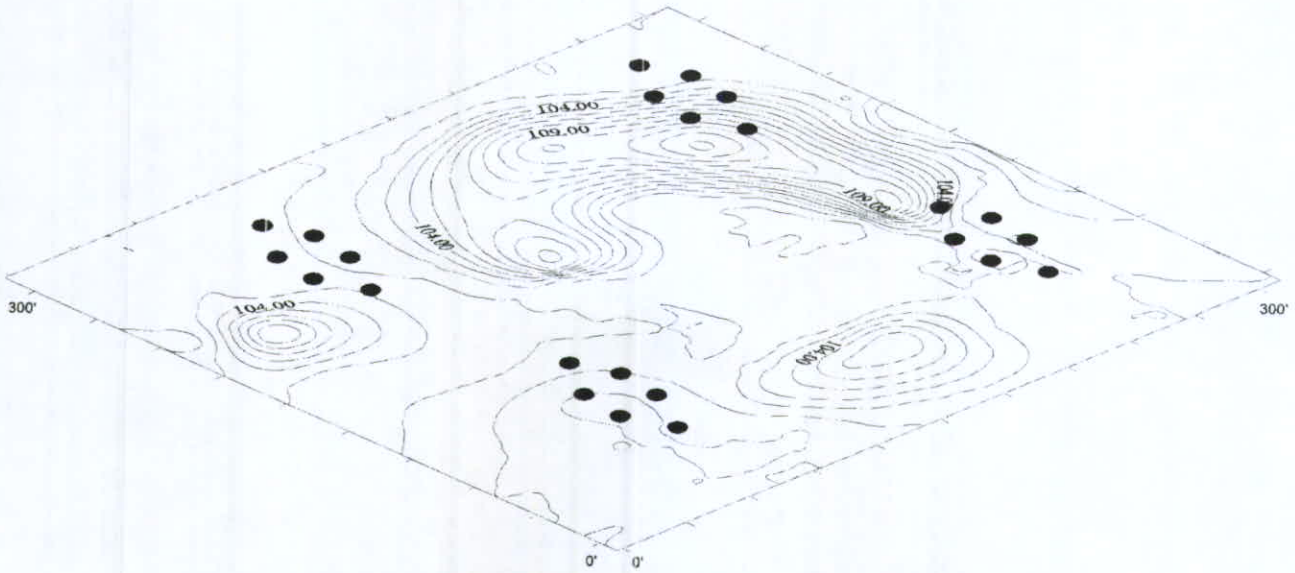


FIG. X1.5 Topographic View of the Slag Pile Showing Sampling Locations

points on the compass (45° intervals) equidistant from the center point. Ten percent of the grids were designated for vertical as well as surface (0 to 15 cm or 0 to 6 in.) sample collection. Additionally, 10 % of the grids were designated randomly for duplicate sampling (using a different aliquot pattern) to check the preliminary estimate on the variability. Additional environmental sampling was conducted but will not be covered in this discussion.

X1.3.2 *Results*—The results supported the initial Phase 1 investigation with lead consistently exceeding the regulatory level. Cadmium consistently was below the regulatory level.

X1.3.3 *Conclusion*— The waste pile was characteristic for lead and classified as hazardous according to the applicable regulations. There was no significant variability with depth, although several gradients were noticed across the grid based on lead concentration (scan) results.

X1.4 Phase 3:

X1.4.1 *Objective*—The objective is to determine thevolume of the waste pile in order to estimate both the disposal cost and the total amount of the civil penalty to be charged to the owner of the pile. The waste pile was surveyed using standard surveying techniques.

X1.4.2 *Results*—The results were used to calculate the volume using geometric principles. Also, a computer program was utilized which constructs contours based on the surveying information. The computer program was used as a check of the manual method, which produced a result that was 10 % higher in volume than the computer program.

X1.4.3 *Conclusion*— For penalty calculation purposes, the smaller estimate was utilized; however, the actual treatment and disposal costs could reflect the larger estimate.

REFERENCES

(1) U.S. Environmental Protection Agency (EPA). 1986. *Test Methods for Evaluating Solid Waste*; SW-846, 3rd Edition. EPA/530/SW-846 (NTIS PB88-239223); First update, 3rd edition. EPA/530/SW-846.3-1 (NTIS PB89-148076). Current edition and updates available on a subscription basis from U.S. Government Printing Office, Stock #955-001-00000-1.

(2) Gilbert, R. O., "Statistical Methods for Environmental Pollution Monitoring," Van Nostrand Reinhold Co., 1987.

(3) Ford, P. J., and Turina, P. J., *Characterization of Hazardous Waste Sites—A Methods Manual, Vol 1: Site Investigations*, EPA 600/4-84/075, (NTIS PB85-215960), 1985.

(4) U.S. Environmental Protection Agency (EPA), *Compendium of ERT Waste Sampling Procedures*, Section 5.0 Waste Pile Sampling, SOP No. 2017, EPA 540/P-91/008, OSWER Directive 9360.4-07, January 1991.

(5) Pitard, F., *Pierre Gy's Sampling Theory and Sampling Practice*, Vol 1: Heterogeneity and Sampling, Chemical and Rubber Company (CRC) Press, 1989.

(6) U.S. Environmental Protection Agency (EPA), *Characterizing Heterogeneous Wastes: Methods and Recommendations*, EPA 600/R-92/033, (NTIS PB92-216894) February 1992. [Also published as hardback Smoley Edition: Rupp and Joens (1993).]

(7) Keith, L., *Principles of Environmental Sampling*, Ed. ACS, 1988.

(8) U.S. Environmental Protection Agency (EPA). *Guidance Document on the Statistical Analysis of Ground Water Monitoring Data at RCRA Facilities*, Office of Solid Waste, 1993.

(9) McCoy and Associates, Inc., "Soil Sampling and Analysis—Practices and Pitfalls," Hazardous Waste Consultant, Vol 10, No. 6, Lakewood, CO, 1992.

(10) PEI Associates, 1991, *Survey of Materials-Handling Technologies Used at Hazardous Waste Sites*, EPA 540/2-91/010, June 1991. (NTIS PB91-186924), 225 pp.

Designation: D 6044 – 96

Standard Guide for Representative Sampling for Management of Waste and Contaminated Media[1]

This standard is issued under the fixed designation D 6044; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide covers the definition of representativeness in environmental sampling, identifies sources that can affect representativeness (especially bias), and describes the attributes that a representative sample or a representative set of samples should possess. For convenience, the term" representative sample" is used in this guide to denote both a representative sample and a representative set of samples, unless otherwise qualified in the text.

1.2 This guide outlines a process by which a representative sample may be obtained from a population. The purpose of the representative sample is to provide information about a statistical parameter(s) (such as mean) of the population regarding some characteristic(s) (such as concentration) of its constituent(s) (such as lead). This process includes the following stages: (*1*) minimization of sampling bias and optimization of precision while taking the physical samples, (*2*) minimization of measurement bias and optimization of precision when analyzing the physical samples to obtain data, and (*3*) minimization of statistical bias when making inference from the sample data to the population. While both bias and precision are covered in this guide, major emphasis is given to bias reduction.

1.3 This guide describes the attributes of a representative sample and presents a general methodology for obtaining representative samples. It does not, however, provide specific or comprehensive sampling procedures. It is the user's responsibility to ensure that proper and adequate procedures are used.

1.4 The assessment of the representativeness of a sample is not covered in this guide since it is not possible to ever know the true value of the population.

1.5 Since the purpose of each sampling event is unique, this guide does not attempt to give a step by step account of how to develop a sampling design that results in the collection of representative samples.

1.6 Appendix X1 contains two case studies, which discuss the factors for obtaining representative samples.

1.7 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 3370 Practices for Sampling Water from Closed Conduits[2]

D 4448 Guide for Sampling Groundwater Monitoring Wells[3]

D 4547 Practice for Sampling Waste and Soils for Volatile Organics[3]

D 4700 Guide for Soil Sampling from the Vadose Zone[4]

D 4823 Guide for Core-Sampling Submerged, Unconsolidated Sediments[5]

D 5088 Practice for Decontamination of Field Equipment Used at Nonradioactive Waste Sites[6]

D 5792 Practice for Generation of Environmental Data Related to Waste Management Activities: Development of Data Quality Objectives[3]

D 5956 Guide for Sampling Strategies for Heterogeneous Wastes[3]

D 6051 Guide for Composite Sampling and Field Subsampling for Environmental Waste Management Activities[3]

3. Terminology

3.1 *analytical unit, n*—the actual amount of the sample material analyzed in the laboratory.

3.2 *bias, n*—a systematic positive or negative deviation of the sample or estimated value from the true population value.

3.2.1 *Discussion*—This guide discusses three sources of bias—sampling bias, measurement bias, and statistical bias.

There is a sampling bias when the value inherent in the physical samples is systematically different from what is inherent in the population.

There is a measurement bias when the measurement process produces a sample value systematically different from that inherent in the sample itself, although the physical sample is

[1] This guide is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.01.01 on Planning for Sampling.

Current edition approved Nov. 10, 1996. Published January 1997.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 11.04.

[4] *Annual Book of ASTM Standards*, Vol 04.08.

[5] *Annual Book of ASTM Standards*, Vol 11.02.

[6] *Annual Book of ASTM Standards*, Vol 04.09.

itself unbiased. Measurement bias can also include any systematic difference between the original sample and the sample analyzed, when the analyzed sample may have been altered due to improper procedures such as improper sample preservation or preparation, or both.

There is a statistical bias when, in the absence of sampling bias and measurement bias, the statistical procedure produces a biased estimate of the population value.

Sampling bias is considered the most important factor affecting inference from the samples to the population.

3.3 *biased sampling, n*—the taking of a sample(s) with prior knowledge that the sampling result will be biased relative to the true value of the population.

3.3.1 *Discussion*—This is the taking of a sample(s) based on available information or knowledge, especially in terms of visible signs or knowledge of contamination. This kind of sampling is used to detect the presence of localized contamination or to identify the source of a contamination. The sampling results are not intended for generalization to the entire population. This is one form of authoritative sampling (see *judgment sampling*.)

3.4 *characteristic, n*—a property of items in a sample or population that can be measured, counted, or otherwise observed, such as viscosity, flash point, or concentration.

3.5 *composite sample, n*—a combination of two or more samples.

3.6 *constituent, n*— an element, component, or ingredient of the population.

3.6.1 *Discussion*—If a population contains several contaminants (such as acetone, lead, and chromium), these contaminants are called the constituents of the population.

3.7 *Data Quality Objectives, DQOs, n*—qualitative and quantitative statements derived from a DQO process describing the decision rules and the uncertainties of the decision(s) within the context of the problem(s) (see Practice D 5792).

3.8 *Data Quality Objective Process*—a quality management tool based on the Scientific Method and developed by the U.S. Environmental Protection Agency to facilitate the planning of environmental data collection activities. The DQO process enables planners to focus their planning efforts by specifying the use of data (the decision), the decision criteria (action level), and the decision maker's acceptable decision error rates. The products of the DQO process are the DQOs (see Practice D 5792).

3.9 *error, n*—the random or systematic deviation of the observed sample value from its true value (see *bias* and *sampling error*).

3.10 *heterogeneity, n*— the condition or degree of the population under which all items of the population are not identical with respect to the characteristic(s) of interest.

3.10.1 *Discussion*—Although the ultimate interest is in the statistical parameter such as the mean concentration of a constituent of the population, heterogeneity relates to the presence of differences in the characteristics (for example, concentration) of the units in the population. It is due to the presence of fundamental heterogeneity (or fundamental error)[7] in the population that sampling variance arises. Degree of sampling variance defines the degree of precision in estimating the population parameter using the sample data. The smaller the sampling variance is, the more precise the estimate is. See also *sampling error*.

3.11 *homogeneity, n*— the condition of the population under which all items of the population are identical with respect to the characteristic(s) of interest.

3.12 *judgment sampling, n*—taking of a sample(s) based on judgment that it will more or less represent the average condition of the population.

3.12.1 *Discussion*—The sampling location(s) is selected because it is judged to be representative of the average condition of the population. It can be effective when the population is relatively homogeneous or when the professional judgment is good. It may or may not introduce bias. It is a useful sampling approach when precision is not a concern. This is one form of authoritative sampling (see *biased sampling*.)

3.13 *population, n*— the totality of items or units of materials under consideration.

3.14 *representative sample, n*—a sample collected in such a manner that it reflects one or more characteristics of interest (as defined by the project objectives) of a population from which it is collected.

3.14.1 *Discussion*—A representative sample can be a single sample, a collection of samples, or one or more composite samples. A single sample can be representative only when the population is highly homogeneous.

3.15 *representative sampling, n*—the process of obtaining a representative sample or a representative set of samples.

3.16 *representative set of samples, n*—a set of samples that collectively reflect one or more characteristics of interest of a population from which they were collected. See *representative sample*.

3.17 *sample, n*—a portion of material that is taken for testing or for record purposes.

3.17.1 *Discussion*—Sample is a term with numerous meanings. The scientist collecting physical samples (for example, from a landfill, drum, or monitoring well) or analyzing samples considers a sample to be that unit of the population that was collected and placed in a container. A statistician considers a sample to be a subset of the population, and this subset may consist of one or more physical samples. To minimize confusion, the term *sample*, as used in this guide, is a reference to either a physical sample held in a sample container, or that portion of the population that is subjected to in situ measurements, or a set of physical samples. See *representative sample*.

3.17.1.1 The term *sample size* also means different things to the scientist and the statistician. To avoid confusion, terms such as sample mass/sample volume and number of samples are used instead of sample size.

3.18 *sampling error*— the systematic and random deviations of the sample value from that of the population. The

[7] Pitard, F. F., "*Pierre Gy's Sampling Theory and Sampling Practice: Heterogeneity, Sampling Correctness and Statistical Process Control*," 2nd ed., CRC Press Publishers, 1993.

systematic error is the *sampling bias*. The random error is the *sampling variance*.

3.18.1 *Discussion*—Before the physical samples are taken, potential sampling variance comes from the inherent population heterogeneity (sometimes called the "fundamental error," see *heterogeneity*). In the physical sampling stage, additional contributors to sampling variance include random errors in collecting the samples. After the samples are collected, another contributor is the random error in the measurement process. In each of these stages, systematic errors can occur as well, but they are the sources of bias, not sampling variance.

3.18.1.1 Sampling variance is often used to refer to the total variance from the various sources.

3.19 *stratum, n*—a subgroup of the population separated in space or time, or both, from the remainder of the population, being internally similar with respect to a target characteristic of interest, and different from adjacent strata of the population.

3.19.1 *Discussion*—A landfill may display spatially separated strata, such as old cells containing different wastes than new cells. A waste pipe may discharge into temporally separated strata of different constituents or concentrations, or both, if night-shift production varies from the day shift. In this guide, strata refer mostly to the stratification in the concentrations of the same constituent(s).

3.20 *subsample, n*— a portion of the original sample that is taken for testing or for record purposes.

4. Significance and Use

4.1 Representative samples are defined in the context of the study objectives.

4.2 This guide defines the meaning of a representative sample, as well as the attributes the sample(s) needs to have in order to provide a valid inference from the sample data to the population.

4.3 This guide also provides a process to identify the sources of error (both systematic and random) so that an effort can be made to control or minimize these errors. These sources include sampling error, measurement error, and statistical bias.

4.4 When the objective is limited to the taking of a representative (physical) sample or a representative set of (physical) samples, only potential sampling errors need to be considered. When the objective is to make an inference from the sample data to the population, additional measurement error and statistical bias need to be considered.

4.5 This guide does not apply to the cases where the taking of a nonrepresentative sample(s) is prescribed by the study objective. In that case, sampling approaches such as judgment sampling or biased sampling can be taken. These approaches are not within the scope of this guide.

4.6 Following this guide does not guarantee that representative samples will be obtained. But failure to follow this guide will likely result in obtaining sample data that are either biased or imprecise, or both. Following this guide should increase the level of confidence in making the inference from the sample data to the population.

4.7 This guide can be used in conjunction with the DQO process (see Practice D 5792).

4.8 This guide is intended for those who manage, design, and implement sampling and analytical plans for waste management and contaminated media.

5. Representative Samples

5.1 Samples are taken to infer about some statistical parameter(s) of the population regarding some characteristic(s) of its constituent(s) of interest. This is discussed in the following sections.

5.2 *Samples*—When a representative sample consists of a single physical sample, it is a sample that by itself reflects the characteristics of interest of the population. On the other hand, when a representative sample consists of a set of physical samples, the samples collectively reflect some characteristics of the population, though the samples individually may not be representative. In most cases, more than one physical sample is necessary to characterize the population, because the population in environmental sampling is usually heterogeneous.

5.3 *Constituents and Characteristics*—A population can possess many constituents, each with many characteristics. Usually it is only a subset of these constituents and characteristics that are of interest in the context of the stated problem. Therefore, samples need to be representative of the population only in terms of these constituent(s) and characteristic(s) of interest. A sampling plan needs to be designed accordingly.

5.4 *Parameters*—Similarly, samples need to be representative of the population only in the parameter(s) of interest. If the interest is only in estimating a parameter such as the population mean, then composite samples, when taken correctly, will not be biased and therefore constitute a representative sample (regarding bias) for that parameter. On the other hand, if the interest happens to be the estimation of the population variance (of individual sampling units), another parameter, then the variance of the composite samples is a biased estimate of the population variance and therefore is not representative. (It is to be noted that composite samples are often used to increase the precision in estimating the population mean and not to estimate the population variance of individual sampling units.)

5.5 *Population*—Since the samples are intended to be representative of a population, a population must be well defined, especially in its spatial or temporal boundaries, or both, according to the study objective.

5.6 *Representativeness*—The word "reflects" in this guide is used to mean a certain degree of low bias and high precision when comparing the sample value(s) to the population value(s). This is a broad definition of sample representativeness used in this guide. A narrower definition of representativeness is often used to mean simply the absence of bias.

5.6.1 *Bias*—Bias is sometimes mistakenly taken to be "a difference between the observed value of a physical sample and the true population value." The correct definition of bias is "a *systematic* (or consistent) difference between an observed (sample) value and the true population value." The word "systematic" here implies "on the average" over a set of physical samples, and not a single physical sample. Recall that sampling error consists of the random and systematic deviations of a sample (or estimated) value from that of the population. Although random deviations may occur on occasions due to imprecision in the sampling or measurement processes, or both, they balance out on the average and lead to no systematic difference between the sample (or estimated)

value and the population value. The random deviation corresponds to the observation of "a random difference between a single physical sample value and the true population value," which can be randomly positive or negative, and is not a bias. On the other hand, a persistent positive or negative difference is a systematic error and is a bias.

5.6.1.1 In order to assess bias, the true population value must be known. Since the true population value is rarely known, bias cannot be quantitatively assessed. However, this guide provides an approach to identifying the potential sources of bias and general considerations for controlling or minimizing these potential biases.

5.6.2 *Precision*—Precision has to do with the level of confidence in estimating the population value using the sample data. If the population is totally homogeneous and the measurement process is flawless, a single sample will provide a completely precise estimate of the population value. When the population is heterogeneous or the measurement process is not totally precise, or both, a larger number of samples will provide a more precise estimate than a smaller number of samples.

5.6.2.1 In the case of bias, the goal in environmental sampling is its absence. In the case of precision, the goal in sampling will depend on factors such as:

(1) The precision level needed to achieve the desired levels of decision errors, both false positive and false negative errors,

(2) If the true value is known or suspected to be well below the regulatory limit, high precision in the samples may not be needed, and

(3) The study budget.

5.6.2.2 Note that the second item applies similarly to bias as well.

5.6.2.3 Since bias, especially during sampling, can be very large when proper procedures are not followed, it is considered to be the first necessary condition for sample representativeness. On the other hand, precision can be more or less controlled, for example, by increasing the number of samples taken or by decreasing the sampling or measurement variabilities, or both.

5.6.2.4 The optimal number of samples to take to achieve a desired level of precision is typically an issue in optimization of a sampling plan. Therefore, the precision issue will be covered only briefly in this guide.

6. A Systematic Approach to Representative Sampling

6.1 A systematic approach is one that first defines the desired end result and then designs a process by which such a result can be obtained. In representative sampling, the desired end result is a sample or a set of samples that achieves desired levels of low bias and high precision.

6.2 A representative sampling process is described in Fig. 1. The key components in the process are described in this section.

6.3 *Study Objective*— A sampling plan is designed according to a defined problem or a stated study objective. The samples are then collected according to the sampling plan. Generally, the study objective dictates that representative samples be taken for the purpose of inference about the population. In that case, these samples will need to be collected according to this guide in order for the inference to be valid.

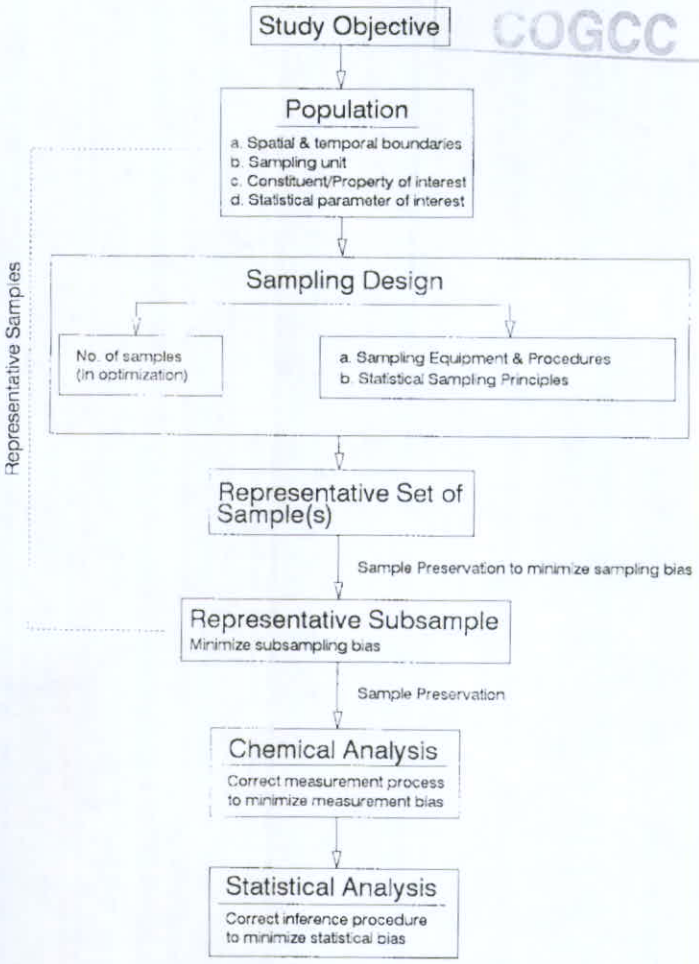


FIG. 1 A Systematic Approach to Representative Sampling

Occasionally, the objective is merely to detect the presence of a contaminant or to obtain a "worst case" sample. In that case, an authoritative sampling approach (biased sampling or judgment sampling) may be taken and this guide does not apply.

6.4 *Population*—A population consists of the totality of items or units of materials under consideration (Compilation of ASTM Standard Definitions, 1990). Its boundaries (spatial or temporal, or both) are defined according to the problem statement. This population is usually called the *target population*. In order to solve the stated problem, samples must be taken from the target population.

6.4.1 *Sampled Population*—Sometimes some parts of the target population may not be amenable to sampling due to factors such as accessibility. The boundaries of the target population actually sampled due to factors such as incomplete accessibility define the sampled population.

6.4.1.1 Although the samples taken from the sampled population may be representative of the sampled population, they may not be representative of the target population. In this case, potential exists that the samples taken from the sampled population may systematically deviate from the true value of the target population, thereby introducing bias when making inference from the samples to the target population.

6.4.1.2 When the boundaries of the target and sampled populations are not identical, some possible solutions are:

(1) The parties to the decision-making may agree that the

sampled population is a sufficient approximation to the target population. A sampling plan can then be designed to take representative samples from the" sampled population,"

(2) Qualifications on the sampling results are made based on the differences between the two populations. Some professional judgment may have to be exercised here, and

(3) Redefine the problem by considering what problem is solvable based on the observed differences between the two populations.

6.4.1.3 Occasionally, the sampled population is chosen on purpose to be different from the target population. For example, an investigator may be interested in the lead content in the sludge of a surface impoundment (the target population). He may decide to take samples from the sludge near the inlet (sampled population). Thus, the impoundment is the target population, while the inlet area is the sampled population. If the interest is in the target population, then this is an example of a biased sampling approach. On the other hand, the involved parties may decide to redefine the target population to include only the inlet area. Then the target population and the sampled population are identical. Again, the definition of a population depends on the problem statement.

6.4.1.4 In yet other circumstances, an investigator may take only a sample from the population. The following cases are possible:

(1) This one physical sample can be a sample from a biased sampling approach, for the purpose of detecting the presence of a contaminant or identifying the source of contamination. Therefore, it is not a representative sample due to its bias,

(2) This one physical sample can be a sample from judgment sampling, for the purpose of estimating the average condition of the population. Bias may or may not exist depending to some degree on the expertise of the sampler,

(3) This sample can be viewed as a population itself if the investigator is interested in the sample alone and a result from this sample is not to be used to infer to areas outside the sample. In this case, no bias exists, and

(4) If this sample is the composite of a few samples taken from the population, bias is likely to be minimal if the original samples are carefully taken.

6.4.2 *Decision Unit*— Often a population may be divided into several exposure units, cleanup units, or strata. If the environmental management decision is to be made for the entire population as a whole, representative samples can be obtained by designs such as a stratified random sampling design. Here the entire population is the decision unit. On the other hand, if the decision is to be made on each unit or stratum, then each unit or stratum is the decision unit. In this case, representative sample(s) need to be taken from each unit or stratum as if the unit or stratum is the population.

6.4.2.1 If the units or strata are relatively small in size or too numerous to take many samples per unit or stratum, composite sample(s) can be taken from each unit or stratum to increase precision without introducing bias. Alternatively, if precision is not a concern and there is sufficient professional expertise to avoid bias, a judgment sample(s) can be taken from each unit or stratum.

6.4.3 *Heterogeneity*— Heterogeneity is discussed in greater detail in Guide D 5956.

6.4.3.1 The degree and extent of population heterogeneity affect potential bias and precision in the samples. Population heterogeneity can be viewed at least in three different ways:

(1) When the population is heterogeneous in a random manner in only the distribution of the concentration, but not in the physical materials such as particle sizes, designs such as a simple random sampling design will generally produce samples with minimal bias. Its precision will then depend on the number of samples taken,

(2) When the population is randomly heterogeneous in concentrations due to large differences in the materials such as particle size, a simple random sampling design may still be effective if the sample volume/weight and sampling equipment are chosen to accommodate the largest particles and thereby prevent introduction of bias, and

(3) If the population is systematically heterogeneous, such as the presence of stratification in concentrations, then a simple random sampling design may not be biased, but will be less precise than an alternative design such as stratified random sampling.

6.4.3.2 Heterogeneity in the population affects the sampling variance. Sampling variance is a function of factors such as the population heterogeneity and the sample volume or weight. It is clear that the more heterogeneous the population is, the larger the inherent sampling variance is. It is also clear that samples of smaller volume or weight will have a higher sampling variance than those with greater volume or weight. However, the reduction in sampling variance due to increased volume or weight may eventually reach a limit. Determination of the optimal sample volume or weight is beyond the scope of this guide.[7]

6.4.3.3 The proper procedure is to first determine the right sample volume or weight, then to determine the number of samples needed for the chosen sample volume or weight.

6.4.3.4 Since stratification as a phenomenon of population heterogeneity is fairly common, it is discussed in greater details as follows.

6.4.4 *Stratification*— There are generally three types of stratification affecting sample representativeness. One is a stratification in the distribution of the contaminant concentration distribution alone. The second is a stratification in sampling materials or matrices alone. The third is a combination of both types. Stratification of any type is not a big problem regarding sample representativeness if each stratum is a decision unit. In that case, the units in a stratum are by definition relatively similar, apart from the random variations in concentrations. A simple random sampling design can be used to obtain representative samples (unbiased) for each stratum. The question of sample representativeness becomes more complicated when a decision is to be made over all the strata in the population.

6.4.4.1 *A Single Representative Sample in A Stratified Population*—When the objective is to obtain a single (physical) representative sample of all the strata, the sample must be a composite of individual samples from the strata (for example, at least one individual sample per stratum). Here the volumes or weights of the individual samples should be proportional to

the relative stratum sizes. The composite sample so obtained would be unbiased. However, since there is only one composite sample, precision of the composite sample cannot be estimated. If there are existing data on the precision of the individual samples in the strata, then the precision of the composite sample can be inferred from the precision of the individual samples by theoretical or empirical relationship. See Guide D 6051.

6.4.4.2 *A Representative Set of Samples*—When the population is stratified, a set of samples obtained by statistical designs such as stratified random sampling, where the number of samples to be taken from the strata are proportional to the relative sizes of the strata, is unbiased and more precise than a set of samples taken without considering the stratification.

6.4.5 *Parameter(s) of Interest*—This refers to the statistical parameter such as mean or variance of the population. It is often used with a characteristic such as concentration of a constituent(s) of the population. An example is the mean (parameter) concentration (characteristic) of lead (constituent). Another example is a population of mixture of silt-size calcium carbonate particles and large cobble-size particles of calcium carbonate. The interest here could be in the mean (parameter) particle size or chemical composition (characteristic) of calcium carbonate (constituent), depending on the study objective.

6.5 *Develop A Sampling Design*—The objectives of a sampling design are to minimize bias and achieve a desired level of precision. Precision and bias are an issue at various stages of the process of inferring from the samples to the population. The first stage is the act of obtaining the physical samples. The second stage is the act of analyzing the physical samples and translating them into data. The third stage is the use of statistical method to infer from the sample data to the population. At the first stage, the main concerns are sampling precision and bias. At the second stage, the concerns are measurement of precision and bias. At the third stage, the concern is statistical bias.

6.5.1 At the first stage of obtaining physical samples, the issues of precision and bias are sometimes grouped together as sampling design issues.

6.5.2 Bias at this stage is often called the sampling bias. Sampling bias is the systematic difference between the value inherent in the physical samples and the true population value. The word "inherent" is used because at this point the physical samples have not been translated into data.

6.5.3 The phrase "systematic difference" implies a persistent difference in long-term average or expectation, not the occasional random difference. Representative samples, apart from the issue of precision, are obtained when this long-term expected difference is zero or nearly so.

6.5.4 Since the true population value is typically not known, sampling bias cannot be assessed. However, efforts to minimize sampling bias can be attempted in at least two areas:

6.5.4.1 *Proper Statistical Sampling Design*—Statistical sampling design has to do with where and how samples are to be taken, where equal probability of selecting any of the units or items in the population is often a primary requirement. If the probability of selection is not equal, it is highly likely that bias will have been introduced into the physical samples so obtained. Depending on the layout of the population, designs such as simple random sampling or stratified random sampling can be used.

6.5.4.2 *Proper Sampling Procedures and Sampling Equipment*—This includes proper procedures for compositing, subsampling, sample preparation and preservation, and proper use of the chosen sampling equipment. This is a major source affecting precision and bias, especially bias.

6.5.5 In the case of precision, it can be controlled by things such as the number of samples taken, the use of composite samples, or more precise sampling techniques. Often, the number of samples to take is considered the key design issue. Some considerations regarding precision are:

6.5.5.1 If a population is relatively small compared to the sample mass/volume and the distribution of the characteristic of interest is random, it may be appropriate to collect a smaller number of samples by a random or systematic sampling approach, and

6.5.5.2 If a population is relatively large compared to sample mass/volume and the characteristic of interest is not randomly distributed (for example, stratified), a greater number of samples and a stratified sampling approach may be needed.

6.5.6 *Compositing*— Compositing is the combination of two or more individual physical samples into a single sample. It is often used to reduce the analytical costs, while maintaining or increasing precision relative to the individual samples (see Guide D 6051). Bias may or may not be introduced in compositing, depending on the study objective and the physical means of compositing. For example:

6.5.6.1 If the study calls for the estimation of the population variance (or standard deviation) of individual samples, then composite samples will surely underestimate the population variance, and

6.5.6.2 If the physical means of compositing changes the characteristics of the samples, then bias may have been introduced (unless such changes are part of the study design).

6.6 *Subsampling*— Sampling bias can be introduced in subsampling unless the same proper sampling protocol is followed as in taking samples from the original population.

6.6.1 *Discussion*—After the physical samples have been obtained and before they are measured, bias can be prevented by following proper sample preservation and preparation procedures. It is not important whether these procedures are viewed as part of the sampling process or as part of the measurement process. It is only important in following the proper procedures to prevent bias.

6.7 *Measurement of Precision and Bias*:

6.7.1 The measurement process, like the sampling process, also consists of a random error and a systematic error. The random errors define the degree of measurement precision, and the systematic error defines the degree of measurement bias.

6.7.2 Like sampling precision, measurement precision is controlled by things such as the number of replicate analyses performed per sample and refinements of the analytical method.

6.7.3 Measurement bias is a systematic difference between the sample value produced by the measurement process and the true population value, assuming that the physical samples are

unbiased before the analysis. The bias can come from contamination, loss or alteration of the sample materials, systematic errors in the measurement device, or from systematic human errors.

6.7.4 Often the measurement bias can be reasonably estimated in a laboratory testing setting when the true value is known. Laboratory samples spiked with known quantities of a chemical or certified reference standard can often be used to assess potential measurement bias. Minimization or adjustment for such estimable bias in the measurement process is essential in order to obtain data that are unbiased. When estimation of bias is not possible, care in measurement protocol and training is probably the only recourse.

6.7.4.1 *Discussion*—It is important to note that, when inferring from the sample data to the population, all the sources of imprecision, including sampling, subsampling, and measurement, need to be combined. The process of accumulating these sources of variation is sometimes called the "propagation of errors." The determination of the optimal numbers of samples, subsamples, and replicates are an issue of optimization and is not covered in this guide.

6.8 *Statistical Bias*— Statistical bias can result from an inappropriate sampling design or inappropriate estimation procedures, or both:

6.8.1 *Selection Bias from Sampling Design*—In the course of taking the sample, if the population units do not have the same probability of being selected, bias can be introduced. This bias can be prevented or minimized when a statistical sampling design is carefully selected, based on the study objective and the layout of the population. Some possible designs are the simple random sampling design and the stratified random sampling design.

6.8.2 *Estimation of Bias from Estimation Procedures*—This bias occurs when the expected value of the statistical estimator does not equal the true value.

6.8.2.1 Estimation bias can occur when the wrong statistical distribution of the data is used. For example, if the normal distribution assumption is used when the true data distribution is lognormal, the interval estimate of the mean concentration will be an biased estimate against the true interval. Thus, the expected value of the estimator will not be equal to the true value. To avoid this potential bias, it is wise to check the data distribution.

6.8.2.2 Estimation bias can also occur when a wrong statistical estimator is used. For example, if the sum of squares of deviations from the sample mean divided by the number of samples (that is, $\Sigma_{i=1,n}\ (x_i - \bar{x})^2/n$) is used to estimate the population variance, then this estimator is biased (its mathematical expected value is not equal to the population variance). If its denominator is modified to be $(n-1)$, then it is an unbiased estimator. For an unbiased statistical estimator, the reader is advised to check with a statistician.

7. Attributes of Representative Samples

7.1 The attributes of a representative (physical) sample or a representative set of (physical) samples can be described in the chronological order in which samples are taken. Note that these attributes apply only to how representative the physical samples are of the population. This corresponds to the upper half of Fig. 1.

7.2 *Design Considerations*:

7.2.1 A well-defined target population. The target population includes all the population units as determined from the stated problem.

7.2.2 The sampled population equals the target population in their spatial or temporal boundaries, or both. The sampled population consists of the population units directly available for measurement.[8]

7.2.2.1 When all the population units in the target population are accessible and directly available for measurement, then the sampled population is identical to the target population in its spatial or temporal boundaries, or both.

7.2.2.2 When not all the population units are directly available for measurement, then the inference from the sample is made to the sampled population, not the target population.

7.2.3 Size (weight or volume) of the sampling unit is well defined.

7.2.3.1 The population can be divided into various sizes (weight or volume) of population units. The size of the sampling unit is the size of the population unit most appropriate for the sampling purposes.

7.2.3.2 The appropriate size of the sample is determined by degree of heterogeneity of the materials to be sampled, such as particle size or shape.

7.3 *Sampling and Measurement Considerations*:

7.3.1 Correct sampling procedures are followed to minimize sampling bias.

7.3.1.1 Absence or minimization of bias is a key attribute of representative samples. Sampling bias can be minimized by following correct sampling procedures. Correct sampling procedures have two components.

(1) A sampling procedure that maximizes the potential of population units having equal probability of selection as sampled, and

(2) Correct sampling procedures. This includes the selection of appropriate equipment and proper use of that equipment.

7.3.2 Sample integrity is maintained during sampling and before chemical analysis.

7.3.3 If subsampling is performed, correct sampling procedures are followed to minimize sampling bias.

7.3.4 Sample preparation errors such as contamination and loss or alteration of constituents are prevented or minimized.

7.3.5 The samples, in the end, collectively reflect the target population within the context of the problem.

7.3.6 These attributes can be summarized into three broad categories:

7.3.6.1 A well-defined population,

7.3.6.2 Correct sampling procedures, and

7.3.6.3 Samples collected in the context of the stated problem.

[8] Gilbert, Richard O., *Statistical Methods for Environmental Pollution Monitoring*, Van Nostrand Reinholt Co., New York, NY 1987.

8. Practical Considerations

8.1 *Sampling Equipment*—The choice of appropriate sampling equipment can be crucial to the task of collecting a representative sample or a representative set of samples. Depending on the goals of the sampling activity, the sampling device used should minimize bias by having certain characteristics and capabilities, such as:

8.1.1 The ability to access and extract from every location in the target population,

8.1.2 The ability to collect a sample of proper shape,

8.1.3 The ability to collect a sufficient mass or volume of sample such that the distribution of particle sizes in the population are represented, and

8.1.4 The ability to collect a sample without the addition or loss of contaminants of interest.

8.2 *Equipment Design*— The improper design of sampling equipment may result in the collection of samples that are not representative of the population.

8.2.1 An example of equipment design influencing sampling results is samplers which exclude certain sized particles from a soil matrix or waste pile sample. The shape of some scoops may influence the distribution of particle sizes collected from a sample. Dredges used to collect river or estuarine sediments may also exclude certain sized particles, particularly the fines fraction which may contain a significant percentage of some contaminants such as polynuclear aromatic hydrocarbons (PAHs). Specific considerations in equipment design are outlined as follows.

8.2.1.1 *Sample Volume Capabilities*—Most sampling devices will provide adequate sample volume. However, the sampling equipment volumes should be compared to the volume necessary for all required analyses and the additional amount necessary for quality control (QC), split and repeat samples. Taking more than one aliquot to obtain an adequate sample volume can impact the representativeness of a sample.

8.2.1.2 *Compatibility*— It is important that sampling equipment, other equipment that may come in contact with samples (such as gloves, mixing pans, knives, spatulas, spoons, etc.) and sample containers be constructed of materials that are compatible with the matrices and analytes of interest. Incompatibility may result in the contamination of the sample and the degradation of the sampling equipment.

8.2.1.3 *Decontamination (see Practice D 5088) and Reuse*—Inadequate decontamination of sampling equipment can result in contamination of the sample and affects its representativeness. Due to design, some equipment is very difficult to adequately decontaminate. In some instances, it may even be desirable to either dispose of sampling equipment after use or to dedicate the equipment to a sampling point.

8.3 *Sampling Procedure*—Inappropriate use of sampling equipment is one of the largest sources of sampling bias. While it is beyond the scope of this guide to discuss it in depth, examples of how bias can be introduced during the sampling procedure are discussed in the following paragraphs. This guide does not provide comprehensive sampling procedures. It is the responsibility of the user to ensure that proper and adequate procedures are used.

8.3.1 *Ground Water*— For a more comprehensive discussion of sampling ground water refer to Guide D 4448.

8.3.1.1 Ground-water samples are usually collected through an in-place well, either temporarily or permanently installed. The following is a list of concerns that should be considered when collecting a ground-water sample.

(1) The well should be purged before collecting samples in order to clear the well of stagnant water which is not representative of aquifer conditions. Purging and sampling rates can cause chemical or physical changes in the water.

(2) Purging can be performed in such a way that the entire column of water is not removed. The best method for avoiding this situation is by lowering a pump or bailer into the top of the column of water.

(3) Bailing may stir up sediment in the well if conducted too vigorously. Increased turbidity can result in a higher metal content in the sample than in a non-turbid sample.

(4) Samples for volatile organic analysis should be collected in a fashion that minimizes agitation of the sample.

(5) Wells with in-place plumbing must also be purged. Samples should be collected immediately following purging. In order to collect a sample representative of ground water, samples should be collected before the water travels through any hoses or in-line treatment devices.

8.3.2 *Surface Water and Sediment*—For a more comprehensive discussion of sampling surface water and sediment, refer to Practice D 3370 and Guide D 4823. General and specific sampling concerns for collection of surface water and sediment samples are as follows:

8.3.2.1 *General Considerations*:

(1) Although bridges and piers may provide access for water and sediment sampling, these structures can also alter the nature of water flow and thus influence sediment deposition or scouring. Depending on the construction materials, these structures can contaminate samples collected in the immediate vicinity.

(2) Wading for water samples should be done with caution since bottom deposits are easily disturbed resulting in increased sediment in surface water samples and a removal of fines from the sediment sample.

8.3.2.2 *Rivers, Streams, and Creeks*:

(1) A good location to collect a vertically mixed surface water sample is immediately downstream of a riffle area. This location is also a likely area for deposition of sediment since the greatest deposition occurs where stream velocity slows down.

(2) Horizontal (cross-channel) mixing occurs in constrictions in the channel. However, this is a poor sediment sample collection area because of scouring.

(3) Surface water samples will be affected by point sources, such as tributaries and industrial and municipal effluents.

(4) Locations immediately upstream or downstream from the confluence of two streams or rivers may not immediately mix, and at times, due to possible back flow, can upset the normal flow patterns.

(5) Unless a stream is extremely turbulent, it is nearly impossible to measure the effect of a waste discharge or tributary immediately downstream of the source. Inflow frequently "hugs" the stream bank with very little cross-channel

mixing for some distance. Samples from quarter points across a stream may miss the wastes altogether and reflect only the quality of water upstream from the waste source. Samples collected within the portion of the cross section containing the wastes would indicate excessive effects of the wastes with respect to the river as a whole.

(6) When sampling tributaries, care should be exercised to avoid collecting water from the main stream that may flow into the mouth of the tributary on either the surface or bottom.

8.3.2.3 *Lakes, Ponds, and Impoundments*:

(1) Stratification of surface water is of greater concern in standing water. For example: A turbidity difference may occur vertically where a highly turbid river enters a lake, and each layer of the stratified water column may need to be considered. In addition, stratification may be caused by water temperature difference; cooler, heavier river water is beneath the warmer lake water.

(2) Dredges used to collect sediment samples can displace and miss lighter materials if allowed to drop freely.

(3) Core samplers used to sample vertical columns of sediment are useful when there is a need to know the history of sediment deposition. Coring devices also minimize the disturbance of fines at the sediment-water interface. However, coring devices can only sample a relatively small surface area. Depending on the core diameter, larger particles may be excluded and a single aliquot may not be sufficient for analytical needs.

8.3.3 *Soils*—For more detailed information, refer to Practice D 4547 and Guide D 4700. General areas of concern for sampling soils are as follows:

8.3.3.1 Soil samples for purgeable organic analyses should be collected with a minimum disturbance of the sample.

8.3.3.2 Samples for VOA analysis should not be mixed.

8.3.3.3 Two potential problems are associated with compositing soil samples. Low concentrations of contaminants present in individual aliquots may be diluted to the extent that the total composite concentration is below the minimum quantification limit. In addition, depending on the soil type, it can be very difficult to produce a homogeneous mixture.

8.3.4 *Waste*—Wastes referred to in this section include any liquid, solid, or sludge from pits, ponds, lagoons, waste piles, landfills, and open or closed containers such as drums, tank trucks, and storage tanks.

8.3.4.1 Any of these units may have multiple phases (floating solids, different density liquid phases, and sludge) and one or all of them may need to be sampled.

8.3.4.2 If sampling from access valves or ports on an open or closed container, care should be taken to be sure that the desired layer is sampled. For example, bottom sampling ports would allow only the heavier contents to be sampled while surface or top sampling would allow only sampling of the lighter layers.

8.4 *Subsampling (Field)*:

8.4.1 Different analyses require different types of bottles and preservation. For multiple analyses of the same waste stream, this may require subsampling in the field. Subsampling in the laboratory may require many of the same procedures; however, laboratory subsampling is beyond the scope of this guide.

8.4.1.1 Samples for organic analyses should always be taken from the first material collected. This minimizes loss of volatile organics during handling of the material.

8.4.1.2 If necessary, place the appropriate volume of material in a tray or other suitable container to composite. The volume is dependent on the needed analyses, and should be specified by the analytical laboratory.

8.4.1.3 Transfer the material into the required containers for analyses. If subsampling takes place, then the analytical sample is the final portion of the material subsampled from the original sampling unit and analyzed in the laboratory.

8.4.2 In subsampling, the original sampling unit can be considered as the population and the correct sampling procedures must be followed to ensure a representative subsample.

9. Keywords

9.1 bias; contaminated media; precision; representative; sample; waste; waste management

APPENDIX

(Nonmandatory Information)

X1. TWO CASE STUDIES OF REPRESENTATIVE SAMPLING

X1.1 Case Study One—Waste Pile Investigation

X1.1.1 *Background*— An industrial facility has managed recovery furnace slag and baghouse dust in a waste pile located on the site. No active management was occurring with the waste pile. No buried containers or extremely heterogenous material (debris) was suspected of being present in the waste pile based on facility records and interviews of personnel.

X1.1.1.1 Lead and cadmium were the constituents of concern based on process knowledge, and the possibility for the waste being hazardous by means of the Toxicity Characteristic (TC) Rule was the regulatory consideration. No preliminary information on the variability of lead and cadmium within the piles was available. The potential for off-site migration of contaminants by means of a drainage ditch that leads to a stream adjacent to the facility was an immediate concern.

X1.1.2 *Phase 1: Objective*—The primary objective of the initial investigation was to determine if the slag and baghouse dust in the waste piles were characteristic for lead via the Toxicity Characteristic Rule. A secondary objective was to provide preliminary information on potential migration and transport of contaminants from the waste piles off site.

X1.1.2.1 The sampling design for this initial investigation

utilized a judgmental sampling strategy to provide a preliminary estimate of the lead and cadmium concentrations in the waste pile, the variability of contaminant concentrations in the pile, and the potential for leaching using the TCLP. Four areal composite samples were collected from the surface (0 to 6 in.) at the four quadrants of the waste pile. Borings were completed at the center of each area that was sampled on the surface. Each four-foot interval was analyzed to assess vertical variability.

X1.1.2.2 The following environmental samples were also collected using a judgmental approach:

(1) Several soil samples in the vicinity of the waste pile,

(2) Sediment upstream and downstream in a stream that borders the facility,

(3) Sediment in a ditch which contained run-off from the pile, and

(4) Two background soil samples.

X1.1.2.3 *Results*—Zinc, copper, cadmium, and lead were all elevated (compared to background) in the samples collected from the waste piles. Since lead and cadmium are TC Rule constituents, the TCLP was completed, and the lead results exceeded the regulatory level of 5 mg/L. Cadmium was just under the regulatory level of 1.0 mg/L. Lead and cadmium concentrations in the soil near the waste piles were 2 to 3 times above background, and the drainage ditch and downstream sediment sample also had elevated lead and cadmium levels.

X1.1.2.4 *Conclusion*— The waste piles contain slag and baghouse dust that is hazardous for lead. The waste pile requires further characterization to determine the variability in the pile. The presence of lead and cadmium in soils and the stream sediment downstream of the facility was confirmed and should be further investigated to determine the extent of contaminant transport.

X1.1.3 *Phase 2: Objective*—The sampling design utilized a systematic grid approach. This design will delineate horizontal and vertical variability in lead and cadmium concentrations. The Phase 1 investigation also provided a good estimate of the anticipated variability in the waste pile.

X1.1.3.1 The number of samples required to adequately characterize the waste pile was calculated based on the anticipated variability, the regulatory level of concern, and the specified confidence interval. The grid sizes were then adjusted to accommodate the projection on the required number of samples. Composite samples were collected within each grid cell based on one center point and eight points on the compass (45 deg intervals) equidistant from the center point.

X1.1.3.2 Twenty percent of the grids were designated for vertical characterization (at the grid center) at four-foot intervals, as well as surface (0 to 6 in.) sample collection. Additionally, ten percent of the grids were randomly designated for duplicate sampling (using a different aliquot pattern within the cell) to check the preliminary estimate on the variability.

X1.1.3.3 Additional environmental sampling was conducted that included a systematic sampling design for the stream adjacent to the facility with sediment samples collected at 100-ft intervals. A systematic approach was also used for the drainage ditch (50-ft intervals), with judgmental samples being collected at any location where visible staining was observed.

X1.1.3.4 *Results*—The results supported the initial investigation with lead consistently exceeding the TC Rule regulatory level; cadium was consistently below the regulatory level. Vertical differences in the lead and cadmium concentrations were not significant. Lead and cadmium were detected at elevated concentrations (relative to background) in the adjacent stream at a point downstream of the confluence with the drainage ditch.

X1.1.3.5 *Conclusion*— The waste pile was characteristic for lead and subject to Subtitle C of RCRA. There was no significant variability with depth, although several gradients were noticed across the grid (horizontally) based on lead concentration (scan) results.

X1.2 Case Study Two—Drum Sampling

X1.2.1 *Background*— An industry has two areas where drums of waste have been stored. One area is a warehouse adjacent to an off-line plating process that contains less than 25 drums (55 gal). The drums have manufacturers' labels indicating they contain an acid solution, and all of the drums are similar in appearance. A second area is a covered shed that has an estimated 100 drums from a variety of processes, several of which are no longer in use at the facility. Information on the content of these drums is not available.

X1.2.2 *Objective*—The objective of the initial investigation was to survey both of the storage areas for safety purposes, assess and record information on the drums, and open drums that were candidates for screening. All drums that were opened were surveyed using an organic vapor analyzer (PID, FID), pH paper, halogen detector, cyanide detector, and radiation meter.

X1.2.2.1 A judgmental sampling design was utilized in the warehouse where the anticipated variability was low. Based on the site screening (pH measurement), six samples were collected for pH analysis from the warehouse.

X1.2.2.2 The drums in the shed were screened in a similar fashion. A variety of results were obtained which included elevated pH, high organic vapor readings, and so forth. A simple random sampling design was used which called for the collection of 15 samples, with five from each major group of drums based on the screening (five corrosives, five potential ignitables with no halogens, and five with elevated halogen readings).

X1.2.2.3 *Results*—The warehouse samples were all corrosive with pH values from 1 to 2 S.U. The shed samples resulted in the collection of five corrosive wastes, three that were both ignitable and characteristic for non-halogenated TC Rule constituents, and two that were ignitable and characteristic for halogenated constituents. In summary, of the 15 drums sampled, 10 contained hazardous waste.

X1.2.2.4 *Conclusions*— All of the drums in the warehouse are subject to Subtitle C of RCRA. The drums in the shed require further assessment due to the fact that several of those sampled did not contain hazardous waste.