

**ATTACHMENT II-PCB WAP**  
**(CELL B/6 PCB WASTE ANALYSIS PLAN)**  
**Clean Harbors Grassy Mountain Facility, LLC**  
**September 28, 2012**



1.0	INTRODUCTION	1
2.0	ANALYTICAL PARAMETERS AND RATIONALE	3
2.1	FINGERPRINT ANALYSES	3
2.2	ADDITIONAL ANALYSES	4
2.2.1	UNIQUE ADDITIONAL ANALYSES	5
2.2.2	ANALYSES USING STANDARD TECHNIQUES	5
3.0	ANALYTICAL PROCEDURES	7
3.1	FINGERPRINT ANALYSES	8
3.2	ADDITIONAL ANALYSES	9
3.2.1	UNIQUE ADDITIONAL ANALYSES	9
3.2.2	ANALYSES USING STANDARD TECHNIQUES	9
4.0	SAMPLING METHODOLOGY	10
4.1	METHODOLOGY	10
4.2	TYPES OF CONTAINERS	11
4.2.1	CONTAINERS AND TANKS	11
4.3	GENERAL CONSIDERATIONS	12
4.3.1	DISPOSITION OF SAMPLES	12
4.3.2	CLEANING OF SAMPLING APPARATUS	12
4.3.3	FROZEN SAMPLES	13
4.3.4	SAMPLING SAFETY PRECAUTIONS	13
4.3.5	REMOTE SAMPLING AND/OR ANALYSIS	13
5.0	WASTE SCREENING PROCEDURES	13
5.1	PROCEDURAL REQUIREMENTS	14
5.2	EVALUATION	16
6.0	INCOMING LOAD PROCEDURES	18
6.1	RECEIVING PROCEDURES	18
6.2	DECISION EVALUATION LOGIC	21
7.0	PROCESS OPERATIONS PROCEDURES	23
7.1	STORAGE	24
7.1.1	STORAGE TANKS	24
7.1.2	STORAGE CONTAINERS	24
7.2	TREATMENT OPERATIONS	25
7.2.1	SOLIDIFICATION	25
7.3	LANDFILL DISPOSAL	26
7.3.1	WASTE AND LEACHATE/LINER COMPATIBILITY	26
7.3.2	IGNITABLE, REACTIVE AND INCOMPATIBLE WASTES	26
7.3.3	WASTES CONTAINING FREE LIQUIDS	26
7.3.4	WASTES CONTAINING ASBESTOS	27
8.0	UNIQUE ANALYSIS PROCEDURES	29
9.0	QUALITY ASSURANCE AND QUALITY CONTROL	30
9.1	PROJECT DESCRIPTION	30
9.1.1	DATA USE	30
9.1.2	PROJECT DIAGRAMS	30
9.2	PROJECT ORGANIZATION	30
9.3	QUALITY ASSURANCE OBJECTIVES	30
9.3.1	QA OBJECTIVES FOR MEASUREMENT DATA IN TERMS OF PRECISION, ACCURACY, COMPLETENESS, REPRESENTATIVENESS, AND COMPARABILITY	31
9.4	SAMPLING PROCEDURES	32

9.4.1	INITIAL CONSIDERATIONS	32
9.5	SAMPLE CUSTODY	37
9.5.1	PRE-ACCEPTANCE SAMPLES	37
9.5.2	LOAD SAMPLES	37
9.5.3	OUTSIDE ANALYSIS	37
9.6	CALIBRATION PROCEDURES AND FREQUENCIES	38
9.6.1	CALIBRATION	38
9.6.2	pH/SELECTIVE ION METERS	38
9.6.3	BALANCES	39
9.6.4	HYDROGEN SULFIDE ANALYSIS	39
9.6.5	TLV SNIFFER	39
9.6.6	FLASHPOINT	39
9.6.7	SPECIFIC GRAVITY	40
9.6.8	NORMALITY	40
9.7	DATA REDUCTION, VALIDATION, AND REPORTING	40
9.7.1	DATA REDUCTION	40
9.7.2	DATA VALIDATION	41
9.8	INTERNAL QUALITY CONTROL CHECKS	42
9.8.1	PURPOSE OF LABORATORY QUALITY CONTROL	42
9.8.2	OVERVIEW OF SPECIFIC QUALITY CONTROL PROCEDURES	42
9.8.3	INTERNAL QUALITY CONTROL	43
9.9	REPLICATE SAMPLES	43
9.10	BLANKS	44
9.11	CALIBRATION MATERIALS	44
9.12	SPIKE SAMPLES	45
9.13	QUALITY ASSURANCE PROGRAM REVIEW	45
9.13.1	PERFORMANCE AUDITS	45
9.13.2	SYSTEM AUDITS	46
9.14	PREVENTIVE MAINTENANCE	46
10.0	SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS	46
10.1	PROCEDURE FOR ASSESSMENT OF PRECISION MEASUREMENTS:	47
10.2	PROCEDURES FOR ASSESSMENT OF ACCURACY	47
10.3	PROCEDURES FOR THE ASSESSMENT OF SURROGATE RECOVERY	47
10.4	INSTRUMENT CALIBRATION	48
10.5	GENERAL LABORATORY PARAMETERS	48
10.6	CORRECTIVE ACTION	49
10.6.1	BLANKS	49
10.6.2	LABORATORY CONTROL SAMPLES	49
10.6.3	DUPLICATES	50
10.6.4	SURROGATE AND MATRIX SPIKES	50
10.6.5	CONTINUING CALIBRATION CHECKS	50
10.7	INSTRUMENT CALIBRATION CHECKS	51
10.8	SPIKE RECOVERIES	51
10.9	DUPLICATE	51
10.10	PERFORMANCE AUDITS	52
10.11	QUALITY ASSURANCE REPORTS TO MANAGEMENT	52

**APPENDICES**

**APPENDIX 1 - OTHER FACILITIES HANDLING PCB CONTAINING WASTES (INFORMATION PURPOSES ONLY)**

**APPENDIX 2 - LINER/LEACHATE COMPATIBILITY REPORT**

**FIGURES**

<b><u>FIGURE</u></b>	<b><u>PAGE</u></b>
<b>FIGURE 5-1 Waste Profile Sheet for PCB Waste Shipment .....</b>	<b>-17-</b>
<b>FIGURE 6-1 FINGERPRINT ANALYSES .....</b>	<b>-24-</b>

**TABLES**

<b><u>TABLE</u></b>	<b><u>PAGE</u></b>
<b>TABLE 2.1 Analyses Run on Specific Waste Types .....</b>	<b>-5-</b>
<b>TABLE 9-1 PRECISION AND ACCURACY REQUIREMENTS FOR FINGERPRINT ANALYSIS .....</b>	<b>-38-</b>
<b>TABLE 9-2 PRECISION AND ACCURACY REQUIREMENTS FOR LOAD AND PROFILE ANALYSIS PARAMETERS .....</b>	<b>-39-</b>



## 1.0 INTRODUCTION

Clean Harbors Grassy Mountain Facility, LLC (Grassy Mountain) has established the following procedures to govern the acceptance of PCB containing wastes destined for Cell B\6, the TSCA\RCRA disposal cell. The procedures established in this Waste Analysis Plan (WAP) will assure that Cell B\6 will be in compliance with all the requirements of 40 CFR 761.75. The most recent approved revision of this plan shall be maintained at the facility as part of the operating record. In the event that there is a difference between the RCRA Waste Analysis Plan and the Cell B\6 Waste Analysis Plan, the more stringent of the two plans shall be applicable.

PCB containing wastes will hereafter be referred to as " PCB waste" or "waste." The WAP establishes the following:

- The parameters for which each PCB waste will be analyzed and the rationale for the selection of these parameters (See Section 2.0);
- The test methods which will be used to test for these parameters (See Section 3.0);
- The sampling method which will be used to obtain a representative sample of the PCB waste (See Section 4.0);
- The frequency with which the initial analysis of the PCB waste will be reviewed or repeated (See Section 5.0); and
- The PCB waste analyses that waste generators have agreed to supply (See Section 5.0).

PCB containing wastes, which are to be landfilled, shall be segregated as follows at the facility:

- The following PCB waste types are examples of the wastes that will be disposed of in Cell B\6 (**after examination for physical appearance**): (Note: Similar wastes with a PCB concentration of <50 ppm PCB's are also included here, if applicable)
  - a. Contaminated debris and/or rags;
  - b. PCB-contaminated or small PCB capacitors; (Note: Generator origin of small capacitors is critical to acceptance!)
  - c. Drained or drained and flushed PCB hydraulic machines per 40 CFR 761.60(b)(3);
  - d. Drained PCB articles per 40 CFR 761.60(b)(5)(I)(B) and (ii) or containers per 40 CFR 761.60(c)(1)(ii) and (2); and

- e. Flushed PCB transformers per 40 CFR 761.60(b)(1)(I).
  - f. Asbestos or asbestos-containing materials contaminated with PCB's.
- The following PCB waste types shall be **sampled** and **analyzed** as per the WAP prior to ultimate disposal in Cell B: (Note: Similar wastes with a PCB concentration of <500 ppm PCB's are also included here)
    - a. Contaminated soils;
    - b. All free liquids and sludges contaminated with <500 ppm PCB provided, that those wastes do not contain more than 10% Total Organic Carbon (TOC) prior to solidification. (e.g. Dredged materials, industrial sludges, municipal sewage, and treatment sludges.
  - The following special PCB waste types include materials from on-site and off-site activities and are **exempt from the above sampling and analysis** requirements but shall be physically inspected prior to disposal, to verify the contents.:
    - a. Contaminated Trash and Debris. Consisting of rags, clothing, sampling/analysis apparatus, contaminated lab debris, glassware, pallets, etc.
    - b. "Empty" containers contaminated with PCB's. This applies to a portable container which has been emptied, but which may hold residuals of PCB's. Examples of containers are: portable tanks, drums, barrels, cans, bags, liners, etc. A container shall be determined "empty" according to the criteria specified in 40 CFR 261.7;
    - c. PCB equipment removed from service provided that adequate information is available from the generator to determine regulatory status (e.g., PCB origin and concentration; manufacturer's status; etc.). Examples: fluorescent light tubes, microwave ovens and fixtures, electronic equipment, etc.
    - d. PCB waste produced from the demolition or dismantling of industrial process equipment of facilities contaminated with PCB's. For these exceptions, the generator shall supply GRASSY MOUNTAIN with sufficient chemical and physical characteristics information for proper management of the waste.

Containers requiring storage prior to landfill disposal shall be stored in the container storage area of the drain and flush building at the PCB treatment facility or in an area in compliance with 40 CFR 761.65. (Wastes which are to be handled at the PCB treatment facility are discussed in Appendix 1 "Other Facilities".) "Staging" of PCB waste is only authorized for a period of five (5) calendar days or less in an area permanently designated outside of the TSCA disposal cell. Staging becomes storage after this period and must occur in a TSCA-approved storage area.

## 2.0 ANALYTICAL PARAMETERS AND RATIONALE

Analyses are provided by the laboratory to augment or verify pre-existing PCB waste identifications and to comply with facility acceptance criteria. Analytical methods are classified as "Fingerprint Analyses" and "Additional Analyses." Applicable wastes are subjected to the "Fingerprint Analyses" as referenced in Section 3.1 as a first step in the analytical protocol. "Additional Analyses" are performed according to need or, in some cases, may also be required analyses, as described in the Process Operations Procedures (Section 7.0). The Laboratory Manager or his/her designee may select these additional analyses to augment the mandatory screening or to provide operational control. Future references to Laboratory Manager refer to Laboratory Manager or his/her designee. The rationale for the "Fingerprint Analysis" and "Additional Analyses" are given in Section 2.0 and 3.0. Analyses are not necessarily repeated for sequential activities or movement of the same PCB waste within the facility, unless required by changes in the character of the waste or in order to document compliance with permit.

## 2.1 FINGERPRINT ANALYSES

Fingerprint Analyses include up to six (6) basic screening procedures that are performed to provide a general characterization of the PCB waste and used to indicate the type of treatment, storage, or disposal that is most suitable for that particular waste. Fingerprint Analyses are run on pre-acceptance samples and incoming load samples to allow for an expedient identification of PCB waste movements. This procedure serves as a check that the PCB waste shipped to the facility actually matches the waste originally profiled by the generator.

The parameters and associated rationale for the Fingerprint Analyses are as follows:

- **Physical Appearance** is used to determine the general identity of the waste. This facilitates subjective comparison of the waste with prior waste descriptions. It is also used to verify the presence or absence of free liquids. This is applicable to all PCB waste types. Drained or drained and flushed PCB items, as applicable, shall be inspected for free liquids as follows: The bottom drain valve shall be opened. Free-flowing liquids, if present, will be drained into containers approved for PCBs. Alternatively, the item or load may be rejected if liquids are present. The liquids shall be handled per this WAP. If no drain valve exists, then prior to the items being sent to the landfill the top cover shall be removed and an inspection for free flowing liquids will be done. The liquids shall be removed and handled as indicated above. PCB items represented as needing to be flushed are not subject to the above; they are transferred to a PPM or similar facility for

flushing prior to landfilling.

- **pH** is undertaken to indicate the corrosive nature of the waste. pH does not apply to certain wastes (equipment, transformers, debris, etc.).
- **Specific Gravity** is important in determining the sedimentation rate or buoyancy of wastes, as well as treatability. This is not applicable or required for all wastes (e.g. tyveks, equipment, debris, etc.).
- **Reactive Cyanides Screen** indicates whether the PCB waste produces hydrogen cyanide upon acidification below pH 2. It is not required if the pH of the waste is less than 6.0, if the waste is not water-soluble, or if the waste is not aqueous. Wastes containing total releasable cyanide with concentrations less than 250 ppm are considered non-reactive. This is not applicable or required for all wastes (e.g. tyveks, equipment, debris, etc.).
- **Reactive Sulfides Screen** indicates whether the PCB waste produces hydrogen sulfide upon acidification below pH 2. It is not required if the pH of the waste is less than 6.0, if the waste is not water-soluble, or if the waste is not aqueous. PCB waste containing total releasable sulfide with concentrations less than 500 ppm are considered non-reactive. This is not applicable or required for all wastes (e.g. tyveks, equipment, debris, etc.).
- **Explosivity Meter Vapor Test (TLV Sniff)** is used to indicate the fire-producing potential of the waste, and to indicate whether the PCB waste might be an ignitable waste or is regulated as flammable or combustible by the US DOT. This test is applicable only to liquids, soils, or sludges. The screen will be supplemented with the flash point test or ignitable solids screen if the TLV sniff is greater than 500.

Table 2.1 presents in tabular form the analyses which are run on specific PCB waste types.

## 2.2 ADDITIONAL ANALYSES

Additional Analyses may be performed to further identify wastes, as deemed appropriate by the Laboratory Manager, or his/her designee hereinafter referred to as the "Laboratory Manager". The results of these analyses provide the Laboratory Manager with another level of confidence concerning the proper means of treatment, storage and disposal. Some of these additional analyses utilize unique procedures and protocol formulated specifically for PCB waste management. Others are standard analytical techniques recognized by the US EPA and/or ASTM. The determination of PCB concentration in liquids is an example of the additional analyses performed by Grassy Mountain.

When a liquid PCB waste or PCB sludge is received at the facility, a Halogenated Organic Compound (HOC) screen shall be run on the load or batch after treatment in the Stabilization Tanks. This test will be conducted when the waste, upon arrival, fails the Paint Filter Test. The facility has the ability to

run the HOC screen without running the paint filter test. The HOC test will detect the presence of HOCs that might adversely affect the cell liner. Any one of the three site-specific methods may be used. This is required only for wastes destined for land disposal at GMF in Cell B16 or determined by the Laboratory Manager or by the State-issued Part B Permit.

### 2.2.1 UNIQUE ADDITIONAL ANALYSES

Unique Additional Analyses are derived procedures which were developed to provide operational control over on-site processes. These analyses are performed to further identify wastes and are run if the Laboratory Manager determines that further waste characterization is necessary.

The applicability of these analyses, as described below, is based on procedures and protocol designated in this plan.

- **Fixation Requirements** are run to determine if the solidification agent will eliminate the free liquids.
- Halogenated Organic Compounds is a special analytical method approved by both EPA Region VIII and UDEQ to determine the total concentration of organic compounds containing halogens, including PCBs. Any of the three methods specified in the State-issued Part B Permit may be used.

### 2.2.2 ANALYSES USING STANDARD TECHNIQUES

Analyses Using Standard Techniques are accepted procedures which provide operational control over on-site processes. These analyses are performed to further identify wastes and are run if the Laboratory Manager determines that further waste characterization is necessary.

The applicability of the following additional analytical procedures is based on ASTM and/or "Standard Methods" approved by EPA.

- Flashpoint further characterizes potentially ignitable wastes to establish proper management methods and to assure conformance with approval conditions (i.e. waste is not RCRA; ignitable)
- Paint Filter Liquids Test (PFLT) is used to determine if PCB waste materials possess free liquids. The test verifies the presence or absence of free liquids as an additional verification determination. (It is also utilized as a "Post-solidification" verification analysis).
- PCB Concentration is utilized to determine if PCB's are present in liquids (except leachate or water) (visible sheen or oil layer fractions) at less than 500 ppm and therefore, amenable to solidification and ultimate landfilling. It is one of the two "Organic Analyses" required for determining the Acceptability of these PCB waste types.
- Total Organic Carbon (TOC) is used to determine if a liquid (except leachate and water) contains organic compounds in concentrations which would allow solidification of the waste prior to landfilling. Liquids or sludges containing <10% TOC must be solidified prior

to landfilling, or shipped off-site for disposal.

**Table 2.1  
Analyses Run on Specific PCB waste Types**

<b><u>PCB waste Type</u></b>	<b><u>Analyses Run</u></b>
• Contaminated debris/ trash, etc. (including demolition materials)	Physical Appearance
• Empty containers, tanks, drums, barrels, liners, etc.	Physical Appearance
• PCB Contaminated or small capacitors*	Physical Appearance
• Drained or drained and flushed PCB Hydraulic machines	Physical Appearance
• Drained PCB articles or containers and article containers/electrical equipment	Physical Appearance
• Flushed PCB Transformers	Physical Appearance
• Asbestos <sup>1</sup> /PCB-Contaminated Waste	Physical Appearance
• Contaminated soils or sludges (e.g. dredged materials, industrial sludges, municipal sewage treatment, sludges) which <u>Do Not</u> require organic analyses per this WAP	Physical Appearance pH TLV SNIFF Specific Gravity Reactive Cyanides <sup>(1)</sup> Reactive Sulfides <sup>(1)</sup> PCB Concentration <sup>(2)</sup> Leachable TOC (Total Organic Carbon) <sup>(2)</sup>
• Liquids (leachate, tire wash water, groundwater, etc.) from on-site TSCA operations <u>and</u> liquids for which organic analyses (other than PCBs and HOC)are <u>not</u> required. <sup>(5)</sup>	Paint Filter Liquids Test (PFLT) <sup>(3)</sup> Physical Appearance pH TLV SNIFF Specific Gravity Reactive Cyanides <sup>(1)</sup> Reactive Sulfides <sup>(1)</sup> Leachable TOC (Total Organic Carbon) <sup>(2)</sup>  PFLT <sup>(4)</sup> Carbon <sup>(2)</sup>

NOTES:

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Asbestos requires special handling procedures. See also Section 7.4 of this WAP.

- (1) Reactive Cyanides Screen is run to indicate whether the PCB waste produces hydrogen cyanide upon acidification below pH 2. It is not required if the pH of the waste is less than 6, if the waste is not water-soluble, or if the waste is not aqueous. Reactive Sulfides Screen is run to indicate whether the PCB waste produces hydrogen sulfide upon acidification below pH 2. It is not required if the pH of the waste is less than 6, if the waste is not water-soluble, or if the waste is not aqueous.
- (2) If the liquid is water with no visual indication of oil being present, the PCB concentration need not be determined. If a phase separation is indicated, the oil may be analyzed for PCB concentration and the water is not analyzed; any free oil phase shall be removed and handled separately as a PCB-contaminated oil. If there is doubt as to whether the liquid is water, specific gravity is run on the liquid. If the specific gravity is not 1.0 within +/- 0.10 accuracy, the waste profile sheet shall be reviewed and a determination as to the type of liquid shall be made.
- (3) If there is doubt as to the presence or absence of free liquids via the physical appearance determination, then the PFLT may be run.
- (4) PFLT Test is run to determine if solidified materials are suitable for TSCA landfill management (disposal).
- (5) If the liquids appear to be an oil/water emulsion, a water/soil slurry or suspension, or a similar aqueous-based liquid, a specific gravity test shall be conducted, and a TOC test shall be conducted. Liquids which have a specific Gravity of  $1.0 \pm 0.1$ ; which also contain less than 10% TOC; may be approved for solidification and landfill. Any liquids outside of these parameters will be handled separately as PCB contaminated oil.

\*Defined as <100 inches<sup>3</sup> in size, or between 100 inches<sup>3</sup> and 200 inches<sup>3</sup> in size and weighing less than 9 lbs..

### **3.0 ANALYTICAL PROCEDURES**

Analytical methods described herein are grouped in accordance with the categories identified in Section 2.0. It should be noted that the information presented in this section is generic in character. As new analytical procedures are required they shall be adopted and this WAP will be updated appropriately.

### 3.1 FINGERPRINT ANALYSES

Fingerprint Analyses are analytical procedures designed to identify or screen specific PCB waste characteristics. Although fingerprinting has become an industry standard procedure to verify proper waste identification, it has also been developed to provide a rapid and effective means for establishing key decision parameters pertinent to proper PCB waste management in addition to helping ensure that incoming wastes match pre-acceptance information.

**Physical Appearance** - The waste is inspected and the physical appearance of the waste is recorded, including:

- Color
- Physical state (solid, semi-solid, or liquid)
- Presence of free standing liquid

**pH Screen** - Full-range pH paper or a pH meter is used directly on liquid samples and on the free liquid portion of liquid/solid samples. For solid samples, a 10:1 water-to-solid mixture can be made and tested.

**Specific Gravity** - The weight of a volumetric sample is compared to an equal volume of water and expressed to the nearest tenth of a point. This test is not applicable to mixed phase loads or loads of debris.

**Reactive Cyanides and Reactive Sulfides Screens** - To a beaker containing approximately 25 ml of waste, 1 to 5 N acid is slowly added to bring the pH down to 2.0 or less. The atmosphere directly above the sample is tested using a gas detector. The reading is taken directly from the tube.

**Explosivity Meter Vapor Test (TLV Sniff)** - The TLV probe is held over the surface of the sample. A reading over 500 ppm indicates the possibility of flammability.

Based on these results additional testing may be necessary. The methods to be used for treatment, storage, and disposal are normally determined during the pre-acceptance analysis and confirmed during incoming load analysis.

### 3.2 ADDITIONAL ANALYSES

Additional Analyses consist of Unique Additional Analyses (which have been developed to provide information on a PCB waste stream where an adequate standard technique could not be found) and Additional Analyses Using Standard Techniques. These unique techniques are summarized in this section and the full procedure is provided in Section 9. Additional Analyses using standard techniques are summarized and the standard reference is provided. Modifications and/or substitutions of those analytical techniques/methods may be substituted if and when those agencies improve or substitute more current methods.

#### 3.2.1 UNIQUE ADDITIONAL ANALYSES

These are analytical procedures found to provide important quantitative or qualitative information pertinent to certain processes. In some cases, these tests provide information not available from standard techniques in Section 3.2.2, below. In other cases, these tests are substituted for standard techniques where they provide information sufficient enough on which to base a management decision.

**Fixation Requirement** - Solidification agent is added to a sample of PCB waste to determine if the liquids can be eliminated as measured in the Paint Filter Liquids Test.

#### 3.2.2 ANALYSES USING STANDARD TECHNIQUES

The procedures and protocol for these standard analyses are referenced as follows:

PARAMETER	METHOD*
<b>Gas Chromatographic Methods for:</b>	
Organochloride Pesticides	8081 <sup>1</sup>
PCB's	8082
<b>Miscellaneous Analytical Methods:</b>	
Ignitability (Pensky-Martens closed-cup method)	1010 <sup>1</sup>
Ignitability (Setaflash closed-cup method)	1020 <sup>1</sup>
Test Method to Determine Hydrogen Cyanide Released from Wastes (Reactive Cyanides)	7.3.3.2 <sup>1</sup>
Flash point (Closed cup)	D93 <sup>3</sup>

Flash point (Open cup)	D92 <sup>3</sup>
Paint Filter Liquids Test	9095 <sup>1</sup>
pH Electrometric Measurement	9040 <sup>1</sup>
pH Paper Method	9041 <sup>1</sup>
Soil pH	9045 <sup>1</sup>
Specific Gravity	213E <sup>2</sup>
Test Method to Determine Hydrogen Sulfide Released from Wastes (Reactive Sulfides)	7.3.4.1 <sup>1</sup>
Total Organic Carbon	9060 <sup>1</sup>

\* The above referenced procedures are described in the following publications:

- <sup>1</sup> "Test Methods for Evaluating Solid Waste," SW-846, U.S. Environmental Protection Agency, Office of Water and Waste Management, Washington, D.C. 20406.
- <sup>2</sup> "Standard Methods for the Examination of Water and Waste Water," 16th edition, American Public Health Association.
- <sup>3</sup> "Annual Book of ASTM Standards", Parts 15, 19, 31, American Society for Testing Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.

#### **4.0 SAMPLING METHODOLOGY**

Sampling is performed by Grassy Mountain and/or by the waste generator. Specific sampling procedures are dependent on both the nature of the material and the type of containment. This section presents sampling methodologies to be utilized by Grassy Mountain personnel for purposes of preacceptance or fingerprinting acceptance situations as outlined in this WAP. In some instances (e.g.; clean-up projects, remote sampling requested by the generator, etc), Grassy Mountain personnel may perform sampling off-site under these conditions. PCB waste generators are referred to 40 CFR 261, Appendix I for sampling procedures.

#### **4.1 METHODOLOGY**

Representative samples shall be taken using methods outlined in "Test Methods for the Evaluation of Solid Waste, Physical/Chemical Methods," SW 846, EPA; "Handbook for Sampling and Sample Preservation of Water and Wastewater," (EPA-600/4-82-029); or "Samplers and Sampling Procedures for Hazardous Waste Streams," (EPA-600/2-80-018); or 40 CFR Part 261, Appendix I.

## **4.2 TYPES OF CONTAINERS**

In addition to the sampling procedures noted above, Grassy Mountain has instituted specific methodologies for ensuring that samples taken from various types of containers are representative. The containers may be transportable such as drums, portable transport units (e.g., tanks, roll-off boxes, lugger boxes) and tanker or dump-type trucks; or stationary; sampling devices are selected depending on the size and type of the containment and on the specific material involved.

Access to a container will influence the number of samples that can be taken, and the location within the container from which samples can be taken. Where appropriate, samples will be taken from locations displaced both vertically and horizontally. The number of samples required for reliable sampling varies depending on the distribution of the waste components in the container. If needed, a sufficient number and distribution of samples will be taken to address horizontal variations in the waste, since there is a greater tendency for heterogeneity in a horizontal rather than a vertical direction. If examination indicates stratification in the waste, then each layer may be composited in proportion to its estimated volume. In cases where the horizontal dimension is large relative to the vertical (e.g., large end-dump trucks, tankers), multiple vertical sections may be sampled.

### **4.2.1 CONTAINERS AND TANKS**

Sampling of small containers (e.g., drums, cartons, and other small units) varies with the nature of the PCB waste material. For flowable materials, the sampling device of choice is either a Coliwasa unit or tubing to draw a full vertical section. For non-flowable wastes, tubing or a trier is used to obtain a representative sample.

Large containers and tanks (include railcars) for flowable materials and bulk containers (including gondola rail cars) for solid materials may be either stationary or mobile. Liquids are sampled with a Coliwasa or tubing to obtain a vertical section, or by weighted bottle or bomb sampler to allow for sampling at various depths. Light, dry powders and granules are sampled with a tube to obtain a vertical core.

Composite samples of dry powders or granules are sampled with a thief. Heavier solids are sampled by

trier or shovel, or by coring with heavy tubing. Depending upon access to the material and its consistency, a smaller shovel or scoop may be used; extensions to the handle may be used to allow further "reach" for the sampling crew. Tank sediments are sampled from the bottom sampling valve when not readily sampled from above.

Where appropriate and access allows, multiple vertical sections are sampled to represent variations in the horizontal dimension. Sampling across the horizontal dimensions may be limited to access ports or to sampling during unloading.

### **4.3 GENERAL CONSIDERATIONS**

In the operation of a waste management facility a number of issues become obvious, which are not necessarily anticipated in regulations or in standard methods. Below are sections addressing several issues of this nature. It is Grassy Mountain's intention to address these issues in this forum to provide insight into the developed techniques.

#### **4.3.1 DISPOSITION OF SAMPLES**

Samples of PCB waste streams are disposed in the same fashion as the waste stream itself. Samples received which are unacceptable for management on-site may be returned to the generator (or representative) or sent to an approved facility. To facilitate this process (sample disposal), samples approved for the same management processes may be consolidated in containers.

#### **4.3.2 CLEANING OF SAMPLING APPARATUS**

Sampling tools shall be kept clean of materials that will interfere with future analyses. Those analyses being conducted for gross qualification will be kept free of loose material which would enter the sample, whereas visually clean or new sampling equipment may be required where the sampling is being performed to determine constituents in the parts per million range.

#### **4.3.3 FROZEN SAMPLES**

Samples of frozen loads are maintained in the lab until the sample is greater than 5°C. In some cases, it may be required to allow entire loads or, for drums loads, 10% of the load to warm up to facilitate sampling or to inspect for free standing liquids. As an alternative, and if conditions warrant (e.g., anticipated freezing conditions) a sample of PCB waste being delivered may be taken at the point of generation for the purpose of satisfying the requirements of this plan. Upon arrival, the load shall be visually inspected for free liquids.

#### **4.3.4 SAMPLING SAFETY PRECAUTIONS**

Safety equipment and protective clothing shall be worn by personnel while sampling. Samplers will wear safety glasses or goggles (as appropriate), chemically resistant coveralls, steel-toed boots, PCB-resistant gloves (e.g., Viton, neoprene, nitrile, etc.), hard hat, and a respirator with organic vapor/acid gas cartridge and pre-filter. A 30-minute self-contained breathing apparatus (SCBA) is also available.

#### **4.3.5 REMOTE SAMPLING AND/OR ANALYSIS**

For preacceptance samples taken at the remote location and transferred to the facility or off-site lab for analysis, chain of custody shall be followed. Fingerprint (final acceptance) analyses must be performed at the facility. Preacceptance analyses may be performed on-site or at an off-site facility.

### **5.0 WASTE SCREENING PROCEDURES**

A series of control procedures has been developed to determine the acceptability of specific wastes at the facility. The waste screening procedure dictates what information a potential customer should provide to enable Grassy Mountain to determine the acceptability of the waste for treatment, storage, or disposal.

Waste screening is a mechanism for deciding to reject or accept a particular type of waste--prior to its acceptance for disposal at the facility--based on the conditions or limitations of existing permits,

existing regulations, and its compatibility with other wastes being treated, stored, or disposed at the facility.

## 5.1 PROCEDURAL REQUIREMENTS

For each new waste stream that is a candidate for management at the facility, except for transformers, other equipment, and large debris such as Tyvek, gloves, wood planks, concrete and asphalt chunks, rags, empty containers, steel pieces, miscellaneous building materials, etc., which cannot be representatively sampled and analyzed in the lab, the following procedures are implemented:

- The generator shall provide Grassy Mountain with:
  - (1) chemical and physical data requested on the Waste Profile Sheet for PCB Waste Shipments (WPS). A typical form and the instruction sheet is included in Section 5.2 of this WAP;
  - (2) a representative sample; and
  - (3) other supporting documentation, as necessary.
- Grassy Mountain will ensure the WPS contains the critical information or run any missing critical analyses, compare certain WPS data by utilizing the representative sample(s) of the waste and performing requisite confirming analyses. Sampling and analyses shall be performed in accordance with the methods outlined in Sections 2.0, 3.0, and 4.0.
- After comparing the data provided by the generator with that obtained from the preacceptance sample, Grassy Mountain shall determine the acceptability of the waste based on:
  - (1) the approval conditions for the facility, and
  - (2) existing disposal restrictions.
- As a minimum, the pre-acceptance evaluation shall be repeated when a generator notifies Grassy Mountain that the process generating the waste has changed (e.g.; when the raw materials to the process have changed), or if the Laboratory Manager determines that the waste does not match the pre-acceptance documentation.
- The following materials will be handled as detailed below:
  - (1) Mineral oil dielectric fluid containing PCBs.
  - (2) Kerosene flushing fluid contaminated with PCBs.
  - (3) Commercially graded oil contaminated with PCBs.
  - (4) Other oils with <500 ppm PCB,

- (5) Flushed PCB transformers with original PCB concentration >500 ppm per 40 CFR 761.60(b)(1)(i),
- (6) Asbestos<sup>2</sup>/PCB-Contaminated Waste
- (7) Contaminated soil, debris and/or rags,
- (8) Non-PCB (<50 ppm), PCB-contaminated, or small PCB capacitors,
- (9) Drained or drained and flushed PCB hydraulic machines,
- (10) Drained PCB articles or containers,
- (11) Liquids <500 ppm PCB concentration (including solidified liquids),
- (12) Dredged materials, industrial sludges, and municipal sewage treatment sludges,
- (13) Drained PCB-contaminated electrical equipment,
- (14) Sludge-like chemical materials, and
- (15) Other PCB materials accepted for storage and transferred to off-site facilities.

Items 1, 2, 3, 4, and 15 may go to the PCB storage facility on-site for eventual shipment off-site to a suitable facility under 40 CFR 761. Refer to Appendix 1. Items 5, 6, 7, 8, 9, 10, and 13 may go directly to Cell B16 after physical appearance is examined and required inspections occur. Items 11, 12, and 14 are sampled as part of fingerprint analyses prior to acceptance for final disposal at the facility. Fingerprint analyses (including any confirming additional analyses (i.e. PCB, TOC) are performed either in the Grassy Mountain laboratory or in a Utah Bureau of Laboratory Improvement-approved laboratory. (See Section 2.0). Waste meeting the description of Item 11 and 14 will be evaluated as described in Section 2.0. Aqueous-based liquids having a Specific Gravity of  $1.0 \pm 0.1$ , have a less than 10% separable oil phase, and which demonstrate a TOC value of less than 10% may be solidified and landfilled. Liquids or sludges containing > 10% TOC, must be handled as PCB-contaminated oil and shipped offsite to a facility properly approved to handle PCBs of these types. In addition, if there is evidence to believe that the oil phase was diluted from > 500 ppm PCBs, the waste shall be treated under Item 3, above.

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Asbestos requires special handling procedures. See also Section 7.4 of this WAP.\*There is no Section 7.4.

## 5.2 EVALUATION

The Laboratory Manager is responsible for pre-acceptance evaluation decisions (i.e., whether to accept or reject wastes). Samples under consideration for acceptance are subject to Fingerprint Analyses as detailed in Section 2.1. The Laboratory Manager may require Additional Analyses to screen samples for other contaminants or properties. The bases for requiring these additional analyses are:

- Laboratory Manager's experience and judgement;
- WPS description of the chemical and physical properties of the wastes;
- WPS description of the process generating the waste; and
- Results of the Fingerprint Analyses.

The pre-acceptance evaluation is concluded with documentation of the decision regarding the acceptability of the waste and the proposed method of management.

The Waste Profile Sheet for PCB Waste Shipments and instructions for its completion follow.

**Figure 5-1 Waste Profile Sheet for PCB Waste Shipment**

**\*INSERT CLEAN HARBORS PCB WASTE PROFILE SHEET**

**Figure 5-1  
(continued)**

**6.0 INCOMING LOAD PROCEDURES**

Each load of waste, upon arrival at the facility, shall be inspected, sampled and analyzed, except as otherwise noted in other sections of this WAP, and managed. This fingerprinting procedure serves two purposes. First, it compares the actual waste characteristics with those determined in the pre-acceptance phase and those listed on the waste manifest. Second, it confirms the characteristics that would indicate the proper disposition of the waste to storage, treatment, or disposal method.

**6.1 RECEIVING PROCEDURES**

Incoming load identification begins upon arrival of the waste at the facility. The inspection, sampling and analysis of the incoming waste shall be performed in accordance with the methods described in Section 2.0, 3.0, and 4.0.

All bulk liquid and solid waste deliveries (this does not include debris, transformers, and capacitors) shall be sampled and analyzed, except where large volumes of a single waste character are received from a single source (e.g., a major site clean-up of contaminated material, or a large volume generator, or a railcar). In such cases, all loads shall be inspected by utilizing the physical appearance; and at least 10 percent of such loads shall be sampled and analyzed.

In the case of loads of drums or portable tanks, at least 10 percent of the containers in each waste stream shall be sampled prior to final disposal. After the load has been accepted, but before further processing, all containers shall be opened for visual inspection for physical appearance. Container samples that are related to one generator and one process may be composited prior to analysis, providing the individual samples are similar in physical appearance.

This procedure may be varied, under certain circumstances, for container shipments defined as solid on the manifest or in pre-acceptance evaluation. If the initial 10 percent sampling (prior to accepting the load) shows similarity in physical appearance, an additional 10 percent of the containers shall be

same), the entire load will be considered as like-type waste exempted from additional inspection/sampling. If any apparent discrepancies (e.g., free liquids) are discovered, all the containers shall be opened and inspected.

It is standard procedure to sample one in every ten loads of a given waste stream, rather than 10% of the total loads received of that waste stream; it is rarely possible to predict the total number of loads to be received. Therefore, once ten loads have been received (and one of them has been sampled), the count starts over, and at least one of the next ten loads must be sampled. The sampling crew is responsible for determining whether a given waste stream has been sampled within the last 10 loads. This may be done by either manual (paper) or electronic (computer database) tracking of the number of loads of a particular waste stream have been received. The samplers are allowed to pick any of the ten loads for sampling, at their discretion, and may sample additional loads if they deem it advisable.

Electrical equipment which must be drained and flushed prior to landfill disposal is sent to the commercial storage facility. Upon completion of drain and flush inspections and operations, electrical equipment is transported to Cell B\6 for ultimate disposal. Refer to Appendix 1.

Generally, most PCB-containing equipment which is received for disposal at the facility will already have been drained and flushed as needed. However, some equipment is received which must be drained (and flushed, if needed) prior to disposal. All draining and flushing of equipment occurs at the commercial storage area of the facility.

Drained electrical equipment, prior to landfiling, shall be inspected to determine if it has indeed been drained. As with PCB containers, at least 10% of the articles on each line item of each manifest shall be opened to check for the presence of free-flowing liquids. For equipment such as transformers, the "opening" will be done by removal of inspection plates or access hatchways, and then visually inspecting the interior area exposed. The inspection will also verify that a sorbent material has been adequately added to blot up any remaining liquids. A quantity of sorbent approximately equal to 5% of the fluid capacity of the article should be present. If liquids are present, or if sorbent has not been adequately added to blot up any remaining liquids (> 5% of the fluid capacity of the article), in any of the 10% of articles opened, then 100% of the articles represented by that line item of the manifest for the shipment

shall be opened and inspected; the articles shall be further drained and sorbent added as needed.. If all of the 10% of articles inspected have indeed been drained (i.e., no free-flowing liquids are observed, and sorbents have been adequately added), then an additional 10% of articles represented by the line item will be opened and inspected as before. If all of these articles have been drained and had sorbents adequately added, then the remainder of the articles on that line item will be declared as "conforming", and all will be routed to the landfill for disposal.

Equipment and articles, which are to be drained, shall be taken to the Commercial Storage area, and drained within designated areas, all of which are within secondary containment. Once in place in the drainage location, all valves or petcocks will be fully opened and allowed to stand open for at least 30 minutes over a catch tank. If there are drain plugs or caps, rather than valves, they will be removed to allow drainage to occur. Articles may be inverted, elevated at a slant, rotated, or otherwise moved to enhance drainage out of complex internal geometries. Articles shall be allowed to drain until any "stream" or "flow" of liquid has ceased, and only an occasional drip remains. Flushing shall then take place as specified by 40 CFR Part 761.

Finally, all valves shall be closed and drain plugs replaced, to insure no further dripping or leakage after the article is removed from the drainage area. Sorbents shall be added through the inspection ports, to blot up liquids which may remain in the article, in an amount approximately equal to 5% of the fluid capacity of the article.

Not all articles have inspection ports or drain valves or plugs, however. For such equipment, such as large, detachable ceramic bushings which are known or suspected to contain liquids, other more-destructive means of opening may be employed. All articles which are known or suspected to contain free liquids (except for *de minimis* quantities such as vials) will be opened by drilling, cracking or breaking a hole in the ceramic with a hammer, or other means to open up cavities which may contain liquids. As stated above, articles will be allowed to drain for at least 30 minutes, and rotated, inverted, etc. to assist in draining complex geometries. If multiple, isolated cavities are apparent, then multiple openings will be made. Where possible, sorbent will be added to blot up liquids which could not be drained. The articles will then be routed to the landfill for disposal.

For all articles described above, any free liquids remaining around the drain ports or openings created, which might drip off of the article while it is in storage, will be wiped off with absorbent toweling. However, those articles which have been forcefully opened cannot be re-sealed, so some long-term weepage from the openings may be unavoidable; also, some oil staining of the exterior surface of the articles in the immediate area of the drain ports is considered normal and may remain. Sorbent pads or toweling will be wrapped or packed around any unsecured openings or valves known to be incompetent, to prevent releases to the environment while present at the facility; such wrapped articles shall be routed to a landfill cell for disposal within 24 hours after completion of the inspection/draining process, or be stored in a PCB container for disposal. Grassy Mountain will use best efforts to complete the inspection/draining process, including the resolution of disputes with generators, as quickly as practicable.

For those unsecured articles described in the preceding paragraph (i.e., those wrapped or packed with sorbent pads), the 24-hour period allowed for disposal will be deemed to start when the article is removed from within the secondary containment area within the PCB Storage warehouse. The time and date of removal from secondary containment shall be recorded on the records described below; unless a specific notation is made otherwise, the "time of removal from secondary containment" will be considered as the time recorded when the article is removed from the PCB drain vats.

A record shall be kept indicating the actions taken on each line item (e.g., group of articles), the technician performing those actions, and the date the article(s) was/were handled. Also, the initials of the technician performing the inspection, draining and flushing, etc. of each article shall be placed on each article using indelible markers, prior to transport to the landfill.

## **6.2 DECISION EVALUATION LOGIC**

The Laboratory Manager decides whether additional analyses are required for a particular waste based on but not limited to the following:

- Results of Fingerprint Analyses;
- Knowledge of generator and/or waste-generating process; and
- Results of pre-acceptance evaluation.

Further testing may be required if the results indicate unexpected characteristics with respect to pre-acceptance analytical results, or if the Laboratory Manager determines that the waste composition has changed.

The effectiveness of the waste identification step is dependent on the following components:

- Inspection;
- Sampling;
- Analytical results;
- Waste Profile Sheet;
- Waste Manifest;
- Waste screening analytical results; and
- Laboratory Manager's judgement.

To facilitate the waste identification process, the fingerprint analytical data is recorded (See Attachment 1) and compared to the corresponding pre-acceptance analysis. The chemist or technician performing the analysis will be indicated on the load sheet. The fingerprint analysis verifies that the waste is indeed the same waste as represented by the pre-acceptance analysis. Grassy Mountain has incorporated the procedure into a computer program for waste acceptance. Information from the generator's waste profile sheet can be stored in memory. When a load is received at the facility, the program identifies the waste stream profile and displays the data obtained for the pre-acceptance analysis. Figure 6-1 lists the parameters and notes the range of each parameter within which a waste is considered in conformance. If the fingerprint analytical data is not within the listed tolerances relative to the pre-acceptance analysis, the discrepancy is recorded. The analysis may be rerun to rule out laboratory error. Resolution of any discrepancy exceeding the tolerance ranges shall be explained in the "Notes" section of the record. The Laboratory Manager should classify the waste as being in nonconformance if it is significantly different in composition from the information shown in the WPS, the pre-acceptance results, or on the manifest, unless the discrepancy can be clarified by the generator, transporter, or by the Laboratory Manager's judgement.

Wastes found to be in nonconformance may be rejected immediately, or they may be reevaluated for possible acceptance by the facility despite the variance. The reevaluation will be based on the following criteria:

- Permit authorization;
- Disposal restrictions;
- Discussions with the generator;

- Facility conditions; or
- Laboratory Manager's judgement.

The Laboratory Manager or customer service representative attempts to resolve with the generator or transporter any significant discrepancies between the actual waste and that shown on the manifest. Significant changes to the manifest may be made at the request of the generator. Any corrections or other changes made to the manifest will be initialed by the person making the change.

**FIGURE 6-1**  
**FINGERPRINT ANALYSES**  
**ACCEPTABLE TOLERANCE RANGES (FOR DISPOSAL)**

PARAMETER	TOLERANCE RANGE
pH +	2.5 pH units
Specific Gravity	±20% of pre-acceptance analysis
Reactivity:	
CN <sup>-(1)</sup>	positive to negative, only
S <sup>2-(2)</sup>	positive to negative, only
Explosivity Meter Vapor Test (TLV Sniff)	± 500 ppm

**Notes:**

1. If this material is to be disposed of directly into the landfill, an increase across 250 ppm will require an explanation or different handling.
2. If this material is to be disposed of directly into the landfill, an increase across 500 ppm will require explanation or different handling.

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**7.0 PROCESS OPERATIONS PROCEDURES**

Each movement of a waste within the facility, during which a significant change in its characteristics may occur, may make it subject to additional inspection, sampling and analysis to determine appropriate handling and management of the waste. Many of the analyses needed for the storage, treatment and disposal functions are performed during incoming load verification. Solid Wastes that have been solidified or that have had absorbent added are subjected to the PFLT Test.\*

Existing and anticipated process operations at the facility, for which current and periodic sampling and analyses is important, include the following:

- Treatment, consisting of solidification;
- Disposal, consisting of landfilling.

\* Where applicable, and as specified in this WAP.

## **7.1 STORAGE**

PCBs are stable. PCBs are not reactive and are not flammable. Compatibility of PCBs during storage is not a problem.

### **7.1.1 STORAGE TANKS**

Liquids arriving by tanker are currently not transferred to storage tanks pending solidification, but rather remain within the tanker. After inspection, sampling, and analysis are complete, the tanker usually transfers the liquids directly into a solidification tank for treatment.

### **7.1.2 STORAGE CONTAINERS**

Storage containers typically consist of fifty-five (55) gallon drums of waste which are received from off-site for storage prior to on-site treatment or disposal. Containers are subject to the fingerprint analysis to ensure conformity with pre-acceptance documentation.

Containers which contain free liquids (or are being held for additional waste management information retrieval) may be staged for a period not to exceed five (5) calendar days in an area designated by the facility outside of Cell B\6 prior to solidification and/or final placement. The liquid contents of such drums shall be solidified. A sample of the solidified wastes must pass the PFLT Test prior to landfilling. If a small amount (less than 2 inches) of free liquid is present, solidification agent may be added to the drum. A sample of the contents of 10% of the solidified drums will be subjected to the PFLT Test. The samples must pass the PFLT Test prior to landfilling.

#### **7.1.2.1 IGNITABLE, REACTIVE, AND INCOMPATIBLE WASTES**

Ignitable, reactive, or incompatible wastes are identified and not accepted for disposal under

TSCA. The fingerprint procedures detailed previously check for pH, reactivity (CN<sup>-</sup> and S<sup>2-</sup>), and flammability.

#### **7.1.2.2 ON-SITE MANAGEMENT PROCEDURES**

Wastes received in containers are also subject to the analytical work for the proposed management process. For example, a drum to be disposed in the landfill would be subject to an inspection for the fingerprint parameters (including free standing liquids). Further, depending on what, if any, treatment process it might be subject to, it might be subject to the analytical protocol for that process (e.g., PFLT Test for post-solidification verification). Finally, it would also be subject to the protocol for the landfill.

### **7.2 TREATMENT OPERATIONS**

The proper and complete treatment (solidification) of a particular waste depends upon the appropriate sampling and analysis.

#### **7.2.1 SOLIDIFICATION**

In this process, waste containing free liquids will be mixed with pozzolanic material (e.g. lime, fly ash, cement kiln dust), carbon, or other reagent to assure all free liquids are chemically fixed.

Pre-treatment analyses consist of tests necessary to determine the type of solidifying material to be utilized. As stated previously, only liquids containing less than 500 ppm PCB and less than 10% total organic carbon (TOC) as described under Section 2 of this WAP shall be solidified, unless otherwise authorized by EPA. PCB and TOC are not determined for leachate or water. In-process analyses are not required for this treatment process. A post-treatment analysis is necessary to assure that all free liquids have been reacted and that the mixture is suitable for land disposal. The PFLT Test is checked in order to monitor the success of this process.

The entire contents of a drum containing greater than 2 inches of free liquids will be solidified.

Free liquids are normally not decanted prior to solidification. If only a small amount (less than 2 inches) of

free liquids is present, solidification agent may be added to the drum prior to landfilling.

### **7.3 LANDFILL DISPOSAL**

Important considerations in landfill operations are waste/liner compatibility and leachate/leachate collection system materials compatibility. Other considerations include preventing incompatible wastes from contacting each other. Further assurance is required to prevent free liquids from being disposed without solidification.

#### **7.3.1 WASTE AND LEACHATE/LINER COMPATIBILITY**

A report from the Gundle Lining Systems, Inc., which addresses waste and liner compatibility, is incorporated as Appendix 2. The report concludes that waste and leachate accepted for landfill disposal will not damage the liner or leachate collection system. PCBs are not reactive, flammable, or incompatible.

#### **7.3.2 IGNITABLE, REACTIVE AND INCOMPATIBLE WASTES**

Pre-acceptance information about the process generating the waste provides information which can be used to detect the presence of ignitable, reactive, and incompatible wastes. Flammability and reactivity (cyanides, sulfides) are screened on incoming waste. Wastes determined to exhibit these characteristics will not be accepted for direct landfill disposal under TSCA. These wastes will be rejected, shipped off-site or treated, if possible, in compliance with RCRA to render them acceptable for disposal.

Soils contaminated by acid and having a pH <2 will be mixed with lime to adjust the pH prior to landfill disposal. Soils contaminated with a base will be landfilled without a pH adjustment.

#### **7.3.3 WASTES CONTAINING FREE LIQUIDS**

The placement of free liquids containing >10% TOC (and/or PCBs) in a landfill cell must be avoided. Grassy Mountain believes that the procedures specified in Section 6.1 of the WAP can reasonably prevent such liquids from being placed into the landfill cells.

The heavy equipment used for landfill operations, such as loaders, bulldozers, and trackhoes, all operate hydraulically, and thus may leak hydraulic oil on occasion. (These leaks often cannot be differentiated from PCB oils.) If a spill of hydraulic oil occurs that has an apparent volume in excess of 250 cubic centimeters (approximately one (1) cup), or causes pooled or ponded free liquids of any kind that are visible on the surface of the cell, the spilled material and all materials visibly contaminated by the spill will be removed, placed in a suitable DOT-specification shipping container, and disposed of at an off-site TSCA-approved disposal facility. Removal of the spill will occur within eight (8) working hours of discovery of the spill; the date of the spill will be used as the "Out-of-Service Date" for the waste. Spills requiring removal will be documented in the landfill operating record, and be included in the quarterly reports submitted to EPA. Equipment which is actively leaking will be taken out of service until it is repaired.

#### **7.3.4 WASTES CONTAINING ASBESTOS**

The handling and disposal of asbestos is regulated under the Clean Air Act, under 40 CFR 61 Subpart M. Specifically, 40 CFR 61.154 contains requirements for periodic cover, dust control, recordkeeping, fencing, signs, etc. All asbestos-containing PCB wastes are also subject to these requirements. Specific requirements include:

- Posting of specified warning signs on security fences;
- Daily cover (or once each 24-hours if operating continuously) of the asbestos waste, if there are any visible emissions air from the site. Cover must be non-asbestos-containing material, at least six inches (6") thick, or a sprayed-on resinous material.
- Certain reporting and recordkeeping requirements also apply.

##### **7.3.4.1 CONTAINERIZED FRIABLE ASBESTOS HAVING FREE LIQUIDS OBSERVED**

This section concerns the potential for receipt of drummed, bagged or boxed friable asbestos which could contain a free liquid from the application of water as wetting agent during asbestos abatement activities of the generator. The purpose of this section is to provide the guidelines for handling a friable asbestos container with obvious free liquids. It is intended to minimize the random or systematic opening

of containers of friable asbestos. For regulatory and health and safety considerations it is not wise to open containers of friable asbestos, even when utilizing personal protective equipment (PPE). For any management of containers of friable asbestos at the Grassy Mountain Facility, employees will be wearing the standard level C PPE.

1. Loads received of containerized friable asbestos will be visually observed by site samplers or container management unit personnel to ascertain if any of the containers obviously contain free liquids. This means that any bag or box visible to the samplers or container management personnel will be observed to ascertain if any indication of liquids is present. Wet cardboard on the bottom of boxed waste or "pooled" liquid at the bottom of bagged wastes are examples of visual indications of free liquids in the container.

2. If no containers which can be physically observed for a load visually indicate the presence of liquids associated with the load, the load will be released to disposal. This includes bagged material received in bulk containers, e.g. dump trucks, roll-off boxes.

3. If any container is identified as "potentially" containing free liquids through the visualization process described in paragraph 1., the container will be opened, with care taken to assure no visible emissions occur, and the liquid will be drained into a drum, spill pan or other collection device. The drained container will be properly disposed and the liquid will be analyzed under the Waste Analysis Plan provisions (see Table 2.1) to confirm that it is water. A successful confirmation of water will allow the liquid to be solidified and disposed of the TSCA Cell. A failed confirmation of water (i.e. specific gravity greater than 1.1 or less than 0.9 or an observed oil sheen) will result in the collected liquid fraction being shipped off-site for alternate disposal.

## ATTACHMENT 1

### Load Sheet

- A) Waste Load Number
- B) Date of Arrival
- C) Generator Name
- D) Profile Number
- E) Waste Description and Process
- F) Quantity (volume)
- G) Weight
- H) Disposal Cell Number and Grid Coordinates
- I) Final Treatment or Disposal Date
- J) Stabilization Process, if performed
- K) Final Treatment or Disposal of Free Liquids, if Discovered (Narrative)
- L) If Inconsistency Discovered, Resolution of Such (Narrative)

All data, including any re-analyses is recorded on the Load Sheet, and becomes part of the permanent file describing all wastes received by the facility. If data on the generator's description of the waste load agrees with Grassy Mountain's examination at the facility, the waste will be accepted for management.

Load sheet described in this attachment must be signed as "accepted" or "rejected" by the laboratory manager or his/her designee i.e.)TSD Chemist or Technical Manager.

### 8.0 UNIQUE ANALYSIS PROCEDURES

Refer to the Waste Analysis Plan (WAP) associated with the RCRA Part B permit for the Grassy Mountain facility. The following unique analysis procedures, among others, are described in detail in the RCRA WAP, or are otherwise described in the application correspondence of May 20, 1992 from Grassy Mountain to EPA:

- explosivity meter vapor test (TLV Sniff);
- fixation requirement (recipe).

## **9.0 QUALITY ASSURANCE AND QUALITY CONTROL**

This section provides the Quality Assurance and Quality Control Plan for laboratory data.

### **9.1 PROJECT DESCRIPTION**

As required by 40 CFR 264.13, before an owner or operator treats, stores, or disposes of any hazardous waste, he/she must obtain a detailed chemical and physical analysis of the waste. The analysis must contain all the information necessary to treat, store, or dispose of the waste in accordance with 40 CFR 264 and 268 and the permit issued. The Quality Assurance/Quality Control (QA/QC) measures described within this plan will help assure the user of these data the best true value.

#### **9.1.1 DATA USE**

The data generated by the laboratory in conjunction with the waste screening will be used to determine the characteristics of the wastes and residues to be managed. The handling procedures and final waste disposal will be based on these data.

#### **9.1.2 PROJECT DIAGRAMS**

The main function of the laboratory is waste screening and acceptance.

### **9.2 PROJECT ORGANIZATION**

For the basic organization chart of the laboratory at the facility refer to page 4 of the Quality Assurance Plan in Attachment II-WAP.

### **9.3 QUALITY ASSURANCE OBJECTIVES**

The tests required for ensuring that the load matches the profile will be as follows:

- pH Screen;
- Physical Appearance;
- Specific Gravity;

- Reactive Cyanides and Reactive Sulfides Screen;
- Explosivity Meter Vapor Test (TLV Sniff).

Other support tests may be deemed appropriate (See Table C-2 of Attachment II-WAP) to further qualify/quantify the waste due to its nature or source.

### **9.3.1 QA OBJECTIVES FOR MEASUREMENT DATA IN TERMS OF PRECISION,**

#### **ACCURACY, COMPLETENESS, REPRESENTATIVENESS, AND COMPARABILITY**

The overall quality assurance objective is to ensure that data generated are technically sound, statistically valid, and properly documented.

It is the goal of Grassy Mountain to produce reliable and accurate assessments of the quality of data generated. These assessments will include the following:

Accuracy: Accuracy means the nearness of a result of the mean (X) of a set of results to the true value. Accuracy is assessed by means of reference samples and percent recoveries.

Precision: Defined as a measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions. Precision is best expressed in terms of relative percent difference. Various measures of precision exist depending upon the prescribed similar conditions.

Completeness: Defined as a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions.

Representativeness: Expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.

Comparability: Defined as the confidence with which one data set can be compared to another.

Table 9-1 lists the precision and accuracy goals for the fingerprint analysis parameters which will

be used to verify loads.

Table 9-2 lists the precision and accuracy requirements for the load and profile analysis parameters which will be used as support tests. Completeness which states "as required" indicates the result will not be required on all loads.

#### **9.4 SAMPLING PROCEDURES**

The main goal in a chemical analysis is to make a measurement of some property of an article. Decisions then are made about the article based on the results of this measurement. More often than not, it is impractical or impossible to analyze the entire article. Therefore, it is imperative to have a sample which globally represents the articles universe of concern.

##### **9.4.1 INITIAL CONSIDERATIONS**

The first item will be to protect personnel from possible exposure to hazardous material. Most of the incoming loads will contain some level of harmful matter. At a minimum, the sampling personnel will wear safety glasses, gloves, and boots. Additional protective items will be required as dictated by the suspected contaminants involved. Sampling personnel will be required to check the manifest or any shipping documents so that they are familiar enough with the waste stream to take all of the necessary safety precautions in collecting a representative sample of the waste stream.

**TABLE 9-1**

**PRECISION AND ACCURACY REQUIREMENTS FOR FINGERPRINT ANALYSIS**

Measurement Parameter	Reference	Experimental Conditions	Precision	Accuracy	Completeness
pH	SW 846 9040	Industrial Waste Samples	±2 pH units	±.5 pH units	90%
Paint Filter Test	SW 846 9095	Industrial Waste Samples	Pass/Fail	Pass/Fail	90%
Reactivity (Cyanide)	GMF 4	Industrial Waste Samples	Pass/Fail	Pass/Fail	90%
Reactivity (Cyanide)	SW 846 7.3.3.2	Industrial Waste Samples	± 20%	50 - 125%	90% when screen is positive
Reactivity (Sulfide)	GMF 4	Industrial Waste Samples	Pass/Fail	Pass/Fail	90%
Reactivity (Sulfide)	SW 846 7.3.4.2	Industrial Waste Samples	± 20%	50-125%	90% when screen is positive
TLV Sniff	GMF 5 GMF-5-mod	Industrial Waste Samples	± 20%	80-120%	90%
PCBs	SW 846 8082	Industrial Waste Samples	± 25% (see section 9.7.2)	±20% (see section 9.7.2)	100% as required

**TABLE 9-2**

**PRECISION AND ACCURACY REQUIREMENTS FOR  
LOAD AND PROFILE ANALYSIS PARAMETERS**

Measurement Parameter	Reference	Experimental Conditions	Precision	Accuracy	Completeness
Specific Gravity (5)	SM 213E 9010	Industrial Waste Samples	±20%	---	90%
Normality	GMF 1	Industrial Waste Samples	±30%	±30%	100% as required
Solids	GMF 3	Industrial Waste Samples	±10%	±10%	90%
PCBs	SW 846 8081	Industrial Waste Samples	±25% (see section 9.7.2)	±20% (see section 9.7.2)	100% as required

The next item is to define what is to be sampled: liquid, homogeneous solid, or irregular solid. This will dictate what type of sample and procedure is to be used. There are four basic types of samplers used for collecting waste load samples: Coliwasa, Thief, Probe, shovel or scoop, and Trier.

#### **9.4.1.1 SLUDGES**

A probe will be used to collect a composite sample of sludges. The probe is especially useful for sludges that form a number of phases. One probe will be used for sampling oily sludges, and a second will be used to sample other sludges. Incoming loads of sludge are sampled with a probe as follows:

1. Prior to sampling, inspect the apparatus to ensure it is clean. If it is not, wash with clean water or appropriate cleaning solution and rinse with water.
2. Slowly insert the probe (metal, glass, or plastic) into the container ensuring that the tube reaches the bottom of the container.
3. Seal the top of the tube by placing thumb or hand over the tube.
4. Keeping the tube sealed, remove it from the container and empty the contents back into the container. Repeat this rinsing action twice more before collecting the actual sample.
5. After rinsing tube, collect a portion of the material and transfer it into sample container. Insure adequate volume is collected.
6. Rinse probe at the sample rinsing station.

#### **9.4.1.2 FREE FLOWING LIQUIDS**

A coliwasa sampler will be used to collect a sample of free flowing liquids. The coliwasa is especially useful for liquids that form a number of phases. One coliwasa will be used for sampling oils, and a second will be used to sample other liquids. Incoming loads of sludge will be sampled with a coliwasa as follows:

1. Prior to sampling, inspect apparatus to insure it is clean. If it is not, wash with clean water or appropriate cleaning solution and rinse with water.
2. Push down inside rod, lower the sampler into the container, and allow to fill with liquid.
3. Push outside rod down to seal the stopper in the bottom, and remove the sampler from the container.

4. Empty the contents back into the container. Repeat this rinsing action twice more before collecting the actual sample.
5. After rinsing the colliwasa, withdraw the sample and transfer it into sample container. Insure adequate volume is collected.
6. Rinse colliwasa at the sample rinsing station.

#### **9.4.1.3 MOIST GRANULES/SOLIDS**

Composite samples of moist granules and solids will be collected with a trier. This type of sampler is made by cutting a tube in half lengthwise and sharpening the tip so that it allows the sampler to cut into sticky solids. The procedure for using a trier is as follows:

1. Insert trier into bottom of container.
2. Withdraw with a slow twisting motion.
3. Return contents back into container. Repeat two more times.
4. Collect a sample and transfer contents of trier to sample container.
5. Clean debris from trier and return to sampler storage.

Alternatively, a scoop or shovel may be used provided the sampler can be assured of obtaining a representative sample. When using a scoop or shovel, the sampler is to attempt to obtain as nearly as possible, equally sized portions of a representative cross section of the waste in the container. Each portion is to be delivered to the sample container, then the all loose material shaken, scraped, or otherwise removed from the scoop, before the next portion is taken. Complete decontamination of the scoop between portions is not required, providing sample integrity is maintained; if the representativeness of the sample is jeopardized by multi-phasic or extremely heterogenous wastes, then decontamination of the scoop, or the use of separate scoops, is necessary.

#### **9.4.1.4 DRY POWDER/GRANULES**

Composite samples of dry powders or granules will be sampled with a thief. It consists of two slotted concentric tubes. The outer tube has a pointed tip that permits the thief to be pushed into the material being sampled. Incoming loads are sampled with a thief as follows:

1. Insert thief into bottom of container.

2. Rotate inner tube to open thief.
3. Rotate inner tube to close thief.
4. Empty contents back into container. Repeat twice more.
5. Collect a sample and transfer contents of thief to sample container.
6. Clean debris from thief and return to sampler storage.

As with the moist granules, a shovel or scoop may also be used.

## **9.5 SAMPLE CUSTODY**

The following sections explain the procedures which will be used for entering arriving samples in the log book.

### **9.5.1 PRE-ACCEPTANCE SAMPLES**

Upon arrival, the pre-acceptance sample will be entered into the sample log and assigned a GB number. This number will be used to track the sample information through the record keeping system. The sample container and any analytical results are to have this GB number to help track the information about the sample.

### **9.5.2 LOAD SAMPLES**

Upon arrival, the hauler will present Receiving with the pertinent documentation. The load is entered onto the load log and assigned a load number. This number is in conjunction with the GB number and is used to track the load information through the record keeping system. The sample container and any analytical results are to have this load number to help track the information about the sample.

### **9.5.3 OUTSIDE ANALYSIS**

Pre-acceptance Samples, which have analysis performed for Grassy Mountain by an outside laboratory, will have chain-of-custody documentation provided by either Grassy Mountain lab staff or a representative of the outside laboratory.

## **9.6 CALIBRATION PROCEDURES AND FREQUENCIES**

The following sections outline calibration procedures and frequencies for instrumentation.

### **9.6.1 CALIBRATION**

Any instrumentation used to generate data must be calibrated to assure that its accuracy is within acceptable limits. The calibration must be performed with appropriate standards. The laboratory analyst assures that the proper standards are used to meet the requirements of the tests.

The frequency of calibration will be determined by several factors: instrument stability, accuracy of data required, and methodology employed. Calibration schedules are determined around a nominal period and increased/decreased to fit the requirements of a given test. The calibration schedule will always be biased to the increased side.

Calibration of tests which will be required to determine load acceptance do not allow room for variance from a specified schedule. Other tests will follow schedules as outlined.

### **9.6.2 pH/SELECTIVE ION METERS**

The pH meter will be standardized to at least two pH levels (generally pH 7 and pH 10). The calibration will be checked with another buffer (such as 4; this buffer will be called the Continuing Calibration Verification (CCV) buffer) and the reading documented in the operations log. The calibration will be checked every two hours in the operating day. The reading should be  $\pm 0.5$  pH units.

Other buffers may be used as appropriate for the pH range of the samples being measured. The buffers used should be within 3 pH units of the samples.

The pH electrode will be inspected daily and cleaned as required. Records of all cleaning will be maintained in the operation log. The electrode(s) will be filled with the proper electrolyte solution when the level is inadequate. Such fillings will be noted in the maintenance records.

### 9.6.3 BALANCES

Balances will be of a type appropriate for the accuracy of the weighing performed. Balances available include single pan analytical and single pan top-loading. The analytical balances will be used on a stable surface and the level checked and adjusted as necessary.

The balance calibration will be checked twice weekly with class "S" weights at 1, 10, and 100 grams (or as applicable with balance range). The results of these checks will be recorded in a log book. Any service required will be recorded in the same log book in the section reserved for the particular instrument in question.

### 9.6.4 HYDROGEN SULFIDE ANALYSIS

The H<sub>2</sub>S test is performed weekly using a detector tube (range 100-2000 ppm), and a control sample of Na<sub>2</sub>S is placed in a disposable 55ml cup. 10ml of HNO<sub>3</sub> is added and the gas is allowed to evolve for 10 seconds. A reading is taken (this tube requires 1 pump of the sampler) and recorded. The reading should be approximately 500 ppm ±20%.

### 9.6.5 TLV SNIFFER

The TLV sniffer will be checked with a calibration gas supplied by the manufacturer **or equivalent** (500 ppm Hexane in Air). Allow the meter to warm up for at least 15 minutes. Place the range setting on 10x. Zero the instrument in a contaminant free environment. Attach the pressure gauge assembly to the bottle of calibration gas. Continue calibration procedure as specified by the manufacturer. Reading should be 500 ppm ±20%.

### 9.6.6 FLASHPOINT

The flashpoint will be tested weekly using Chlorobenzene (FP 86°F) as a control. The fingerprint analysis will report >140°F or <75°F. Place a small amount in the setaflash tester.

Measure the flashpoint of chlorobenzene and record the range at which it flashes (such as 75°F - 85°F).

### **9.6.7 SPECIFIC GRAVITY**

On a weekly basis place a given volume of de-ionized water in a tared disposable beaker. Divide the weight in grams by the sample volume to get the specific gravity of the sample. The specific gravity analysis must be run with the waste sample greater than or equal to 5°C.

### **9.6.8 NORMALITY**

The normality of the Sodium Hydroxide solution is checked weekly against Potassium Acid Phthalate (KHP). The normality of the hydrochloric acid is checked against the Sodium Hydroxide. The endpoint is determined using the pH meter at pH 7 or alternately a phenolphthalein and/or methyl red endpoint (as appropriate).

The normality of the solutions are checked weekly and when a new solution is created.

## **9.7 DATA REDUCTION, VALIDATION, AND REPORTING**

The following sections explain data reduction, validation, and reporting procedures for samples.

### **9.7.1 DATA REDUCTION**

The amount and scheme of data reduction will be dependent on the specific method of analysis. Analytical procedures specify the means by which raw data obtained during analysis is reduced to produce the final analytical result.

In the process of taking a measurement and relating that to the level of analyte in the sample, certain guidelines must be followed to avoid distortion of the analytical value through the calculation process.

Calculations will follow accepted rounding and significant figure rules.

Raw data and calculations are recorded by the analyst on a data sheet. Minimum recording requirements are:

- the test performed (method references included);
- sample information such as volumes, weight, and dilutions;
- analyst's identity;
- date samples were prepared and/or analyzed; and

- sample results along with correct units.

Calculations are checked by the analyst as well as another analyst familiar with the test before the data is submitted for final reporting.

The checker spot checks several calculations (at least 10%), and inspects the data for completeness and consistency. This includes checking for transcription errors.

Once the data has past the checker, it is ready to be reported.

Errors will be corrected with a single line through the erroneous result. The correct result will be placed in proximity to the old result. Care will be taken as not to obscure the old result. The analyst or checker will place his/her initials next to the correction. Obliteration of the error with correction fluid is not allowed.

### **9.7.2 DATA VALIDATION**

Data integrity generated during sample collection, laboratory analysis, and reporting will be validated using the following systematic approach.

Field and sample information will be audited for completeness and accuracy including specific evaluation of the chain-of-custody procedures and documentation, sample request documentation, and the sample log-in procedure.

Operational QA procedures including calibration and calibration documentation, instrument maintenance, standard preparation, and reagent preparation will be reviewed quarterly.

All data and supporting documentation generated for reporting purposes will be examined for accuracy, precision and comparability according to the following framework.

The QC acceptance criteria specified in EPA SW 846 Test Methods for Evaluating Solid Waste, Third Edition, will be used where listed. In the absence of SW 846 QC acceptance criteria, the criteria listed below will be used.

- The relative percent difference between duplicate analysis must range between 0 and 20%.
- Percent spike recoveries range between  $\pm 2$  of standard deviations (SD), the historical percent recovery.
- Laboratory control standards must agree within  $\pm 2$  standard deviations of the historical data base or no greater than  $\pm 20$  percent of the true value.

- Quality control data for reagent blanks, Laboratory control standards, accuracy, and precision must be recorded in notebooks.
- Data validation will be a joining effort of the analyst, the group leader, and the Quality Control Coordinator under the review of the Laboratory Director with the following established responsibilities.
- The analyst will be responsible to review personally-generated data against historical and established reporting criteria. Any data not meeting requirements will be subject to corrective action.
- The group leader will be responsible for all data generated in his or her section to assure that all operational criteria for data reporting have been met prior to sending data for report generation.
- The Quality Control Coordinator will be responsible for the upkeep and ongoing review of all operational QA procedures to ensure that they are current, appropriate, and meet client, regulatory and laboratory needs.

## **9.8 INTERNAL QUALITY CONTROL CHECKS**

The following sections describe laboratory and specific quality control procedures.

### **9.8.1 PURPOSE OF LABORATORY QUALITY CONTROL**

The overall effectiveness of a quality control program is dependent upon laboratory operations in accordance with a program which systematically assures the precision and accuracy of analyses. This system must detect errors and either prevent the reoccurrence of error or measure the degree of error inherent in the system.

### **9.8.2 OVERVIEW OF SPECIFIC QUALITY CONTROL PROCEDURES**

Routine parts of the quality control program include the following procedures: All instrumentation and equipment will be calibrated daily/prior to use with standards traceable to NBS Certified Standards, or equivalent. Type II ASTM grade water will be monitored to assure its quality for use in analytical procedures. Laboratory consumables such as reagents, glassware, and standards must meet ACS Standards. Reagents and solutions will be labeled to indicate the contents, hazards, data of preparation, analyst, and expiration date. Preparation of

primary standards, working standards, and solutions will be documented in appropriate log books noting stock solutions from which they were prepared. Class A volumetric glassware will be utilized in any volumetric procedures involving standard preparation. Glassware will be cleaned according to established procedures.

### **9.8.3 INTERNAL QUALITY CONTROL**

Internal quality control checks during sample analysis will include the following:

- Standard calibration of each analytical procedure will be developed by preparing known concentrations of standards. Initial and continuing factors will be checked against historical calibration factors.
- Instrumental calibration will be verified both initially and during sample analysis (once every 10 samples). The continuing calibration is made with standards independent from that used for instrumental calibration (but may be from the same source supplier). The calibration check must agree within established limits with the calibration of the instrument recalibrated. Continuing calibration standards must agree within established limits of calibration. If not, the cause of the discrepancy is identified and corrected. Any affected samples will be reanalyzed.
- The analyst will prepare and analyze reagent blanks at a rate of 5% or one per sample set.
- For some analytical procedures, internal standards will be used as part of the quantitation scheme.
- For certain analytical approaches, surrogates will be spiked into samples before preparation.
- When available, certified EPA or NIST certified SRMs or CRM's will be introduced into the analysis scheme.
- Duplicate samples will be analyzed at a rate of 10% or one per sample set.
- Spiked samples will be analyzed at a rate of 10% or one per sample set, where applicable. Spike solutions will be added prior to sample preparation procedure. The spiking solution must be independent from that used for instrument calibration.

### **9.9 REPLICATE SAMPLES**

Replicate samples help evaluate the precision of a method. They can help us quantify the uncertainty of an analytical value.

Replicates can exist in two forms: replicate sample analysis or replicate spiked sample analysis. If no analytes are expected to be found in an analysis it is better to choose to do replicate spiked samples.

Replicate samples, usually a duplicate, are to be analyzed at a minimum frequency of 10% (1 for every 10 samples). For tests which are run infrequently (once a month), duplicates will be analyzed with each batch.

Replicates which exceed the control limits indicate the need to reanalyze the associated sample batch. Exceptions may be documented by respiking/reanalysis and written comment on laboratory bench sheet.

#### **9.10 BLANKS**

Blanks demonstrate that the method is free from interferences or, alternately, allow the analyst to monitor the background and keep it from reaching levels which would interfere with the detection and quantification of the target analytes.

Blanks also serve to inspect the reagents used for contamination. If a reagent is found to be injecting unacceptable quantities of interference into the measurement system, it needs to be replaced with a higher grade/interferant free material.

If the level of blank contamination is constant and can be controlled, appropriate control limits can be established. Blank values must be recorded on an ongoing basis in this case.

#### **9.11 CALIBRATION MATERIALS**

Quantifying the amount of an analyte in a sample is dependent on the reference used. A value can only be as good as the standard to which it is compared. Calibration materials must be of known purity and composition.

When a standard is created it must be compared to an existing standard to insure it is within acceptable tolerances. This will involve the use of a Standard Reference Material (SRM) or Certified Reference Material (CRM) from NIST, U.S. EPA, or another source which can be traced back to NIST or U.S. EPA.

The Quality Control samples from U.S. EPA can be used to evaluate the accuracy of a standard and/or instrument calibration.

Inhouse calibration standards must agree with reference material within the acceptance criterion as noted in each method. Data documenting this fact will be kept on file. Standards which exceed this limit will be discarded.

## **9.12 SPIKE SAMPLES**

Spike samples are used to demonstrate the accuracy of a method. Spike samples involve the introduction of an artificial concentration of an analyte of interest. The amount of analyte detected versus the amount added is called recovery.

Each method should be tested using method spikes. This involves the analysis of a sample matrix (DIW, etc) and the target analyte(s) (or a subset thereof) to demonstrate that the analytes can indeed be seen and their concentration accurately determined.

After the method has been proven effective, the ongoing process is monitored with matrix spikes. This involves the introduction of an artificial concentration of an analyte of interest to a sample. The sample is first analyzed to ascertain the native level of analyte present. Then a known quantity of analyte is added and the difference noted. This helps to evaluate the method accounting for random matrix effects.

Spike samples should be analyzed at a minimum frequency of 10% (1 for every 10 samples). For tests which are run infrequently (once a month), spikes should be analyzed with each batch. Also, spiking solutions should be independent of calibration solutions.

Recoveries which exceed the control limits indicate the need to reanalyze the associated sample batch. Exceptions may be documented by respiking/reanalysis and written comment on laboratory bench sheet.

## **9.13 QUALITY ASSURANCE PROGRAM REVIEW**

The following sections outline the procedures for performance and system audits.

### **9.13.1 PERFORMANCE AUDITS**

These audits will involve the use of blind samples given to an analyst to evaluate the accuracy of an analysis. The term blind (sometimes called single blind) means the analyst is aware that these samples are spikes, but is not aware of the concentration. These studies will be coordinated by the laboratory manager and/or Quality Assurance Coordinator on a quarterly basis.

Major defects (e.g., a chemist not analyzing quality control samples, improper calibration procedures being used) which are brought to light by these studies are investigated and appropriate corrective action will be applied.

### **9.13.2 SYSTEM AUDITS**

System audits will evaluate the laboratory staff's capability to produce good data. They judge whether or not the quality control practices are being followed and are effective.

The system audits are conducted by the Quality Assurance Coordinator at least semi-annually.

The results of these audits require response from the laboratory staff listing corrective actions taken to remove defects.

### **9.14 PREVENTIVE MAINTENANCE**

All instruments and equipment will be maintained according to specific manufacturer's requirements.

Regularly scheduled maintenance will be performed by trained analysts or manufacturer service representatives.

Major analytical instrumentation may be serviced and maintained under manufacturer's service contracts.

Instrumental maintenance shall include as needed repairs, parts, replacements, and cleaning.

To minimize instrument downtime, laboratories will maintain a wide inventory of parts and supplies for all instrumentation.

Instrument and equipment maintenance log books will accompany all instruments and equipment. Each log book will contain the following information:

- preventive maintenance - scheduled and unscheduled;
- repairs - scheduled and unscheduled; and
- operator's comments.

Maintenance and repair log book entries must include date, time, analyst, and when applicable, invoice number for charges incurred by service representative.

## **10.0 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS**

**10.1 PROCEDURE FOR ASSESSMENT OF PRECISION MEASUREMENTS:**

Percent difference between duplicate analysis

$$\text{Percent Difference = } \frac{\frac{|D_1 - D_2|}{D_1 + D_2} \times 100}{2} \text{ (\% of DIF)}$$

or

$$\frac{|D_1 - D_2|}{D_1 + D_2} \times 200$$

Where D<sub>1</sub> = Sample Value

D<sub>2</sub> = Duplicate Sample Value

**10.2 PROCEDURES FOR ASSESSMENT OF ACCURACY**

% Spike Recovery for Control Standards, and Spiked Samples

$$\% \text{ Recovery} = \frac{\text{SSR} - \text{SR}}{\text{SA}} \times 100$$

Where SSR = Spiked Sample Result

SR = Sample Result

SA = Spike Added

**10.3 PROCEDURES FOR THE ASSESSMENT OF SURROGATE RECOVERY**

Calculation:

$$\% \text{ Surrogate Recovery} = \frac{C \times V_s}{Q_s} \times 100$$

**Where:** C<sub>s</sub> = Measured concentration of surrogate compound in sample.

V<sub>s</sub> = Total volume (or weight) of sample to which surrogate was added.

Q<sub>s</sub> = Quantity of surrogate compound added to sample.

## Control Chart Generation from QC Data

Quality Control will be maintained to monitor the analysis of duplicates, control results and spike recoveries for given method and parameters.

Control charts are constructed as follows:

The control limit is set by program limit or in their absence, the standard deviation (SD) is used to calculate and set the control limit.

$$SD = \{ (X - X_2)^2 / (n - 1) \}^{1/2}$$

Where  $X_1$  = the  $i^{\text{th}}$  result of the function of interest (RPD, Spike Recovery, etc.)

$X$  = Mean of function of interest

$n$  = Total number of measurements

## CONTROL LIMITS

Control Limit (CL) =  $X \pm 3$  (SD)

### 10.4 INSTRUMENT CALIBRATION

Instruments will be calibrated with at least 3 standards (where applicable). Linear regression is the preferred technique to analyze calibration data. In this case the correlation coefficient must meet or exceed the critical value for a 95% confidence interval.

In cases where it might be deemed more suitable to use another method for determining calibration precision (such as computing % RSD of Relative Response Factor values), appropriate confidence limits will set per method specifications.

### 10.5 GENERAL LABORATORY PARAMETERS

Within the laboratory there are numerous other parameters to be evaluated. The objective for any control limits based on historical data and will be the 95% confidence interval (mean  $\pm 2s$ ). Other values may have set limits based on method specification as applicable (such as sample storage =  $4 C \pm 2$ ).

## **10.6 CORRECTIVE ACTION**

The need for corrective action in an analysis can be indicated by a QC check value exceeding established control limits. The analyst must stop analysis, investigate the cause, and correct the problem. If an assignable cause cannot be determined (i.e., matrix effects), all samples analyzed since the last in control QC check must be reanalyzed. All corrective actions must be documented. If an assignable cause can be determined, all of the data generated since the last QC check is suspect. If the cause can be attributable to a specific cause or event, the data succeeding the cause must be reevaluated (or in the case of calculated errors the data must be recalculated), if necessary. Data immediately preceding the cause or event must be checked to confirm precision and accuracy.

### **10.6.1 BLANKS**

When method blanks do not fall within the acceptance range as noted in the method and the level of contamination will effect the quality of the data, the following corrective action scheme will be followed:

- Check calculations for accuracy;
- Verify method of cleaning glassware;
- Reinject the blank;
- Reextract the batch associated with the blank or;
- Reject the waste associated with the blank.

### **10.6.2 LABORATORY CONTROL SAMPLES**

When laboratory control samples (LCS) do not fall with in the acceptance range, as noted earlier, the following corrective action scheme will be followed:

- Check calculations for accuracy;
- Verify that correct reagents and methods were utilized in analysis;
- Reinject the LCS;
- Reextract the batch associated with the blank, or;
- Reject the waste associated with the LCS.

### 10.6.3 DUPLICATES

When duplicate sample analyses do not fall within the acceptance range as calculated earlier, the following corrective action will be taken:

- Check calculations for accuracy;
- Verify that the same methods were utilized in analysis;
- Reinject the duplicate;
- Reextract the duplicate;
- Resample the waste, or
- Reject the waste associated with the batch.

If matrix effects are determined to be the cause of outlier, the problem is noted and the analyst may continue.

### 10.6.4 SURROGATE AND MATRIX SPIKES

When matrix spikes do not fall within the acceptance range, as calculated earlier, the reason for the outlier must be determined. The following corrective actions will be taken to determine the cause:

- Check calculations for accuracy;
- Verify that the proper methods were used;
- Verify that the spike solution added was made correctly and the correct amount of spike solution was added to the sample;
- Reinject the spiked sample;
- Reextract the sample to determine whether or not matrix effects have caused the anomaly;
- Resample, reextract, and reinject the waste, or;
- Reject the waste associated with the batch.

If matrix effects are determined to be the cause of outlier, the problem is noted and the analyst may continue.

### 10.6.5 CONTINUING CALIBRATION CHECKS

When continuing calibration checks fail to meet the acceptance criterion as noted earlier, the analyst must stop and note the reason before continuing. The analyst should look at the following possibilities :

- Have operating conditions been changed (gases, pressures, etc...);
- Has the volume of injection been altered;
- Has the calibration check solution been prepared correctly;
- Have the standard curve solutions been properly prepared and injected correctly;
- Have all data from standards been added to the curve, as necessary.

If, following these steps, the cause can not be determined, the analyst must stop and prepare a new calibration curve.

### **10.7 INSTRUMENT CALIBRATION CHECKS**

An instrument calibration check is the repetitive analysis of a given standard to ensure consistent instrument operation from day to day. Some methods utilize specific procedures to evaluate this check. If the value exceeds control limits, the analysis must be stopped and corrective action implemented.

### **10.8 SPIKE RECOVERIES**

Percent recoveries for matrix spikes and/or surrogate spikes are monitored by the analytes and must be within the control limits established by multiple analyses under similar conditions. Outlier values must be documented with an explanation. Some problems may be deemed an unavoidable part of normal operation. Other problems are indicative of the need for re-extraction/re-analysis. All corrective actions are documented on the lab data sheets.

If matrix effects are suspected as the cause of low spike recovery, the same sample is to be re-spiked once. If the recovery is still low, below method accept criterion, matrix interference can be assumed.

### **10.9 DUPLICATE**

Relative Percent Difference criteria are used to evaluate duplicate analyses. The analyst must monitor this value and document the cause of outliers. The spiking levels for MS/MSD should be at least 2 times the native levels and at least 10 times the Method Detection Limit. Poor precision at low concentrations can contribute significantly to the inaccurate determination of RPD.

#### **10.10 PERFORMANCE AUDITS**

Performance audits involve the use of blind samples given to an analyst to evaluate the accuracy of an analysis. Major defects which are brought to light by these studies are investigated and appropriate corrective action is taken (i.e. retraining and reanalysis of spike samples). Corrective actions are reported to the laboratory director.

#### **10.11 QUALITY ASSURANCE REPORTS TO MANAGEMENT**

Oral and/or written reports of the results of inspections and other major problems will be provided to the laboratory director. Other reports will be provided at his/her request.