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MMSC-ESH-95070

# *U.S. DOE Pinellas Plant Exemption Package for Disposal at the LES Grassy Mountain Facility*

August 1995

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**LOCKHEED MARTIN**

*Lockheed Martin Specialty Components is the Management and Operating Contractor for the Pinellas Plant Under U.S. Department of Energy Contract No. DE-AC04-92AL73000.*

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# EXECUTIVE SUMMARY

The issue facing the Pinellas Plant and the Department of Energy (DOE) is to determine the disposition of any Mixed Waste (MW) that may be generated during shutdown. Any MW must be treated to the applicable Land Disposal Requirements (LDR) standards in order to be in compliance with the Environmental Protection Agency (EPA) regulations. Further, DOE Order 5820.2A states that "DOE low-level waste shall be disposed of on the site at which it is generated, if practical, or if onsite disposal capability is not available, at another DOE disposal facility". The purpose of this document is to request and provide support documentation for an exemption to DOE Order 5820.2A requirements so that a specific MW stream can be treated and disposed at a commercial facility.

Management of MW is regulated under the RCRA regulations, Atomic Energy Act (AEA), and DOE orders. The RCRA LDR prohibit long-term storage and ban land disposal of these wastes without prior treatment to meet regulatory standards. As a result, the Pinellas Plant must determine the most feasible method of treatment and disposal for any MW generated during shutdown at the site. As a means to determine the most appropriate disposition for contaminated F006 Sludge, an analysis of alternatives was conducted including the option of sending the waste to a commercial facility. A NEPA analysis (see Appendix A) must be completed prior to final evaluation of the commercial facility option. At this time, LES Grassy Mountain Facility has a RCRA Part B permit for land disposal of hazardous waste and will also accept very low levels of radioactive contaminated material. A letter documenting the acceptable level from the Utah Department of Health will be provided. This facility is located near Clive, Utah, approximately 85 miles west of Salt Lake City.

Based on this alternative analysis, the exemption required is to allow approximately 189 cubic meters of MW that is listed, and is contaminated with tritium, to be land disposed at the LES Grassy Mountain facility. This alternative offers the combination of lowest cost coupled with the highest assurance of success. Detailed supplemental information is provided in this report. Table 1 is a summary of the options analysis for F006 Sludge.

Table 1. Summary of Alternatives for Sludge Disposal

| | Option | Cost[1] | Feasibility | Comments |
|---|---|---|---|---|
| 1. | Onsite Storage | NA | Not Feasible | The Pinellas Plant is in shutdown and transition. No permit for disposal in Florida can be obtained. |
| 2. | Shipment and Storage at Hanford | $1,075.3 | Feasible with HQ Authorization | This postpones the problem. Costs are for shipping, treatment and storage only. Disposal would be required at a later time. The option has unknown expenses that will increase the ultimate cost. |
| 3. | Shipment and Disposal at NTS | NA | Feasible | NTS authority to dispose of MW is pending EPA and state agreement. This is not an option at this time. |
| 4. | Disposal at Envirocare | $496.1 | Feasible | Requires an exemption to use non-DOE disposal facility. Following shipment and disposal, no additional costs would be incurred. This would be a "final" solution. |
| 5. | Disposal at LES Grassy Mountain[2] | $165.9 | Feasible | Requires exemption to use facility. |

[1] Costs are in thousands of dollars.

[2] Laidlaw Environmental Services (LES), US Pollution Control, Inc. (USPCI), Grassy Mountain Facility near Clive. Utah.

# 1.0 SITE WASTE MANAGEMENT PROGRAM

## 1.1 Introduction

The Pinellas Plant is owned by the U.S. Government and operated by Lockheed Martin Specialty Components (Specialty Components) under contract with the U.S. Department of Energy (DOE). The DOE's Albuquerque (AL) Operations Office administers the contract through the Pinellas Area Office (PAO) and is the Programmatic Office of the plant.

In operation since 1956, the Pinellas Plant currently employs about 850 people on a site occupying 100 acres in Pinellas County, Florida (just north of St. Petersburg). Neutron generators, thermal batteries, lightning arrestor connectors, capacitors and other nonnuclear products that are the principal components of nuclear weapons were built at the plant until production was terminated in September 1994. The by-products of the manufacture and cleaning of these parts and components included hazardous and radioactive wastes.

Waste management activities at the plant are controlled by Federal, State and local requirements and regulations, including Florida Department of Environmental Regulation Operating Permit No. HO52-159339. The Waste Management/Minimization Department has primary responsibility for identifying, storing and shipping hazardous, radioactive and mixed waste. Waste Management is part of the Environmental Management Division. Authority for Waste Management flows from the plant's President to the Environmental Management Division Director to the Manager, Waste Management/Minimization.

The Pinellas Plant generated a variety of hazardous and radioactive wastes as by-products of its production processes and continues to generate a smaller quantity in support of Reconfiguration and Technology Transfer Initiatives. The predominant hazardous waste streams include halogenated solvents, flammable solvents, flammable solids, contaminated oils (halogens), plating bath streams and miscellaneous lab pack wastes. Flammable solids are disposed of at the BDT facility in Clarence, New York. Halogenated solvents, flammable solvents and oils are recycled by Laidlaw Environmental Services. Laidlaw also disposes of plating bath wastes and lab packs.

The primary radionuclide of concern is tritium. Tritiated waste may be produced during the tritium loading of neutron generator components, laboratory operations, and contamination control efforts. Tritium may also be found in scrapped equipment and effluent control waste. Low Level tritiated waste is disposed of at the Savannah River burial site in Aiken, South Carolina in compliance with the Savannah River Waste Acceptance Criteria.

Radioactive Materials Management Areas (RMMAs) have been designated in plant locations that have the potential to produce radioactive hazardous (that is, mixed) waste. This potential is indicated by the presence of unencapsulated or unconfined radioactive material, or the use of subatomic particles or beams that may cause activation. Radioactive Materials Management Areas are identified on the basis of the processes performed in the area; particularly the hazardous chemicals and radioactive materials used. The designation is made by the Manager, Health Physics and the Specialist, Radioactive Waste in consultation with the Manager, Waste Management. The plant has an ongoing program to assess potential to produce mixed waste in areas throughout the facility.

In keeping with the Performance Objective for the Certification of nonradioactive hazardous waste, all hazardous wastes generated in an RMMA are considered suspect.

The DOE Moratorium has been lifted (see MMSC-ESH-0016, "Moratorium Documentation Manual for the Pinellas Plant"), and hazardous waste from an RMMA can be shipped to a licensed commercial facility only if Waste Management determines, on the basis of radioassay and/or radiological survey, that: 1) no radioactivity has been added as a result of DOE operations, and 2) the waste does not exceed the surface contamination criteria set in DOE Order 5400.5, "Radiation Protection of the Public and the Environment." The Pinellas Plant Radiological Control Manual, "Unconditional release of nonradioactive materials or items," uses the DOE Order 5400.5 removable contamination limit. Waste Management requires every effort to be made to meet the Pinellas Plant As Low As Reasonably Achievable (ALARA) objective of <220 dpm/100cm$^2$. To release any item over the ALARA limit requires clearance from the Health Physicist. Any mixed waste will be stored in the generating area pending the development of proper disposal procedures and the identification of commercial facilities that are licensed to handle mixed waste containing tritium.

## 1.2 Waste Characterization and Certification

Background - The radiation in our natural environment, including cosmic rays and radiation from the naturally radioactive elements, both outside and inside the human body. Radioassay techniques are useful for measuring added radioactivity only if they can detect the background radiation in a virgin sample of the material being assayed.

Mixed Waste - Radioactive waste that is also regulated under Subtitle C of the Resource Conservation and Recovery Act (RCRA); in other words, waste that is both radioactive and hazardous. Radioactive Toxic Substances Control Act (TSCA) waste, although technically not mixed waste, is also subject to the Performance Objective for Certification of Nonradioactive Hazardous Waste.

Nonradioactive Waste - Material which: 1) was not radioactive when it was received by the Pinellas Plant, 2) has had no radioactivity added to it during storage or use, and 3) does not exceed the surface contamination criteria in DOE Order 5400.5.

Potentially Contaminated Material - Any item or substance which, in the opinion of Health Physics, could be reasonably expected to have become contaminated as a result of direct or indirect contact with controlled radioactive material.

Radioactive Materials Management Area - An area in which the potential exists for contamination because of the presence of unencapsulated or unconfined radioactive material, or of beams or other sources of particles (neutrons, protons, etc.) capable of causing activation. The following areas (Figures 1-1 and 1-2) of the Pinellas Plant have been designated as RMMAs:

- Tube Assembly
- Tube Exhaust
- Product Analysis
- Gas Analysis Laboratory
- 182C, Tube Assembly
- 182G, Tube Exhaust
- Building 200, Environmental Test
- Building 800, Accelerator
- Neutralization Facility
- Radioanalytical Laboratory

Note: For non-routine projects or processes, temporary RMMAs may be delineated with the concurrence of Health Physics and Waste Management.

Radioactive Waste - Any waste managed because of its radioactive content, which is not otherwise regulated for that radioactive content i.e., Clean Air Act (CAA), Clean Water Act (CWA), etc.

Restricted Release - The release of material for use, treatment, or disposal under the control of the DOE or according to the provisions of a Nuclear Regulatory Commission (NRC) or State radioactive materials license.

Unrestricted Release - The release of property (that is, waste) to any party for use, treatment, or disposal without concern for radioactive content, following a decision that is documented and based on risk-based standards and associated procedures.

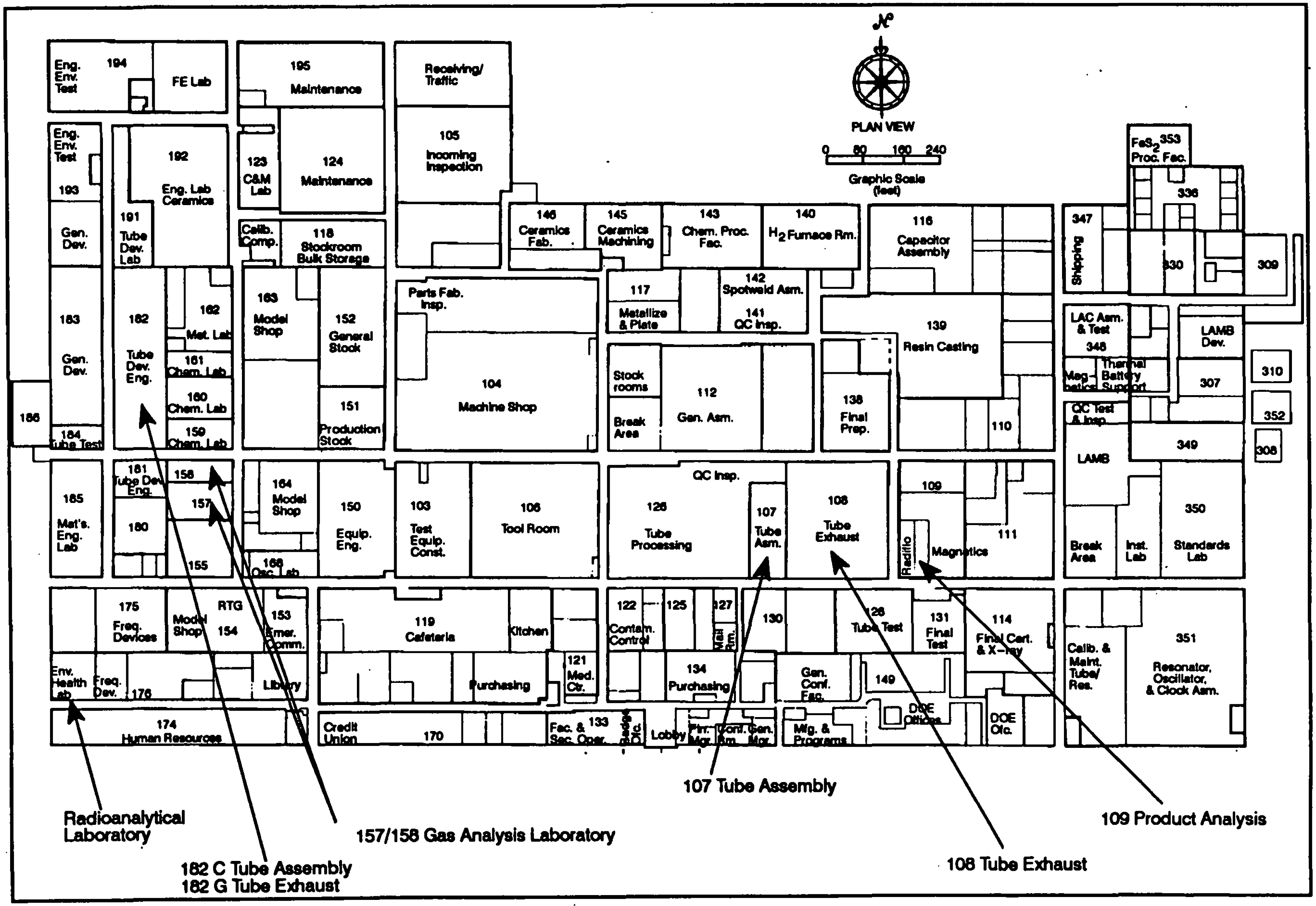

Figure 1-1. Pinellas Plant RMMAs in Existing First Floor Layout

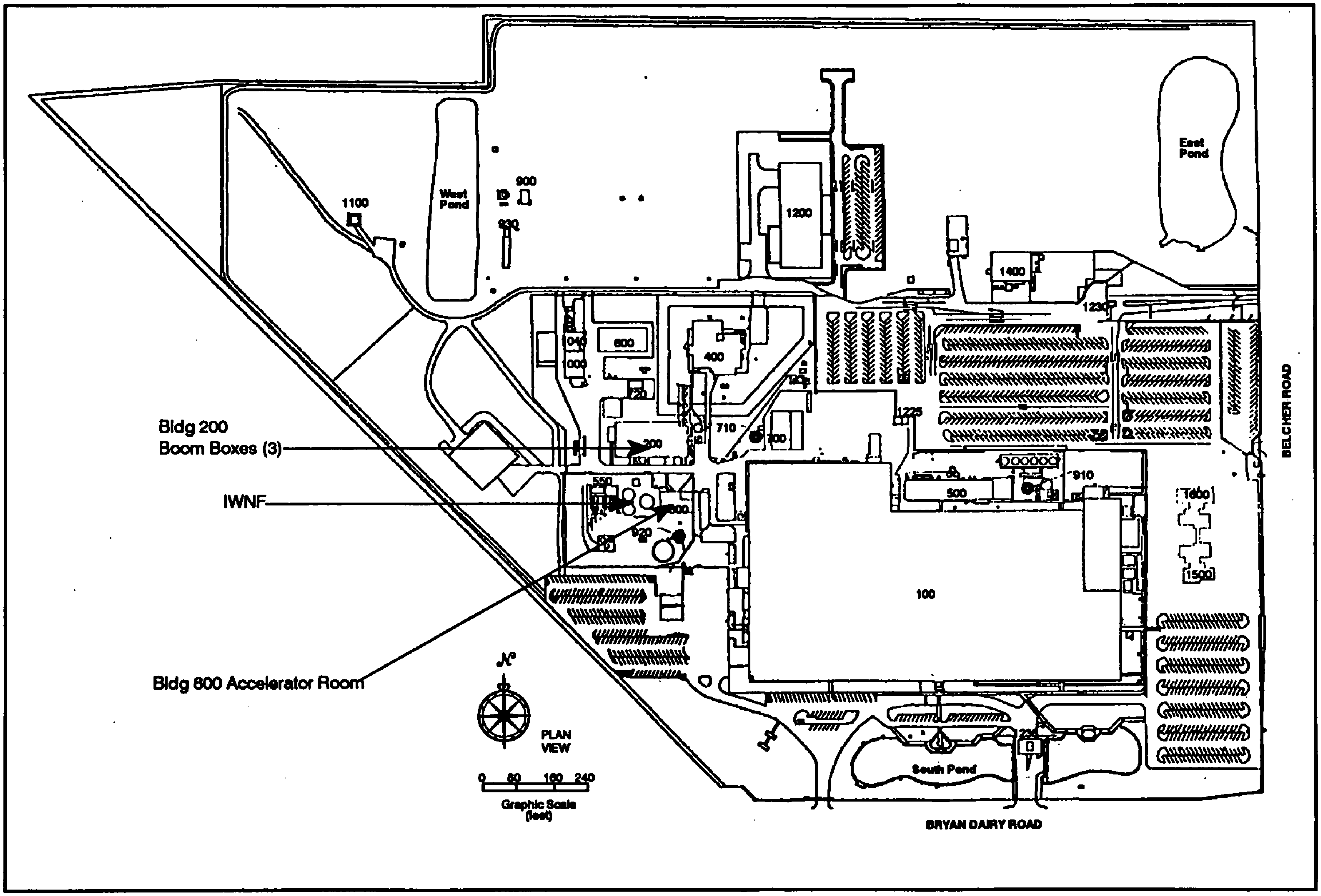

Figure 1-2. Pinellas Plant RMMAs - Site As-Built

Without access to a commercial MW disposal facility, there are three possible scenarios for handling potentially contaminated material:

**Case 1** **The waste is found to be mixed waste and is stored, treated or disposed of at another DOE facility according to applicable orders and regulations.**

**Case 2** **No radioactive component is detected; the waste is simply hazardous. Hazardous waste is shipped off-site for treatment and disposal.**

No hazardous waste produced in RMMAs is being shipped to off-site commercial Treatment, Storage and Disposal (TSD) facilities. Hazardous wastes from other areas of the plant are being shipped to pre-approved off-site commercial TSD facilities in accordance with Title 40 Code of Federal Regulations (CFR), "Protection of Environment."

The "Hazardous Waste Management Quality Program Plan", (MMSC-QPP-0017) outlines quality assurance requirements for hazardous waste handling. These requirements are implemented by means of step-by-step instructions in Operating Procedures. Waste Management uses the procedures in the "Waste Management Operating Procedures Manual", (MMSC-OSP-0027) to collect and prepare hazardous waste for disposal.

Environmental, Safety and Health (ES&H) Standards 4.08 and 8.01 also provide guidance for the handling and disposal of hazardous waste.

**Case 3** **The waste is found to be mixed waste and is shipped off-site to a licensed, RCRA-permitted commercial facility for treatment of the hazardous component, then returned to a DOE facility for treatment and/or disposal of the radioactive component.**

The classification of an RMMA is based on materials (radioactive/chemical) used in the area, surface contamination, and the possibility of unencapsulated or unconfined radioactive material creating a potential for mixed waste production.

### 1.2.1 Determining the Radioactivity Status of Waste

Waste is considered mixed waste if either of the following conditions exists.:

- Radioanalysis of the waste reflects a greater activity level than the established Minimum Detection Limit (MDL) and Detection Limit (DL) for that specific waste matrix. Waste Management must assume that the greater activity level has been caused by a process or by exposure to activating beams or particles at the Pinellas Plant.

- The waste is found to exceed the surface contamination criteria stated in DOE Order 5400.5.

Process Knowledge

The designation of an RMMA is the starting point for identifying mixed waste. A waste may be determined not to be a mixed waste based on the Pinellas Plants' knowledge of the origin, storage, use, and potential exposures of the waste material (see Figure 1-3, Waste Flow Diagram). After the establishment of an RMMA as defined in the Performance Objective, waste originating outside of the defined RMMA is considered nonradioactive. If a waste originates from within an established RMMA, process knowledge can still be used to determine that the waste is nonradioactive (i.e., sealed container that has never been opened). The surface of the container would still have to meet requirements set forth in DOE Order 5400.5.

Specific plant locations are delineated as RMMAs on the basis of the processes, and radioactive materials used in those locations. An RMMA is indicated if a potential for contamination exists because of the presence of unencapsulated or unconfined radioactive material (principally tritium), or because chemicals or other supplies used in the area are exposed to subatomic particles or beams capable of causing activation. The classification of an RMMA must be based on the Potential for Contamination during routine operations. Paragraph 1.2.8 of this manual describes the RMMAs identified at the Pinellas Plant.

An FC-294, Chemical Certification Disposal Log, (see Figure 1-4) has been developed for use by hazardous waste generators to enhance existing tracking procedures and to comply with DOE initiatives to maintain traceable records of hazardous waste movement from the point of generation through disposal.

Waste Management Operators have been instructed to only accept hazardous waste from generators that provide a properly completed FC-294 certifying that hazardous waste requiring disposal was not generated in an RMMA and is not a mixed waste. If the waste was generated in an RMMA, it requires the generator to call Waste Management for further guidance.

In addition, Waste Management has developed the FC-2766, "Certification of Conformance of the DOE Performance Objective," (see Figure 1-5) for the purpose of certifying that any waste that is generated in an RMMA has been analyzed in accordance with approved procedures, and meet all release criteria established by the DOE for certifying the waste as containing no DOE-added radioactivity.

The operator will maintain a drum record on each bulk hazardous waste drum using the FC-294 form provided by generators that will list the source of wastestream contained in the drum. In some instances, comparable wastestreams from various areas are consolidated into one drum. The operators have been trained to list the source of each wastestream on the drum record.

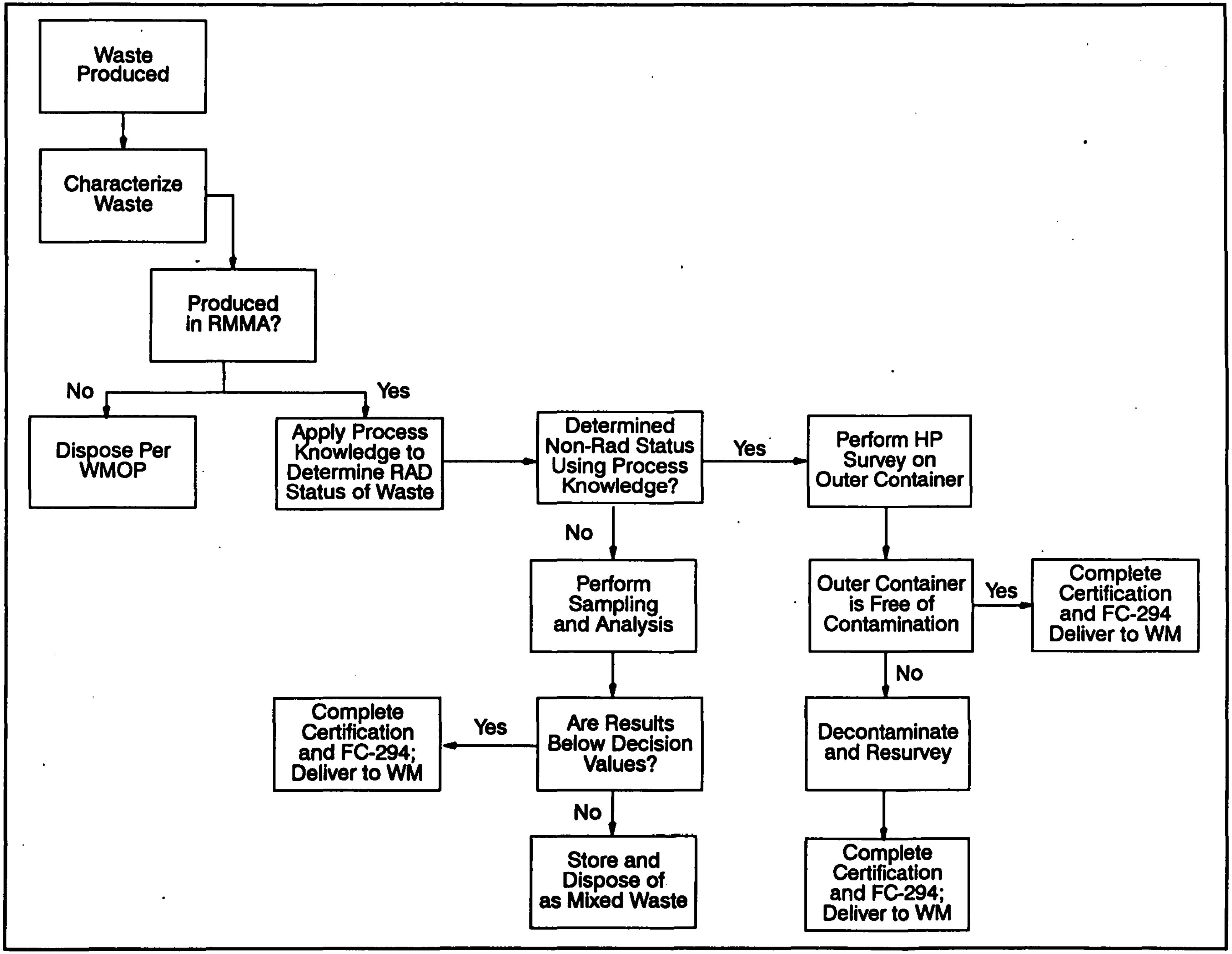

Figure 1-3. Waste Flow Diagram

**FC-294 WP** **CHEMICAL WASTE DISPOSAL LOG** **(11/17/94)**

| | | | | | | |
|---|---|---|---|---|---|---|
| **Generator Badge ID** | **To the best of my knowledge the information provided on this form is correct.**<br>**Generator Signature:** | **Generator Unit ID** | **Supervisor Badge ID** | **To the best of my knowledge the information provided on this form is correct.**<br>**Supervisor Signature:** | **Date MM/DD/YYYY** | **Log Number** |

**CALL WASTE MANAGEMENT AT EXT. 6464 IF YOU ANSWER "YES" TO QUESTIONS 1 OR 2.**

| | | | |
|---|---|---|---|
| **1) Was the waste generated in a radioactive materials management area (RMMA)? *** | **Yes/No**<br>______ | **2) Do you suspect that the waste could have come in contact with (or near) any radioactive material?**<br>**Some examples are: ~The waste was used in conjuction with neutron test equipment or particle accelerator.**<br>**~The waste came in contact with sealed or unsealed radioactive calibration sources.** | **Yes/No**<br>______ |

***Radioactive Materials Management Areas (RMMA)s: 107 Tube Assembly, 108 Tube Exhaust, 109 Product Analysis, 157/158 Gas Analysis Lab, 182C Tube Assembly, 182G Tube Exhaust, Bldg 200 Boom Box room, Bldg 800 Accelerator, Wastewater Neutralization Facility**

| **Drawing Number** | **Sufix** | **Material Description** | **Unit ID** | **Area** | **Qty** | **Unit of Measure** | **Volume %** | **Disposal Code** | **Disposal Reason** | **Comments** |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | |
| | | | | | | | | | | |
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| | | | | | | | | | | |

Figure 1-4. FC-294, Chemical Certification Disposal Log

### Instructions for completing the FC-294 (Rev. 04/94)

For the purpose of completing the FC-294, the **requester** is defined as the employee completing the FC-294. The **supervisor** is the employee with direct supervision of the requester.

1. **Requestor Badge ID**: Enter the requester's badge number.
2. **Requester Signature**: Self explanatory
3. **Requester Unit ID**: Enter the unit number to which the requester is assigned.
4. **Supervisor Badge ID**: Enter the badge number of the requester's direct supervisor. When completing the FC-294 on-line over the local area network, this block will be filled in automatically.
5. **Supervisor Name**: Print the name of the requester's direct supervisor. When completing the FC-294 on-line over the local area network, this block will be filled in automatically.
6. **Date MM/DD/YYYY**: Enter the date the waste will be delivered to Waste Management.
7. **Log Number**: Automatically filled in when completing the FC-294 on-line over the local area network.
8. **Questions 1 and 2**: Read and answer these questions. If you are uncertain of the answer, or if you answered yes to either question, call Ext. 6464 and request assistance before proceeding.
9. **Drawing Number**: Enter the full drawing, stock or diamond label number of the waste material. (e.g., 46AXXXXXX, 124AXXXXX, SSXXXXXX, etc.) Entry of an invalid drawing number on the FC-294 will result in the return of the form to the requester's supervisor for correction.
10. **Suffix**: Enter the full suffix for the waste material if one exists. (e.g., -200, -201, P001, etc.)
11. **Unit ID**: Enter the unit number of the unit which generated the waste described in the entry.
12. **Area**: Enter the area number where the waste was generated. (e.g., 158A, 190B, 200, 400, etc.)
13. **Qty**: Enter the numerical quantity of the waste being transferred to Waste Management. (e.g., 1, 1.5, 100.5, etc.)
14. **Unit of Measure**: Enter the two character abbreviation for the unit of measure of the waste material being transferred to Waste Management. (e.g., BG=bag, BT=bottle, GM=gram, etc.)(See table below) Entry of an invalid unit of measure on the FC-294 will result in the return of the form to the requester's supervisor for correction.
15. **Volume%**: Enter an estimate of the fullness of the container of waste being transferred to Waste Management. (e.g., 10%, 20%, 50%, 100%, etc.)
16. **Disposal Code**: Enter the disposal code from the diamond label for the waste material.
17. **Disposal Reason**: Enter the code corresponding to the reason for the disposal of the material. (See table below)
18. **Comments**: Enter remarks which may be of importance to Waste Management in the safe and proper handling of the waste which may not be communicated by the other information provided for the material.

**Commonly Used Units of Measure**

| | | | |
|---|---|---|---|
| BG | Bag | KG | Kilogram |
| BT | Bottle | KT | Kit |
| BX | Box | LB | Pound |
| CN | Can | LI | Liter |
| CR | Cylinder | ML | Milliliter |
| JR | Jar | TU | Tube |
| CS | Case | PG | Package |
| CT | Carton | PK | Pack |
| DR | Drum | PT | Pint |
| EA | Each | RO | Rolls |
| FT | Foot | SA | Sack |
| GL | Gallon | SI | Square Inch |
| GM | Gram | TO | Troy Ounce |
| GR | Gross | CI | Curies |

(Note: Requests for additional unit of measure codes may be submitted in writing to Waste Management at MS 001.)

**Common Disposal Reasons - Code**

| | |
|---|---|
| Contaminated | CONTAMINAT |
| Contaminated-Heavy Metal | HEAVY MTL |
| Contaminated-Mercury Waste | HG-WASTE |
| Contaminated-Asbestos | ASBESTOS |
| Contaminated-Radioactive | RADWASTE |
| Cured | CURED |
| Dead | DEAD |
| Dirty | DIRTY |
| Empty | EMPTY |
| Equipment Replacement | EQUIPMENT |
| Excess | EXCESS |
| Expired | EXPIRED |
| Flammable Waste | FLAMMABLE |
| Hardened | HARDENED |
| No Longer Needed | NOT NEEDED |
| No Longer Used | NOT USED |
| Rusted | RUSTED |
| Samples | SAMPLES |
| Scrap | SCRAP |
| Slurry Residue | SLURRY |
| Solvent Laden Wipes | SOLVENT |
| Trash | TRASH |
| Used Up in Process | USED UP |
| Waste | WASTE |

(Note: Requests for additional disposal reason codes may be submitted in writing to Waste Management at MS 001)

Please refer any questions regarding completion of the FC-294 to Waste Management at ext. 6484.

Figure 1-4 (Continued). FC-294, Chemical Certification Disposal Log

FC-2766 (10/94M)

# *Certification of "Conformance to the DOE Performance Objective"*

The undersigned certifies that the identified wastes contained within the container conform to the performance objectives established by the Department of Energy for the removal of hazardous waste from a Radioactive Materials Management Area. Based upon process knowledge, operational procedures, and radiological analysis, this waste meets all release criteria established by the DOE for certifying the wastes as containing no DOE-added radioactivity.

Container Number ______________________________

Radiation Survey Report ________________________________

Uniform Hazardous Waste Manifest Number ____________________________

☐ Process Knowledge

☐ Radioanalysis

**Certification of meeting DOE performance objective:**

Waste Management: ____________________________

Health Physics: ____________________________

Date: ________

Figure 1-5. FC-2766, Certification of Conformance to the DOE Performance Objectives

The Specialist, Waste Management will ensure that miscellaneous chemicals packaged using secondary packaging (lab packs) have certifications for each waste container packaged in the drum. In some instances, one certification may apply to several containers in a drum. A packing slip will also be completed listing each container packaged in the drum. The Specialist will maintain the certification record and packing slip as part of the shipping manifest file currently maintained for all hazardous waste shipments.

In some areas of the Pinellas Plant, special processes are performed on products or equipment. These processes may entail cutting or special cleaning operations. A description of the products is listed below.

Neutron Tube - Neutron tubes contain a small amount of tritium. The tritium is hydrided to occluder films in the tube under vacuum. Tritium hydrides used in neutron tubes are in a stable physical form. The tritium is contained within the tube envelope. This has been confirmed historically through smear survey data and product life.

The neutron tube envelope is used in non-destructive operations.. It is very durable and is considered a sealed source at the Pinellas Plant. Incidents of tritium leakage or permeation with current neutron tubes are not common. Aqueous and non-aqueous liquids are used in non-destructive operations (cleaning and degreasing) at the Pinellas Plant. The liquids are sampled for tritium prior to disposal to determine if radioactivity has been added.

Destructive testing of neutron tubes is conducted dry. Areas where neutron tubes are dry destructive tested are controlled as RMMAs per ES&H 8.05.

Neutron Generator - Neutron generators contain a neutron tube and associated electronics depending on the requirements. Neutron generators are coated and sealed to increase its strength and further prevent damage in handling. Neutron generators are considered a sealed source of tritium and leakage and permeation is not expected.

Destructive testing of neutron generators is conducted dry. Areas where neutron generators are dry destructive tested are controlled as RMMAs per ES&H 8.05.

The Radioanalytical Laboratory (RAL) is a counting laboratory who's primary function is to provide analytical as well as technical support to the Environmental Management and Health Physics groups. Environmental sample analysis includes surface and subsurface waters, air filters, and columns, as well as soil samples collected from various locations on- and off-site. Health Physics performs radiological surveys in a number of areas throughout the plant. Surveys are performed in radiological controlled areas, as well as uncontrolled areas. The RAL also performs bioassay analysis on a daily basis.

Analysis is performed for tritium, uranium, and plutonium. Environmental and Health Physics analysis for tritium is performed by liquid scintillation counting using a nonhazardous, biodegradable cocktail. Plutonium and uranium analysis is performed in a fume hood reserved for plutonium and uranium analysis only. This area of the RAL has been designated as an RMMA. All glassware and laboratory equipment dedicated to plutonium and uranium analysis is kept separate from other labware. The plutonium and uranium analysis process involves the use of acids, plutonium and uranium tracers. The tracer materials are of a low activity (5-10 dpm). Acids ($HNO_3$ HCl, HF, HI, and $BO_3$) are also used. A waste beaker is set aside for solutions which may contain traces of plutonium and/or uranium activity. Prior to disposal, these solutions are heated to dryness.

The RAL has completed a study and developed a procedure for the analysis of solvents (alcohol and acetone). Minimum detection activities (MDAs) for sample matrices consisting of 100% alcohol and 100% acetone have been determined. A DL has been established for these matrices in order to determine whether a waste contains radioactivity above background levels. The RAL will design studies and develop analysis procedures based on their initial study for all waste matrices suspected of containing any added radioactivity.

Radioactive Materials Management Areas are designated by the Manager, Health Physics and the Specialist, Radioactive Waste in consultation with the Manager, Waste Management. These professionals have the following qualifications:

- **Manager, Waste Management/Minimization** - This individual has nine years of experience in the hazardous waste field. She has attended RCRA courses and seminars, as well as Department of Transportation (DOT) classes for hazardous and radioactive waste transport. She is a certified Environmental Protection Agency (EPA) Trainer and has been instructed in NQA-1.
- **Specialist, Radioactive Waste** - This member of the Waste Management staff has eight years of experience in the health physics (HP) and radioactive waste fields. He worked as an HP Technician with responsibility for radioactive waste before becoming a Specialist, Radioactive Waste. He has attended a DOT hazardous waste transportation class and numerous radiation training courses, including the Advanced Radioactive Training course offered by the DOT. In addition, he has completed RCRA training and has been instructed in NQA-1.

- **Manager, Health Physics** - A Certified Health Physicist, has over 11 years of experience in the field of radiation protection. He has attended various DOT courses covering hazardous and radioactive waste transport and has been instructed in NQA-1.

The Pinellas Plant has an ongoing program to assess areas throughout the plant for their potential to produce mixed waste.

Waste would be considered contaminated on the basis of process knowledge alone if the waste originated in a contaminated area. However, Health Physics might still perform a radiological survey for informational purposes.

Radiological Survey and Radioassay

A radiological survey is performed by Health Physics to determine if waste contamination exceeds the site's surface contamination criteria. The site's limits are below those set in DOE Order 5400.5.

The Pinellas Plant relies primarily on radioassays to identify mixed waste [ES&H Std. 8.05].

The plant has established a plan for sampling and analyzing hazardous wastes that have been generated in RMMAs. This plan consists of the following steps:

1. All RMMAs are assessed for their potential to produce mixed wastes. Waste Management makes this assessment on the basis of the processes and chemicals used in each area. This information is obtained from the area manager or supervisor.
2. The manager of each RMMA area supplies Waste Management with a list of the hazardous chemicals used in the RMMA. In addition, the Specialist, Radioactive Waste compiles his own list based on an area assessment and observation of the processes that the chemicals are used in.
3. Virgin chemicals and waste materials from an RMMA are analyzed by the Radioanalytical Laboratory. Waste is stored in the generating area while the analysis is being performed.

The RAL analyzes the samples using a Beckman Liquid Scintillation Counter. The calibration of this device is routinely verified to meet the manufacturer's specifications. The analysis uses quench curves to determine efficiency of the instrument on the sample. This is based on U.S. National Institute of Science and Technology (NIST) tritiated water standards and water blanks.

Quality assurance samples are also analyzed to verify the results. The Laboratory reports the results to Health Physics and to Waste Management [ES&H Std. 8.05, Paragraph 6.2].

Sampling and analysis follow accepted practices that employ valid statistical methods to define confidence levels. The Laboratory develops procedures as required to analyze each new chemical or material it receives. These procedures are prepared on the basis of current, published chemical literature as well as practices accepted by government regulators, including the following guidelines:

- NTIS Accession No. PB88-239223/XAB (EPA/SW-846), Test Methods for Evaluating Solid Waste. Field Manual, Physical/Chemical Methods (3rd Edition), Volume 2. Office of Solid Waste and Emergency Response, Environmental Protection Agency: Washington, DC.
- NTIS Accession No. DE88000215/XAB (DOE/EH-0053-V.4), Environmental Survey Manual, Volume 4. Office of Environmental Audit, Department of Energy: Washington, DC.
- NTIS Accession No. EMSL-LV-0539-17, Radiochemical Analytical Procedures for Analysis of Environmental Samples, F.B. Johns et. al. (authors). Environmental Monitoring and Support Laboratory: Las Vegas, NV.

- NTIS Accession No. DE91010178/XAB (HASL-300-ED.27-VOL.1), Environmental Measurements Laboratory (EML) Procedures Manual, N.A. Chieco et. al. (authors). Environmental Measurements Laboratory, Department of Energy: New York, NY.
- NTIS Accession No. PB84-215581 (EPA-520/5-84-006), Eastern Environmental Radiation Facility Radiochemistry Procedures Manual, R. Lieberman, Ed. Eastern Environmental Radiation Facility: Montgomery, AL.
- Section 6, Radioanalytical Laboratory procedures.

To determine if any radioactivity has been added to a material, A DL for the sample matrix will be used. The DL is based on blanks prepared from virgin chemicals.

The results of the waste radioassay are kept by Health Physics and the Specialist, Radioactive Waste. The Specialist retains copies of the virgin chemical analysis.

4. If any radioactivity has been added during DOE operations, the waste is considered mixed waste and is not released to a commercial TSD facility; it is stored on-site pending the development of proper disposal procedures and the identification of a commercial TSD facility that is licensed to handle the waste.
5. If no radioactivity has been added during DOE operations, the waste is considered hazardous waste. A tag on a container of waste released from an RMMA and signed by Waste Management attests that the

waste is free from added radioactivity. Because the moratorium is lifted, this waste will be shipped to a licensed commercial facility for recycling or disposal [WM.2.07, WM.2.08, and ES&H Stds. 8.01, 8.05 Paragraph 6.4]. The certification of nonradioactivity for RMMA hazardous waste can be traced to the waste shipment. Hazardous waste in drum shipments can be traced by comparing the analytical results to the drum log description and sampling records. For bulk shipments of hazardous waste, such as flammable liquids, Waste Management can compare dated inspection forms that indicate the amount of waste stored in each tank with the date the waste was certified as nonradioactive. Shipment papers identify the date the bulk waste was shipped.

### 1.2.2 Shipment of Mixed Waste to a Licensee

In accordance with DOE Order 5820.2A, "Radioactive Waste Management," radioactive waste is disposed of at a DOE site.

If any waste produced at the plant is determined to be mixed waste, it will be stored in the generating area until proper storage and disposal procedures can be written and mixed waste disposal facilities can be identified.

### 1.2.3 Department of Transportation Shipping Requirements

The Pinellas Plant does not ship mixed waste for disposal at present.

Radioactive waste shipping procedures are part of the Environmental, Safety and Health Manual, ES&H Std. 8.04, and WM 7.04. These procedures conform to the requirements in Title 49 CFR, "Transportation."

The Nuclear Materials Representative from the plant's Facilities and Security Division coordinates between the Pinellas Plant and DOE radioactive waste disposal facilities for shipments of radioactive waste.

### 1.2.4 Quality Assurance

Quality Program Plans (QPPs) are quality assurance documents that identify the Federal, State and local requirements and regulations that DOE facilities must follow in their daily operations. NQA-1, Quality Assurance Program Requirements for Nuclear Facilities, and DOE Order 5700.6C, "Quality Assurance," outline the proper format and content of a QPP.

At the Pinellas Plant, QPPs are written and maintained by personnel who are well acquainted with the functions of a division or department, and the QPP must be approved by the Division Director or Department Manager. In addition, all QPPs are reviewed and approved by a representative of the addition, all QPPs are reviewed and approved by a representative of the PAO, who ensures that the QPPs satisfy the requirements in NQA-1 and DOE Order 5700.6C.

The QPPs cited in this document pertain to waste management and characterization, including requirements for designating RMMAs and for determining whether DOE operations have added any radioactivity to a material. A PAO representative has determined that the QPPs comply with the Performance Objective.

Operating Procedures and Standards apply the quality assurance requirements in the QPPs to day-to-day operations. A PAO representative has determined that the procedures that Waste Management employs to process hazardous waste conform to the Performance Objective. The PAO periodically audits Waste Management activities to ensure that personnel are following the Operating Procedures and Standards.

Environmental Oversight and Quality Assurance (EO&QA), an independent group in the Audits and Ethics Division, conducts compliance audits and reviews and quality audits of environmental management activities to ensure compliance with environmental laws, regulations, requirements, permits, DOE Orders, Martin Marietta Command Media, and Corporate Environmental Management and Specialty Components policy and procedures.

Any future changes to the Operating Procedures, Standards or QPPs which may affect the plant's compliance with the Performance Objective will be reviewed and approved by line management and a PAO representative, as described in this section.

The Technical Support Department coordinates the review and approval of new and modified QPPs, Standards and Operating Procedures. This group also maintains a Master copy of each document, oversees the distribution of current manuals and plans, and keeps a record of past revisions.

### 1.2.5 Training

A performance-based RMMA training program has been developed based on the requirements in the Performance Objective. The RMMA training consists of a computer-based portion along with area-specific classroom training. Personnel required to complete this training include radiation workers, maintenance workers, calibration personnel, members of the ES&H Department, and the Waste Management Operation employees. Certain Directors, Managers, and DOE employees are also included in the training program.

Waste Management staff are retrained annually. The Manager, Waste Management assesses each person's training needs at the start of employment and on the basis of periodic self-audits. Waste Management personnel who designate work areas as RMMAs must complete radiological training and hazardous waste training.

Health Physics personnel attend off-site radiological training, as well as on-site training by a Health Physicist. Only a Health Physicist may certify hazardous waste as nonradioactive.

All personnel who handle radioactive material receive annual radiological training every two years [Section II]. A training procedure for handling radiological waste is being developed.

Training records are maintained by the Manager, Transportation in the plant's Procurement and Distribution Department. Training effectiveness is evaluated by comparing performance on the job with Operating Procedures and Standards.

### 1.2.6 Records

All records associated with procedures incorporated in this document and covered by the Performance Objective are quality records and are maintained as permanent records, pending the incorporation of retention guidelines into DOE Order 1324.2A, "Records Disposition."

Records related to radioactive and mixed waste disposal are retained by the Specialist, Radioactive Waste and by Security.

Records related to nonradioactive hazardous waste disposal are retained by the Manager, Waste Management and the Specialist, Hazardous Waste.

Revision records for the procedures and plans themselves are maintained by the Technical Support Department.

### 1.2.7 Review and Approval of Criteria and Procedures

The following manuals contain Operating Procedures and Standards covered by the Performance Objective:

Waste Management Operating Procedures Manual

Waste Management Operating Procedures are developed and maintained by Waste Management specialists and approved by the Manager, Waste Management. Procedures for nonroutine operations are also reviewed by the Director, Environmental Management and by Health Physics.

The Manager, Waste Management ensures that the Operating Procedures include performance criteria for subcontractors, as outlined in the Hazardous Waste Management Quality Program Plan. The Manager also checks to make sure that the Operating Procedures satisfy government regulations and requirements including, but not limited to, the following:

- Florida Department of Environmental Regulation Operating Permit Number HO52-159339.
- Title 40 CFR, "Protection of Environment," Parts 260-266, U.S. Environmental Protection Agency.
- Title 49 CFR, "Transportation," Parts 100-177, U.S. Department of Transportation.
- SW-846, Test Methods for Evaluating Solid Waste, U.S. Environmental Protection Agency.
- ASME NQA-1, Quality Assurance Program Requirements for Nuclear Facilities.
- DOE Order 5400.5, "Radiation Protection of the Public and the Environment."
- DOE Order 5480.3, "Safety Requirements for the Packaging and Transportation of Hazardous Materials, Hazardous Substances, and Hazardous Wastes."
- DOE Order 5700.6C, "Quality Assurance."
- DOE Order 5820.2A, "Radioactive Waste Management."

Environmental, Safety and Health Manual

ES&H Standards related to Waste Management are prepared by Waste Management specialists, reviewed by the Manager, Waste Management, and approved by the Director, ES&H.

Radiological Control Manual

The Radiological Control Manual is reviewed and approved by the Manager, Industrial Hygiene and Health Physics (IH&HP) Department. The Manager, IH&HP reports to the Director, ES&H.

Laboratory Procedures

Radioanalytical Laboratory procedures have been developed by a Senior Developmental Chemist and approved by the Manager, Chemical Technology.

### 1.2.8 Descriptions of Pinellas Plant Radioactive Materials Management Areas

107 Tube Assembly and 182C Tube Assembly

The processes performed in the Tube Assembly areas include vacuum firing, evaporation, welding, brazing and particle blow-off.

The primary contamination concern in these areas is the brazing operation. Tritium-loaded domes are brazed; although the domes are sealed there is always a possibility of leakage. In addition, the Tube Exhaust areas are located next to, and work with, the assembly areas, so there is a possibility of contamination's spreading to those areas.

108 Tube Exhaust and 182G Tube Exhaust

The Tube Exhaust areas are the locations in which tritium is actually loaded into the tubes or domes. The areas contain several tritium beds. Tubes are loaded, exhausted and baked in these areas.

Contaminated molecular sieves and contaminated oils are stored in Area 108.

157/158 Gas Analysis Laboratory

Thin-film hydrides that may contain tritium are analyzed in the Gas Analysis Laboratory by using mass spectrometers. Tritium standards are used to calibrate the sensitivity in these instruments.

Tritium targets from the Engineering areas are also analyzed here. Tubes are disassembled and the targets, sources and getters are analyzed.

Building 200 Environmental Test

In Building 200 Environmental Test, tubes and generators are destructively tested. This effort includes exploding some units in "boom boxes."

Area 109 Product Analysis

The Area 109 Product Analysis Laboratory analyzes defects in ferroelectric neutron generators. Also, debris from the destructive testing process in Building 200 is sent to the Product Analysis Laboratory, where it is analyzed for defects in the manufacturing process.

Building 800 Accelerator

The accelerator in Building 800 is used to irradiate battery compound samples (such as lithium and silicon) with high-flux neutrons. The accelerator beams on tritium-loaded targets.

Neutralization Facility

The Pinellas Plant operates a neutralization facility for the purpose of maintaining a pH balanced effluent prior to discharge to the Publicly Owned Treatment Works (POTW). Wastewater is piped through a system of drains which are separated into three types of streams. Sanitary wastewater effluents are pumped into a series of liftstations and then discharged to the POTW. Industrial wastewater consisting of deionized water, air handler condensate, acid and caustic discharges, and plating bath rinse waters are pumped into liftstations and then conveyed to the Industrial Wastewater Neutralization Facility (IWNF). Wastewater effluents from radiological areas are piped to three above ground holding tanks prior to tritium analysis, then discharged to the POTW. Prior to 1995, the radiological holding tanks were discharged to the IWNF. This lead to contamination of the F006 Sludge. Occasionally, the IWNF tanks require cleaning to remove accumulate solids and precipitants (sludge). It is this sludge that is a potential concern related to this document. However, this situation will not occur again since the radiological drains cannot flow to the IWNF.

Radioanalytical Laboratory

The RAL is a counting laboratory who's primary function is to provide analytical as well as technical support to the Environmental Management and Health Physics groups. Analysis is performed for tritium, uranium, and plutonium. Plutonium and uranium analysis is performed in a fume hood reserved for plutonium and uranium analysis only. All glassware and laboratory equipment dedicated to this process is kept separate from other labware. The plutonium and uranium analysis process involves the use of acids, plutonium, and uranium tracers. A waste beaker is set aside for solutions which may contain traces of plutonium and/or uranium activity. Prior to disposal, these solutions are heated to dryness.

### 1.2.9 Referenced Documents

DOE Order 1324.2A, Record Disposition

DOE 5400.5, Radiation Protection of the Public and the Environment.

DOE order 5700.6C, Quality Assurance

DOE 5480.3, Safety Requirements for the Packaging and Transportation of Hazardous Materials, Hazardous Substances, and Hazardous Wastes.

DOE Order 5820.2A, Radioactive Waste Management

Vermont State Nuclear Advisory Panel, 1979, A Primer on Radiation, p. 14.

MMSC-ESH-92010, "Radiological Control Manual", December 1, 1992

MMSC-QPP-0017, "Hazardous Waste Management Quality Program Plan," February 3, 1993

MMSC-OSP-0027, "Waste Management Operating Procedures Manual," June 14, 1993

CHM 2201-4, Environmental Chemistry Laboratory Procedure, "General Liquid Scintillation Analysis for Tritium," January 21, 1992.

MMSC-QPP-0019, "Quality Program Plan for Radioactive and Mixed Waste Management," December 21, 1992.

Waste Management Procedures:

- WM.2.01 Hazardous Waste Drum Preparation, Removal and Storage
- WM.2.02 Daily Chemical Waste Pickup
- WM.2.04 Bulk Chemical Waste Storage
- WM.2.07 Bulk Chemical and Drum Waste Shipments
- WM.2.08 Hazardous Waste Transportation and Loading for Off-Site Shipment
- WM.3.01 Sampling Methods for Nonradioactive Waste and Environmental Sites
- WM.3.02 Shipping Samples for Analysis

MMSC-ESH-0006, "Environmental, Safety and Health Manual", July 30, 1993

ES&H Standards:

- Std. 4.08 Storage, Handling, and Use of Hazardous Materials
- Std. 8.01 Chemical Waste Disposal
- Std. 8.04 Radioactive Waste Handling and Packaging
- Std. 8.05 Control of Potentially Contaminated Hazardous Wastes (PCHW)

Tritium Removable Contamination Standards

### 1.3 Shipment Forecasts

Historically, tank sludge build up has been removed every two years. The last shipment occurred in April 1992. However, with lower production activity at the Plant, it is estimated that three years will produce approximately the same quantity. In August 1995 it is estimated that 189 cubic meters (liquid volume) of sludge will require disposal. This will constitute the final shipment of contaminated sludge.

## 2.0 SHIPMENT CAMPAIGN AND TSD FACILITY

### 2.1 Industrial Wastewater Neutralization Facility

Prior to 1995, a potential source of MW generation was the Industrial Wastewater Neutralization Facility (IWNF). Wastewater from plant operations was generated as three separate types of effluents:

1) Sanitary wastewater effluents were pumped into a series of lift stations and then discharged directly to the Publicly Owned Treatment Works (POTW) without receiving treatment.
2) Industrial wastewater, which consists of deionized water, air handler condensate, acid and caustic discharges, plating bath rinse waters, and water from various cleaning operations entered the northwest lift station and then on to the IWNF.
3) Wastewater effluents from radiological areas (rad drain) were piped to three above ground health physics holding tanks and held there until the tritium analysis is complete. Once released by the radiological laboratory, the wastewater was then discharged to the IWNF.

The combined wastewater entered the equalization tank where it was agitated to thoroughly mix the components and obtained a stable pH. The pH was adjusted at this time if necessary with either sodium hydroxide or sulfuric acid. The wastewater was then transferred to the neutralization tanks.

The wastewater entered neutralization Tank No. 1 and/or Tank No. 2, depending on operating conditions and capacity needed at the time. The pH could be adjusted in the neutralization tanks, if necessary. Much of the particulates and suspended solids settled out at this time and result in a sludge accumulation at the bottom of the neutralization tanks. Because the system received rinse water from plating operations the sludges are classified as a hazardous waste. They are assigned the EPA hazardous waste code No. F006.

The Neutralization tanks are approximately 25 feet in diameter and 20 feet high with a cat-walk around the outside and across the center. The Neutralization tanks typically contain about 10 feet of water overlying the sludge. This water continuously overflows an inner wall in the neutralization tank and flows down a drain between the inner and outer wall of the tank and is discharged to the Pinellas County POTW.

All wastewater discharges are based on compliance with the Pinellas Plant's Industrial Wastewater Discharge Permit (No. 018-IE) issued by the Pinellas County Sewer System. This permit establishes limits for metal concentrations, biochemical oxygen demand, pH and total suspended solids. Tritium limits for effluent discharges are based on compliance with 49 CFR and DOE orders. The plant has never had an excursion of tritium since the IWNF began operation.

When the sludge accumulates to a depth of approximately 2 to 3 feet the decision is made to have the sludge removed. The sludge must be sampled and analyzed for tritium activity and RCRA constituents before removal to recognize any safety precautions that need to be taken during the removal process and to choose the appropriate disposal methods and disposal site.

The sludge has been removed in the past by means of an industrial sized pump and is accumulated into tanker trucks. Once a disposal site has been approved, this is the method that will be utilized to transport the sludge.

## 2.2 Waste Characterization

### 2.2.1 Project Description

In accordance with DOE requirements, a sampling and analysis plan must be submitted for approval prior to any sampling activity or sludge shipments to a disposal facility. This plan provides the procedures utilized for the sampling and analysis of the accumulated sludge in neutralization Tank No. 2. This information is essential to the accurate identification of any tritium activity or RCRA constituents and for choosing the proper disposal method.

### 2.2.2 Sampling and Analysis Schedule

Sampling and analysis will begin upon approval of this sampling and analysis plan. The initial sampling and analysis will encompass the sludge at the bottom of neutralization Tank No. 2. In the future, this sampling and analysis plan may be used to sample neutralization Tank No. 1. After the initial sampling and analysis, periodic samples will be analyzed based on the amount of accumulated sludge at the bottom of the neutralization tanks.

### 2.2.3 Waste Identification

This sampling plan specifically addresses the sampling and analysis of the accumulated sludge on the bottom of neutralization Tank No. 2. The waste is a sticky mud-like material with a foul odor. Tritium could have contaminated the sludge through the rad drain as a result of small liberations during routine plant operations. RCRA constituents could possibly contaminate the sludge during routine plant operations such as electroplating, chemical etching, and cleaning processes.

### 2.2.4 Objectives of Sampling and Analysis

The objectives of the sampling and analysis plan are: 1) to provide enough information to make the analysis results representative of the waste sludge that has accumulated in the past, as well as improve generator knowledge of the waste; 2) to determine that the waste meets DOE and 10 CFR tritium limits for effluent discharges; 3) to identify the presence and concentrations of all parameters listed in the following "Laboratory Analysis" section.

### 2.2.5 Analysis Plan

Laboratory Analysis

The sludge samples will be submitted for analysis to Specialty Components radiological laboratory to determine tritium activity. They will also be submitted for analysis to a State of Utah certified laboratory for full characterization including the follow parameters:

A) Identify the presence and concentrations of the Toxicity Characteristic Leaching Procedure (TCLP) metals:

| Analyte | SW846 Test Method |
|---|---|
| As | 7061 |
| Ba | 7080 |
| Cd | 7131 |
| Cr | 7191 |
| Pb | 7421 |
| Hg | 7471 |
| Se | 7740 |
| Ag | 7760 |

B) Identify the present and concentration of the following organic compounds:

| Analyte | SW-846 Test Method |
|---|---|
| TCLP Volatiles | 8010, 8020, 8040, 8080, 8240 |

C) Identify the presence and concentrations of the following organic compounds:

| Analyte | SW-846 Test Method |
|---|---|
| TCLP Semivolatiles | 8250, 8270, 8280 |

D) Identify the presence and concentrations of the following parameters:

| Analyte | SW-846 Test Method |
|---|---|
| Total Cyanides | 9010 |
| Amenable Cyanides | 9010 |

E) Quantify the following parameters:

| Parameter | SW-846 Test Method |
|---|---|
| pH | 9040 |

| Parameter | EPA Test Method |
|---|---|
| Percent Solids | 160.3 |
| Percent Water | 160.3 |

A sludge sample has been submitted to a state of Utah certified laboratory for testing.

Quality Objectives

Quality objectives are designed to meet the requirements of SW-846, Test Methods for Evaluating Solid Waste, EPA. The procedures for sampling and analyzing the waste sludge are included in this document.

Use of Process Knowledge and Existing Analytical Data

Based on process knowledge and past analytical data, it is known that the waste sludge is near background levels for tritium. However, process knowledge cannot be provided for other possible hazardous constituents. It is through the use of sampling and analysis that any other hazardous constituents will be identified.

Use of Results

Result of the sampling and analysis will be used to demonstrate compliance with 10 CFR and DOE orders. They will also be used to ensure compliance with RCRA hazardous waste regulations. In the event that the analysis reveals hazardous constituents, the results will be used to develop a treatment plan to facilitate land disposal.

### 2.2.6 Personnel and Responsibilities

In order to implement a quality sampling and analysis plan, a number of personnel and disciplines are involved. Below is a list of the personnel and their responsibilities within the sampling and analysis plan:

**Health Physics** - Health Physics will issue a special work permit (SWP) allowing for the sampling of the waste sludge.

**Waste Facility Operator** - A Waste Facility Operator will sample the waste sludge under the supervision of specialist, waste management.

**Environmental Technician** - The Technician, Hazardous Waste assists in the sampling and supervises the completion of the related documents such as NFC1-156, Certification of Representative Sample.

**Advanced Specialist, Waste Management** - The Advanced Specialist, Waste Management directs, coordinates, and supervises the waste sludge sampling. The specialist writes the sampling and analysis plan. The specialist arranges for the transportation of the samples to a qualified laboratory for analysis, completes the shipping paperwork, and approves the chain of custody.

**Packaging Engineer** - The Packaging Engineer will supervise the packaging of the waste sludge samples in accordance with DOT regulations.

**Receiving and General Stock (R&GS)** - R&GS ships samples that are transported by a designated carrier. R&GS also inputs information from the shipper Purchase Order (PO) into the Shipment Mobility/Accountability Collection (SMAC) System.

**Purchasing** - Purchasing provides the shipper POs used to ship samples of waste sludge for analysis. It also issues a PO number in response to a general purchase requisition (GPR)

**Specialist, Quality Assurance** - The Specialist, Quality Assurance evaluates vendor qualifications and monitors the performance of outside laboratories on the basis of supplier surveys and scope of work submitted with the GPR.

### 2.2.7 Supplies Needed

<u>Safety Equipment</u>

- Safety glasses
- Rubber gloves

- Tyvex suit
- Plastic apron

Materials Checklist

- Sample log book
- Sampling Plan
- FC-2604, Sample Information Form
- FC-209, Chain of Custody Record
- Sampling device, such as a stainless steel corer
- Precleaned glass sample jars
- Precleaned one-gallon glass jar
- Precleaned glass stirring rod
- Precleaned glass coliwasa
- Identification labels
- Safety equipment

### 2.2.8 Sample Documentation

A sample is tracked from its collection point through analysis by using several types of documentation.

Log Entry

The Advanced Specialist, Waste Management maintains a sample log book. The sample log book is used to record and number all waste samples as they are collected.

Sample Labeling

Every sample is required to have a label that indicates the name of the material sampled, sampling date and time, area in which the sample was collected, and the sampler's signature.

Sample Information Form, FC-2604

Sample information is used to communicate sampling and analysis instructions and to certify that the samples collected are representative of their source. The sample information form is filled out by the person who requests sampling and by the people who collect the samples. Multiple samples can be recorded on one form as long as they come from the same source. Split samples share the same ID number and are distinguished by a letter suffix (-A, -B, etc.). Before giving the sample information form to the Advanced Specialist, Waste Management, the sampler photocopies the analysis parameters on page two and attaches them to the chain of custody record.

Chain of Custody Record, NFC-209

The chain of custody record is used to track samples from their source, through analysis, and back to Waste Management. The chain of custody record is filled out by the sampler, approved by the Advanced Specialist, and is signed over to the person who is delivering the samples to the laboratory. This delivery person signs the chain of custody indicating possession of the samples. The delivery person then signs the chain of custody over to the person taking possession of the samples at the laboratory. The person receiving the samples at the laboratory signs the chain of custody indicating he has possession of the samples. The samples are kept in a locked cabinet until they are analyzed and the results are reviewed. The laboratory returns the chain of custody with the analysis results attached, as well as any remaining sample to Waste Management.

### 2.2.9 Sampling Discussion

Sampling Purpose

The purpose of this procedure is to direct the collection of waste sludge samples for analysis. the waste sludge will be sampled and analyzed for tritium activity and RCRA hazardous constituents prior to shipment to a disposal facility.

Definitions

**Representative Sample** - A sample that reflects the entire contents of the sampled vessel. This procedure requires the collection of representative samples of the accumulated waste sludge.

**Sample Custodian** - The person who has physical possession of the sample, or the person who last possessed it if it has not been formally transferred to another person or facility, such as a laboratory. The transfer of a sample from one custodian to another must be recorded on a Chain of Custody record.

Number of Samples

The neutralization tank will be sampled in ten random locations. A core will be taken from the top to the bottom of the sludge at each of the ten locations. Each core will be placed into a precleaned one-gallon glass jar and thoroughly stirred with a precleaned glass stirring rod or coliwasa. The stirred sample will then be split by pouring sample into four precleaned, 8 oz glass sample jars and one 30 ml VOC vial. The samples will be allocated according to the following chart.

| Sample Description | Test Parameter |
|---|---|
| No. 1 8-oz. jar | Library sample |
| No. 2 8-oz. jar | TCLP Metal, Solid, |
| moisture | |
| No. 3 8-oz. jar | Tritium Analysis, pH |
| No. 4 8-oz. jar | TCLP semivolatiles |
| 30 ml VOC vial | TCLP Volatiles |

In order to avoid confusion during the sampling process, the allocation of the samples cannot be changed without a rewrite of the sampling and analysis plan by the Advanced Specialist, Waste Management. The samples will be shipped to the appropriate laboratories for analysis.

Sampling Rationale

The rationale in sampling is to collect a representative sample of the accumulated waste sludge which contains all of the substances that are present in neutralization Tank No. 2. This sample will then be analyzed for tritium activity as well as RCRA constituents to determine the appropriate disposal method.

Sampling Strategy

The strategy in sampling the neutralization Tank No. 2 will be to core down through the sludge until the bottom of the concrete tank is reached in 10 randomly chosen locations with a stainless steel coring device. Coring from top to bottom will negate any affect of stratification or layering of the sludge in the neutralization tank. This core of material will be thoroughly stirred with a precleaned glass stirring rod or coliwasa. The stirred sample will be used to fill four 8 oz. glass sample jars and one 30 ml VOC vial.

### 2.2.10 Sampling Procedure

The Advanced Specialist, Waste Management will obtain a special work permit (SWP) from health physics allowing for the sampling of the accumulated waste sludge.

The water is drawn down in the neutralization tank(s) within a few inches of the sludge to allow Waste Management personnel to retrieve composite samples.

The Advanced Specialist assigns personnel to perform the sampling. All sampling must be done by two members of Waste Management: one collects the samples while the other directs according to this procedure.

The Waste Facility Operator will put on safety glasses, rubber gloves, tyvex suit and a plastic apron. Under the supervision of the Advanced Specialist, the Waste Facility Operator will determine the sampling locations in the neutralization Tank No. 2 as designated by the sampling plan.

Using a stainless steel coring device, the Operator will core down through the sludge at each of the designated locations until the bottom of the concrete tank is reached (approximately 2 to 3 feet). The material removed from the tank with the coring device will be placed into a precleaned one-gallon glass jar.

The material in the one-gallon glass jar will be thoroughly mixed by stirring with a precleaned glass stirring rod or coliwasa. The mixed sample will be placed into four precleaned, 8 ounce, glass sample jars and one 30 ml VOC vial. The lids will be placed on the sample jars and the sample labels will be attached to the sample jars.

One 8 oz. sample jar will be held by Waste Management as a back-up (library sample) and the three other 8 ounce sample jars along with the 30 ml VOC vial will be shipped to the appropriate laboratories for analysis.

### 2.2.11 Reference Document

Title 40 CFR, Parts 260 through 266.

Title 49 CFR, Parts 100 through 177.

SW-846, Test Methods for Evaluating Sold Waste, Environmental Protection Agency (EPA)

NVO-325 (Rev. 1) Nevada Test Site (NTS) Defense Waste Acceptance Criteria, Certification, and Transfer Requirements.

## 2.3 Assessment of LES Grassy Mountain Facility

LES Grassy Mountain is a disposal facility for hazardous waste located in Tooele County, Utah. The facility, which can accept low level radioactive waste, is located on the eastern edge of the Great Salt Lake Desert, 85 miles west of Salt Lake City and approximately ten miles north of Interstate 80. The disposal site is in an area set aside by the county and zoned for hazardous waste disposal.

The LES Grassy Mountain facility is near the DOE Vitro Uranium Mill Tailings disposal facility. The DOE facility contains several million cubic yards of low-level radioactive mill tailings generated by the Vitro Company at their former uranium mill near Salt Lake City. An Environmental Impact Statement (EIS) was completed by DOE on the suitability for using the site for the disposal of radioactive material at the time the mill tailings were placed there.

The climate is typically desert arid conditions, with average annual rainfall of less than 5 inches and evaporation rate of greater than 70 inches. The groundwater levels under the site vary from 20 to 30 feet below ground surface. The groundwater at the site is classified as "briny". The EIS conclusion on the site was, "Given existing technologies, however, development of the area for any purpose appears unlikely because of it unproductive soil, and its remoteness from population centers". There are no residential and agricultural activities within a 30 mile radius of the facility.

In August 1995, the Lockheed Martin Specialty Components Waste Management personnel conducted an audit of the LES Grassy Mountain facility in Clive, Utah. LES Grassy Mountain passed the audit in all aspects and a report will be issued in the near future and will be issued as an Addendum to this Exemption Request.

LES Grassy Mountain has a Part B permit, EPA Identification No. UTD991301748, to dispose of hazardous waste at their facility. This permit was issued June 30, 1988, by the Utah Bureau of Solid and Hazardous Waste.

Each state licensee must meet financial responsibility requirements as a license condition, and the financial responsibility requirements for commercial radioactive waste burial facilities provide assurance that funds are available for closure and monitoring of the site prior to the termination of the license. There are similar financial responsibility requirements under RCRA. LES Grassy Mountain has met these financial requirements.

Waste can be received by the LES Grassy Mountain facility in a number of ways. It can be shipped via trucks using Interstate 80 or by rail using Union Pacific's main rail line that runs east and west just south of the interstate. Waste can be received by the site in a number of forms ranging from barrels, boxes, bags, to bulk containers and rail cars.

The material is placed in the cell in discrete lifts. Each lift is compacted and its compaction checked prior to the next placement of waste. The compaction criteria required does preclude the placement of soil while the material is frozen so the soil received in the winter is typically stored onsite until spring. Accurate records are maintained on the location of the waste in the cell. The wastes placed in the cell are segregated by waste generator to build a waste specific cell for the exclusive use of that client.

LES Grassy Mountain has an established relationship with the Utah Bureau of Solid and Hazardous Waste. There is currently no state opposition to bringing out-of-state waste to the site.

## 2.4 Comparison of DOE and NRC Requirements

This section is a comparison of the requirements of DOE Order 5820.2A with the NRC requirements at 10 CFR 61.

## COMPARISON OF REQUIREMENTS

## DOE ORDER 5820.2A AND NRC REQUIREMENTS AT 10 CFR 61

| DOE Order 5820.2A Requirements (Paragraph/Content Summary) | | 10 CFR 61 Requirements (Part Number/Content Summary) | |
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| Charter III: Policy Statements | | 61.23 | A license for the receipt, possession and disposal of waste containing or contaminated with source, special nuclear, or by-product material will be issued by the Commission upon finding that the issuance of the license will not be inimical to the common defense and security and will not constitute an unreasonable risk to the health and safety of the public, and: . . . |
| 1.0 | DOE-low-level waste operations shall be managed to protect the health and safety of the public, preserve the environment and ensure that no legacy requiring remedial action remains after operation. | | |
| 2.b | DOE-low-level waste operations shall be managed on a systematic basis using the most appropriate combination of waste generation reduction, segregation, treatment, and disposal practices so that the radioactive components are contained and the overall system cost effectiveness is maximized. | | No directly comparable requirement. |
| 2.c | DOE-low-level waste shall be disposed of on the site at which it is generated if practical, or if on-site disposal capability is not available, at another DOE disposal facility. | | No directly comparable requirement. |
| 2.d | DOE-low-level waste shall conform to the requirements of DOE Order 5820.2A, applicable EH Orders and shall also be regulated by the appropriate regional authorities under RCRA. | 61.23 | See previous description. |

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|  | Requirements | 61.23 | See Previous description. |
| 3.a.1 | Protect public health and safety in accordance with standards specified in applicable EH Orders and DOE Orders. |  |  |
| 3.a.2 | Assure that external exposure to the waste results in an effective dose equivalent (EDE) that does not exceed 25 mrem/yr to any member of the public. | 61.41 | Concentrations of radioactive material which may be released to the general environment in ground water, surface water, air, soil, plants, or animals must not result in an annual dose exceeding an equivalent of 25 millirems to the whole body, 75 millirems to the thyroid, and 25 millirems to any other organ of any member of the public.<br>Reasonable effort should be made to maintain releases of radioactivity in effluents to the general environmental as low as is reasonably achievable. |
| 3.a.2 | Releases to the atmosphere shall meet the requirements of 40 CFR 61. | 61.41 | See previous description. |
| 3.a.3 | Assure that committed effective dose equivalents received by intruders after loss of institutional control (100 years) will not exceed 100 mrem/yr for continuous exposure or 500 mrem for a single acute exposure. | 61.42 | Design, operation, and closure of the land disposal facility must assure protection of any individual inadvertently intruding into the disposal site and occupying the site or contacting the waste at any time after institutional controls over the disposal site are removed. |
| 3.a.4 | Protect ground water resources consistent with Federal, State and local requirements. |  | No directly comparable requirement. |

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| 3.b.1 | Field organizations with disposal sites shall prepare and maintain a site specific radiological performance assessment for the disposal of waste to demonstrate compliance with the performance objectives stated in para. 3a. | 61.40 | Land disposal facilities must be sited designed operated, closed, and controlled after closure so that reasonable assurance exists that exposures to humans are within the limits established in the performance objective in 61.41 through 61.44. |
| 3.b.2 | Each field organization shall, for each DOE reservation within it cognizance, prepare and maintain an overall waste management systems performance assessment supporting the combination of waste management practices being used. | | No directly comparable requirement. |
| 3.b.3 | Where practical, monitoring measurements to evaluate actual and prospective performance should be made at locations as required within and outside each facility and disposal site. | 61.53(c) | During the land disposal facility site construction and operation the licensee shall maintain a monitoring program. Measurements and observation must be made and recorded to provide data to evaluate the potential health and environmental impacts during both the construction and the operation by the facility and to enable the evaluation of long-term effects and the need for mitigative measures. The monitoring system must be capable of providing early warning or radionuclides from the disposal site before they leave the site boundary. |
| 3.c.1 | Technical and administrative controls shall be directed to reducing the gross volume of waste generated and/or the amount of radioactivity requiring dose. | | No directly comparable requirement. |

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| 3.c.2 | All DOE-low-Level waste generators shall establish auditable programs to ensure that the amount of low-level waste generated and/or shipped for disposal is minimized. | 61.52a(11) | Only wastes containing or contaminated with radioactive materials shall be disposed of at the disposal site. |
| 3.c.3 | Each DOE-low-level waste generator shall separate uncontaminated waste from low-level waste to facilitate cost effective treatment and disposal. | 61.52a(11) | See previous description. |
| 3.c.4 | Each DOE-low-level waste generator preparing a design for a new process or process change shall incorporate principles into the design to minimize generation of low-level waste. | | This appears to be a generator requirement. |
| 3.d.1 | Low-level waste shall be characterized with sufficient accuracy to permit proper segregation, treatment, storage, and disposal. | | This appears to be a generator requirement. |
| 3.d.2 | Waste characterization data shall be recorded on a waste manifest, and include: a) physical and chemical characterization of the waste, b) volume of waste, c) weight of waste, d) major radionuclides and concentrations, e) packaging date, package weight, and external volume. | | This appears to be a generator requirement. |
| 3.d.3 | The concentration of a radionuclide may be determined by direct methods or by indirect methods such as use of scaling factors which relate the inferred concentration of one radionuclide to another that is measured. | 61.55a(8) | The concentration of a radionuclide may be determined by indirect methods. |

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| 3.e.1 | Waste shipped from one field organization to another for treatment, storage or disposal shall be done in accordance with the requirements established by the operations office having responsibility for operations of the receiving facility. | No directly comparable requirements. |
| 3.e.2 | Waste acceptance criteria shall be established for each low-level waste treatment, storage, and disposal facility, and submitted to the cognizant field organization. | No directly comparable requirements. |
| 3.e.3 | Generators of waste shall implement a low-level waste certification program to provide assurance that the waste acceptance criteria for any low-level waste treatment, storage, or disposal facility used by the generator are met. Generators and facilities receiving the waste are jointly responsible for assuring compliance with waste acceptance criteria. | This appears to be a generator requirement. |
| 3.e.4 | Generator low-level waste certification programs shall be subject to a periodic audit by operators of facilities to which the waste is sent by the generator. | This appears to be a generator requirement. |

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| 3.e.5 | Waste acceptance criteria for storage, treatment, or disposal facilities shall address the following issues: a) allowable quantities/concentrations of specific radioisotopes to be handled, processed, stored or disposed of; b) criticality safety requirements; c) restrictions regarding low-level waste classified for security reasons; d) external radiation and internal heat generation; e) restrictions on the generation of harmful gases, vapors, or liquids in waste; f) chemical and structural stability of waste packages, radiation effects, microbial activity, chemical reactions, and moisture; g) restrictions for relating and complexing agents having the potential for mobilizing radionuclides; and h) quantity of free liquids. | 61.56 | The following requirements are minimum requirements for all classes of waste and are intended to facilitate handling at the disposal site and provide protection of safety and health of personnel at the site . . . |
| 3.f.1 | Waste shall be treated by appropriate methods so that the disposal site can meet the performance objectives. | 61.40 | Land disposal facilities must be sited, designed, operated, closed and controlled after closure so that reasonable assurance exits that exposures to humans are within the limits established in the performance objectives 61.41 through 61.44. |
| 3.f.2 | Waste techniques shall be implemented as necessary to meet performance requirements. | | No directly comparable requirement. |

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| 3.f.3 | The development of large scale waste treatment facilities shall be supported by NEPA documentation in addition to the following: a) a document that analyzes waste streams needing treatment, b) a construction design report, c) a safety analysis report. | 61.10 | An environmental report prepared in accordance with Subpart A of Part 51 of this chapter must accompany the application. Please note Part 51 Subpart A is NEPA regulations implementing section. |
| 3.f.4 | Operation of waste treatment facilities shall be supported by adequate documentation including: a) operation and maintenance procedures, b) personnel training and qualification procedures, c) monitoring and emergency response plans, d) records shall be maintained for each package of low-level waste that enters and leaves the treatment facility. | 61.80(a) | Each licensee shall maintain any records and make any reports in connection with the licensed activities as may be required by the conditions of the licensee of the rules, regulations, and orders of the NRC. |
| 3.g.1 | Off-site shipment of low-level waste shall be in compliance with DOE 1540.1. | 10 CFR 71 | Packaging and transportation of Radioactive Material. |
| 3.g.2 | Generators shall provide an annual forecast in the third quarter of the fiscal year to the field organization managing the off-site disposal facility to which the waste is to be shipped. | | No directly comparable requirement. |
| 3.g.3 | Generators must receive advance approval from the receiving facility and shall certify prior to shipment that waste meets the receiving facility waste acceptance criteria. | | No directly comparable requirement. |

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| 3.g.4 | Each package of waste must comply with the labeling requirements of DOE 1540.1. | 61.57 | Each package of waste must be clearly labeled to identify whether it is Class A waste, Class B waste, or Class C waste in accordance with 61.55. |
| 3.h.1 | Low-level waste shall be stored such that the performance objectives stated in paragraph are met. | | No directly comparable requirement. |
| 3.h.2 | Records shall be maintained for all low-level waste that enters and leaves the storage facility. | 61.80(e) | Licensees shall record the location and quantity of radioactive wastes contained in the disposal site . . . |
| 3.h.3 | The development and operation of a waste storage facility shall be supported by the following documentation; a) an analysis which identifies the need for the storage facility, b) a Construction Design Report, including projected waste planned for storage, construction and operating cost estimates, c) a safety analysis report and appropriate NEPA documentation, and d) operational procedures and plans. | 61.3 | No person may receive, possess, and dispose of radioactive waste containing source, special nuclear, or by-product material at a land disposal facility unless authorized by a license issued by the NRC pursuant to Part 61, or unless exemption has been granted by the NRC under 61.6. |
| 3.i.2 | Engineered modifications for specific waste types and for specific waste compositions for each disposal site shall be developed through the performance assessment model. | 61.54 | NRC may upon request or on its own initiative authorize provisions other than those set forth in 61.51 through 61.53 for the segregation and disposal of waste and for the design and operation of a land disposal facility on a specific basis, if it finds reasonable assurance of compliance with the performance objectives of Subpart C of this part. |

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| 3.i.4 | Disposition of waste designated as greater-than-class C as defined in 10 CFR 61.55 must be justified by a specific performance assessment through the NEPA process. | | No directly comparable requirement. |
| 3.i.5a | Waste must not be packaged for disposal in cardboard or fiberboard boxes, unless such boxes meet DOT requirements and contain stabilized waste with a minimum of void space. | 61.56(a)(1) | Waste must not be packaged for disposal in cardboard or fiberboard boxes. |
| 3.i.5b | No freestanding liquids shall exceed 1% of the volume of the waste when the waste is in a disposal container, or 0.5% of the volume of the waste processed to a stable form. | 61.56(a)(3) | Solid waste containing liquid shall contain as little freestanding and noncorrosive liquid as is reasonably achievable but in no case shall the liquid exceed 1% of the volume. |
| 3.i.5c | Waste must not be readily capable of detonation or of explosive decomposition or reaction at normal pressures and temperatures, or of explosive reaction with water. | 61.56(a)(4) | Waste must not be readily capable of detonation or of explosive decomposition or reaction at normal pressures and temperatures, or of explosive reaction with water. |
| 3.i.5d | Waste must not contain, or be capable of generating, quantities of toxic gases, vapors, or fumes harmful to persons transporting, handling, or disposing of the waste. | 61.56(a)(5) | Waste must not contain, or be capable of generating, quantities of toxic gases, vapors, or fumes harmful to persons, transporting, handling, or disposing of the waste. This does not apply to radioactive gaseous waste packaged in accordance with paragraph (a)(7) of this section. |
| 3.i.5e | Waste in a gaseous form must be packaged at a pressure that does not exceed 1.5 atmospheres at 20°C. | 61.56(a)(7) | Waste in a gaseous form must not be packaged at a pressure that does not exceed 1.5 atmospheres at 20°C. Total activity must not exceed 100 curies per container. |

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| 3.i.5f | Waste must not be pyrophoric. | 61.56(a)(6) | Waste must not be pyrophoric. Pyrophoric materials contained in waste shall be treated, prepared, and packaged to be nonflammable. |
| 3.i.6 | Waste containing amounts of radionuclides below regulatory concern, as defined by Federal regulations, may be disposed without regard to radioactivity content. | | No directly comparable requirement. |
| 3.i.7a | Disposal site selection criteria shall be developed for establishing new low-level waste sites. | 61.7(c) | The licensing process (1) during the preoperational phase, the potential applicant goes through a process of disposal site selection by selecting a region of interest, examining a number of possible disposal sites within the area of interest and narrowing the choice to the disposal site. |
| 3.i.7b | Disposal site selection shall be based on an evaluation of the prospective site in conjunction with planned waste confinement technology and NEPA. | 61.7(c)(1) | See previous description. |
| 3.i.7c | The disposal shall have hydrogeologic characteristics which will protect the groundwater resources. | 61.50(a)(7) | The disposal site must provide sufficient depth to the water table that ground water intrusion, perennial or otherwise, into the waste will not occur. The commission will consider exception if certain conditions are met 6150(a)(8). The hydrogeologic unit used for disposal shall not discharge ground water to the surface within the disposal site. |

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| 3.i.7d The potential for natural hazards such as floods, erosion, tornadoes, earthquakes, and volcanoes shall be considered in site selection. | 61.50(a)(5) The disposal site must be generally well drained and free of areas of flooding or frequent ponding. Waste disposal shall not take place in a 100 year flood plain coastal high-hazard area or wetland, as defined in Executive Order 11988, "Floodplain Management Guidelines." |
| | 61.50(a)(6) Upstream drainage areas must be minimized to decrease the amount of runoff which could erode or inundate waste disposal units. |
| | 61.50(a)(9) Areas must be avoided where tectonic processes such as faulting, folding, seismic activity, or volcanism may occur with such frequency and extent to significantly affect the ability of the disposal site to meet the performance objectives (a)(10) - Areas must be avoided where surface geologic processes such as mass wasting, erosion, slumping, landsliding, or weathering occur with such frequency and extent to significantly affect the ability of the disposal site to meet the performance objectives. |
| 3.i.7e Site section criteria shall address the impact on current and projected populations, land use resource development plans and nearby public facilities, accessibility to transportation routes and utilities, and the location of waste generation. | 61.50(a)(3) Within the region of state where the facility is to be located, a disposal site should be selected so that project population growth and future developments are not likely to affect the ability of the disposal facility to meet the performance objectives. (a)(4) - Areas must be avoided having known natural resources which, if exploited, would result in failure to meet the performance objectives. |

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| 3.i.8a | Design criteria shall be established prior to selection of new disposal facilities, new disposal sites, or both. | | The licensing process (1) During the operational phase, the potential applicant goes through a process of disposal site select by selecting a region of interest, examining a number of possible disposal sites and narrowing the choice to the proposed site. Through a detailed investigation of the disposal site characteristics, the applicant obtains data on which to base an analysis of the site's suitability. |
| 3.i.9a | Field organizations shall develop and implement operating procedures for low-level waste disposal facilities. | 61.23(b) | The applicant is qualified by reason of training and experience to carry out the disposal operations. |
| 3.i.9b | Permanent identification markers for disposal excavations and monitoring wells shall be emplaced. | | No directly comparable requirements. |
| 3.i.9c | Operating procedures shall include training for disposal facility operating personnel, emergency response plans and a system of reporting unusual occurrences. | 61.23(b)<br>61.11(b)(3) | See previous description.<br>A description of the applicants personnel training program. |
| 3.i.9d | Waste placement into disposal units should minimize void spaces. | 61.52(a)(4) | Wastes must be emplaced in a manner that maintains the package integrity during emplacement, minimizes the void spaces between packages, and permits the void spaces to be filled. |
| 3.i.9e | Operations are to be conducted so that active waste disposal operations will not have an adverse effect on filled disposal units. | | No directly comparable requirement. |

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| 3.j.1 Field organizations shall develop site-specific stabilization measures comprehensive closure plans for new and existing operating low-level waste disposal sites. The plan shall address closure of disposal sites within a 5 year period after each is filled and meet EPA requirements. Performance objectives for disposal sites shall be developed on a case-by-case basis as part of the NEPA process. | 61.52(a)(9) Closure and as set forth in the approved site closure plan must be carried out as each disposal unit (e.g., each trench) if filled and covered. (7) - The boundaries and locations of each disposal unit (e.g., trenches) must be accurately located and mapped by means of a land survey. Near-surface disposal units must be marked in such a way that the boundaries of each unit can be easily defined. Three permanent survey marker control points, referenced to the United States Geological Survey (USGS) or National Geodetic Survey (NGS) control stations, must be established on the site to facilitate surveys. The USGS or NGS control stations must provide horizontal and vertical controls as checked against USGS or NGS record files. 61.52(8) - A buffer zone of land must be maintained between any buried waste and the disposal site boundary and beneath the disposed waste. The buffer zone shall be of adequate dimensions to carry out environmental monitoring activities specified in 61.53(d) of this part and take mitigative measures if needed. (1) - Active waste disposal operations must not have an adverse effect on completed closure and stabilization measures. |
| 3.j.2 During closure and post closure, residual radioactivity levels for surface soils shall comply with existing DOE decommissioning guidelines. | No directly comparable requirement. |

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| 3.j.4 | Inactive disposal facilities, disposal sites, and disposal units shall be managed in conformance with RCRA, CERCLA, and the Superfund Amendments and Reauthorization Act, or, if mixed waste is involved, may be included in permit applications for operation of contiguous disposal facilities. | | No directly comparable requirement. |
| 3.j.5 | Closure plans for new and existing operating low-level information must waste disposal facilities shall be reviewed and approved by the appropriate field organization. | 61.12 | Specific Technical include (9) a description of the disposal site, closure plan, including those design features which are intended to facilitate disposal site closure and to eliminate the need for ongoing active maintenance. |
| 3.j.6 | Termination of monitoring and maintenance activity at closed facilities or sites shall be based on an analysis of site performance at the end of the institutional control period. | | No directly comparable requirement. |
| 3.k.1 | Each operational or non-operational low-level waste disposal facility site treatment, storage, and disposal facility should be monitored by an environmental monitoring that conforms with DOE 5484.1 and, at a minimum, meet the requirements of paragraph 3.k.2 through 3.k.4. | 61.53(c) | During the land construction and operation, the licensee shall maintain a monitoring program . . . |

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| 3.k.2 | The environmental monitoring shall be designed to measure: a) operational effluent releases, b) migration of radionuclides, c) disposal unit subsidence, d) changes in disposal facility and disposal site parameters which may effect long-term site performance. | 61.53(c) | Measurements and observations must be made and recorded to provide data to evaluate the potential health and environmental impacts during both the construction and the operation of the facility and to enable the evaluation of long-term effects and the need for mitigative measures. The monitoring system must be capable of providing early warning of radionuclides from the disposal site before they leave the site boundary. |
| 3.k.3 | The environmental monitoring program may include, but not necessarily be limited to, monitoring surface soil, air, surface water, and in the subsurface, soil and water, both in the saturated and the unsaturated zones. | | No directly comparable requirement. |
| 3.k.4 | The monitoring program shall be capable of detecting changing plans for taking trends sufficiently in advance to allow application of any necessary corrective action prior to exceeding performance objectives and ascertain whether or not the requirements of applicable EH orders are being met. | 61.53(b) | Licensee must have corrective measures if migration of radionuclides would indicate that the performance objectives may not be met. |
| 3.1 | Consistent with DOE 5700.6B, the low-level waste operational and disposal practices shall be conducted in accordance with applicable requirements of ANSI/ASME NQA-1 and other appropriate national consensus standards. | 61.53(c) | See previous summary. |

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| 3.m.1 | Each field organization shall develop and maintain a record system that records the following: historical record of waste generated, treated, stored, shipped, and/or disposed of. | 61.89\ | Following receipt and acceptance of a shipment of radioactive waste the licensee shall record the date of disposal of the waste, the location in the disposal site, the condition of the waste. |
| 3.m.2 | A waste manifest shall be kept and accompany each waste package from generator through final disposal. At a minimum the following data should be included: a) waste physical and chemical characteristics, b) quantity of each major radionuclide present, c) weight of the waste, d0 volume of the waste. |  | See 10 CFR 20.311(b). |
| Chapter IV: Policy Statement | DOE waste containing NARM material or 11e(2) by-product material is defined in the Atomic Energy Act, as amended, or similarly contaminated material derived from DOE remedial actions, shall be stored, stabilized in place, and/or disposal of consistent with the requirements in 40 CFR 192. Small volumes of such DOE waste may be managed as low-level waste in accordance with the requirements of Chapter III of this Order. If the waste is classified as mixed waste RCRA requirements must also be met. |  | See 10 CFR 40 - Appendix A. |

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| | Requirements | See 10 CFR 40 - Appendix A. |
| 3.a.1 | Wastes covered under this chapter in quantities too large for acceptance at DOE low-level waste disposal site shall be managed according to the requirement of 40 CFR 192 and disposed of at specially designated DOE sites or tailing disposal sites established under UMTRCA of 1978 (Public Law 95-604). These sites will normally be located in the State in which the wastes are generated. | |
| 3.a.2 | With the approval of the appropriate field organization, small volumes of 11e(2) and NARM material may be disposed of at DOE low-level waste sites in accordance with the requirements of Chapter III DOE 5820.2A. | 61.1(b) The regulations in this part do not apply to (1) disposal of high-level waste as provided for in part 60 of this chapter, (1) disposal of uranium or thorium tailings or wastes (by-product material as defined in 40.4(a-1). . . in quantities greater 10,000 kg and containing more than mCi or Ra-226. |
| 3.a.3a | NARM material mixed with RCRA hazardous chemicals shall be managed as hazardous waste under RCRA. | No directly comparable requirement. |
| 3.b | Consistent with DOE 5700.6B, waste management practices shall be conducted in accordance with applicable requirements of ANSI/ASME NQA-1 and other appropriate national consensus standards. | No directly comparable requirement. |

## 2.5 Quality Assurance and Waste Certification

When packaging and shipping of MW, Pinellas personnel will adhere to the Pinellas Plant Quality Plan for Radioactive Waste Shipment (WM 7.11). The plan describes the certification program for radioactive wastes generated at the Pinellas Plant and defines the Plant's Quality Assurance (QA) program as it applies to waste certification and characterization. The relevant elements of American National Standards Institutes/American Society of Mechanical Engineers (ANSI/ASME) NQA-1, Quality Assurance Program and Supplements (NQA-1), are addressed, and implementation of plant standards and procedures are identified.

Further, the Project Plan establishes a management plan that will demonstrate compliance of MW streams generated at the Pinellas Plant with the RCRA LDR. MW streams generated at the plant are analyzed to ensure restricted wastes meet the applicable treatment standards. Characterization involves a combination of process knowledge, existing analytical data, and additional sampling and analysis. The sampling and analysis process was developed to acquire information necessary for proper characterization of mixed waste streams while maintaining data quality objectives.

Records that furnish documentary evidence of quality shall be specified, prepared, and maintained. Records shall be legible, identifiable, and retrievable. Records shall be protected against damage, deterioration, and loss. Requirements, responsibilities for record transmittal, distribution, retention, maintenance and disposition shall be established and documented.

LES Grassy Mountain maintains QA procedures and control, as well as, procedures for performance and systems audit. The procedures are contained in the LES Grassy Mountain Waste Analysis Plan that is attached as Appendix E. The operational QA for the LES Grassy Mountain facility is also included in Appendix E.

## 2.6 Mixed Waste Transportation

Interstate and intrastate transportation of mixed waste is simultaneously controlled at the Federal and state levels by several government agencies. At the Federal level, the Department of Transportation (DOT) regulates mixed waste transportation through regulations under the Hazardous Materials Transportation Act (HMTA) and the Hazardous Material Transportation Uniform Safety Act (HMTUSA). The regulations applicable to mixed waste transportation are contained in 49 CFR 171-180.

The EPA regulates mixed waste transportation under the RCRA regulations. Transporters of mixed waste are regulated under 40 CFR 263. Mixed waste generators must identify their wastes, obtain EPA identification numbers, comply with the hazardous waste manifest system, and properly package, label, mark, and placard mixed wastes slated for transport. Under 40 CFR 263, mixed waste transporters must also obtain EPA identification numbers. Storage of containerized waste at unpermitted transfer facilities is limited to 10 days or less. Mixed waste transporters must comply with the hazardous waste manifest system, and they are required to report and clean up transportation-related discharges. The Pinellas Plant will adhere to all applicable DOT, EPA and state regulations concerning the shipment of MW to an offsite commercial disposal facility.

## 2.7 Options and Cost Analysis

The purpose of this section is to discuss the alternatives available for the disposal of tritium contaminated F006 Sludge by the Pinellas Plant. Each alternative has its advantages and disadvantages. Costs are derived by analysis of the identified alternative. This section provides a breakdown of cost estimates and associated assumptions.

Table 2. Summary of Alternatives

| Waste Classification | Evaluation Factors | Permanent Disposal at LES Grassy Mtn | Permanent Disposal at Envirocare | Interim Storage at Hanford | Treatment and Storage Onsite |
|---|---|---|---|---|---|
| 1. Tritium Contaminated F006 Sludge (treated before land disposed) | Cost | $165,940 | $493,333 | $1,075,300 | |
| | Likelihood of Success | High | High | See Note | Not Feasible |

Note: Probability of success is high, provided permission can be obtained to store Pinellas MW at Hanford. It is very important to note, however, that this alternative only postpones the permanent disposition of the waste to a later date. Hanford has no capability for disposal of this type of waste. Final disposal costs are not included in this alternative.

### 2.7.1 IWNF Sludge

An approximate quantity of 189 cubic meters of F006 Sludge at the Pinellas Plant is classified as potential MW. The physical and chemical matrix of the waste is a relatively homogeneous mixture of precipitates and water containing contaminated with heavy metals. The low-level radiological contaminant is tritium. For purposes of this analysis, one waste stream was considered.

The process of dewatering the waste sludge and then packaging and shipping the waste to LES Grassy Mountain for final disposal was reviewed but resulted in a significant additional cost and increased schedule.

The following assumptions were made in the preparation of this estimate:

1. Solid waste consists of liquid sludge. The liquid volume of 189 cu meters was used in the calculations.
2. Adequate utilities exist at the TSDF to treat and solidify the sludge at their site and will be furnished.
3. Solidification or treatment will be performed by the TSDF.
4. Shipping will be done via truck, with no more than ten tanker trucks (liquid). Shipping costs are $7838 per tanker to LES Grassy Mountain, $2.30/mile for Envirocare and $2.09/mile for Hanford. These are base costs only and do not include any additional handling costs.
5. Disposal costs for waste at LES Grassy Mountain are $286 per ton of liquid. Envirocare charges $57 per cubic foot.
6. Storage costs for mixed waste at Hanford are $350/per cubic foot.

Calculations:

### LES Grassy Mountain Option

| | | | |
|---|---|---|---|
| Pump sludge to tanker (8 hours) | X10 | = | $24,000.00 |
| Confined space entry and training | | = | 3,500.00 |
| Transportation costs to Clive, Utah per tanker ($7838.00) | X10 | = | 78,380.00 |
| Treat and Dispose at LES Grassy Mountain ($286.00/ton liquid) | | = | 60,060.00 |
| Total | | | $165,940.00 |

### Envirocare Option A

| | | | |
|---|---|---|---|
| Pump sludge to tanker (21 tons per tanker at $150/ton) | X10 | = | $31,500.00 |
| Confined space entry | | = | 3,500.00 |
| Transport to Clive, Utah (2300 miles at $2.30/mile) | X10 | = | $52,900.00 |
| Solidify at Envirocare Specialty Components estimate) | | = | 25,000.00 |
| Disposal at Envirocare ($1,538.88/CY X247.2 CY) | | | $380,430.00 |
| Total | | | $493,333.00 |

### Hanford Option

| | | |
|---|---|---|
| Dewatered Sludge (approx 20,000 gal) | = | $96,000.00 |
| Load into 55 gallon drums (360 drums) | = | $5,000.00 |
| Transport to Richland, WA in six trucks (3,200 miles at $2.00/mile) | = | 38,400.00 |
| Store at Hanford ($350.00/cu ft X 2674 cu ft) | = | 935,900.00 |
| Total | | $1,075,300.00 |

### 2.8 Procurement Method

Specialty Components will provide project funds for LLW disposal at the LES Grassy Mountain Facility through contract DE-AC04-9 2AL73000. Specialty Components will prepare a purchase order based on a minimum of 50,000 gallons of estimated waste volume, consistent with the prevalent charge per cubic foot.

### 2.9 NEPA Analysis

When the determination is made that the F006 Sludge contains above background radioactive material (tritium) a NEPA evaluation will be conducted. An example of the NEPA checklist is in Appendix A.

## 3.0 AUDITS

### 3.1 Pinellas Plant Requirements

Surveillances at the Pinellas Plant are performed by the Environmental Oversight and Quality Assurance throughout the waste management process. Surveillances are performed at the point of segregation on a random basis determined by an accepted method. Points have been established for surveillance activity including pre-use of containers, contents before closure and after closure before final shipment.

Internal audits are also conducted on at least an annual basis by a team of auditors, including at least one "lead auditor", qualified to NOA-1. The formal program includes establishing long-term plans of functions, system, and activities to be audited, detailed plans regarding personnel, procedures and scope for individual audits. All audits are conducted using written checklists. Results of all audits are documented in an audit reports addressed to responsible management for written corrective audit responses to identified observations and findings. All nonconformances are documented in a report format. Verification of completion on all accepted corrective responses are documented prior to close out of the audit.

Procedures governing Quality Audits are as follows:

- Planning and Scheduling of Audits
- Administration of Audits
- Conducting Audits

Environment, Safety and Health Audit/Appraisal Program establishes policy and procedures for appraisal operations at the Pinellas Plant to ensure management that Environment, Safety and Health (ES&H) Program objectives are met, specifically that:

- Desired results are being achieved,
- Contractual ES&H requirements and approved policies and procedures are being effectively maintained,
- Internal ES&H controls by management are effectively maintained,
- Applicable laws and regulations pertaining to safety are significant to both U.S. DOE and Pinellas.

Internal audits and surveillance will be performed, as required, at the LES Grassy Mountain Facility according to procedures outlined in the Waste Analysis Plan that is attached as Appendix E. Laboratory QA is also described in the operational QA for the LES Grassy Mountain facility in Appendix C.

## 3.2 Audit of LES Grassy Mountain Facility

On July 31 through August 3, 1995, a Pinellas Plant Waste Management team was on-site at the LES Grassy Mountain Facility. State records were also reviewed at the Utah Department of Environmental Quality Bureau of Solid and Hazardous Waste in Salt Lake City, UT. No issues were found which would indicate that the site was not in compliance with all State and Federal regulations.

# APPENDIX A

## NEPA DOCUMENTATION FOR PINELLAS PLANT MIXED WASTE DISPOSAL PROJECT

An example of the NEPA checklist that will be followed is given in the following pages. In accordance with DOE requirements, this evaluation will be carried out prior to making arrangements for shipping material.

# Interoffice Memo

MARTIN MARIETTA
MARTIN MARIETTA SPECIALTY COMPONENTS, INC.

Date: January 31, 1995

To: **Bob O'Connell** MS027
**Dick Rossmeisl** MS001 ✓
From: Andrew DeWeerd MS040

Subj: SHIP LOW LEVEL MIXED WASTE FROM NEUTRALIZATION TANKS TO UTAH

The attached Preliminary Change Decision Form **(95-011)** has been reviewed by the Risk Management Department. This change concerns the subject listed above, "Ship Low Level Mixed Waste from Neutralization Tanks to Utah".

The following actions items must be completed . Upon completion of each action item, the **PCDF Change Originator (Dick Rossmeisl)** shall obtain the department contact person's signature and date. Once completed, return this letter to Risk Management for project approval prior to starting work on the project.

| **Signature/Date** | **Action Item** |
|---|---|
| N. Schermerhorn | •Contact Nancy Schermerhorn (x6840) for NEPA approval or categorical exclusion status. |
| M.Findley/P.Neri/R. Rowland | •Contact Industrial Hygiene for Special Work Permit prior to cleaning equipment.<br>•Contractor will need to have proper training to handle mixed waste.<br>•Contractor will need to submit confined space entry work plan, including training certification and hazards, one week prior to start of work. |
| A. Weaver | •Obtain Health Physics Special Work Permit and sample prior to draining tank. |

The following action items do not require sign-offs, but must be implemented/completed prior to project implementation:

1. **If changes to the original scope of work are necessary contact Risk Management immediately.** Please contact me if I may be of assistance.

A. DeWeerd 31 JAN 95
Andrew DeWeerd (x6272)
Engineer, Risk and Emergency Management

Risk Management project approval: ______________________
Safety Engineer

| | | | | |
|---|---|---|---|---|
| cc: | M. Lisenby | 035 | B. Burkhart | 017 |
| | J. Mentzer | 050 | S. Hartson | 015 |
| | M. Locklear | 034 | J. Santa Cruz | 015 |
| | J. Havelka | 050 | R. Simonton | 015 |
| | J. Moore | 050 | C. Eberle | 015 |
| | N. Schermerhorn | 001 | | |

79 21

# U.S. DEPARTMENT OF ENERGY
## Albuquerque Operations Office
## ENVIRONMENTAL CHECKLIST/ACTION DESCRIPTION MEMORANDUM

| | |
|---|---|
| Project/Activity Title:<br>Safety and Health Program Activities - FY 1994 | NEPA ID Number: Date: |
| Program Office: Defense Programs | B/R Code: |
| AO/Contractor:<br>Martin Marietta Specialty Components, PAO | Project/Activity Number: |
| AO/Contractor Contact Name:<br>Sarah Hartson/Charles Biedermann | Signatures:<br>Charles Bied |

A .BRIEF PROJECT/ACTIVITY DESCRIPTION: Include category (experiment, test, modification, maintenance, etc.), location, schedule, cost, etc.

This action includes routine safety and health program activities to be performed at the Pinellas Plant during FY 1994. These actions are consistent with documentation requirements as published in 10 CFR 1021 - National Environmental Policy Act Implementing Procedures, Appendix B to Subpart D, subsection B2, dated April 24, 1992. Examples of the types of activities to be performed as part of this action are presented in Attachment 1.

B. ENVIRONMENTAL CONCERNS: Will the project/activity, either during construction or operation, affect or involve any of the following considerations? If the proposed project/activity represents a commitment to a course of actions that would ultimately require a positive response to one or more of the considerations below, identify consideration numbers and provide explanation.

| | YES | NO |
|---|---|---|
| 1. Air emissions | ✓ | |
| 2. Liquid effluents | | ✓ |
| 3. Solid waste | ✓ | |
| 4. Radioactive materials/soils | ✓ | |
| 5. Hazardous constituents | | ✓ |
| 6. Mixed waste (rad & haz) | | ✓ |
| 7. Chemical storage/use | | ✓ |
| 8. Petroleum storage/use | | ✓ |
| 9. Asbestos materials | ✓ | |
| 10. Utility system | | ✓ |
| 11. Clearing or excavation | ✓ | |

| | YES | NO |
|---|---|---|
| 12. Activity outside area fence/wildlife | | ✓ |
| 13. Archaeological/cultural resources | | ✓ |
| 14. Noise levels | | ✓ |
| 15. Radiation/toxic chemical exposures | ✓ | |
| 16. Pesticide/herbicide use | | ✓ |
| 17. High Energy source/explosives | | ✓ |
| 18. Transportation issues | | ✓ |
| 19. Special status species/environment | | ✓ |
| 20. Environmental restoration site | | ✓ |
| 21. Other | | ✓ |

Explanation and qualification of specific "yes" responses.
Explanation - See Attachment 2

C. Permits: Does the proposed project/activity require any local, state or federal permits/notifications or potential violation of laws, regulations, or DOE Orders?

___ Yes ✓ No

Explain:

The intent of the Pinellas Plant is to perform this proposed action in accordance with all applicable statutory and regulatory requirements, permits, and DOE Orders. This project/activity has been reviewed with respect to regulatory compliance requirement at the Pinellas

Plant. No permits have been determined to be needed for this project at this time.

Future activities performed at the site in fulfillment of this action will be reviewed for regulatory compliance prior to their initiation. This review will include:

1. Solid Waste Regulations review
2. Resource Conservation and Recovery Act (RCRA) permit review
3. Hazardous Solid Waste Amendments permit review
4. Clean Air/Water Act compliance review
5. State of Florida Air/Water Regulations compliance review
6. Southwest Florida Water Management District (SWFWMD) approval to withdraw groundwater
7. Pinellas County Air/Sewer Use permit review
8. Toxic Substances Control Act compliance review

D. NCO CLASSIFICATION/RECOMMENDATION: ____________________

Signature: ____________________ Title ____________________ Date ____________________

E. PSO DETERMINATION/RECOMMENDATION: ____________________

Signature: ____________________
Title: ____________________

F. EH OBJECTION/DETERMINATION: ___YES ___No

Signature: ____________________
Title: ____________________

# ATTACHMENT 1

## Block A: Project/Activity Descriptions
## Routine Safety and Health Activities at the Pinellas Plant Site - FY 1994

Activities listed below are examples of the types of safety and health activities to be performed at the Pinellas Plant during FY 1994. They are provided for clarification and are not intended to be all-inclusive of the anticipated FY 1994 Safety and Health actions.

1. Modifications of existing Plant facilities to enhance workplace habitability.

    - Modifications to existing heating and air conditioning systems
    - Installations of shielding to reduce sound levels in production areas
    - Installation/modification of interior and exterior lighting systems

2. Installation of and improvement to building and instrumentation control systems including alarm and security systems, fire detection and protection systems, emergency warning systems, etc.

3. Installation of and improvements to health and safety systems.

    - Installation, relocation, or replacement of eye wash stations
    - Installation, internal reconfiguration, or replacement of fume hoods resulting in no net new emissions
    - Installation of radiation monitoring equipment and instrumentation including Kanne chambers
    - Modification/installation of fire suppression systems including wet spray systems

4. Safety and environmental improvements of a facility, including replacement and upgrade of facility components, that do not result in a significant change in the expected useful life, design, or function of the facility.

5. Removal of Asbestos-containing materials from buildings in accordance with EPA NESHAPS (National Emissions Standards for Hazardous Air Polllutants), and other applicable EPA and/or OSHA Standards.

# ATTACHMENT 2

## Environmental Concerns
## Routine Safety and Health Activities
## at the Pinellas Plant - FY 1994

| Concern Number | Response |
|---|---|
| 1. Air emissions: | As a result of the planned activity, some minor changes to the plant's air emissions profile may result. All such changes will be properly addressed according to existing air pollution regulations and air permit conditions. |
| 3. Solid waste: | Some activities, such as installation of improved air conditioning or ventilation systems, may produce uncontaminated solid waste which will be disposed of in a approved off-site landfill. All debris will be screened for the presence of hazardous constituents prior to removal from the site in accordance with DOE and Pinellas Plant procedures. |
| 4. Radioactive waste/soil: | Small amounts of low-level radioactive waste may result from routine modification of tritium removal and monitoring systems. All radioactively contaminated waste and potentially radioactive waste will be handled in accordance with MMSC procedures for identification, segregation, labeling , packaging, transport, and disposal. Radiologically contaminated waste will be disposed of off-site in accordance with established DOE and Pinellas Plant procedures. |
| 9. Asbestos materials: | As a result of facility health and safety system modifications, asbestos containing insulation, floor tiles, and other materials may be encountered. All asbestos removal will be conducted by trained personnel according to applicable EPA NESHAPS and OSHA requirements using appropriate monitoring and personal protective and containment measures. Waste materials will be disposed of in accordance with regulatory requirements and DOE Orders and Pinellas Plant procedures. |
| 11. Clearing/excavations: | Small areas may be excavated for the installation and/or removal of utility lines, fences, security stations, etc. All excavation activities are reviewed for potential impacts to site environmental resources. Activities that involve impacts to wetlands, or threatened or endangered species are not encompassed by this action. Any clearing or excavation activity, however, has the potential to encounter previously buried or contaminated materials. Activities will be monitored and appropriate action will be taken under applicable programs, if required. |
| 15. Radiation/toxic chemical exposures: | Activities in which asbestos, radiologically or hazardous contaminated materials are encountered will be conducted in accordance with appropriate monitoring and personnel protective measures to protect worker health and safety, and prevent environmental releases. |

# APPENDIX B

# GENERAL AUDIT INFORMATION FOR LES GRASSY MOUNTAIN FACILITY

# LAIDLAW ENVIRONMENTAL SERVICES
# AUDIT INFORMATION

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## GRASSY/GRAYBACK MOUNTAIN FACILITY

LAIDLAW
ENVIRONMENTAL
SERVICES

# I. FACILITY IDENTIFICATION

| | |
|---|---|
| Facility Name | Grassy Mountain (RCRA) and<br>Grayback Mountain (TSCA) |
| Site Location: | 3 miles east, 7 miles north, off I-80,<br>Exit 41 (Knolls) |
| Mailing Address: | P. O. Box 22750<br>Salt Lake City, Ut 84122-9998 |
| Phone: | (801) 595-3900 |
| EPA I.D. No.: | UTD991301748 |

## A. Grassy and Grayback Mountain Facility Management

| | |
|---|---|
| General Manager: | Randy Miller |
| Environmental Manager: | Don Hensch |
| Laboratory Director: | Adam Ganz |

# II. COMPANY ORGANIZATION

U.S. Pollution Control, Inc. (USPCI), and Oklahoma corporation, is a wholly-owned subsidiary of USPCI, Inc., with both based in Columbia, South Carolina, and with field offices throughout the United States. USPCI, is a Laidlaw Company.

## A. Corporate Sales Office

| | |
|---|---|
| Address: | P. O. Box 210799<br>Columbia, SC 29221 |
| Phone: | (803) 798-2993 / (800) 845-1019 |
| Fax: | (803) 551-4348 |

## B. U.S. Pollution Control, Inc. Officers

| | |
|---|---|
| President | William E. Stilwell, Jr. |
| Director: | William E. Stilwell, Jr. |
| Officers: | |
| President | William E. Stilwell, Jr. |
| Vice President | David M. Sprinkle |
| Vice President | Michael A. Faucett |
| Vice President | William H. Shea, Jr. |
| Vice President | Gary McCuistion |
| Vice President | Ken Shuler |
| Vice President | Jeff Zelik |
| Vice President | Randy Miller |
| Vice President | Robert Arquilla |
| Secretary | Henry H. Taylor |
| Assistant Secretary | Barbara J. Hamilton |
| Assistant Secretary | Susan A. Whittaker |
| Treasurer | Paul R. Humphreys |
| Assistant Treasurer | William D. Ridings |
| Assistant Treasurer | Thomas W. Ritter |

## III. PERMITS-AUTHORIZATIONS

### A. Chronology

In 1982, the Grassy Mountain facility opened and began limited operations. The facility received its Part A permit on August 3, 1983. A time frame was then established for submittal and review of individual sections. The State of Utah declared the Part B application complete and developed a draft permit for public review and comment, which was released on November 19, 1987. The Grassy Mountain facility received its Part B permit on June 30, 1988 for RCRA operations.

Under the Toxic Substances Control Act (TSCA), the U.S. Environmental Protection Agency (EPA) also first approved, in December 1985, landfill disposal of PCB solid wastes at this site.

### B. Authority-Agency Contacts

The U.S. EPA, the Utah State Department of Environmental Quality, Division of Solid and Hazardous Waste, and Tooele County are the authorities governing the generation, treatment, and disposal of hazardous wastes within the State of Utah. EPA Region VIII (Denver, Colorado) is the governing authority with regard to wastes regulated by TSCA.

AGENCY CONTACTS

Utah State Department of
Environmental Quality
Division of Solid and Hazardous Waste
288 North 1460 West
Salt Lake City, UT 84114-4880
(801) 538-6170

U.S. Environmental Protection Agency
Region VIII
999 18th Street, Suite 500
Denver, CO 80202
(303) 293-1509

## IV. FACILITY SITE CHARACTERISTICS

### A. Geographic Location

The facility is located in the Great Salt Lake Desert, approximately 85 miles west of Salt Lake City, Utah, in Tooele County. Its location is within a 100 square mile zone set aside by the Tooele County Commission for hazardous waste activities. The site's approximate location is indicated on Exhibit No. 1. Actual directions to the facility are included as Exhibit No. 5.

The nearest residential neighbor is approximately 40 miles west of the facility in Wendover. The land within a 30-mile radius of the site is used as bombing ranges by the U.S. Air Force and for desert warfare training by the U.S. Army. Ninety-five percent (95%) of the land in the surrounding 30 miles is owned by the Bureau of Land Management. Magcorp owns an evaporation pond located approximately two miles south-southwest of the facility. The total acreage owned by USPCI, Inc. is 640 acres (one square mile); approximately 400 acres is utilized for active waste management by the Grassy and Grayback Mountain facilities. A site plan of the facility is included as Exhibit No. 2.

## V. TREATMENT AND DISPOSAL SERVICES

Total Number of Employees: 88

- Administrative
- Professionals/Engineers/Technicians
- Operational/Production Personnel
- Clerical
- Laboratory Personnel

### A. General Operations

#### 1. Two Treatment and Disposal Facilities:

**Grassy Mountain Facility**

| | |
|---|---|
| a) Disposal: | Landfill cells for solid hazardous (RCRA) waste<br>Landfill cell for solid industrial waste |
| b) Storage: | Containers–110-gallon or less; bulk–10 day staging |
| c) Treatment: | Neutralization<br>Stabilization<br>Oxidizer deactivation |

**Grayback Mountain Facility**

| | |
|---|---|
| a) Disposal: | Landfill cells for solid PCB waste |
| b) Storage: | Containers—110-gallon or less; bulk–10 day staging<br>Tanks |
| c) Treatment: | Transformer drain and flush |

#### 2. Transportation

As of March 1994, USPCI's transportation division owned and operated 81 tractors with more then 195 vans, tankers, and flatbed trailers for transportation of hazardous wastes and PCB solids and liquids. Terminals are located in Los Angeles, CA; San Jose, CA; Houston, TX; San Antonio, TX; Kansas City, MO; Woodward, OK; Lakepoint, UT; Philadelphia, PA; Atlanta, GA; and Tucker, GA.

All tractor-trailers are provided with required personal protective and emergency spill clean-up equipment. Drivers participate in driver safety training programs as well as annual hazardous materials and waste training programs.

Wastes are also accepted from common carriers.

To accommodate rail shipments, a rail siding located at the site allows for unloading of gondolas, intermodal containers, and tank cars. Bulk solids, bulk liquids and drums shipped by truck also have direct access into the facility.

## B. Disposal Cells

The various cells are constructed and operated to exceed applicable state and federal regulations. All PCB, industrial, and hazardous waste cells are constructed above ground level.

### 1. PCB (TSCA) Disposal

Cell X and Cell Y were designed for landfill disposal of TSCA-regulated PCB solid wastes.

**Cell X (Final Closure)**
In early 1986, USPCI completed construction of a disposal cell for PCB-contaminated solids at the Grayback Mountain facility. This cell is constructed similarly to RCRA Cell Two and meets the HSWA requirements in effect at that time.

In ascending order, Cell X consists of: a compacted clay liner with a permeability of less than $10^{-7}$cm/sec., a secondary 60 mil HDPE liner, a secondary leachate detection/collection system, a primary 60 mil HDPE liner, a primary leachate detection/collection system, a non-woven geotextile fabric, and a two-foot protective soil layer. Both of the leachate collection and detection systems drain to sumps for the removal of any leachate. No free-flowing liquids are placed in this cell. Cell X contains approximately 200,000 cubic yards of waste. The final closure for this cell is complete.

**Cell Y**
Cell Y, for PCB landfill disposal, was completed in January 1990, and permitted in February, 1990. It was placed in service in May of that year. This cell measures 532 feet by 587 feet, with a designed capacity of 230,300 cubic yards, 250,300 cubic yards with GCL closure.

Cell Y is constructed as follows in ascending order: a compacted clay liner with a permeability of less than $10^{-7}$cm/sec., a secondary 60 mil HDPE liner, a secondary leachate detection/collection system, a primary 60 mil HDPE liner, a primary leachate detection/collection system, a non-woven geotextile fabric, and a two-feet thick protective soil layer. Both the leachate collection and detection systems drain into sumps for the removal of any leachate. This cell also has a stabilization tank which is used to solidify liquid PCB wastes. The PCB concentration of wastes must be known prior to disposal. Neither industrial lagoon sludges nor surface impoundment sludges or solids can be placed in Cell Y.

**Cell Z**
Cell Z was completed in June 1992, and final operating approval was issued in October 1992. This cell measures 1098 feet long, 460 feet wide at the north end, opening to 514 feet on the south end. It has a capacity of 443,000 cubic yards, 482,000 with GCL closure.

Cell Z is constructed as follows in ascending order: a compacted clay liner with permeability of less than $10^{-7}$cm/sec., a secondary 60 mil HDPE liner, a secondary leachate detection/collection system, a primary 60 mil HDPE liner, a primary

Notification of impending closure was filed in mid-December, 1990. When filled and closed, this cell will contain approximately 350,000 cubic yards of waste.

**RCRA Cell Four**

RCRA Cell Four was completed in May 1990, permitted on December 20, 1990, and placed into service on December 27, 1990. This cell is rectangular in shape, measuring 470 feet by 1187 feet, with a capacity of 376,500 cubic yards, 418,200 cubic yards with GCL closure.

In ascending order, this cell consists of a 3-foot compacted clay liner with a permeability of less than $10^{-7}$cm/sec., a secondary 60 mil HDPE liner, a secondary leachate detection/collection system, a primary 60 mil HDPE liner, a primary leachate detection/collection system, a non-woven geotextile fabric, a two-foot soil protective cover, a tertiary 80 mil HDPE liner, a tertiary leachate detection/collection system, a non-woven geotextile fabric, and a two-foot protective soil layer. Both of the leachate collection and detection systems drain into sumps for the removal of any leachate.

**RCRA Cell Five**

RCRA Cell Five was completed in June 1992, and permitted for service on July 17, 1992. This cell measures 374 feet by 356 feet, with a capacity of 363,000 cubic yards, 402,500 cubic yards with GCL closure.

In ascending order this cell consists of a 3-foot compacted clay liner with a permeabiity of less than $10^{-7}$cm/sec., a secondary 60 mil HDPE liner, a secondary leachate detection/collection system, a primary 60 mil HDPE liner, a primary leachate detection/collection system, a non-woven geotextile fabric, a two-foot soil protective cover, a tertiary 80 mil HDPE liner, a tertiary leachate detection/collection system, a non-woven geotextile fabric, and a two-foot protective soil layer. Both of the leachate collection and detection systems drain into sumps for the removal of any leachate.

**RCRA Cell Six**

RCRA Cell Six was completed in October 1993 and has received it final operating permits. This cell measures 1370 feet by 544 feet, with a capacity of 558,600 cubic yards, 613,900 cubic yards with GCL closure.

In ascending order, this cell conists of: a 3-foot compacted clay liner with a permeability of less than $10^{-7}$ cm/sec., a secondary 60 mil HDPE liner, a secondary leachate detection/collection system, a primary 60 mil HDPE liner, a primary leachate detection/collection system, a non-woven geotextile fabric, a two-foot soil protective cover, a tertiary 80-mil HDPE liner, a tertiary leachate detection/collection system, a non-woven geotextile fabric, and a two-foot protective soil layer. Both of the leachate collection and detection systems drain into sumps for the removal of any leachate.

### 3. Industrial (non-hazardous) Waste Disposal

USPCI's industrial cells are designed and built to provide an increased level of security for waste that, although not classified as hazardous under RCRA, should be disposed of in a more secure manner than a sanitary (municipal) landfill. Wastes destined for USPCI's industrial cells are subject to the same evaluation procedure as hazardous wastes (i.e., pre-shipment samples, waste profiles, certificates, load arrival samples, and analytical data).

**Industrial Cell One (pending closure)**
This non-hazardous waste landfill cell was completed in August of 1985 and constructed as follows (ascending order): a clay liner, a 60 mil HDPE liner, a primary leachate collection system, a non-woven geotextile fabric, and a three-foot sand layer. This cell is undergoing final closure.

**Industrial Cell Two (pending closure)**
This cell is constructed as follows, in ascending order: subgrade, a secondary 60 mil HDPE liner, a secondary leachate detection/collection system, a 60 mil HDPE primary liner, a primary leachate detection/collection system, a non-woven geotextile fabric, and a two-foot soil protective cover.

Industrial Waste Cel! Two measures 306 feet by 1066 feet, with a capacity of 245,900 cubic yards.

**Industrial Cell Three**
Cell Three was constructed in 1991 and has a projected life of approximately three years. This cell is shaped in an irregular trapezoid, approximately 1642 feet long, 679 feet wide at the base, and approximately 350 feet wide at the top. It has a capacity of 706,000 cubic yards.

This cell is constructed as follows, in ascending order: subgrade, a 60 mil HDPE secondary liner, a secondary leachate detection/collection system, a 60 mil HDPE primary liner, a primary leachate detection/collection system, a non-woven geotextile fabric, and a two-foot soil protective cover.

## C. Treatment

### Stabilization

Separate stabilization facilities are maintained at this location for PCB wastes and RCRA wastes/industrial wastes. Various stabilization agents are mixed with the waste to eliminate free liquids, immobilize toxic constituents, and/or detoxify toxic constituents. Analytical tests are performed on the stabilized wastes to assure that the appropriate reactions are completed. The stabilized wastes are then placed in the designated landfill cell.

## E. Waste Analysis and Tracking System

The Waste Analysis Plan describes procedures used to obtain the chemical/physical data necessary to determine:

- Acceptance of waste material by Grassy or Grayback Mountain
- Methods of handling
- Treatment methods
- Disposal

### 1. Initial Waste Evaluation

Along with the shipment of a 2-quart representative sample[1] from the customer to the Grassy Mountain laboratory, the following will also be required:

a) USPCI Waste Profile Sheet (Exhibit No. 3).

b) Material safety data sheet pertaining to the primary constituents of the waste.

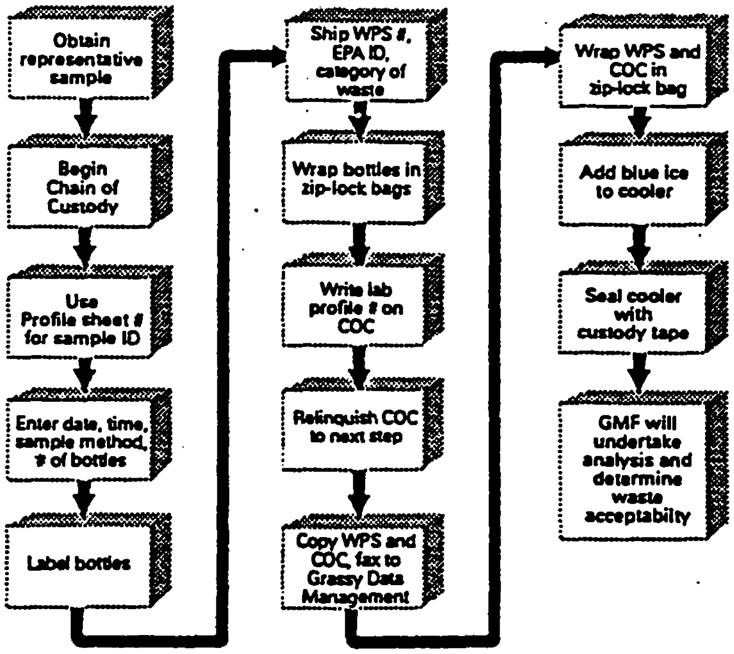
c) Any other analytical data and additional information regarding waste stream composition.

d) Notifications or certifications regarding Land Disposal Restriction (LDR) wastes.

[1] If additional analysis is required such as recipe development, Table HOC analysis and treatability studies, the flow diagram below (Flow Chart One) should be followed to ensure the waste stream is processed as efficiently as possible. Additionally, all samples received should have chain of custody.

From this information, a decision will be made by the Grassy and Grayback Mountain technical staff as to what additional analysis may be required.

Flow Chart One



## VI. TRAINING-INSPECTION-SAFETY-CONTINGENCY PLAN

### A. Employee Training Program

The Grassy/Grayback Mountain facility training program enables employees to understand the processes and materials with which they are working and the safety and health hazards associated with those processes and materials. Both RCRA (Resource Conservation and Recovery Act) and OSHA (Occupational Safety and Health Administration) 1910.120 training requirements are met by facility personnel successfully completing classroom training, on-the-job training, seminars, and/or short courses. The training takes place both on and off-site.

All new employees receive 24 hours of classroom training including: company overview, preparedness and prevention, contingency plan, regulatory review, chemistry of hazardous materials, DOT placarding and hazardous materials recognition, principles of safety, confined space entry, instrumentation, waste identification and segregation, industrial hygiene, toxicology, respiratory protection, personal protective equipment, decontamination, and emergency response. In addition, specific training is given to each employee with regard to his/her job duties. Training is maintained on an annual basis, with a minimum of eight hours per person per year.

All employees learn appropriate procedures for emergency response. In addition, Spill Response, Fire Response, and Medical First Responder groups with approximately 12 members each initially receive at least 40 hours of specialized training. These employees will respond to all emergencies and hazardous materials releases as directed by the site emergency response coordinator designated by the facility contingency plan.

Each employee takes several written tests throughout the training process. The tests, along with attendance sheets, job descriptions, training requirements, training history, personal protective equipment and on-the-job certificates, are kept in employees' files and maintained by the facility training coordinator. A qualified training coordinator insures all training programs are properly administered and recorded at the Grassy/Grayback Mountain facility.

### B. Safety

The Grassy/Grayback Mountain facility conducts a complete program in health surveillance which includes:

- Pre-employment physicals
- Employment physicals upon hiring (Protocol I)
- Exit physicals upon termination

**Protocol I**

The health surveillance program includes:

- Medical health history and general physical exam
- Stress exercises, EKG

- Emergency oxygen
- Spill clean-up and containment material
- Heavy equipment
- Sprinkler protection for the drum dock, fire hydrants for the site (five)
- Spill response trailer

The Grassy/Grayback Mountain facility employs an emergency diesel-driven generator which can supply power for emergency lights and critical pumping equipment in the event of a power outage.

## D. Inspections

Inspections are conducted daily and recorded on appropriate checklists. Any exceptions to standard conditions are recorded and repaired on a schedule which ensures protection of human health and the environment.

The facility inspection plan is meant to address the types of problems which are inherent to the materials the Grassy/Grayback Mountain facility handles and the equipment and structures used in the hazardous waste disposal process. Equipment and structures are generally classified as follows:

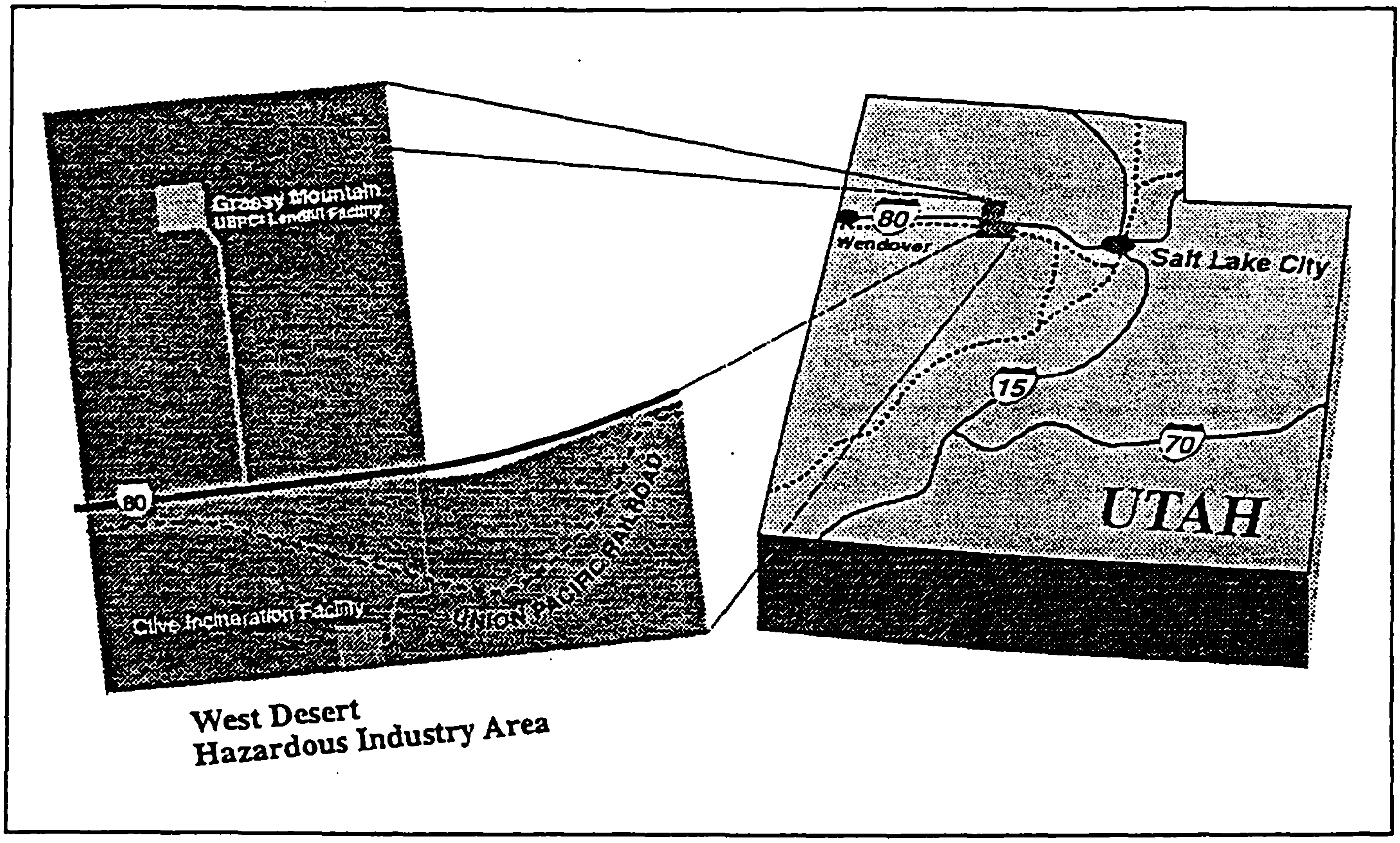
- Safety equipment
- Emergency equipment
- Fire protection equipment
- Security devices
- Heavy equipment
- Treatment facilities
- Housekeeping
- Leachate detection
- Storage tanks

Frequency of inspection is based on expected deterioration rates and the realistic probabilities that any equipment malfunction or failure could impact human health or the environment. Areas subject to spills, i.e., loading/off-loading facilities, are inspected daily. Containers are inspected when off-loaded and daily while in storage.

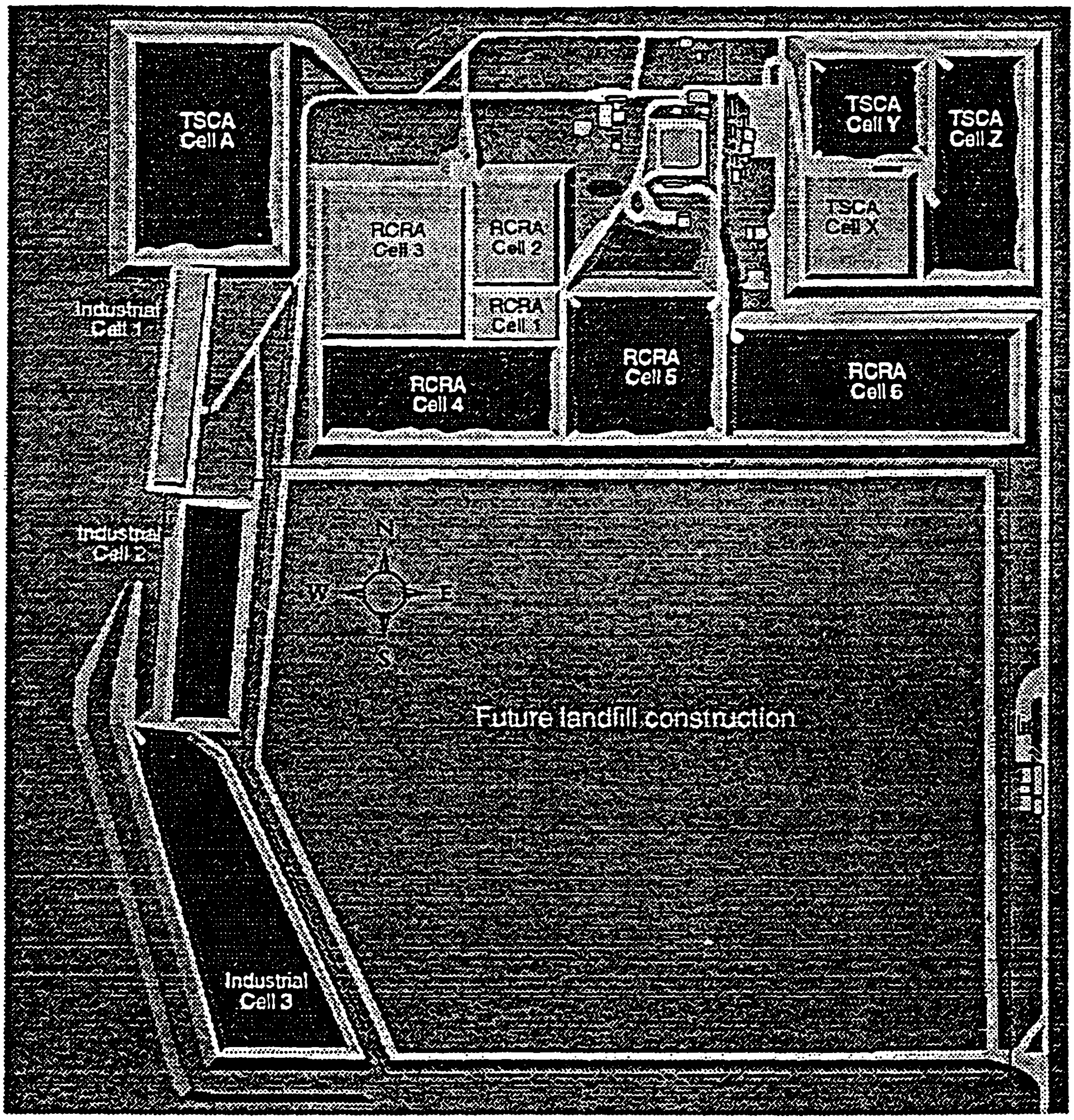
## E. Contingency Plan

The Grassy/Grayback Mountain contingency plan has been developed in accordance with 40 CFR 264, Subpart D, and describes the actions facility personnel will take in response to fires, explosions, or any unplanned sudden or non-sudden release of hazardous waste or constituents to the environment. The plan identifies five emergency coordinators and describes their responsibilities with respect to initiating and implementing the plan. The emergency coordinators designated in the plan are authorized to commit the resources necessary to ensure successful, expedient implementation.

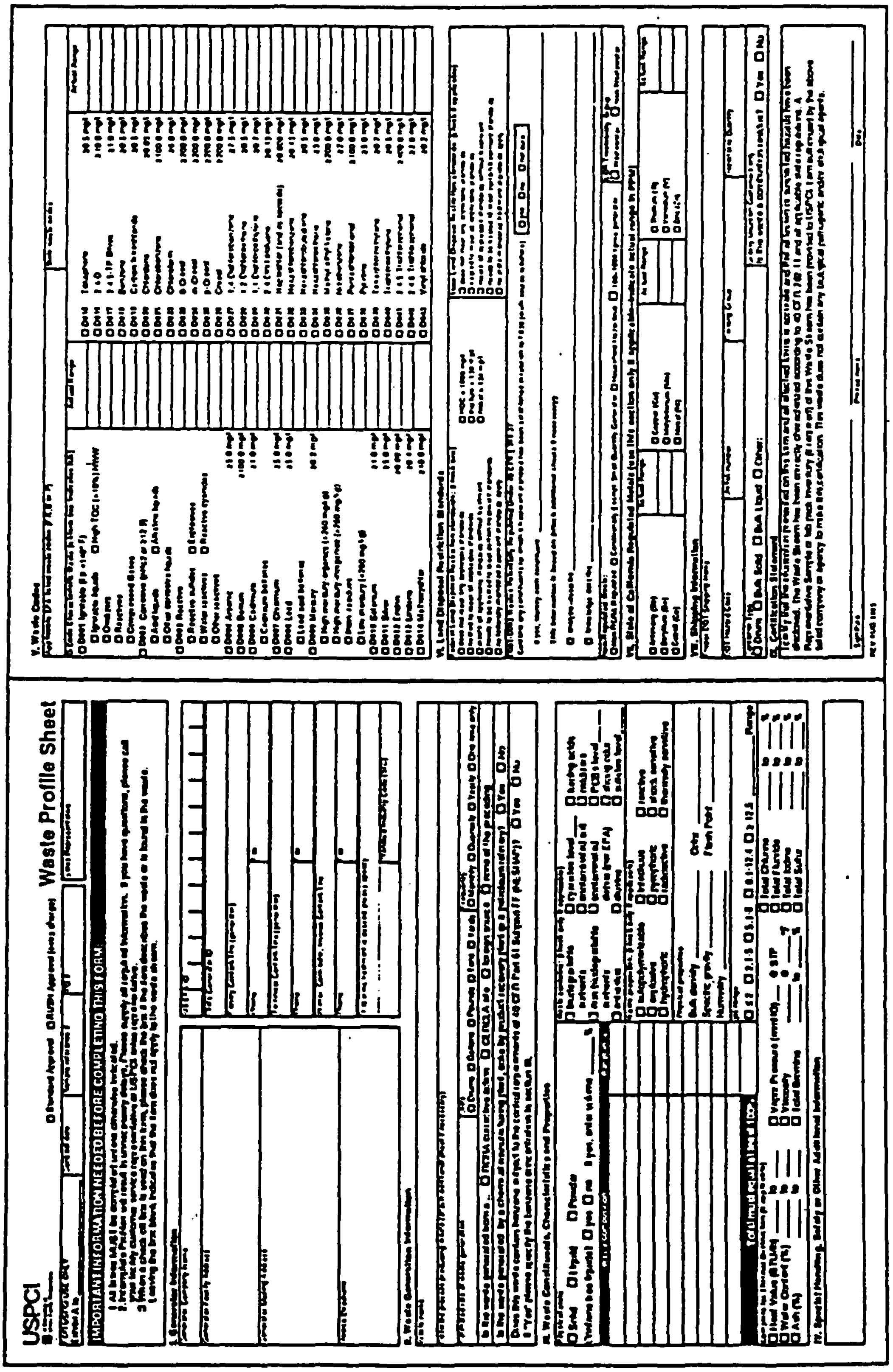
## EXHIBIT 1: FACILITY LOCATION MAP



## EXHIBIT 2: FACILITY SITE PLAN



## EXHIBIT 3: WASTE PROFILE SHEET

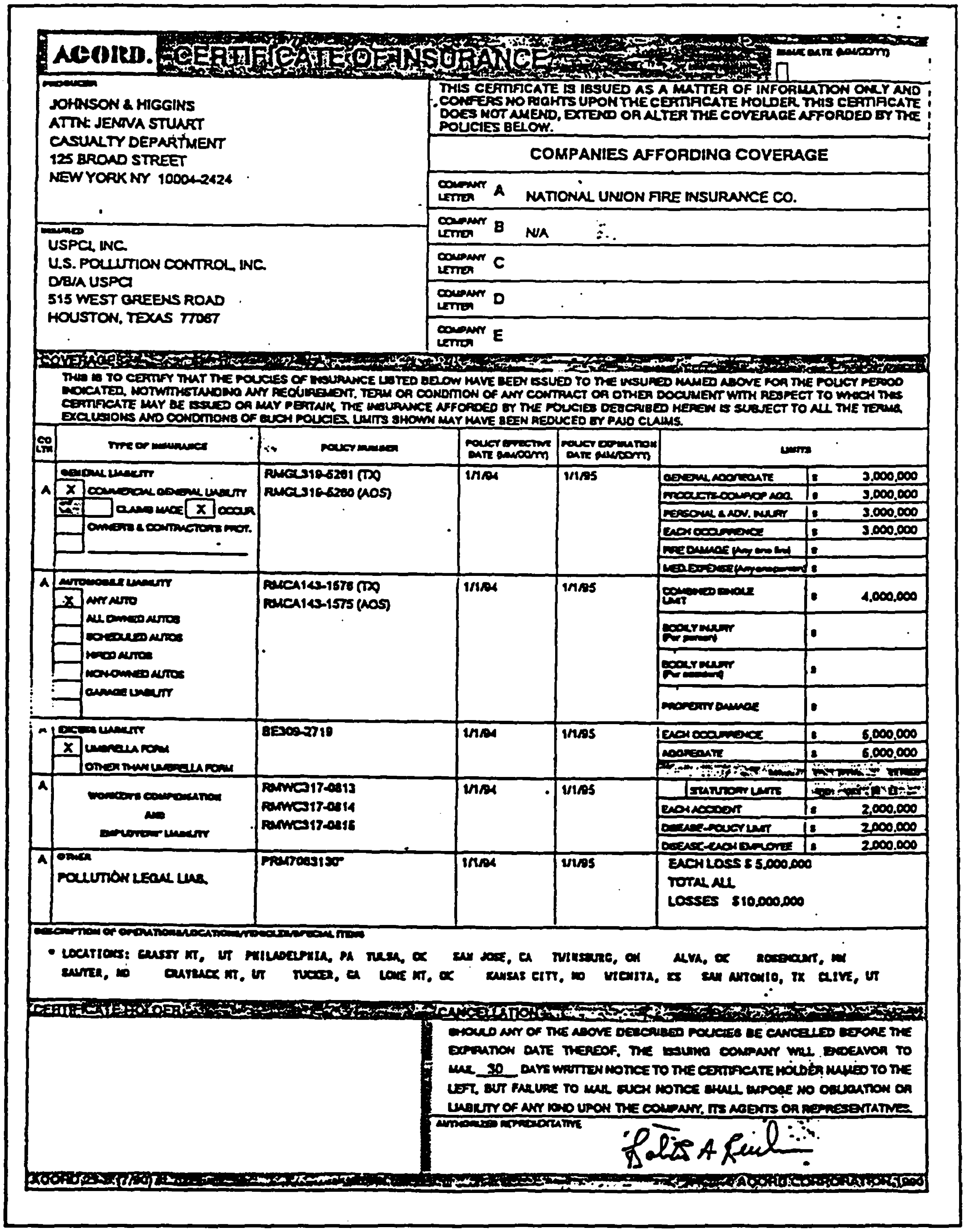


USPCI

Waste Profile Sheet

IMPORTANT INFORMATION: READ BEFORE COMPLETING THIS FORM

# EXHIBIT 4: CERTIFICATE OF INSURANCE



**ACORD. CERTIFICATE OF INSURANCE**

ISSUE DATE (MM/DD/YY)

PRODUCER

JOHNSON & HIGGINS
ATTN: JENIVA STUART
CASUALTY DEPARTMENT
125 BROAD STREET
NEW YORK NY 10004-2424

THIS CERTIFICATE IS ISSUED AS A MATTER OF INFORMATION ONLY AND CONFERS NO RIGHTS UPON THE CERTIFICATE HOLDER. THIS CERTIFICATE DOES NOT AMEND, EXTEND OR ALTER THE COVERAGE AFFORDED BY THE POLICIES BELOW.

COMPANIES AFFORDING COVERAGE

| | |
|---|---|
| COMPANY LETTER A | NATIONAL UNION FIRE INSURANCE CO. |
| COMPANY LETTER B | N/A |
| COMPANY LETTER C | |
| COMPANY LETTER D | |
| COMPANY LETTER E | |

INSURED

USPCI, INC.
U.S. POLLUTION CONTROL, INC.
D/B/A USPCI
515 WEST GREENS ROAD
HOUSTON, TEXAS 77067

COVERAGES

THIS IS TO CERTIFY THAT THE POLICIES OF INSURANCE LISTED BELOW HAVE BEEN ISSUED TO THE INSURED NAMED ABOVE FOR THE POLICY PERIOD INDICATED, NOTWITHSTANDING ANY REQUIREMENT, TERM OR CONDITION OF ANY CONTRACT OR OTHER DOCUMENT WITH RESPECT TO WHICH THIS CERTIFICATE MAY BE ISSUED OR MAY PERTAIN, THE INSURANCE AFFORDED BY THE POLICIES DESCRIBED HEREIN IS SUBJECT TO ALL THE TERMS, EXCLUSIONS AND CONDITIONS OF SUCH POLICIES. LIMITS SHOWN MAY HAVE BEEN REDUCED BY PAID CLAIMS.

| CO LTR | TYPE OF INSURANCE | POLICY NUMBER | POLICY EFFECTIVE DATE (MM/DD/YY) | POLICY EXPIRATION DATE (MM/DD/YY) | LIMITS | |
|---|---|---|---|---|---|---|
| A | GENERAL LIABILITY<br>☒ COMMERCIAL GENERAL LIABILITY<br>☐ CLAIMS MADE ☒ OCCUR.<br>☐ OWNERS & CONTRACTORS PROT.<br>☐ | RMGL319-5281 (TX)<br>RMGL319-5280 (AOS) | 1/1/94 | 1/1/95 | GENERAL AGGREGATE | $ 3,000,000 |
| | | | | | PRODUCTS-COMP/OP AGG. | $ 3,000,000 |
| | | | | | PERSONAL & ADV. INJURY | $ 3,000,000 |
| | | | | | EACH OCCURRENCE | $ 3,000,000 |
| | | | | | FIRE DAMAGE (Any one fire) | $ |
| | | | | | MED. EXPENSE (Any one person) | $ |
| A | AUTOMOBILE LIABILITY<br>☒ ANY AUTO<br>☐ ALL OWNED AUTOS<br>☐ SCHEDULED AUTOS<br>☐ HIRED AUTOS<br>☐ NON-OWNED AUTOS<br>☐ GARAGE LIABILITY<br>☐ | RMCA143-1576 (TX)<br>RMCA143-1575 (AOS) | 1/1/94 | 1/1/95 | COMBINED SINGLE LIMIT | $ 4,000,000 |
| | | | | | BODILY INJURY (Per person) | $ |
| | | | | | BODILY INJURY (Per accident) | $ |
| | | | | | PROPERTY DAMAGE | $ |
| A | EXCESS LIABILITY<br>☒ UMBRELLA FORM<br>☐ OTHER THAN UMBRELLA FORM | BE309-2719 | 1/1/94 | 1/1/95 | EACH OCCURRENCE | $ 5,000,000 |
| | | | | | AGGREGATE | $ 5,000,000 |
| A | WORKER'S COMPENSATION AND EMPLOYERS' LIABILITY | RMWC317-0813<br>RMWC317-0814<br>RMWC317-0815 | 1/1/94 | 1/1/95 | ☐ STATUTORY LIMITS | |
| | | | | | EACH ACCIDENT | $ 2,000,000 |
| | | | | | DISEASE-POLICY LIMIT | $ 2,000,000 |
| | | | | | DISEASE-EACH EMPLOYEE | $ 2,000,000 |
| A | OTHER<br>POLLUTION LEGAL LIAB. | PRM7083130* | 1/1/94 | 1/1/95 | EACH LOSS $ 5,000,000<br>TOTAL ALL LOSSES $10,000,000 | |

DESCRIPTION OF OPERATIONS/LOCATIONS/VEHICLES/SPECIAL ITEMS

* LOCATIONS: GRASSY MT, UT PHILADELPHIA, PA TULSA, OK SAN JOSE, CA TWINSBURG, OH ALVA, OK ROSEMOUNT, MN SAWYER, ND GRAYBACK MT, UT TUCKER, GA LONE MT, OK KANSAS CITY, MO WICHITA, KS SAN ANTONIO, TX CLIVE, UT

CERTIFICATE HOLDER

CANCELLATION

SHOULD ANY OF THE ABOVE DESCRIBED POLICIES BE CANCELLED BEFORE THE EXPIRATION DATE THEREOF, THE ISSUING COMPANY WILL ENDEAVOR TO MAIL 30 DAYS WRITTEN NOTICE TO THE CERTIFICATE HOLDER NAMED TO THE LEFT, BUT FAILURE TO MAIL SUCH NOTICE SHALL IMPOSE NO OBLIGATION OR LIABILITY OF ANY KIND UPON THE COMPANY, ITS AGENTS OR REPRESENTATIVES.

AUTHORIZED REPRESENTATIVE

ACORD 25-S (7/90)

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# APPENDIX C

# LES GRASSY MOUNTAIN FACILITY LABORATORY CERTIFICATION

Utah Department of Health

DIVISION OF LABORATORY SERVICES

# State of Utah

Michael O. Leavitt
Governor
Rod L. Betit
Executive Director
Charles D. Brokopp, Dr. P.H.
Director

46 North Medical Drive
Salt Lake City, Utah 84113-1105
Telephone: (801) 584-8400
Fax: (801) 584-8486

JUN 28 1995

ADAM GANZ
USPCI-GRASSY MOUNTAIN FACILITY
P.O. BOX 22750
SLC, UT 84122

Certificate No.: E-112
Account No: 8015953637

On the basis of your most recent audit results, the laboratory listed is hereby certified for environmental monitoring under the Resource Conservation and Recovery Act and authorized to perform the following analytes, or groups of analytes by method:

METALS
- ANTIMONY 6010A
- * ARSENIC 6010A
- * ARSENIC 7060
- BARIUM 6010A
- BERYLLIUM 6010A
- CADMIUM 6010A
- CHROMIUM 6010A
- COPPER 6010A
- LEAD 6010A
- LEAD 7421
- MERCURY 7470
- NICKEL 6010A
- SELENIUM 6010A
- SELENIUM 7740
- SILVER 6010A
- THALLIUM 6010A
- THALLIUM 7841
- VANADIUM 6010A
- ZINC 6010A

MISCELLANEOUS
- CYANIDE TOTAL/AMENABLE 9010
- PAINT FILTER LIQUID TST 9095
- PH 9040
- PH 9041
- PH 9045
- SULFIDES 9030
- * TCLP METAL 1311

* Provisional Certification

The expiration date for this laboratory's certification is MAY 31 1996.

Page Two

The analytes for which a laboratory is authorized to perform at any given time will be those indicated in the most recent certificate letter. Please review for completeness and accuracy. Any discrepancies must be documented and notice received by this bureau within 15 days of receipt. Copies of this letter will be on file in (1) the Bureau of Laboratory Improvement, Division of Laboratory Services and (2) in the Department of Environmental Quality. The certification will be recalled in the event that your Laboratory's certification is revoked.

Respectfully,

Charles Brokopp, Dr. P.H.
Director

cc. Richard Denton - DEQ (cc. electronic)
Dennis Downs - DEQ (cc. electronic)
U.S. EPA Region VIII QAO
Management Services Coordinator (cc. electronic)

# APPENDIX D

# LES GRASSY MOUNTAIN FACILITY RADIOACTIVE WASTE ACCEPTANCE LIMIT

LAIDLAW
ENVIRONMENTAL
SERVICES

August 16, 1995

John Bosek
Laidlaw Environmental Services
5303 126th Ave. North
Clearwater, FL. 34620

Mr. Bosek

Don Hensch asked me to provide to you a copy of the Grassy Mountain Facility Quality Assurance Plan and a copy of the section of the facility's Waste Analysis Plan which prohibits us from accepting waste with a radioactive reading of $\geq$ 40 uR / hr.

If you require additional information, please feel free to contact me at (801) 595-3946.

Sincerely

Robert E. Waite
QA Coordinator
USPCI/Laidlaw Grassy Mountain Facility

USPCI
P.O. Box 22750 Salt Lake City, Utah 84122
Phone 801.595.3900 Fax 801-595-3990

# APPENDIX E

# LES GRASSY MOUNTAIN FACILITY WASTE ANALYSIS PLAN

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# WASTE ANALYSIS PLAN

## 1.0 INTRODUCTION

As required at 40 CFR Section 264.13, before an owner or operator treats, stores, or disposes of any hazardous waste, he must obtain a detailed chemical and physical analysis of a representative sample of the waste. The analysis must contain all the information which must be known to treat, store, or dispose of the waste in accordance with 40 CFR Parts 264 and 268 and the permit issued. The analysis may include data developed under 40 CFR Part 261, 40 CFR Section 262.11 (including applying knowledge of the hazard characteristic of the waste in light of the materials or the processes used), and existing published or documented data on the hazardous waste or on hazardous waste generated from similar processes.

U.S. Pollution Control, Inc (USPCI) has established the following procedures to govern the acceptance of all hazardous waste at it's Grassy Mountain Facility. USPCI developed this Waste Analysis Plan (WAP) as a requirement of the Part B Permit issued June 30, 1988 for its Grassy Mountain Facility located near Knolls, Utah. The procedures established in this WAP will assure that this facility will be in compliance with all the requirements of 40 CFR 264.13, including the land disposal restrictions in 40 CFR Part 268.

The most recent revision of this plan will be maintained at the facility as a part of the operating record.

The purpose of a waste analysis plan is to establish necessary sampling methodologies, analytical techniques and overall procedures which should be undertaken for hazardous wastes (here-in-after referred to as "waste") which enter the Grassy Mountain Facility for treatment, storage, or disposal. The WAP establishes the following:

- The parameters for which each hazardous waste will be analyzed and the rationale for the selection of these parameters (i.e.; how analysis for these parameters will provide sufficient information on the properties of the waste **(See Section 2.0)**;
- The test methods which will be used to test for these parameters **(See Section 3.0 and 10.0)**;
- The sampling method which will be used to obtain a representative sample of the waste to be analyzed **(See Section 4.0)**;
- The frequency with which the initial analysis of the waste will be reviewed or repeated to assure that the analysis is accurate and up to date **(See Section 5.0)**;

- The waste analyses that hazardous waste generators have agreed to supply (**See Section 5.0**);
- The methods which will be used to meet the additional waste analysis requirements for specific waste management methods as specified in 40 CFR Sections 264.17, 264.314, 264.341, and 268.7 (**See Section 7.0**).
- For surface impoundments exempted from land disposal restrictions under 40 CFR 268.4(a), the procedures and schedules for:
  - (i) the sampling of impoundment contents (**na**);
  - (ii) the analysis of test data (**na**); and
  - (iii) the annual removal of residue which does not meet the standards of 40 CFR Part 268 Subpart D (**na**).
- The procedures which will be used to inspect and, if necessary, analyze each movement of hazardous waste received at the facility to assure that it matches the identity of the waste designated on the accompanying manifest or shipping paper, including:

(i) the procedures which will be used to determine the identity of each movement of waste managed at the facility (**See Section 2.1**); and

(ii) the sampling method which will be used to obtain a representative sample of the waste to be identified (**See Section 4.0**).

## 2.0 ANALYTICAL PARAMETERS AND RATIONALE

Analyses are provided by the laboratory to augment or verify pre-existing waste identifications, to comply with facility acceptance criteria and to assure compliance with land disposal restrictions. Analytical methods are classified as "Fingerprint Analyses" and "Additional Analyses." At a minimum, all wastes are subjected to the "Fingerprint Analyses" 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 General Manager may select these additional analyses to augment the mandatory screening or to provide operational control. This arrangement allows a tiered approach to waste identification, enabling USPCI to structure the analyses to adequately identify the waste or to define operational parameters for various treatment processes. The "Fingerprint Analyses" and "Unique Additional Analyses" have been developed to provide needed operational indications of the characteristics of a waste. The rationale and the procedures for these tests are given in Sections 2.0 and 3.0, respectively. Other "Additional Analyses" described in this section are accepted standard techniques, and are found in the references cited in Section 3.0.

A summary of the analytical parameters within each category and their usage is provided herein. Analyses are not necessarily repeated for sequential activities or movement of the same waste within the facility unless required by changes in the character of the waste, in order to document compliance with the permit, or in order to establish compliance with the land disposal restrictions.

## 2.1 FINGERPRINT ANALYSES

Fingerprint Analyses include eleven basic screening procedures that are performed to provide a general characterization of the waste, and are 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 waste movements. This procedure serves as a check that the waste shipped to the facility actually matches the waste originally profiled by the generator to further ensure that a change in waste type has not occurred.

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

1. **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 standing liquids.
2. **pH Screen** is undertaken to indicate the corrosive nature of the waste and indicate if the material might be a California List waste. pH may not apply to certain wastes (e.g., organic solvent waste, oil waste, or solid waste).
3. **Specific Gravity** is important in determining the sedimentation rate or buoyance of wastes in suspension, as well as treatability in other process units.

4. **Reactive Cyanides Screen** indicates whether the waste produces hydrogen cyanide (and might be a California List Waste) 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.
5. **Reactive Sulfides Screen** indicates whether the 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. Waste containing total releasable sulfide with concentrations less than 500 ppm are considered non-reactive.
6. **Water Reactivity Screen** is used to determine whether the waste has a potential to vigorously react with water to form significant gases or solids, or whether it generates significant

heat. This test is meant for gross characterization of the waste only. This test does not apply to wastes which are already in contact with excess water, or for which sufficient data exist that indicate no potential reactivity with water.

7. **Solids Screen** is selected to determine solids management requirements.
8. **Explosivity Meter Vapor Test (TLV Sniff)** is used to indicate the fire-producing potential of the waste, and to indicate whether the waste might be a RCRA ignitable waste or regulated as flammable or combustible by the US DOT. This test can be applied to all waste liquids, semi-solids or solids. The screen will be supplemented with the flash point test for those liquid materials exceeding 200 ppm if they are destined for the surface impoundment, land treatment, or landfill.
9. **Radioactivity Screen** is used to screen incoming material for signs of radioactivity.

10. **Oxidizer Screen** is a rapid qualitative method for determining the presence or absence of oxidizing materials in liquid or semi-solid materials. It is also used to indicate if a material might be improperly identified (e.g., not identified as D001 material).

11. **Reducer Screen** is a rapid qualitative method for determining the presence or absence of reducing materials in liquid or semi-solid materials. It is also used to indicate if a material might be improperly identified.

## 2.2 ADDITIONAL ANALYSES

Additional Analyses are performed to further identify wastes, as appropriate, and are run if the General Manager, or his designee (e.g., the Laboratory Manager, site chemist, or...), hereinafter collectively referred to as the "General Manager," determines that further waste characterization is necessary or if required in Section 7.0. The results of these analyses provide the General Manager with another level of confidence concerning the proper means of treatment, storage and disposal. Some of these additional analyses utilize unique procedures and protocols formulated particularly for the management of hazardous waste, which are preferable for waste characterization. Others are standard analytical techniques recognized by the US EPA and ASTM.

### 2.2.1. UNIQUE ADDITIONAL ANALYSES

Unique Additional Analyses are derived procedures which were developed to provide operational control over on-site processes. Again, these analyses are performed to further identify wastes and are run if the General Manager determines that further waste characterization is necessary or if required in Section 7.0 (Process Operations Procedures).

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

- **Fixation Requirements** is run to determine the ratio of reaction reagent(s)-to-waste (often referred to as the "recipe") required to effect stabilization.

- **Cyanides - Peroxide Amenability** determines the effectiveness of $H_2O_2$ for cyanide treatment.
- **Cyanides - Chlorination Amenability** (Sodium Hypochlorite or direct Chlorination) is run to determine the effectiveness of hypochlorite for cyanide treatment.
- **Cyanides - Conversion Amenability** is tested to determine the effectiveness of other types of reagents treatment for cyanides.
- **Liquid Waste Compatibility** is tested to determine whether liquid wastes stored or processed together are compatible.
- **Soluble Sulfides** are analyzed to provide quantitative backup to the reactive sulfides screen.
- **Sulfate Screen** is to indicate sulfate presence, since a waste with high dissolved sulfates will have a tendency to precipitate.
- **Sulfide - Peroxide Amenability** determines the effectiveness of $H_2O_2$ for sulfide treatment.

- **Distillation** is used to determine the percent recovery and boiling range of a sample, and to generate a distillate of the material suitable for further testing.
- **Ignitability Test Method For Solids** is used to indicate the fire-producing potential of a sample and is used as a backup or substitute test for the TLV Sniff. This test can be applied to all solid waste samples.
- **Normality** is used to define the amount of reagent or waste required to approach a neutral pH. The test is similar to the percent acidity and percent alkalinity tests, but the end point, to simulate operational practices, is at (or near) a pH of 7.
- **Methanol Extraction** is used to generate an extract for VOA analyses.

### 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 General Manager determines that further waste characterization is necessary or if required in Section 7.0 (Process Operations Procedures).

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

- **Hexavalent Chromium** is to quantify the concentration of this species for treatment control and to indicate if a waste might be a California List waste.
- **Heavy Metals** (As, Ba, Cd, Cr, Pb, Hg, Se, Ag) are run to quantify heavy metals concentration to determine process operating parameters and to indicate if a waste might be a California List waste.
- **Miscellaneous Metals** (Cu, Fe, Ca, Mg, Mn, Ni, Va, Zn) determine the potential for salt precipitation and is used for monitoring certain processes and to indicate if a waste might be a California List waste (in the case of Ni, only).
- **PCBs** are run to indicate whether PCBs are present in oil-bearing wastes, to ascertain their concentration, and to indicate if a waste might be a California List waste or a TSCA waste. (Note: This test uses the

"standard" Gas Chromatographic Method noted in Section 3.2.2.)

- **pH** provides a more precise measurement of pH and an indication of corrosivity when determining process parameters.
- **Sulfates** determines if the major acid component is sulfuric acid or its salts.
- **Phosphates** determines if the major acid component is phosphoric acid or its salts.
- **Chlorides** determines if the major acid component is hydrochloric acid or its salts.
- **Nitrates** determines if the major acid component is nitric acid or its salts.
- **Free Cyanides** measures the cyanides that would be potentially reactive under acid conditions and to indicate if a waste might be a California List Waste.
- **Total Cyanides** (Distillation with $MgCl_2$) quantifies the concentration of all free and complexed cyanides.
- **Total Sulfides** quantifies the concentration of total sulfide.
- **Oil and Grease** test is to quantify the amount of oil and grease so as not to impact certain processes.

- **Phenols** quantifies the concentration of phenols.
- **Total Residue** quantifies the suspended and dissolved solids present and moisture content for selected processes.
- **Filterable Residue** quantifies the suspended solids present to determine if a liquid waste contains filterable solids and if it might be a wastewater for BDAT purposes (e.g.; for the First Third List).
- **Non-Filterable Residue** quantifies the dissolved solids present to determine acceptability for certain processes.
- **Specific Organic Compounds** indicates the concentrations of specific organics and can be used to indicate if a waste might be a land disposal restricted waste.
- **Flash Point** further characterizes potentially ignitable wastes to establish proper management methods and to assure conformance with permit conditions. A closed cup is used for liquids, a Pensky-Martens Closed Cup Tester for solids.

- **Viscosity** determines the pumpability of the waste.
- **Elemental Analyses** (including Cl) determine potential acid gas generation and incineration parameters.
- **Water Content** is to determine the amount of free water or indicate the combustibility of the waste.
- **Heat Value (BTU)** assesses organic wastes suitable as a fuel supplement.
- **Percent Ash** is used to estimate particulate generation, and inorganic solid residue for incineration or supplemental fuel systems control.
- **Priority Pollutant Scan** checks for the presence of EPA priority pollutants (e.g.; volatile organics, acid extracts, and base/neutral extracts).
- **IR Scan** may be run to provide a fingerprint spectrum of organic wastes.
- **Paint Filter Liquids Test** is used to determine if solid materials contain free liquids. Materials containing free liquids must be solidified or have the free liquids otherwise removed prior to landfilling, except as provided by regulation (e.g.; labpacks, very small containers, etc...). Materials

not destined for landfilling (i.e.; surface impoundment, tanks, land treatment), or are known to contain free liquids by visual examination do not require this test and may be noted as failing the test. The test is also used in indicating if a waste might be a California List waste.

* **Toxicity Characteristics Leaching Procedure (TCLP)** is used to determine the constituent concentration in waste extract (CCWE) levels. It is also used to develop leachates from some other waste samples to indicate if the material may potentially be a land disposal restricted material (e.g.; First Third List).
* **Total Organic Halides (TOX)** is run to determine if a waste stream may contain Halogenated Organic Compounds (HOC's) and could be a California List waste, if a waste stream may be unacceptable for landfilling, or if a waste stream may be a candidate for fuels blending or incineration.

- **Total Organic Carbon (TOC)** is run to indicate if a waste may contain CCWE restricted wastes or if a waste may be a wastewater for BDAT purposes (e.g.; First Third List) it may also serve as a surrogate for other analyses (i.e.; TOX, Solvent Scan, etc...). For example, where a TOX analysis is required to be less than a certain value, a TOC analysis below that value may be utilized since the organic carbon analysis is a more conservative value.
- **Alkalinity** determines the acidity in the waste to an endpoint of pH 3.7.
- **Acidity** determines the alkalinity in the waste to and endpoint of pH 8.3.
- **Reactivity of Explosively Contaminated Soil and Sediment (Explosivity Test)** is run to determine whether a contaminated soil or sediment is classified as reactive due to its explosive properties.
- **Total Releasable (Reactive) Cyanides** is run to determine whether a waste is reactive due to releasable cyanides above 250 mg/l.

* **Total Releasable (Reactive) Sulfides** is run to determine whether a waste is reactive due to releasable sulfides above 500 mg/l.

## 3.0 ANALYTICAL PROCEDURES

Analytical methods described herein are grouped in accordance with the three categories identified in Section 2.0. It should be noted that the information presented in this section is generic in character and, therefore, certain methods are discussed which may pertain to several management techniques or even to management processes that are excluded from the facility for which this Waste Analysis Plan is presented. This is necessitated because of the nature of the hazardous waste management business. As the land disposal restrictions change the way management facilities do business, it is often necessary to have additional analyses performed to assure the waste shipped on-site or off-site are acceptable for the proposed management method (e.g., does incinerator ash meet applicable treatment standards, if any). Unforeseen restrictions may occur which require analyses not necessarily required for acceptance at this facility alone. In order to provide the methods for analyses which are currently anticipated, those analytical methods are also included. As new analytical procedures are required (e.g.;

due to HSWA requirements,...) they will be adopted and this WAP will be updated appropriately.

## 3.1 FINGERPRINT ANALYSES

Fingerprint Analyses are analytical procedures designed to identify or screen specific 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 waste management in addition to ensuring 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)
* Layering (single phased, bi-layered, multi-layered)
* 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 such that the result is a dimensionless figure (sample density [mg/ml] / 1 [mg/ml] for $H_2O$). This test is not applicable to mixed loads or loads of debris.

**Reactive Cyanides and Reactive Sulfides Screens** - To a beaker containing approximately 25 ml of waste, 3 N nitric 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.

**Water Reactivity Screen** - Water is mixed in a sample of waste, approximately 2 grams, in a 10:1 ratio of water to waste. If the waste is a solid or oily, it is added to about 20 ml of water under cover of a hood. For liquid wastes, water is added to the waste. If the waste is water-reactive, generation of gases, heat, or turbulence is recorded. If the reaction is questionable, the amount of sample is scaled up with 10 times the amount of water and retested.

**Solids Screen** - Approximately 8 grams of waste are added to a moisture determination balance set at approximately 105°C for 10 minutes. The percent solids is determined comparing the initial weight to the weight after 10 minutes of drying.

**Explosivity Meter Vapor Test (TLV Sniff)** - The TLV probe is held over the surface of the sample. A reading over 200 ppm indicates the possibility of flammability.

**Radioactivity Screen** - All incoming waste loads will be monitored for radioactivity using a micro R meter. All fingerprint analyses samples will be placed within 6 inches of the detector until a steady time weighted average reading is obtained. Any waste load found to have a reading of 40 micro R/hr or greater shall not be accepted without receiving authorization for acceptance from the Utah Bureau of Radiation Control. Results are to be recorded as positive ($\geq$ 40 $\mu$R/hr) or negative (< 40 $\mu$R/hr).

**Oxidizer Screen** - Wet a strip of KI-starch paper in HCL. Dip the wetted strip into the sample and observe the color. A color of light brown to dark purple or black indicates the potential presence of oxidizing material and is noted as a positive test result.

**Reducer Screen** - a small amount of sample is placed in a test tube containing I2 starch solution, if a reducer is present, the blue color of the indicator will fade.

Based on these results additional testing may be necessary. The methods to be used for treatment, storage, and disposal are determined during the preacceptance 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 waste stream where an adequate standard technique could not be found) and Additional Analyses Using Standard Techniques. The unique techniques are summarized in this section and the full procedure is provided in Section 10. Additional Analyses using standard techniques are summarized and the standard reference is provided.

### 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.

**Normality** - Gently stir with a calibrated and rinsed pH probe approximately 10 ml of waste in a beaker. Typically, for use on samples with a pH < 4.5 or a pH > 10.5, titrate slowly with continuous stirring with 3 N base or acid, respectively. The volume of titrant used is read and recorded.

**Fixation Requirement** - To one part of waste, candidate reagent (e.g., fly ash, cement kiln dust, cement, lime, activated carbon, water) is added. Occurrence of unacceptable reactions, such as the generation of considerable heat or vapors, is noted. Reagent is added until the sample passes the Paint Filter Liquids Test. The final ratio of reagent to sample is recorded. Recipes developed to meet treatment standards may vary as appropriate.

**Cyanide - Peroxide Amenability** - Adjust pH of sample to 9-10. Slowly add a stoichiometric amount of $H_2O_2$, keeping the temperature below 50°C. Check for excess $H_2O_2$ with indicator strips. Add $H_2O_2$ until excess remains for 30 minutes. Quench the excess with the enzyme catalase. The standard cyanide analysis is then performed.

**Cyanide - Chlorination Amenability** - To approximately 100 ml of cyanide waste, add at least a stoichiometric amount of hypochlorite, solution while stirring. Check for chlorine excess immediately after hypochlorite addition and every 10 minutes for one hour, maintaining pH in the range 11-12. Add additional hypochlorite if excess disappears, until an excess is maintained for one hour. A running total of hypochlorite additions is kept. After a chlorine excess has been maintained for one hour, add 50-100 mg $Na_2S_2O_3$ to remove the chlorine. Perform a standard cyanide hypochlorite solution addition.

**Cyanide - Conversion Amenability** - To 100 g of waste in a 250 ml beaker, add 2 ml of 10 N NaOH and 5.0 g reagent. If the waste is a solid or a heavy sludge, add 100 ml water, or more, if the sample is difficult to stir. Cover with a 10 cm watchglass and heat to boiling, agitating the sample with a magnetic stirrer. Boil 30 minutes, remove from heat, transfer liquid portion of sample and at least 3 washings to a 1 liter volumetric flask, and dilute to the mark. Shake the 1 liter flask a few times to ensure proper mixing of contents, pour 100 ml into a 250 ml beaker, and drop-wise add 10% $Cd(NO_3)_3\ 4H_2O$ until precipitation ceases. Using a dropper, add 50% $HNO_3$ until the pH is in the range of 5 to 7. Filter the sample through filter paper, and transfer 1 ml of the filtrate to a 50-ml volumetric flask. Add 5 ml $Fe(NO_3)_3$ solution, and dilute to 50 ml. Compare the solution to standards visually, or use a spectrophotometer at 480nm (4800A). If a spectrophotometer is used, prepare a blank containing 5 ml $Fe(NO_3)_3$ solution diluted to 50 ml with distilled water.

**Liquid Waste Compatibility** - Samples of liquid wastes are added to each other in approximate proportion to their final mixed volumes. Upon mixing, the generation of heat, gases, precipitates, increase in viscosity, and layering are noted.

**Soluble Sulfides** - An approximate 5 gram waste sample is diluted to 100 ml with distilled water. The solution/slurry is filtered by suction-filtration through filter paper. The resultant filtrate is then analyzed for sulfide using the Antimony Potassium Tartrate Test (Method 427) as described in "Standard Methods."

**Sulfate Screen** - To 50 ml of sample, hydrochloric acid is added to adjust pH to below pH 2. Then at least 1 ml of 10% $BaCl_2$ solution is added. Formation of any precipitate is noted.

**Sulfide - Peroxide Amenability** - Adjust pH of sample to 8-9 while stirring. Slowly add a stoichiometric amount of $H_2O_2$, keeping temperature below 50°C. Check for excess $H_2O_2$ with indicator strips. Add $H_2O_2$ until excess remains for 30 minutes. Quench the excess with the enzyme catalase. The sulfide standard analysis is then performed, with dissolved iron or copper added as catalysts.

**Distillation** - In an appropriate size stand flask/condenser distillation device, add a known quantity of sample and boiling chips. Apply heat appropriately (e.g.; with an electrically-heated oil bath or steam). During distillation, maintain heat so that a drop of liquid remains on thermometer bulb. Monitor temperature and collected volume of each fraction.

**Ignitability Test Method For Solids** - A prescribed sample volume (fill to mark in tester cup) is packed firmly in tester cup and lid secured. Place cup in "stove" and insert thermometer. Raise stove (and tested material) temperature lower than the estimated flash point. Light the test flame and adjust it to the prescribed diameter. Supply heat to tester and hold temperature for one minute before flashing sample. Record temperature and repeat until a temperature of 140°F is attained. Repeat and perform calculations.

**Methanol Extraction for Solids** - place a small sample in a methanol cap, shake, and extract with a pipet. Nine (9) ml is used for VOA analyses. Determine dry solids to correct to a dry standard basis. (USPCI Procedure 11).

### 3.2.2 ADDITIONAL ANALYSES USING STANDARD TECHNIQUES

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

| PARAMETER | METHOD | REFERENCE |
|---|---|---|
| Sample Work Up Techniques: | | |
| Inorganic Techniques | | |
| Acid digestion procedure for flame atomic absorption spectroscopy | | 1-3005 |
| Acid digestion procedure for flame atomic absorption spectroscopy | | 1-3010 |
| Acid digestion procedure for furnace atomic absorption spectroscopy | | 1-3020 |
| Acid digestion of oils, greases, or waxes | | 1-3030 |
| Dissolution procedure for oils, greases, wax | | 1-3040 |
| Acid digestion of sludges | | 1-3050 |
| Alkaline digestion | | 1-3060 |
| Organic Techniques | | |
| Extraction Procedure for Oily Wastes | | 1-1330 |
| Organic Extraction and Sample Preparation | | 1-3500 |
| Waste Dilution | | 1-3580 |
| Separatory funnel liquid-liquid extraction | | 1-3510 |
| Continuous liquid-liquid extraction | | 1-3520 |
| Acid-base cleanup extraction | | 1-3530 |
| Soxhlet extraction | | 1-3540 |
| Sonication extraction | | 1-3550 |
| Purge and Trap | | 1-5030 |
| Hexadecane Extraction and Screening of purgeable organics | | 1-3820 |
| Inorganic analytical methods: | | |
| Antimony | | |
| | Atomic absorption, direct aspiration method | 1-7040 |
| | Atomic absorption, furnace method | 1-7041 |
| Arsenic | | |
| | Atomic absorption, furnace method | 1-7060 |
| | Atomic absorption, gaseous hydride method | 1-7061 |
| Barium | | |
| | Atomic absorption, direct aspiration method | 1-7080 |
| | Atomic absorption, furnace method | 1-7081 |
| Beryllium | | |
| | Atomic absorption, direct aspiration method | 1-7090 |
| | Atomic absorption, furnace method | 1-7091 |

| PARAMETER | METHOD | REFERENCE |
|---|---|---|
| Cadmium | | |
| | Atomic absorption, direct aspiration method | 1-7130 |
| | Atomic absorption, furnace method | 1-7131 |
| Chromium | | |
| | Atomic absorption, direct aspiration method | 1-7190 |
| | Atomic absorption, furnace method | 1-7191 |
| | Hexavalent chromium: Co-precipitation | 1-7195 |
| | Hexavalent chromium: Colorimetric | 1-7196 |
| | Hexavalent chromium: Chelation-extraction | 1-7197 |
| | Hexavalent chromium: Diff. phase polarography | 1-7198 |
| Copper | | |
| | Atomic absorption, direct aspiration method | 1-7210 |
| | Atomic absorption, furnace method | 1-7211 |
| Lead | | |
| | Atomic absorption, direct aspiration method | 1-7420 |
| | Atomic absorption, furnace method | 1-7421 |
| Mercury | | |
| | In liquid waste (manual cold-vapor technique) | 1-7470 |
| | In solid or semi-solid waste (manual cold-vapor technique | 1-7471 |
| Nickel | | |
| | Atomic absorption, direct aspiration method | 1-7520 |
| | Atomic absorption, furnace method | 1-7521 |
| Osmium | | |
| | Atomic absorption, direct aspiration method | 1-7550 |
| | Atomic absorption, furnace method | 1-7551 |
| Selenium | | |
| | Atomic absorption, furnace method | 1-7740 |
| | Atomic absorption, gaseous hydride method | 1-7741 |
| Silver | | |
| | Atomic absorption, direct aspiration method | 1-7760 |
| | Atomic absorption, furnace method | 1-7761 |
| Thallium | | |
| | Atomic absorption, direct aspiration method | 1-7840 |
| | Atomic absorption, furnace method | 1-7841 |
| Vanadium | | |
| | Atomic absorption, direct aspiration method | 1-7910 |
| | Atomic absorption, furnace method | 1-7911 |
| Zinc | | |
| | Atomic absorption, direct aspiration method | 1-7950 |
| | Atomic absorption, furnace method | 1-7951 |

| PARAMETER | METHOD | REFERENCE |
|---|---|---|
| Organic Analytical Methods: | | |
| Gas Chromatographic Methods | | |
| Halogenated Volatile Organics | | 1-8010 |
| Nonhalogenated Volatile Organics | | 1-8015 |
| Aromatic Volatile Organics | | 1-8020 |
| Acrolein, Acrylonitrile, Acetonitrile | | 1-8030 |
| Phenols | | 1-8040 |
| Phthalate Esters | | 1-8060 |
| Organochloride Pesticides and PCB's | | 1-8080 |
| Nitroaromatics and Cyclic Ketones | | 1-8090 |
| Polynuclear Aromatic Hydrocarbons | | 1-8100 |
| Chlorinated Hydrocarbons | | 1-8120 |
| Organophosphate Pesticides | | 1-8140 |
| Chlorinated Herbicides | | 1-8150 |
| Gas Chromatographic/Mass Spectroscopy Methods for | | |
| Volatile Organics | | 1-8240 |
| Semivolatile Organics: | | |
| Packed Column Technique | | 1-8250 |
| Capillary Column Technique | | 1-8270 |
| Polychlorinated Dibenzo-P-Dioxins and Polychlorinated Dibenzofurans | | 1-8280 |
| Miscellaneous Analytical Methods: | | |
| Acidity | | 2-402 |
| Alkalinity | | 2-403 |
| % Ash | | 3-(*) |
| Compatibility Test for Wastes and Membrane Liners | | 1-9090 |
| Corrosivity towards Steel | | 1-1110 |
| Total and Amenable Cyanides (Free Cyanides) | | 1-9010 |
| Total and Amenable Cyanides (Free Cyanides) | | 1-9012 |
| Test Method to Determine Hydrogen Cyanide Released from Wastes (Reactive Cyanides) | | 1-7.3.3.2 |
| Flash point (Closed cup) | | 3-D93 |
| Flash point (Open cup) | | 3-D92 |
| Heat Value (BTU) | | 3-D240 |
| Ignitability (Pensky-Martens closed-cup method) | | 1-1010 |
| Ignitability (Setaflash closed-cup method) | | 1-1020 |
| $NH_3$-Nitrogen | | 2-417 |
| Nitrate | | 1-9200 |
| Nitrate-Nitrogen | | 2-418 |
| Total Recoverable Oil & Grease | | 1-9070 |

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| PARAMETER | METHOD | REFERENCE |
|---|---|---|
| Miscellaneous Analytical Methods: | | |
| Oil & Grease Extraction Method for Sludge Samples | | 1-9071 |
| Oil & Grease | | 2-503 |
| Total Organic Carbon (TOC) | | 1-9060 |
| Total Organic Halides (TOX) | | 1-9020 |
| Paint Filter Liquids Test (Free Liquids Test) | | 1-9095 |
| pH Electrometric Measurement | | 1-9040 |
| pH Paper Method | | 1-9041 |
| Soil pH | | 1-9045 |
| Phosphorous | | 2-424 |
| Total Solids Dried at 103-105°C | | 2-209A |
| Total Suspended Solids (Filterable Residue) | | 2-209C |
| Fixed & Volatile Solids (Non-filterable Residue) | | 2-209D |
| Specific Gravity | | 2-213E |
| Sulfides | | 1-9030 |
| Test Method to Determined Hydrogen Sulfide Released from Wastes (Reactive Sulfides) | | 1-7.3.4.1 |
| TCLP | | 5-268 |
| EP Toxicity | | 5-261 |
| Viscosity | | 3-(*) |
| Water Content | | 3-(*) |
| Ignitability test method for solids | | 3-D93 |

The above referenced procedures are described in the following publications. The first digit of the reference numbers above are keyed to the numbers shown below:

1 - "Test Methods for Evaluating Solid Waste," SW-846, U.S. Environmental Protection Agency, Office of Water and Waste Management, Washington, D.C. 20406, 3Rd Ed, 1986.
2 - "Standard Methods for the Examination of Water and Waste Water," 16th edition, American Public Health Association, 1985.
3 - "Annual Book of ASTM Standards", Parts 15, 19, 31, American Society for Testing Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103
4 - "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1979.
5 - 40 Code of Federal Regulations, Parts 260-268.

Note: * This analysis is material specific.

## 4.0 SAMPLING METHODOLOGY

Sampling is performed by USPCI 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 on-site by USPCI personnel. Fingerprint sampling is performed at the USPCI - Grassy Mountain Facility, except for the special cases noted in plan or as approved by the Bureau of Solid and Hazardous Waste. In some instances (e.g., clean-up projects, remote sampling requested by the generator, etc), USPCI personnel may perform sampling off-site under these conditions. Waste generators are referred to 40 CFR 261, Appendix I for sampling procedures.

## 4.1 METHODOLOGY

Representative samples will 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. Excerpts from "Samplers and Sampling Procedures for Hazardous Waste Streams" are given for reference in Appendix I.

## 4.2 TYPES OF CONTAINMENT

In addition to the sampling procedures noted above, USPCI has instituted specific methodologies for ensuring that samples taken from various types of containers are representative. The type of container 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; such 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, several 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 waste material. For flowable materials, the sampling device of choice is either a Coliwasa unit or tubing to draw a full vertical section. For nonflowable wastes, tubing or a trier is used to obtain a representative sample.

Large containers and tanks (including railcars) for flowable materials and bulk containers (including box 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. Heavier solids are sampled by trier or shovel, or by coring with heavy tubing. 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 dimension may be limited to access ports or to sampling during unloading.

### 4.2.2 WASTE PILES

Waste accessibility, frequently a function of pile size, is a key factor in the sampling strategy for a waste pile. Piles are sampled by multiple vertical sections using triers, tubing, shovels, or similar devices. Large piles may be sampled with heavy tubing, soil augers, or through the use of excavation equipment such as a backhoe.

In cases where size impedes access to the center or bottom of a waste pile, a set of samples that is generally representative of the entire pile can be obtained by scheduling sampling to coincide with emplacement or removal.

### 4.2.3 SURFACE IMPOUNDMENTS, PITS, PONDS, AND LAGOONS

The representativeness of the samples of the waste in a surface impoundment is dependent on the number of samples collected over the volume of the waste. Additionally, for large surface area impoundments, horizontally displaced sampling may be necessary. Generally, a minimum of three samples are collected, one each from the bottom, the middle, and the top of the impoundment. These samples are composited, if necessary. A weighted bottle or bomb sampler is generally used for this sampling.

## 4.3 PROCESS SAMPLING

The variability of the waster stream at any point in a treatment process is first determined from knowledge of the process producing the stream, or from the results of a preliminary investigation of the waste stream. Sampling frequency is based upon sampling from appropriate in-line sampling points in the process stream, and compositing them, if necessary, for analysis. The samples can be varied in size depending on the flow rate of the stream. For solid or semi-solid streams, samples are taken using a scoop, shovel or similar device.

## 4.4 GENERAL CONSIDERATIONS

In the operation of a hazardous 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 USPCI's intention to address these issues in this forum to provide insight into the development techniques.

### 4.4.1 DISPOSITION OF SAMPLES

Samples of waste samples are disposed in the same fashion as the waste stream itself. If, for example, a waste is approved for stabilization and landfilling, it (the sample) will be stabilized (either in the lab or in the stabilization process) and landfilled. Samples received which are unacceptable for management on-site will be returned to the generator (or representative). To facilitate this process (sample disposal), samples approved for the same management processes may be consolidated in containers managed under the provisions of 40 CFR 262.34

### 4.4.2 CLEANING OF SAMPLING APPARATUS

Sampling tools will be kept clean of materials which will interfere with future analyses. Those analyses being conducted for gross qualification will be kept free of loose material which could 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.

### 4.4.3 FROZEN SAMPLES

Samples of frozen loads are maintained in the lab until the sample is greater than 5°C (Note: to speed up fingerprinting, samples may be heated under the vent hood). In some cases, it may be required to allow entire loads or, for drum 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 waste being delivered may be taken at the point of generation for the purpose of satisfying the requirements of this plan. Such samples will be taken either from the load or the point of generation or accumulation by either the generator's representative or a

USPCI representative and be accompanied by a chain of custody document. If this procedure is utilized, the load will be visually inspected for Physical Appearance.

### 4.4.4 SAMPLING SAFETY PRECAUTIONS

At a minimum, samplers will wear safety glasses, gloves, protective clothing, and protective footwear. Laboratory chemists check the manifest and other shipping documents to be familiar with the material and ensure samplers take all necessary precautions.

### 4.4.5 REMOTE SAMPLING AND/OR ANALYSIS

In cases where USPCI directs off-site sampling or analysis for the purpose of having that sample and/or analysis meet the requirements of the USPCI provisions (e.g.; Fingerprint Testing, Operational Process Specific Testing, etc) a USPCI representative will be at the sampling site to ensure compliance with the provisions of this Waste Analysis Plan. For samples taken at the remote location and transferred to the facility for analysis, chain of custody will be followed.

### 4.4.6 LAB PACKS

Lab pack material is accepted subject to a contents review. That is, material which are proposed to be landfilled are inventoried and sent to the facility for a review of their inventories. The inventories are reviewed for incompatibility of contained materials, land disposal restrictions, and utilization of appropriate absorbents. Since lab packs contain many small quantities of individual materials, they are not sampled, but they are inspected to ensure adequate absorbent is present and the drum is 90% full. (Note: The permit application contained a detailed labpack program which is now superseded by the procedures contained within this revised Waste Analysis Plan.)

### 4.4.7 NON-HAZARDOUS WASTES

The facility accepts unmanifested loads of waste which are not hazardous as defined under RCRA. In order for the load to be accepted, the certification shown in Figure 5-1, or an equivalent, is typically completed and on file at the facility or should accompany the load. These wastes will be inspected for physical appearance, at a minimum. The certification utilized has the generator attest that the material is non-hazardous.

## 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 must provide to enable USPCI to determine the acceptability of the waste for treatment, storage, or disposal.

Waste screening is the mechanism for deciding to reject or accept a particular type of waste--prior to its acceptance for management 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 the special materials noted below, the following procedures are implemented:

* The generator will provide USPCI with:
  (1) chemical and physical data requested on the Hazardous Waste Profile Sheet (HWPS), Typical Form shown as Figure 5-2 (or an equivalent form, such as an MSDS); (2) a representative sample;

(3) other supporting documentation, if necessary (e.g.; to document compliance with the land disposal restrictions,...).

- USPCI will ensure the HWPS contains the critical information or run any missing critical analyses, verify certain HWPS data by utilizing the representative sample(s) of the waste and performing requisite confirming analyses. Sampling and analyses will be performed in accordance with the methods outlined in Sections 2.0, 3.0, and 4.0. For accepted waste streams the representative sample will normally be retained by USPCI for reference for 30 days.
- After comparing the data supplied by the generator with that obtained by identification, USPCI will determine the acceptability of the waste based on:
  - (1) the permit conditions for the facility,
  - (2) existing land disposal restrictions, and
  - (3) the availability of the proper waste management techniques.
- As a minimum, the pre-acceptance evaluation will be repeated when a generator notifies USPCI that the process generating the waste has changed (e.g.; when the raw materials to the process have

changed), or if the General Manager, or his designee (e.g.; the Laboratory Manager, site chemist, etc...), hereinafter collectively referred to as the "General Manager," has reason to suspect that the waste does not match the pre-acceptance documentation.

* Exceptions to the foregoing requirements include the following special materials from on-site or off-site activities, including associated materials (such as contaminated PPE):

1. Contaminated Trash and Debris. This is limited to discarded containers of laboratory chemical, lab equipment, lab clothing, debris from lab spills or clean up, and floor sweepings.

2. "Empty" containers of waste commercial products or chemicals. This applies to a portable container which has been emptied, but which may hold residuals of the product or chemical. Examples of containers are: portable tanks, drums, barrels, cans, bags, liners, etc. a container shall be determined "empty" according to the criteria specified at 40 C.F.R. Section 261.7.

3. Asbestos containing waste from building demolition or cleaning. This applies to asbestos-bearing waste insulation materials, such as wall board, wall spray coverings, pipe insulation, etc.

4. Commercial products or chemicals: off-specification, outdated, contaminated, or banned. This also includes products voluntarily removed from the market place by a manufacturer

or distributor, in response to allegations of adverse health effects associated with product use.

5. Residue and debris from clean-up of spills or releases of a chemical substance(s) or commercial product(s).

6. Animal waste and parts.

7. Chemical-containing equipment removed from service. Examples: cathode ray tubes, batteries, fluorescent light tubes, etc.

8. Waste produced from the demolition or dismantling of industrial process equipment of facilities contaminated with chemicals from the process. For these exceptions, the generator will supply USPCI with sufficient chemical and physical characteristics information for proper management of the waste.

9. Labpacks accepted in accordance with the provisions of Section 4.4.6 (Labpacks).

## 5.2 EVALUATION

The general Manager is responsible for the pre-acceptance evaluation decision (i.e.; whether to accept or reject the waste). All samples under consideration for acceptance are subject to the Fingerprint Analysis (Section 2.1). The General Manager may require Additional Analysis to screen samples for other contaminants or properties which indicate possible treatment or disposal methods. The basis for requiring these additional analysis are:

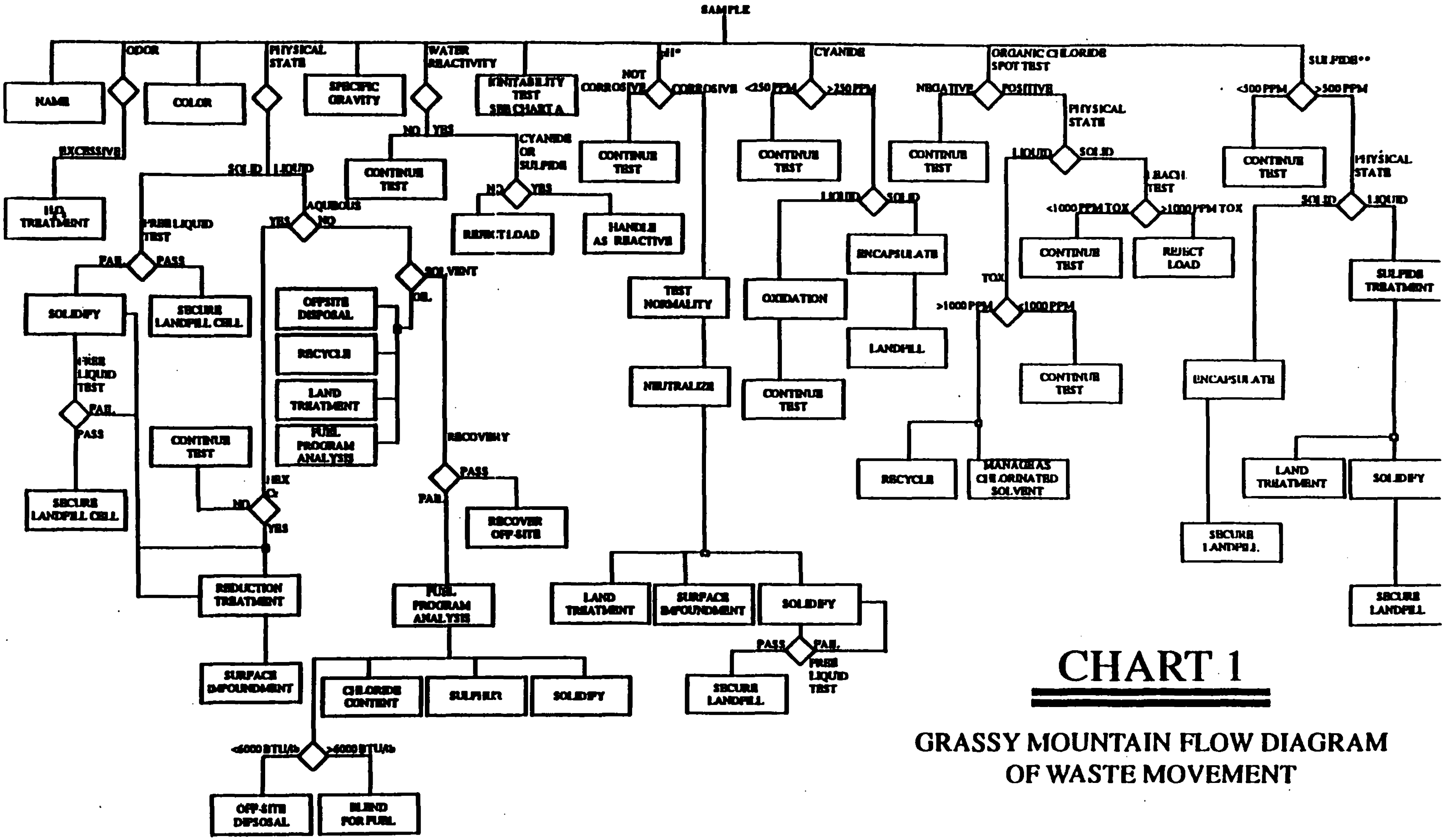
* General Manager's experience and judgement;

- HWPS description of the chemical and physical properties of the waste;
- HWPS description of the process generating the waste; and
- Results of the Fingerprint Analyses.

The pre-acceptance evaluation is aided by the use of Chart 1 and Chart 1A (given as an aid in the decision making process and does not have to be followed exactly), following, and is concluded with documentation of the decision regarding the acceptability of the waste and the proposed method of management. This decision is embodied within an "Acceptance Sheet" (typical form shown in Figure 5-3).

# CHART 1

# GRASSY MOUNTAIN FLOW DIAGRAM
# OF WASTE MOVEMENT



# CHART 1

## GRASSY MOUNTAIN FLOW DIAGRAM OF WASTE MOVEMENT

*pH>12.5 OR <2.0 IS DEFINED AS CORROSIVE
**SULFIDE REACTIVITY TEST NOT PERFORMED

# CHART 1A

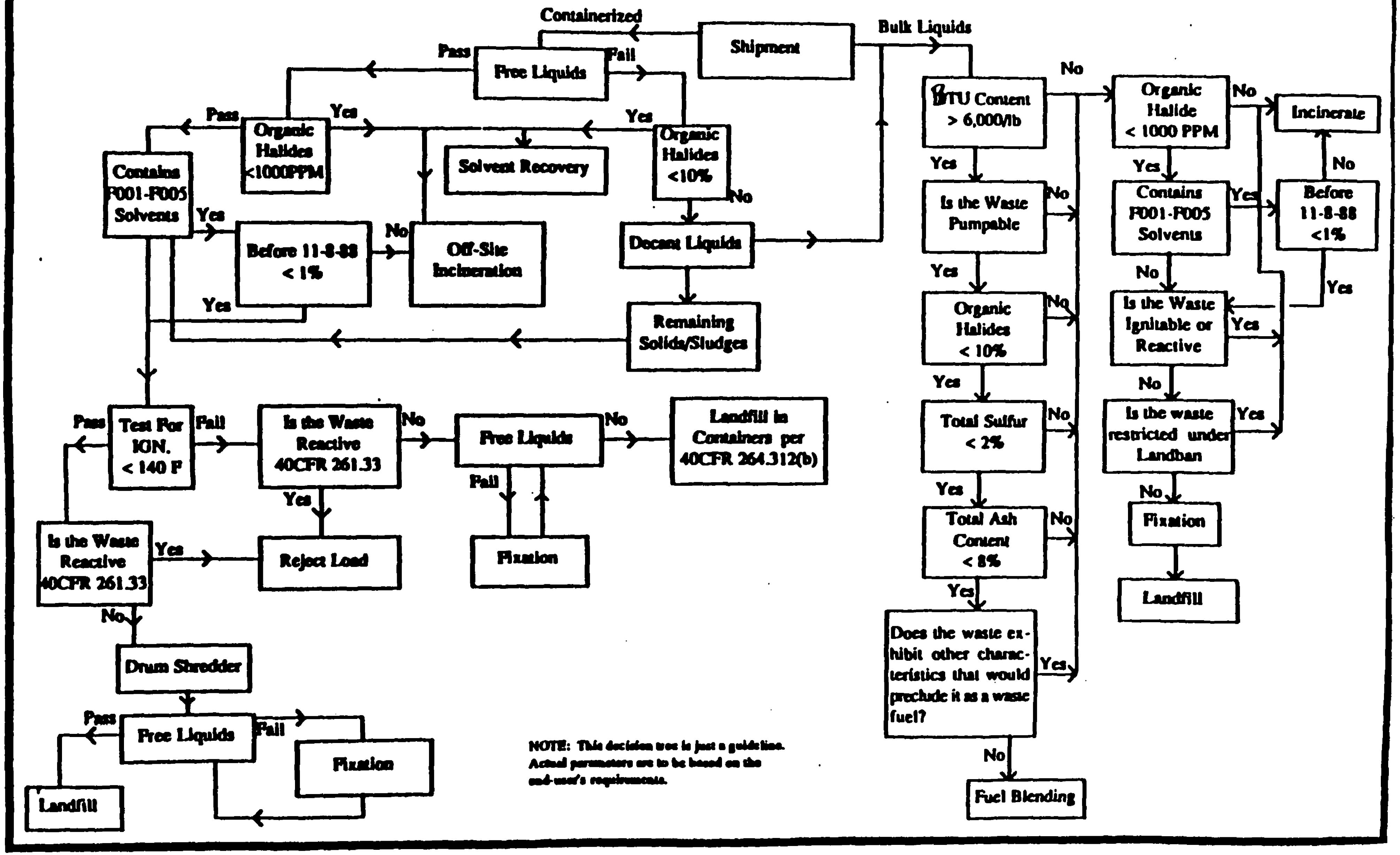
# DECISION TREE

# FOR

# NON-AQUEOUS WASTE

Chart 1A

## DECISION TREE FOR NON-AQUEDUS WASTE



NOTE: This decision tree is just a guideline. Actual parameters are to be based on the end-user's requirements.

## FIGURE 5-1

## NON-HAZARDOUS WASTE CERTIFICATE

## (TYPICAL FORM)

POLLUTION
CONTROL, INC.

# CERTIFICATE OF NON-HAZARDOUS WASTE

______________________________, here called "Generator" has contracted with United States Pollution Control, Inc., here called "Contractor" for transportation, storage, treatment, and disposal of the solid waste described below and Generator hereby certifies and warrants to Contractor that the solid waste to be so disposed of is not "hazardous waste" as that term is defined in Title 42 United States Code §6903, because said waste meets the following conditions:

a) Said waste is exempt from regulation because it meets the provisions of Title 40 Code of Federal Regulations §261.4, and/or,

b) Said waste is not listed as a hazardous waste in Subpart D of Title 40 Code of Federal Regulations Part 261 and said waste is not classified as a hazardous waste in Subpart C of Title 40 Code of Federal Regulations Part 261 as a result of actual testing or knowledge of the hazard characteristics of the waste in light of the materials or processes used.

DESCRIPTION OF NON-HAZARDOUS WASTE:

ORIGIN OF WASTE:

TYPICAL ANALYSIS OF WASTE:

| Compound | Concentration or Volume* |
|---|---|

DATED this______________________________, 19______

______________________________
GENERATOR

By______________________________
Title

ART TRENDS / Form 122

FIGURE 5-2

HAZARDOUS WASTE PROFILE SHEET

(TYPICAL FORM)

**USPCI, INC.**

# HAZARDOUS WASTE PROFILE SHEET

EXHIBIT A TO ______________________ Contract Dated ______________

As prescribed by the Resource Conservation and Recovery Act (Public Law 98-580 Sec. 3004), a detailed chemical and physical analysis must be submitte before we can handle your waste stream. This information is necessary to help us evaluate whether we can safely and economically transport and dispose your hazardous wastes in an environmentally sound manner. Be as complete as possible. If an area is not applicable, mark as such. Should a laboratory analys be available, please attach it to this form. We can arrange analytical laboratory services, if needed, for an appropriate fee. All information we receive will held in strictest confidence to protect your interests. **SAMPLE WILL NOT BE PROCESSED UNLESS ALL AREAS OF THIS SHEET ARE PROPERLY FILLED IN**

Purchase Order No. ______________________ Sales Representative ______________________

**I. Generator Information** ( ) Check if small quantity Generator per CFR-40

Name of Company ______________________ US EPA ID __ __ __ __ __ __ __ __ __ __ __ __

Facility Address ______________________ Invoicing Information ______________________

______________________ ______________________

Mailing Address ______________________ ______________________

______________________ ______________________

Technical Contact ______________________ Title ______________ Phone ( ) ______________

General Contact ______________________ Title ______________ Phone ( ) ______________

**II. Waste Process Information: Industry Category** ______________________

Waste Name ______________________ Rate of Generation ______________________

Process Producing Waste ______________________ Listed Waste Code (F, K, U, P) ______________________

| | | | | | |
|---|---|---|---|---|---|
| **III. Waste Properties** | Layers | Free Liquids | pH ______ | Normality ______ | ( ) Organic |
| Physical State @ 25°C | ( ) Single Phase | ( ) Yes | Type Acid ______ | Type ______ | ( ) Inorganic |
| ( ) Liquid ( ) Powder | ( ) Bi-layered | ( ) No | Density ______ | Caustic ______ | Flash Point |
| ( ) Solid ( ) Sludge | ( ) Multi-layered | | Color ______ | Odor ______ | ______ °F |

Chemical Composition ( )% ( ) PPM (List all known)

| | Range Lower | Range Upper |
|---|---|---|
| ______________ | ( _ ) | ( _ ) |
| ______________ | ( _ ) | ( _ ) |
| ______________ | ( _ ) | ( _ ) |
| ______________ | ( _ ) | ( _ ) |
| ______________ | ( _ ) | ( _ ) |
| ______________ | ( _ ) | ( _ ) |
| ______________ | ( _ ) | ( _ ) |

Metals-EP Tox Test or Total (Circle One) Mg/l or PPM (Circle One)

| | | | | | |
|---|---|---|---|---|---|
| Arsenic (As) | (____) | Lead (Pb) | (____) | Nickel (Ni) | (____) |
| Barium (Ba) | (____) | Vanadium (V) | (____) | Zinc (Zn) | (____) |
| Cadmium (Cd) | (____) | Selenium (Se) | (____) | Thallium (Tl) | (____) |
| Chromium (Cr) | (____) | Silver (Ag) | (____) | Cobalt (Co) | (____) |
| Mercury (Hg) | (____) | Copper (Cu) | (____) | | |

Possible Component (include unit of measure)

Cyanides ______________ 2, 4, D ______________ Dioxin ______________

Sulfides ______________ Phenolics ______________ Organic Chlorides ______________

**IV. Generator's Knowledge** Copies of all waste analysis result used to classify this waste must be attached. If the generator's knowledge is used in leiu of waste analysis the basis of that knowledge must be disclosed here: ______________________

______________________

______________________

**V. Hazardous Characteristics (Circle)** Ignitable D001 Corrosive D002 Reactive D004 EP Toxic List D-Code ______________

Is the waste ( ) Pyrophoric ( ) Infectious ( ) Water reactive ( ) Radioactive ( ) Pathogenic

( ) Explosive ( ) Pesticides/Herbicides ( ) Biological ( ) Shock sensitive ( ) Etiological

**VI. Shipping Information (From CFR-40)**

Proper DOT Shipping Name ______________________

DOT Hazard Class ______________ UN/NA Number ______________ Reportable Quantity ______________

Method of Shipment ( ) Bulk Liquids ( ) Bulk Solids

( ) Drums ( ) Other

Special Handling and Safety Instructions ______________________

______________________

______________________

I certify and warrant that the above information; the information attached, and the waste stream as described is true and correct to the best of my knowledge and ability. Willful or deliberate omissions have not been made. All known and/or suspected hazards have been disclosed. A sample representative of the waste stream has been or is being sent to the proper facility.

______________________ ______________________ ______________

Signature Title Date

ART TRENDS/FORM

FIGURE 5-3

ACCEPTANCE SHEET

(TYPICAL FORM)

## 6.0 INCOMING LOAD PROCEDURES

Each load of waste, upon arrival at the facility, will be visually inspected, sampled and analyzed, except as otherwise noted, as defined herein, before the waste is mixed with other wastes, applied to the land treatment unit, or landfilled. 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.

## 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 will be performed in accordance with the methods described in Section 2.0, 3.0, and 4.0.

All bulk liquid and solid waste deliveries will 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 will be inspected by utilizing the physical appearance; and at least 10 percent of such loads will be sampled and analyzed. Or, in the case of a railcar or remediation project under the control of USPCI (including its associated divisions), the vessel itself may be sampled and analyzed in lieu of individual loads.

In the case of loads of drums or portable tanks, at least 10 percent of the containers in each waste stream will be selected for sampling upon delivery. 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. After the load has been accepted, but before further processing, all containers will be opened and visually inspected for similar physical appearance.

Samples from incoming loads will be subjected to the Fingerprint Analysis identified in Section 2.1. All such samples will normally be retained at the facility laboratory for a period of 7 days.

## 6.2. DECISION EVALUATION LOGIC

There are major decision points regarding the need for evaluation of whether a waste found to be dissimilar to the pre-acceptance evaluation can be still accepted.

The General Manager (or his designee, collectively referred to as the General Manager) decides whether additional analyses are required for a particular waste based on the following:

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

Further testing will probably be required if the results indicate unexpected characteristics with respect to pre-acceptance analytical results, or if the general Manager has reason to suspect that the waste composition has changed.

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

- Inspection;
- Sampling;
- Analytical results;
- Hazardous Waste Profile Sheet;
- Hazardous Waste Manifest;
- Waste Screening Analytical Results;
- General Manager's judgement.

To facilitate the waste identification process, the fingerprinting analytical data recorded in the load sheet (Typical form shown as Figure 6-1) and compared to the corresponding pre-acceptance analysis. The fingerprint analysis verifies that the waste is indeed the same waste as represented by the pre-acceptance analysis. USPCI has incorporated the procedure into a computer program for waste acceptance. Information from the generator's waste profile sheet is 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 preacceptance analysis. Figure 6-2 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 preacceptance analysis, the discrepancy shall be recorded. In an attempt to resolve the discrepancy, the analysis may be rerun to rule out laboratory error. Resolution of any discrepancy exceeding the tolerance ranges will be explained in the "Notes" section of the record. The General Manager must classify the waste as being in nonconformance if it is significantly different in composition from the information shown in the HWPS, the preacceptance results, or on the manifest, unless the discrepancy can be clarified by the generator, transporter, or by the General Manager's judgement.

Waste 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;
- Land Disposal Restrictions;

- Discussions with the generator;
- Facility conditions;
- General Manager's judgement.

Pursuant to 40 CFR Section 264.72, the General Manager must discuss and attempt to resolve with the generator or transporter any significant discrepancies between the actual waste and that shown on the manifest. 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

LOAD SHEET

(TYPICAL FORM)

# LOAD SHEET FOR: GM88-4000-

LOAD#/SAMPLE LETTER : ____________ ID #

IALS OF PERSON CHECKING IN LOAD ____________

GINAL RECORD # ____________

DATE OF ARRIVAL ____________ NAME:

MANIFEST # ____________ LOAD TICKET# ____________ WEIGHT __

WEIGHT TO AREA ____________ QUANTITY ________ (D) (Y) (G) (B) (T) (C) (P)

DRUM SIZES ___5 GAL. ___10 GAL. ____15 GAL. ___20 GAL. ___30 GAL. ___55 GAL. ___85 G

DRUM DOCK OPERATOR INIT. ________ DRUM SHREDDED (Y) (N) DOCK GRIDS: _____

DOCK NOTES ________________________________________

HAULER ID# ________________ TRUCK TYPE ______ UNIT # ______ MILES ________

PROFILE WASTE CODES ____/____/____///

LOAD WASTE CODES / / / / /

## COMMENTS MAY CONTAIN OTHER WASTE CODES

## MGMT AUTHORIZATION/HANDLING INSTRUCTIONS

ACCEPTABLE FOR MANAGEMENT WITH THE FOLLOWING TREATMENT

COMMENTS:

## ---WASTE ANALYSIS INFORMATION---

WPS # WASTE NAME :

COLOR : / PHYSICAL STATE : /

LAYERING: / FREE-STANDING LIQUID: (POS) (NEG)

Temp: Absorbant Free: (Y) (N)

| PARAMETER | | METHOD | PROFILE | LOAD | ANALYTICAL CHEMIST'S INIT. |
|---|---|---|---|---|---|
| Sp. Gravity | 1 | 213 E | | | |
| | 3 | 9040 | | | |
| rmality | 4 | 1 | | | |
| Solids Scr. | 4 | 3 | | | |
| TLV Sniff | 4 | 5 | | | |
| Flash Point | 3 | 1020 | | | |
| TOX Scr. | | Cu Wire | | | |
| Free Liquids | 3 | 9095 | | | |
| Radioact. Scr. | 4 | 7 | | | |
| BTU/LB | 2 | D-240-85 | | | |
| Water RX Scr. | 4 | 2 | | | |
| CN~Tot mg/l | 3 | 9010 | | | |
| PCB's | 3 | 8080 | | | |
| Oxidizer Scr. | 4 | 6 | | | |
| Reducer Scr. | 4 | 10 | | | |
| TOX | 3 | 9020 | | | |
| RX Sulf. Scr. | 4 | 4 | | | |
| RX CN~ Screen | 4 | 4 | | | |
| Fix. Req/Ratio | 4 | 8 | | | |

BULK SOLID LOADS

YES NO INITIAL

(1) Visually checked for free liquids.

(2) Were free liquids present?

(3) Check for free liquids at truck washout.

(4) Check for free liquids at solidification unit.

(5) Gallons of free liquids found.

(6) Door secured after inspection.

Items 1,2,3,4,5,6 apply ONLY to gondola loads.

Items 1 and 2 apply to OTHER bulk loads.

Agent/Ratio: F /O /L /W

Total: F /O /L /W

OPERATOR'S INITIALS

1 - Standard Methods 2 - ASTM 3 - SW-846 4 - USPCI

NOTES:

HANDLING CODES: S01 S02 S05 T07 T24 T31 T39 T47 T50 T63 D79 D80 D81

TSD DATE TSD AREA INITIALS AUTHORIZING TSD

SPECIAL TSD INSTRUCTIONS FOR THIS MATERIAL:

RATOR'S INITIALS CERTIFYING RECEIPT IN ACCORDANCE W/ABOVE INSTRUCTIONS

CELL GRIDS :

BILLING CODES : / / / / /

WASHOUT CERTIFICATION (INT.) (EXT.) TIME REQUIRED

TSD CHEMIST'S SIGNATURE:

PLEASE RETAIN THESE DOCUMENTS
THIS IS THE ONLY COPY YOU WILL
RECEIVE !!

CUSTOMER SAMPLE REFERENCE NUMBER : GM88- UPDATE DATE:
GENERATOR BROKER

ATTENTION : CONTACT:
PLEASE FORWARD TO ENVIRONMENTAL DEPT.! / -

ANALYST NAME :
SALES REP: CUSTOMER SAMPLE # :
SAMPLE ARRIVAL DATE : SAMPLE SITE :
PROFILE ARRIVAL DATE : DATE COMPLETED :

## ---ACCEPTANCE/DENIAL INFORMATION---

ACCEPTABLE FOR DISPOSAL AT THIS FACILITY
COMMENTS:
TYPE OF CONTAINER :

## ---WASTE ANALYSIS INFORMATION---

WASTE PROFILE SHEET #
WASTE NAME :
COLOR :
PHYSICAL STATE :

| PARAMETER | | METHOD | PROFILE | LOAD | PARAMETER | | METHOD | PROFILE | LOA |
|---|---|---|---|---|---|---|---|---|---|
| Sp. Gravity | 1 | 213 E | | | Arsenic | 3 | 7061 | | |
| pH | 3 | 9040 | | | Antimony | 3 | 7040 | | |
| Normality | 4 | 1 | | | Barium | 3 | 7080 | | |
| Solids Sc. | 4 | 3 | | | Beryllium | 3 | 7090 | | |
| LV Sniff | 4 | 5 | | | Cadmium | 3 | 7130 | | |
| lash Point | 3 | 1020 | | | Chromium | 3 | 7190 | | |
| Ash Content | 2 | D-482 | | | Cobalt | 3 | 7200 | | |
| BTU/LB | 2 | D-240-85 | | | Copper | 3 | 7210 | | |
| BTU/GAL | 2 | D-240-85 | | | Lead | 3 | 7420 | | |
| Hex. Chrome | 3 | 7196 | | | Mercury | 3 | 7470,7471 | | |
| Water RX Sc. | 4 | 2 | | | Nickel | 3 | 7520 | | |
| RX Sulf. Scr. | 4 | 4 | | | Selenium | 3 | 7741 | | |
| CN~Tot mg/l | 3 | 9010 | | | Vanaduim | 3 | 7910 | | |
| RX CN~ Screen | 4 | 4 | | | Zinc | 3 | 7950 | | |
| Org. Chloride | 3 | 8020,9020 | | | Nitrogen | 1 | 417 | | |
| TOX | | Cu Wire | | | Phosphorus | 1 | 424 | | |
| TOX Leachate | 3 | 9020 | | | Aromatics | | | | |
| Free Liquids | 3 | 9095 | | | Alaphatics | | | | |
| PCB's | 3 | 8080 | | | Asphalteens | | | | |
| Radioactive | 4 | 7 | | | Parafines | | | | |
| Ox. Mat. Sc. | 4 | 6 | | | Oil/Grease | 3 | 9070 | | |
| Fix. Ratio | 4 | 8 | | | Sulfides | 3 | 9030 | | |
| CN~Free mg/l | 3 | 9010 | | | Redux. Mat Sc. | 4 | 6 | | |

1 - Standard Methods 2 - ASTM 3 - SW-846 4 - USPCI

THE GRASSY/GRAYBACK MT. FACILITY PERMITS ALLOW THE TREATMENT/
STORAGE/HANDLING OF THIS WASTE STREAM BY THE METHODS DESIGNATED ABOVE

APPROVAL DENIAL DATE / /
ROVAL DENIAL DATE / /

## FIGURE 6-2
## FINGERPRINTING ANALYSES
## ACCEPTABLE TOLERANCE RANGES (FOR DISPOSAL)

| PARAMETER | TOLERANCE RANGE |
|---|---|
| pH[1] | ± 2.5 pH units |
| Specific Gravity[2] | ± 20% |
| Reactivity: | |
| $CN^-$(3) | positive to negative, only |
| $S^{2-}$(4) | positive to negative, only |
| Water | positive to negative, only |
| Solids Screen | ± 20%[5] |
| Explosivity Meter Vapor Test (TLV Sniff) | ± 200 ppm |
| Radioactivity Screen | no tolerance[6] |
| Oxidizer Screen | positive to negative, only |
| Reducer Screen | positive to negative, only |

Notes:

1. For pH, the tolerance range decreases as pH of the waste crosses the values 2.0 (from above 2.0) and 12.5 (from below 12.5). Any waste which "crosses" the boundary condition (that is, pH less than 2.0 or greater than 12.5) will be considered to potentially exhibit the characteristics of corrosivity, regardless of the tolerance range, unless the material is a listed waste.

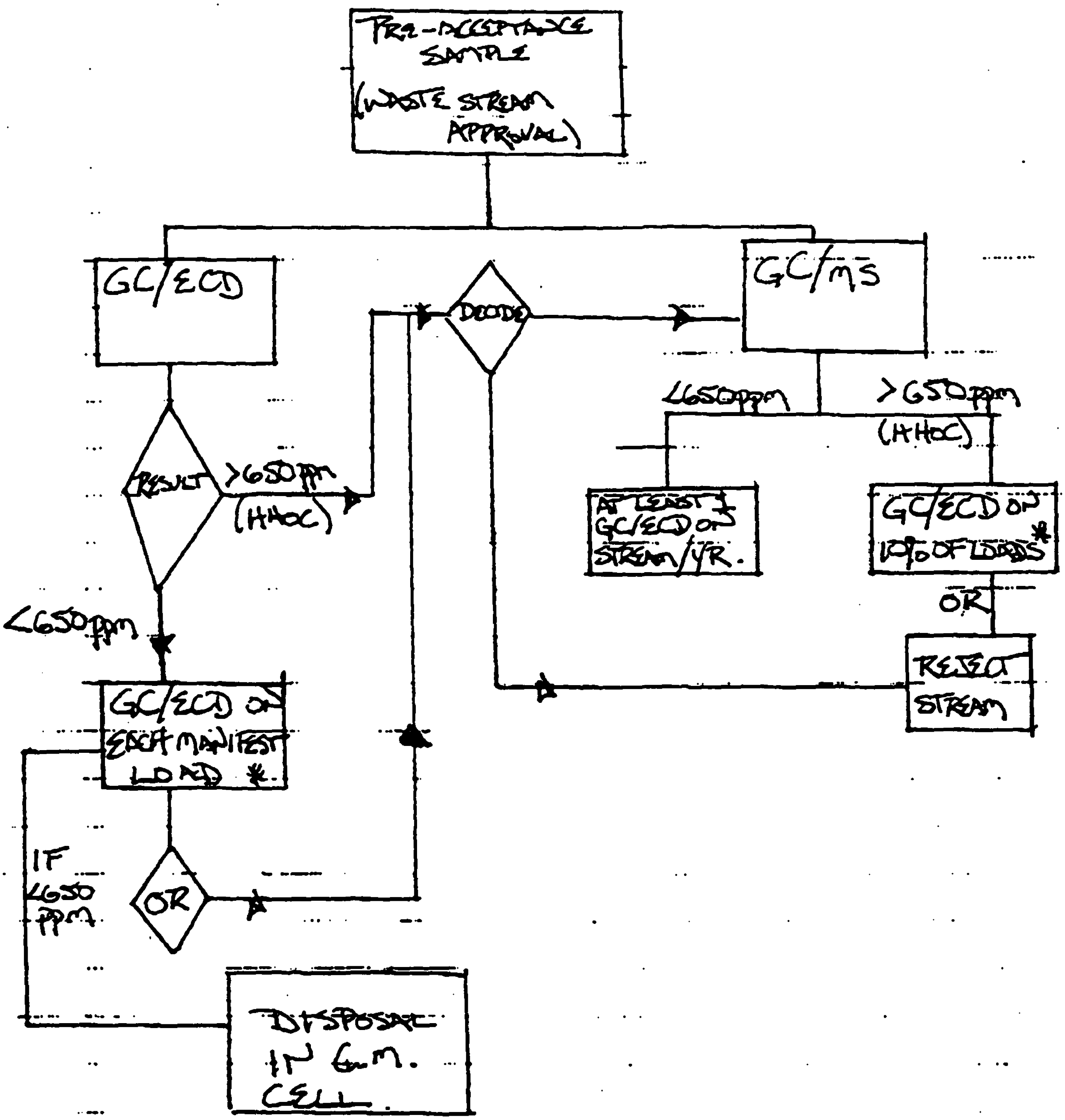
2. Specific gravity is not applicable to bulk loads of debris or other non-homogenous wastes.

3. If the material is to be disposed of directly into the landfill, an increase across 250 ppm will require explanation, further analysis or, potentially, different handling.

4. If the material is to be disposed of directly into the landfill, an increase across 500 ppm will require explanation, further analysis or, potentially, different handling.

5. For example, an initial reading of 50% may vary from 30% to 70%, inclusive.

6. Radioactivity may not exceed 40 microrads/hr without approval from the Utah Bureau of Radiation Control.



(* LOADS AS DEFINED IN WASTE ANALYSIS PLAN)

## 7.0 PROCESS OPERATIONS PROCEDURES

Each movement of a waste within the facility, during which any 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. These are not repeated unless it is known or believed that the waste characteristics may have significantly changed during storage or processing.

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

- Storage, consisting of containers, tanks, and surface impoundments;
- Treatment, consisting of chemical oxidation, neutralization, chromium reduction, blending, land treatment, stabilization;
- Disposal, consisting of landfilling.

The analytical procedures including additional mandatory analyses for each of these processes is described separately below.

## 7.1 STORAGE

Stored containerized liquid wastes are segregated with respect to compatibility. Most liquid wastes which are transferred from drums, portable tanks or tank trucks are placed in bulk storage tanks prior to further treatment. Before any waste are placed in a storage unit, the General Manager will assess the compatibility of the waste with the storage unit materials of construction and with wastes already stored therein. If there is any suspicion of incompatibility, additional liquid compatibility testing will be performed.

### 7.1.1 STORAGE TANKS

#### 7.1.1.1 WASTE/TANK COMPATIBILITY

There are three types of tanks for storing/receiving wastes:

(1) steel tanks (carbon steel) for alkaline, neutral organic or oil waste;

(2) stainless steel tanks for treatment of all wastes; and

(3) reinforced fiberglass tank primarily for acidic wastes.

The previously described "fingerprint" analyses are performed on incoming wastes to ensure compatibility of the waste and tank. The pH of the liquid is the most significant parameter. Liquid acidic waste with a pH $< 3$ are unloaded in the fiberglass-lined tank, reaction tank, or stabilization system. Liquid acidic waste with a pH $\geq$ 3 and < 5 will be unloaded into the fiberglass tank or reaction tank unless the liquid can be stabilized at the time of acceptance. All other liquid waste, pH $\geq$ 5 will go to one of the steel storage or treatment tanks. By this practice, incompatible, (i.e.; acidic and alkaline,) wastes will be segregated.

### 7.1.1.2 IGNITABLE, REACTIVE, AND INCOMPATIBLE WASTES

Liquid ignitable wastes, such as flammable solvents, will be stored in steel tanks. These tanks will be equipped with pressure vacuum breather valves with flame-arresters. No smoking, sparks or a source of ignition will be permitted in the vicinity of the tanks.

Corrosive alkaline wastes will not be mixed with flammable liquids. Tanks will be designed by service, that is, organic containing tanks will not be switched to inorganic (alkaline) waste storage without a thorough cleaning with steam cleaner, followed by a water rinse. After cleaning, the vessel will be opened and inspected and vapor "sniff" test performed to ensure only de minimus organic constituents remain in the vessel. Rinsate will be analyzed for total organic carbon and if a value exceeding 250 ppm is obtained, further cleaning will be required. Rinsate will be transferred to the surface impoundment or tank after checking flash point to ensure it is over 140°F, as appropriate. Reactive wastes containing cyanides and/or sulfides are generally alkaline in nature. These must never come in contact with acidic waste or material. Similar procedures are used on a stream-by-stream basis to ensure that any other combination of incompatible waste are not stored in the same tank.

### 7.1.2 STORAGE SURFACE IMPOUNDMENT

The purpose of this section is to assure waste/liner compatibility and proper management of ignitable, reactive, and incompatible wastes in the surface impoundment.

#### 7.1.2.1 WASTE AND LEACHATE/LINER COMPATIBILITY

USPCI intends to avoid contact by high concentrations of chlorinated hydrocarbons with the clay or HDPE liners. Further, concentrated portions of most organics are to be kept away from the liner. When a generator indicates chlorinated hydrocarbons may be present, pre-acceptance samples of that waste stream will be tested for Total Organic Halides (TOX) or other appropriate surrogate. If the test is negative (i.e.; TOX (or TOC) < 1,000 mg/l), the waste may be accepted for storage in the surface impoundment. To ensure this occurs, each material destined for the surface impoundment (and for which the generator indicates chlorinated hydrocarbons may be present) will be tested for

TOX on the pre-acceptance analysis and for each waste stream with results greater than 500 mg/l TOX, this analysis will be reconfirmed (to ensure it does not exceed 1,000 mg/l TOX upon arrival for approximately 10% of the incoming loads of that waste stream (to ensure it does not exceed 1,000 mg/l TOX) and after close observation and study, if the General Manager finds the nature of this material is such that it is not likely to exceed 1,000, this additional study may be concluded after a minimum of 20 reanalyses. If the material contains less than 1,000 mg/l TOX the material will be considered acceptable. If the TOX test indicates greater than 1,000 mg/l total organic halides are present the material will be rejected for the surface impoundment or total organic chlorine may be determined to ensure it is not above 1,000 mg/l. If the total organic chlorine is above 1,000 mg/l, the material will not be acceptable for the surface impoundment unless evaluation of liner and waste compatibility indicates the waste may be safety managed.

### 7.1.2.2. IGNITABLE, REACTIVE AND INCOMPATIBLE WASTES

Pre-acceptance information about the process generating the waste provide information which can be used to detect the presence of oxidizers and other reactives. Flammability, reactivity (cyanides, sulfides, and water) and other oxidizing potential are checked on incoming waste. Highly acidic waste or alkaline wastes (pH $\leq$ 2.0 or pH > 12.5, respectively), are not placed in the surface impoundment directly, unless the material is a weak acid or base (e.g.; the normality is less than 2 N), is not aggressive, the liquid waste compatibility check does not detect any significant reactivity problems, and/or the resulting mixture is within an acceptable pH range (2.0 < pH < 12.5). Strong oxidizers are also not considered for storage in the surface impoundment without prior treatment. Ignitable wastes are not accepted, unless, if added, the resulting mixture would no longer test positive as evidenced by the ignitability screen. A similar handling procedure is followed for sulfide or cyanide bearing wastes. That is, reactives are not accepted for storage, unless the resultant mixture does not test positive for reactivity.

### 7.1.3 STORAGE CONTAINERS

Storage containers typically consist of 55 gallons drums of waste which are received from off-site for storage prior to an on-site treatment or disposal process. Therefore, the containers are subject to the fingerprint analysis to ensure conformity with the pre-acceptance documentation.

#### 7.1.3.1 IGNITABLE, REACTIVE AND INCOMPATIBLE WASTES

Ignitable, reactive, or incompatible incoming wastes are identified and classified by the sampling and analytical procedures described earlier, and information submitted by the generator.

The fingerprint procedures detailed previously check for pH, reactivity ($CN^-$ and $S^=$) and flammability. The containers are segregated accordingly.

### 7.1.3.2. ON-SITE MANAGEMENT PROCEDURES

Wastes received in containers are also subject to the analytical work for the proposed management process. For example, a drum of F001 waste would be subject to an inspection for the fingerprint parameters (including free standing liquids). Depending on whether it was subject to an extension or petition or was a treatment residue, it might also be subjected to verifications testing (e.g.; TOC analysis, TCLP, etc...). 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.; Paint Filter Liquids test for stabilization,...). Finally, it might also be subject to the protocol for the landfill, if that was the ultimate disposal option on-site.

### 7.1.3.3 LABORATORY PACKS

Drums packed with small quantities of waste are accepted for storage and disposal under special provision listed in Sections 4.4.6 and 5.1. As with all wastes destined for land disposal, all wastes contained within the lab pack must not be prohibited from land disposal.

### 7.1.3.4 MANAGEMENT OF RESIDUES

Management of sump residues, floor sweepings, and other miscellaneous debris originating out of the drum management area will be managed as on-site generated waste under the conditions of Section 5.1(1). However, where the material is identifiable to a specific waste, it shall be managed in accordance with the approved management conditions for that waste (e.g.; a spill of F002 material may be managed as F002), or if precluded by permit, regulation, or operational conditions, it may be reprofiled for alternative management, as appropriate.

## 7.2 TREATMENT OPERATIONS

The proper and complete treatment of a particular waste depends upon appropriate sampling and analysis during selected phases of the operation. The results of this analytical program serve to determine safety constraints, confirm the selection of treatment methods, and identify the process parameters. The treatment sampling/analysis program may be divided into three segments, each with a specific purpose:

- Pre-treatment analyses confirm that the waste falls within the selected process design parameters and allow the fine tuning of the process operational conditions for optimum treatment;

- In-process analyses are performed to control the process and to monitor progress;
- Post-treatment analyses confirm successful treatment and that the characteristics of the process effluent are such that it can be sent to the next step (discharge, disposal, or further treatment) based upon permit or process constraints.

### 7.2.1 CHEMICAL OXIDATION

In this process, cyanide-and/or sulfide-bearing or other oxidizable wastes are treated to convert these compounds to more innocuous species. This conversion may utilize hydrogen peroxide, sodium hypochlorite, calcium hypochlorite, chlorine, or other oxidizing agents.

Pre-treatment analyses establish that the waste is sufficiently alkaline and pumpable, and the identity and quality of the optimum oxidizing reagent.

In-process analyses consist of a check for free cyanides (or sulfides) as a measure of reaction completeness. Samples are taken and analyzed until the remaining cyanide (or sulfide) levels are within acceptable limits (e.g.; total CN < 1,000 mg/l and total releasable CN < 250 mg/l if destined for direct landfilling, etc...). This may involve the addition of more reagents or a longer reaction time.

When these tests indicate that the oxidation reaction has been sufficiently completed, a final sample is taken and the free cyanide (or sulfide) concentration is confirmed. A final pH analysis is part of the post-treatment analyses.

### 7.2.2 NEUTRALIZATION

In this process, aqueous inorganic wastes are neutralized and/or heavy metals precipitated. Most of the waste fed to this system will be acidic in nature. Thus, lime or other reagents, including suitable alkaline wastes, are used for neutralization purposes.

Pre-treatment analyses serve to screen out those wastes that are not acceptable to this process, including wastes that are not pumpable, wastes with significant concentrations of cyanides or sulfides, and wastes that are not primarily aqueous.

In-process analysis are performed to monitor the pH as a means of controlling the reaction process. The chemicals are mixed in a reaction tank then transferred to a treated liquid storage tank.

A final pH is the post-treatment analysis used to determine when the reaction has been completed. The effluent is directed to the appropriate handling area.

### 7.2.3. CHROMIUM REDUCTION

In this process hexavalent chrome is reduced to the less toxic trivalent form. The conversion may utilize sodium metabisulfate, ferric chloride or other suitable agents.

Pretreatment analyses establish that the waste is sufficiently acidic, pumpable, compatible with the tanks and identity and quality of the reducing reagent.

In-process analyses are performed to monitor pH and hexavalent chrome. The pH of the solution directly affects the rate of the reaction. The chemicals are mixed in the reaction tank and then transferred to a treated liquid storage tank.

Post-treatment analyses are performed to assure that the chrome reduction is sufficiently completed and the final pH is acceptable for subsequent handling.

### 7.2.4 BLENDING

Typically, in this process wastes containing sufficient heating values will be blended with other suitable waste. However, waste destined for incineration may require blending, too. The resultant mixtures are used as supplemental fuels for lime kilns, incinerators, or similar operations. Unintentional blending, such as storage of several waste streams in the surface impoundment or storage tanks, is not covered by this section.

Pre-acceptance analyses are used to determine the acceptability of each waste stream for the fuels blending program. Additional analysis for heat value is also required for materials destined for supplemental fuels to ensure sham recycling does not occur in accordance with applicable guidance (e.g.; currently, BTU value > 5,000 BTU/lb). For materials destined for incineration, on the other hand, this analysis is not mandatory.

In-process analyses are performed to assure the blending of wastes is staying within the final product requirements. This is necessary because acceptance criteria are different than final product criteria which are based upon the end user's criteria. Those criteria are, in turn, based upon that user's permits, regulations, or other needs. For example, if a reuser has a minimum requirement for heat value and a maximum requirement for chlorides, then the blend requirements will be a function of the user's requirements for both parameters.

Post-treatment analyses consist of tests necessary to assure that the blend is suitable for use as fuel or incineration.

### 7.2.5 LAND TREATMENT

The demonstration of the land treatability of a given waste and the routine treatment of the waste begin and continue with the characterization of that waste. It is only after thorough characterization of a waste has been completed that enough can be known for the purpose of treatment. Comprehensive waste analytical data are needed to identify and quantify the hazardous and pertinent non-hazardous constituents contained in the waste. If significant concentrations of persistent (in soil) organics are present which are potentially hazardous, the analytical procedures throughout the course of the treatment demonstration and unit operation will necessarily be more extensive (i.e.; analysis for many of the Appendix VIII compounds versus only gross parameters such as total oil and grease). The converse also may be true; thorough characterization may allow the elimination of certain analytical procedures throughout the course of the treatment demonstration, unit operation, and monitoring program.

The characterization phase will also be important in helping to define capacity limiting constituents (CLC) (e.g.; metals), rate limiting conditions (RLC), and the application limiting constituents (ALX).

Waste characterization will be done at three levels. Prior to receipt at the facility, detailed initial characterization of each major waste from its source will be carried out to check for general waste quality and the full range of hazardous constituents present (Table 7.2.5A). Waste received at the facility destined for land treatment unit will be sampled and analyzed (fingerprint analyses) for constituents which indicate: (1) whether the waste is indeed from the source and of the type listed on the manifest and (2) how the waste should be applied to the LT unit. In addition, the residue from facility storage of waste to be land applied will be analyzed prior to calculation and application (Table 7.2.5B)

### 7.2.5.1 INITIAL WASTE CHARACTERIZATION

Sampling of waste is to be in accordance with good scientific methods to produce accurate representative samples of the waste. Specific amounts needed for analysis will depend on the analytical methods used. This analysis assures that wastes accepted generally fall within an acceptable range of quality compared to the waste(s) for which the land treatment demonstration was made.

Before any waste stream is first accepted for land treatment, it will be characterized as in the following discussion for comparison to the waste for which the land treatment demonstration (LTD) was made. Each stream will be analyzed in detail whenever the waste is reprofiled.

### 7.2.5.2 ANNUAL WASTE CHARACTERIZATION

The chemical analysis of the waste to be land treated will include both analysis of the waste alone and analysis of the waste in conjunction with the soil, as well as analyses of soil-pore liquid and ground water. Discussed here are those analyses carried out in characterizing the waste.

# TABLE 7.2.5A
# ANNUAL PHYSICAL AND CHEMICAL ANALYSIS
# (WASTE CHARACTERIZATION)

## MODIFIED "SKINNER LIST" FOR PRINCIPAL HAZARDOUS CONSTITUENTS IN PETROLEUM REFINING WASTES LAND TREATMENT

## A. REQUIRED ANALYSES FOR K048, K049, K050, K051, K052 AND NON-HAZARDOUS WASTES AND D002, D003, D004, D005, D006, D007, D008, D009, D010, D011 AND K062 WASTES.

### 1. METALS (TOTAL)

Antimony
Arsenic
Barium
Beryllium
Cobalt
Cadmium
Chromium
Copper
Lead
Mercury
Nickel
Selenium
Vanadium
Zinc

### 2. Volatiles

Benzene
Carbon disulfide
Chlorobenzene
Chloroform
Cyclohexane
1,2 Dichloroethane
Ethylbenzene
Ethylene dibromide
Methyl ethyl Acetone
Styrene
Toluene
Xylene (m-, o-, & p-)

### 3. Semi-Volatile Base/Neutral Extractable Compounds

Anthracene
Benzo(a)anthracene
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(g,h,i)perylene
Benzo(a)pyrene
Bis(2-ethylhexyl)phthalate
Butyl benzyl phthalate
Chrysene
Dibenz(a,h)anthracene
Dichlorobenzenes
Diethyl phthalate
Dimethyl phthalate
7,12-Dimethylbenz(a)-anthracene
Di(n)octyl phthalate
Di(n)butyl phthalate
Fluoranthene
Fluorene
Idene
Ideno(1,2,3-c,d)pyrene
1-methylnapthalene
2-methylnapththalene
Napthalene
Phenanthrene
Pyridine
Pyrene Quinoline

### 4. Semi-Volatile Acid-Extractable Compounds

o-Cresol
p-Cresol
m-Cresol
2,4-dimethylphenol
2,4-dinitrophenol
4-nitrophenol
Phenol

### 5. Miscellaneous Analyses

% Water
pH
Total Solids
Total oil and grease
Ash content

### B. ADDITIONAL ANALYSES FOR D002, D003, D004, D005, D006, D007, D008, D009, D010, D011, AND K062 WASTES.

1. EP Toxicity

Arsenic
Barium
Beryllium
Cadmium
Chromium
Cobalt
Copper
Lead
Manganese
Mercury
Molybdenum
Nickel
Selenium
Silver
Zinc

2. Miscellaneous Analyses

Total Organic Carbon (TOC)
Purgeable Organic Halogen (POH)
Total Dissolved Solids (TDS)
Total Suspended Solids (TSS)
Sulfide
Gross Cations and Anions

### 7.2.5.3 ADDITIONAL LOAD ANALYSIS

To ensure organic chlorides are not land applied, each material destined for land treatment will be tested for TOX on the pre-acceptance analysis and, for each waste stream with results greater than 500 mg/l TOX, this analysis will be reconfirmed upon arrival for approximately 10% of the incoming loads of that waste stream (to ensure it does not exceed 1,000 mg/l TOX) and, if after close observation and study, the General Manager finds the nature of the material is such that it is not likely to exceed 1,000, this additional study may be concluded after a minimum of 20 reanalyses. If the material contains less than 1,000 mg/l TOX the material will be considered acceptable. If the TOX test indicate greater than 1,000 mg/l TOX are present, the material will be rejected for land treatment or total organic chlorine may be determined to ensure it is not above 1,000 mg/l. If the total organic chlorine is above 1,000 mg/l, the material will not be acceptable for the surface impoundment unless evaluation of liner and waste compatibility indicates the waste may be safety managed.

### 7.2.5.4 ANALYSIS OF RESIDUES

The residue is also characterized before application. The samples used for analysis must come from the tank, impoundments, or other sources where each waste load will be obtained for filling the applicator. This analysis is normally done in addition to any analysis done on the waste off-site unless the off-site waste is to be directly applied to the area without mixing with other residues. A sample is collected and analyzed for the constituents listed in Table 7.2.5B.

**TABLE 7.2.5.B**

Oil Content
Heavy Metals (As, Cd, Co, Cr, Cu, Pb, Hg, Ni, Zn, Se, Ba, Ag)
pH

The residue can then be applied to the land treatment system. All waste is removed and applied before allowing any new residue to be placed in the tank or the pertinent analyses must be run on those new residues.

Loading rate calculations are performed prior to any application of waste to the land treatment area.

For waste to be applied from the surface impoundment, loading calculations will be based on either: (1) a composite analysis from the impoundment, or (2) calculations using individual generator analysis and volumes.

### 7.2.6 STABILIZATION

In the process, waste containing free liquids will be mixed with pozzolanic material to assure all free liquids are chemically fixed.

Pre-treatment analyses consist of tests necessary to insure the waste are compatible with the tanks and the pozzolanic reactant. In-process analyses are not required for this treatment. Post-treatment analyses are necessary to assure that all free liquids have been chemically reacted and the mixture is suitable for final handling or processing. The Paint Filter Test is regularly checked in order to monitor this process.

## 7.3 LANDFILL DISPOSAL

Important considerations in landfill operations are waste/liner compatibility and leachate/leachate collection system materials compatibility. Other considerations include preventing ignitable, incompatible and reactive waste from contacting each other. Further assurance is required to minimize free liquids.

### 7.3.1 WASTE AND LEACHATE/LINER COMPATIBILITY

USPCI intends to avoid contact by high concentrations of chlorinated hydrocarbons with the clay or HDPE liners. Further, concentrated portions of most organics are to be kept away from the liner. When a generator indicates chlorinated hydrocarbons may be present, solid pre-acceptance samples will be subjected to the Toxicity Characteristic Leaching Procedure (TCLP) found in 40 CFR 268, Appendix I. The extract from the TCLP or liquid from liquid streams will be tested for Total Organic Halides (TOX). If the test is negative (i.e., TOX < 1,000 mg/l), the waste will be accepted for disposal in the landfill. To ensure this occurs, each material destined for landfilling (and for which the generator indicates chlorinated hydrocarbons may be present) will be tested for TOX on the pre-acceptance analysis and for each waste stream with results greater than 500 mg/L TOX, in the TCLP extract, this analysis will be reconfirmed (to ensure it does not exceed 1,000 mg/L TOX, in the extract) upon arrival for approximately 10% of the incoming loads of that waste stream and, if after close observation and study, the General Manager finds the nature of the material is such that it is not likely to exceed 1,000, this additional study may be concluded after a minimum of 20 reanalyses. If the material contains less than 1,000 mg/l TOX, in the extract, the material will be considered acceptable. If the TOX test indicates greater than 1,000 mg/l total organic halides are present in the extract, the material will be rejected for landfilling or total organic chlorine may be determined to ensure it is not above 1,000 mg/l, in the extract. If the total organic chlorine is above 1,000 mg/l, the material will not be accepted for direct landfilling unless evaluation of liner and waste compatibility indicates the waste may be safely managed. The sample is checked for Aromatic Hydrocarbons using the TLV Sniffer. Information supplied by the generator will be used in conjunction with the data from USPCI analyses to determine acceptability. A decision is made in the following steps (a) through (j) below.

(a) pH Screen (no limit)

(b) Specific Gravity (no limit)

(c) TLV-Sniff (if > 200, flash point must be run and if < 200, the flashpoint is considered > 140°F)

(d) Flashpoint must be higher than 140°F (if < 140°F, the material must be in non-leaking container).

(e) Solids Screen (no limit)

(f) Free standing liquids - no free standing liquids can be observed or the material must be solidified or the liquids otherwise eliminated.

(g) Reactivity (if above these standards, the material may be encapsulated)

  (1) Sulfides < 500 ppm (Reactive Sulfides)
  (2) Cyanides < 250 ppm (Reactive Cyanides)
  (3) Water (must not be water reactive)

(h) Oxidizer Screen (if positive, the material must be in non-leaking containers or additional information must be ascertained to ensure the material is not an oxidizer under 49 CFR 173.150.

(i) Using the generator supplied information or other developed information, compare the waste to the "Manufacturer's Chemical Resistance charts or other information" (See Figure 7-1). If the waste contains any of the chemicals listed as having a moderate to severe effect on the liner it cannot be considered for disposal in the cell until the leachate test has been performed or unless the total chemical concentration of concern is less than 1% as evidenced by either a TOC or TOX of under 10,000 mg/l; otherwise the chemical of concern must be analyzed for or determined to be less than th the concentration stated in Fig 7-1.

j) Reduction Materials Screen (no limit).

### 7.3.2 IGNITABLE, REACTIVE AND INCOMPATIBLE WASTES

Pre-acceptance information about the process generating the waste provide information which can be used to detect the presence of oxidizers and other reacties. Flammability, reactivity (cyanides, sulfides, and water) and other oxidizing potential are checked on incoming waste. Highly acidic liquid wastes (e.g., pH < 2.0), are not landfilled directly. Reactivity is checked and reactive wastes such as $AlCl_3$ and $POCl_3$ are rejected. Strong oxidizers are also not considered for disposal without treatment. Liquid ignitable wastes in containers are solidified (after removal of free standing liquids) prior to landfilling by either filling the container with a solidification reagent, or placing the contents into the solidification system and crushing or shredding the drum carcus. A similar handling procedure is followed for drums which contain sulfide or cyanide bearing semi-solid or liquids. The only difference being, in the latter case, the container (after solidification, if necessary) is encapsulated in conjunction with landfilling.

Incompatible containerized materials are segregated in the container storage area and are landfilled such that incompatible waste are segregated for final disposal.

### 7.3.3 WASTES CONTAINING FREE LIQUIDS

All materials will be checked for free standing liquids. If free standing liquids are present, they will either be removed, the material will be solidified by either filling the container with a solidification agent or placing the contents into the solidification system and crushing or shredding the drum, or by shredding the drum and its contents and, if necessary, solidifying the shredded material. If free standing liquids are not present and the fingerprint and process specific criteria are met, then the waste may be landfilled directly.

Free standing liquids in drums may be decanted and treated or they may be solidified by either filling the container with an absorbent agent and/or by placing the contents into the solidification system and crushing or shredding associated containers. The remaining semi-solid material will be solidified using fixation agents so that free liquids are no longer present prior to landfilling, if free standing liquids are present. Drums will be inspected in accordance with the procedures specified at Section 6.1. Batched solidified wastes will be tested using the Paint Filter Liquids test.

Part 268.41 restricted waste may be solidified provided the waste extract after solidification does not contain organic compounds in excess of the values listed in Table CCWE as determined in the pre-acceptance characterization.

# 8.0 LAND DISPOSAL RESTRICTIONS

[RESERVED]

## 9.0 QUALITY ASSURANCE AND QUALITY CONTROL

### 9.1 PROJECT DESCRIPTION

As required by Part 40 of Code of Federal Regulations (40 CFR), Section 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 which is necessary to treat, store, or dispose of the waste in accordance with 40 CFR Parts 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 of the best true value.

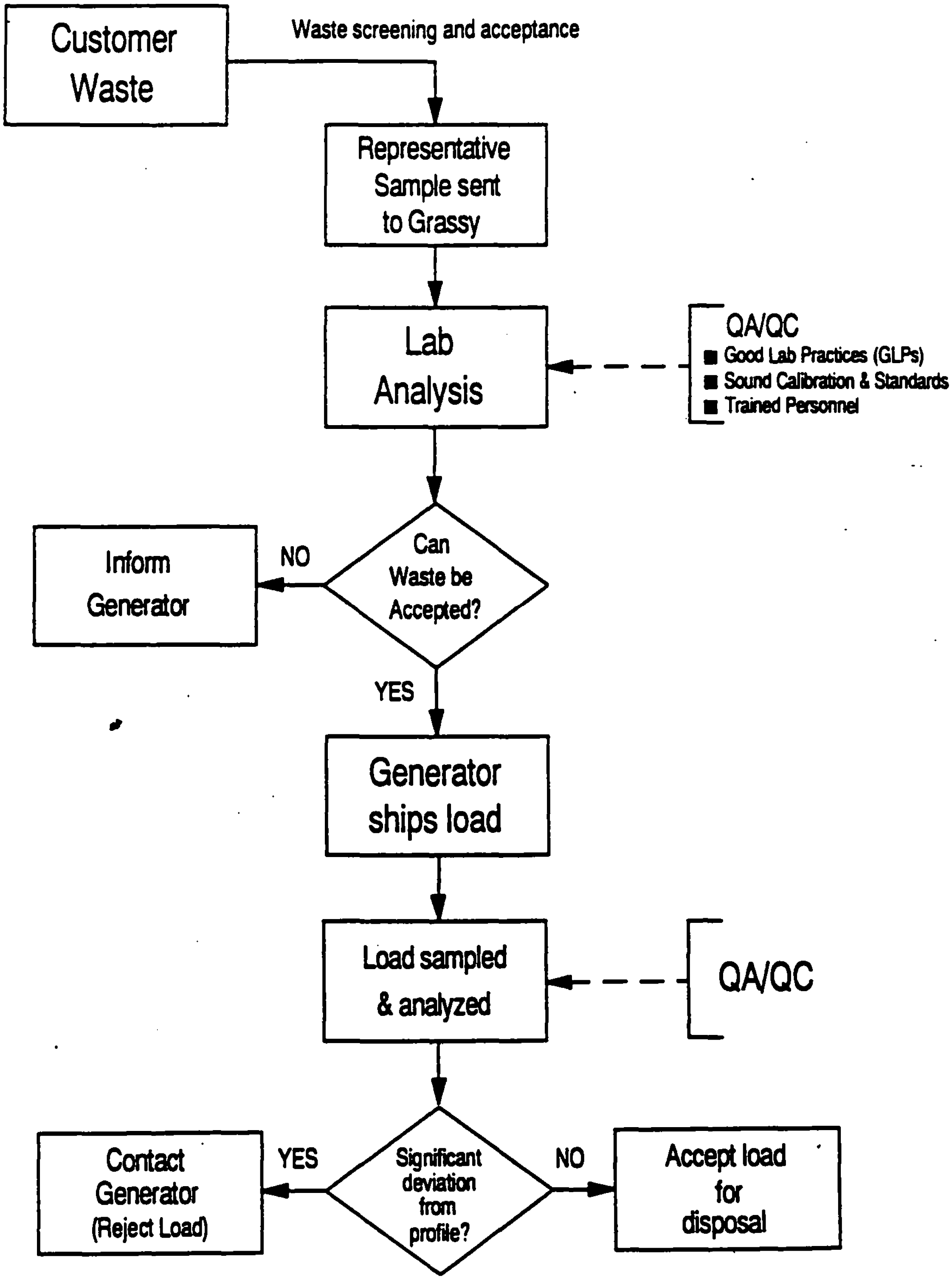
#### 9.1.1 DATA USE

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 at USPCI's - Grassy Mountain Facility. The handling procedures and final waste disposal will be based on these data.

#### 9.1.2 PROJECT DIAGRAMS

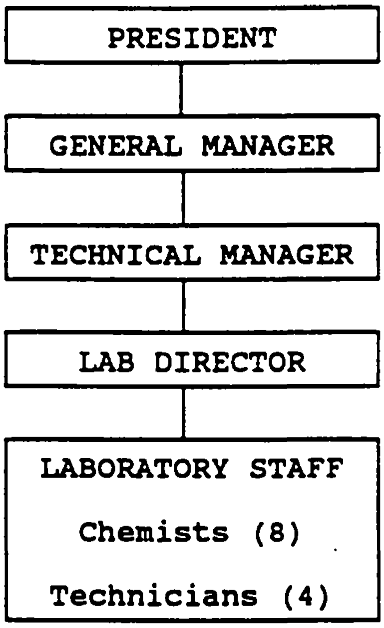
The main function of the laboratory is waste screening and acceptance. The following flow chart outlines the basic course followed in this function.

## WASTE SCREENING AND ACCEPTANCE



## 9.2 PROJECT ORGANIZATION

### 9.2.1 ORGANIZATIONAL CHART



LABORATORY QA Coordinator[1]

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[1] Due to the small number of chemists, the QA Coordinator has additional responsibilities which include conducting analyses.

## 9.3 QUALITY ASSURANCE OBJECTIVES

The tests required for insuring the load matched the profile are as follows:

pH Screen
Paint Filter Liquids Test
Reactivity (includes cyanide, sulfide, and water)
TLV Sniff (flashpoint)
Radioactivity Screen
Oxidizer/Reducer Screens

Other support tests may be deemed appropriate to further qualify/quantify the waste due to it's nature or source.

### 9.3.1 PRECISION AND ACCURACY

The following table lists the precision and accuracy goals for the fingerprint analysis parameters which are used to verify loads. See Glossary of Terms for calculations.

| Measurement Parameter | Reference | Experimental Conditions | Precision | Accuracy | Completeness |
|---|---|---|---|---|---|
| pH | SW 846 9040 | Industrial Waste Samples | ±2 pH units | ±.5 pH units | 100% |
| Paint Filter Test | SW 846 9095 | Industrial Waste Samples | PASS/FAIL | PASS<br>--<br>FAIL | 100% |
| Reactivity (Water) | USPCI 4 | Industrial Waste Samples | PASS/FAIL | PASS<br>--<br>FAIL | 100% |
| Reactivity (Cyanide) | USPCI 4 | Industrial Waste Samples | ±20% | ±25% | 100% |
| Reactivity (Cyanide) | SW 846 7.3.3.2 | Industrial Waste Samples | ±20% | ±25% | 100% when screen is positive |

| Measurement Parameter | Reference | Experimental Conditions | Precision | Accuracy | Completeness |
|---|---|---|---|---|---|
| Reactivity (Cyanide) | SW 846 9010 | Industrial Waste Samples | ±20% | ±25% | 100% when screen is positive |
| Reactivity (Sulfide) | USPCI 4 | Industrial Waste Samples | ±20% | ±25% | 100% |
| Reactivity (Sulfide) | SW 846 7.3.4.2 | Industrial Waste Samples | ±20% | ±25% | 100% when screen is positive |
| Reactivity (Sulfide) | SW 846 9030 | Industrial Waste Samples | ±20% | ±25% | 100% when screen is positive |
| TLV Sniff | USPCI 5 | Industrial Waste Samples | ±20% | ±20% | 100% |
| Radioactivity Screen | USPCI 7 | Industrial Waste Samples | ±20% | (2) | 100% |
| Oxidizer Reducer Screen | USPCI 6 | Industrial Waste Samples | PASS/FAIL | PASS<br>--<br>FAIL | 100% |
| TOX | SW 846 9020 | Industrial Waste Samples | ±20% | ±20% | 100% as required |

[2] The facility cannot evaluate accuracy data on this test due to lack of calibration materials. The meter must have annual calibration

Reactivity could have either the two quantitative tests performed depending on the source of the waste and/or governing regulations.

The following table lists the precision and accuracy requirements for the load and profile analysis parameters which are used as support tests. Completeness which states "as required" indicates the result is not required on all loads.

| Measurement Parameter | Reference | Experimental Conditions | Precision | Accuracy | Completeness |
|---|---|---|---|---|---|
| Specific Gravity (5) | SM 213E<br>9010 | Industrial Waste Samples | ±20% | ±20% | 90% |
| Normality | USPCI<br>1 | Industrial Waste Samples | ±30% | ±30% | 100%<br>as required |
| Solids | USPCI<br>3 | Industrial Waste Samples | ±10% | ±10% | 90% |
| BTU | ASTM<br>D-240-85 | Industrial Waste Samples | ±20% | ±20% | 90%<br>as required |
| PCBs | SW 846<br>8080 | Industrial Waste Samples | ±25% | ±25% | 100%<br>as required |

## 9.4 SAMPLING PROCEDURES

The main goal in a chemical analysis is to make a measurement of some property of any article. Decisions are then 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 universe concern.

### 9.4.1 INITIAL CONSIDERATIONS

The first item is to protect personnel from possible exposure to hazardous material. Most of the incoming loads contain some level of harmful matter. At a minimum, the sampling personnel will wear safety glasses, rubber gloves, and boots. Additional protective items will be required as dictated by the suspected contaminants involved. Sampling personnel are 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.

The next item is to define what is to be sampled; liquid, solid; homogenous or irregular. This will dictate what type of sampler and procedure is to be used. There are four basic types of samplers used for collecting waste load samples:

Coliwasa
Probe
Thief
Trier

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

1. Prior to sampling, inspect the apparatus in insure it is clean. If 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 insuring 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 is used to collect a sample of free flowing liquids. The coliwasa is especially useful for liquids that form a number of phases. One coliwasa is used for sampling oils and a second is used to sample other liquids. Incoming loads of sludge are sampled with a coliwasa as follows:

1. Prior to sampling, inspect the apparatus to insure it is clean. If 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 coliwasa, withdraw the sample and transfer it into sample container. Insure adequate volume is collected.
6. Rinse coliwasa at the sample rinsing station.

**9.4.1.3 MOIST GRANULES/SOLIDS** - Composite samples of moist granules and solids are collected with a trier. This type of sampler is made by cutting a tube in half length wise and sharpening the tip so that it allows the ampler 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 to container. Repeat 2 more times.
4. Collect a sample and transfer contents of trier to sample container.
5. Clean debris from trier and return to sampler storage.

**9.4.1.4 DRY POWDERS/GRANULES** - Composite samples of dry powders or granules are 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.

## 9.5 SAMPLE CUSTODY

### 9.5.1 PREACCEPTANCE SAMPLES

Upon arrival, the preacceptance sample is entered into the sample log and assigned a "GM" number. This number is used to track the load information through the record keeping system. The sample container and any analytical results are to have this GM number to help track the information about the sample.

### 9.5.2 LOAD SAMPLES

Upon arrival, the hauler presents receiving with the pertinent documentation. The load is entered onto the load log and assigned a load number. This number is conjunction with the GM number 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

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

## 9.6 CALIBRATION PROCEDURES AND FREQUENCIES

### 9.6.1 CALIBRATION

Any instrumentation used to generate data must be calibrated to assure that their accuracy is within acceptable limits. The calibration must be done with appropriate standards. The laboratory analyst assures that the proper standard is used to meet the requirements of the test.

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

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

### 9.6.2 TOX ANALYZER

**9.6.2.1 GENERAL OPERATION-** The Mitubishi TOX 10 analyzer is used for the analysis of total organic halides. It will detect chlorine, bromine, and iodine, but not fluorine. It is calibrated using 2,4,6-Trichlorophenol and results are reported as μg/Chlorine.

The manufacturer's manual will be consulted for complete operating instructions. Maintenance schedules as recommended by the manufacturer will be followed as applicable. Maintenance will be properly recorded in the maintenance logbook.

The instrument requires oxygen (prepure) and Argon (prepure) for operation.

**9.6.2.2 INITIAL CALIBRATION** - The instrument requires no initial calibration since it is recalibrated each day of operation.

**9.6.2.3 CONTINUING CALIBRATION** - A single point calibration is run at the beginning of each analytical sequence. Immediately following calibration a Laboratory Control Standard (LCS) is analyzed. The concentration value for the LCS must be with the tabulated acceptance window or ±15% the true value if no windows have been established.

During analysis the calibration standard is analyzed after every 10th sample. If the measured concentration differs from the expected value by more than 10%, the instrument is recalibrated, ant the previous 10 samples are reanalyzed.

**9.6.2.4 DOCUMENTATION** - All worksheets and printouts are part of the analytical records. They should be clearly labeled identifying date, analyst, analyte and the identity of each run.

### 9.6.3 pH/SELECTIVE ION METERS

The pH meter will be standardized at at least two pH levels (generally pH 4 and pH 7). The calibration will be checked with another buffer (such as 10; 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 of operation. The reading should be ±.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 cleanings 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.

### 9.6.4 UV/VIS SPECTROPHOTOMETERS

The Spec 20 is the only UV/Vis spectrophotometer currently in use at the laboratory. It will be run according to the procedure recommended in the manufacturer's manual.

All absorption cells must be kept clean, free of scratches, fingerprints, smudges and evaporated film residues.

Any maintenance items recommended in the manufacturer's manual will be performed and documented as required.

Calibration of the spectrophotometer will be in accordance with the method being run, with a minimum of five standards and a reagent blank. Calibration curve will be re-run with every set of 10 samples (excluding QC samples).

### 9.6.5 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 is 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 logbook. Any service required will be recorded in the same logbook in the section reserved for the particular instrument in question.

### 9.6.6 HYDROGEN SULFIDE ANALYSIS

The $H_2S$ test is performed weekly using a Drager tube (range 100-2000 ppm) and a control sample of $Na_2S$ is placed in a disposable 55ml cup. 10ml of $HNO_3$ is added and the gas is allowed to evolve for 10 seconds. A reading is taken (this tube required 1 pump of the sampler) and recorded. The reading should be approximately 500 ppm ±20%.

### 9.6.7 TLV SNIFFER

The TLV sniffer is checked with a calibration gas supplied by the manufacturer (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%.

### 9.6.8 FLASHPOINT

The flashpoint is 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). If performed on the Pensky-Martens, fill the cup to the mark and report the actual flash point.

### 9.6.9 TOTAL SOLIDS

The solids are determined on an Ohaus Moisture balance. On a weekly basis 10.0 grams of a mixture of 70% fine sand and 30% water is created and added to the balance. The timer is set to 10 minutes. Record the weight. Set the timer for 5 more minutes. If after 5 minutes the weight has changed more than 5% from the first weight, this indicates incomplete drying. Set for another 5 minutes and recheck until weight is constant. The balance reads <u>% moisture</u>. Subtract this value from 100 to get % solids. Record this value.

Place a thermometer on the pan. Set the timer for 10 minutes. Check the temperature at the end of 10 minutes. The temperature should not be above 105°C ±5°C. If it is, turn the watts control down and recheck until the temperature is within the required specification.

### 9.6.10 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.

### 9.6.11 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 METHODOLOGY

Specific Methods are outlined in Section 10.

## 9.8 DATA REDUCTION, VALIDATION, AND REPORTING

### 9.8.1 DATA REDUCTION

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
- 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.

## 9.9 INTERNAL QUALITY CONTROL CHECKS

The laboratory QC checks are in accordance with generally accepted good laboratory practices. However, the laboratory reserves the right to utilize alternative practices which meet or exceed the listed requirements after the Executive Secreatry's approval.

### 9.9.1 METHODOLOGY

The laboratory uses only analytically sound methods which are, or are based on those recognized by source such as the EPA, AOAC, or ASTM.

### 9.9.2 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 verses the amount added is called recovery.

Each method should be tested using method spikes. This involves the analysis of a blank 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.

After a sufficient number of recoveries for a given sample matrix have been accumulated, control limits will be established (see Section 9.12).

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.

### 9.9.3 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.

Replicates 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.

After a sufficient number of replicates for a given sample matrix have been accumulated, control limits will be established (see Section 9.12).

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.

### 9.9.4 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/interferent free material.

Blanks are to be run with each sample batch or 1 for every 10 samples which ever is more frequent.

Analyte concentration in the blank should not exceed 2 times the method detection limit.

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.

### 9.9.5 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 which it is compared to. 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) from NBS, EPA, or another source which can be traced back to NBS or EPA.

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

Inhouse calibration standards must agree with reference material within 10%. Data documenting this fact will be kept on file. Standards which exceed this limit will be discarded.

## 9.10 QUALITY ASSURANCE PROGRAM REVIEW

### 9.10.1 PERFORMANCE AUDITS

These audits 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 laboratory QA chemist on a quarterly basis.

Major defects (e.g. a finding of 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 applied.

### 9.10.2 SYSTEM AUDITS

System audits 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 Facility Manager or his designee at least semi-annually.

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

### 9.11 PREVENTIVE MAINTENANCE

The manufacturer's manual will be consulted for complete operating instructions. Maintenance schedule as recommended by the manufacturer will be followed as applicable. Maintenance will be properly recorded in the instrument maintenance logbook.

The laboratory will keep backup instruments for certain tests where the cost of the instrument permits. In the event cost is prohibitive, monies will be provided for emergency repairs and/or replacement.

All backups will be calibrated in accordance with appropriate procedures before put into service.

### 9.12 ROUTINES TO ASSESS DATA PRECISION, ACCURACY AND COMPLETENESS

If at any point and time USPCI feels they have sufficient data to convert to standard deviation, they may petition for approval.

### 9.12.1 METHOD SPIKE RECOVERIES

Method spike recoveries will be used when testing new procedures and training new analysts. The spikes should be made at levels appropriate for the analysis (approximately ten times the estimated detection limit). The recovery shall be within the 95% confidence interval ± 2s) for historical data (0-10 years) or ± 25% in lieu of historical data.

Matrix spike samples will be analyzed along with sample batches to estimate the accuracy of the analysis. The recovery shall be within the 95% confidence interval (+ 2s) for historical data.

### 9.12.2 REPLICATES (INCLUDES MATRIX SPIKE DUPLICATES)

Replicate will be analyzed along with sample batches to estimate the precision of the analysis. In lieu of historical data, the Relative Percent Difference (RPD) shall be equal to or less than 20% (providing the results are at least 10 times greater than analytical detection limit). If more than two replicates are analyzed (such as triplicates) the Relative Percent Standard Deviation will be computed. Likewise, a 95% confidence interval will be established for this parameter.

### 9.12.3 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 be set per method specifications.

### 9.12.4 GENERAL LABORATORY PARAMETERS

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

## 9.13 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 recaluculated), if necessary. Data immediately preceeding the cause or event must be checked to confirm precision and accuracy.

### 9.13.1 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.

### 9.13.2 SPIKE RECOVERIES

Percent recoveries for matrix spikes and/or surrogate spikes are monitored by the analysts 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. In lieu of historical data, when sufficient data has been collected, USPCI may utilize new control limit criteria after approval of the executive secretary.

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, matrix interference can be assumed.

### 9.13.3 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.

### 9.13.4 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.

### 9.14 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.

# SECTION 10

# UNIQUE ADDITIONAL ANALYSIS PROCEDURES

# USPCI ANALYTICAL PROCEDURE 1

## (USPCI - 1)

## NORMALITY

# USPCI ANALYTICAL PROCEDURE 1

## (USPCI - 1)

## NORMALITY

## (Acidity or Alkalinity)

## 1.0 SCOPE AND APPLICATION

1.1 This method is for determining normality (acidity or alkalinity) of hazardous waste samples that are acidic or caustic liquids.

1.2 It should be noted that the data generated from normality measurements are used at the U.S.P.C.I. Grassy Mountain facility for two purposes. One is as one part of a fingerprint procedure to ensure that pre-shipment samples of waste streams are representative of the loads actually sent. A difference ± 1.25 normality units is considered a discrepancy. The other purpose is to give an indication of the volumes of acidic and caustic waste streams that must be mixed together for neutralization before disposal. Neither of these purposes require accurate values for low normality samples. Therefore, the procedure which is used and given here is one that gives sufficient accuracy for high normality samples.

## 2.0 SUMMARY OF METHOD

2.1 The liquid waste is titrated with a titrant that is the opposite pH (e.g. titrate an acidic waste with a caustic titrant). Solids are added to a nominal volume of DIW for titration, if desired. The normality is calculated by using the amounts of the sample and titrant and the known normality of the titrant. The end point of the titration is determined by a calibrated pH meter.

## 3.0 INTERFERENCES

3.1 Response times for glass pH electrodes may be slowed by oil films on the electrode.

## 4.0 SAFETY

4.1 Wear appropriate gloves and safety glasses when handling acids and caustics.

4.2 Prevent spills and splashes. Wash areas (if spill occurs) thoroughly with water.

4.3 If sample has extremely high normality, splattering may occur when titrating. Therefore, analysis should be performed in the hood.

4.4 Do not breath vapors; keep samples in the hood.

## 5.0 APPARATUS AND EQUIPMENT

5.1 Buret - Pyrex or Kimar, 25 ml, with divisions of 0.1 ml, or equivalent. One each for acid and base titrants.

5.2 10 ml and 50 ml disposable polystrene beakers.

5.3 pH meter

## 6.0 REAGENTS

6.1 pH buffers: Baker Analyzed 5657-1 (pH 4), 5655-1 (pH 10), 5656-1 (pH 7), or equivalents.

6.2 Concentrated hydrochloric acid, 12 N: Baker Analyzed Reagent 9535-3, or equivalent.

6.3 Hydrochloric acid, 3 N: Add slowly, while stirring, 258 ml of HCL acid (6.2) and dilute to 1 liter.

6.4 Sodium Hydroxide, 3 N: Dissolve 120 g of NaOH (6.5) in 800 ml of Type I water while stirring. Dilute to 1 liter.

6.5 Sodium Hydroxide: Mallinckrodt AR# 7708-5, or equivalent.

## 7.0 SAMPLE HANDLING AND PRESERVATION

7.1 Handle sample with extreme caution; wearing gloves and glasses.

7.2 No preservation required.

7.3 If sample fumes, store in refrigerator at 4°C.

## 8.0 pH - METER CALIBRATION AND STANDARDIZATION

8.1 Make up pH 4 and pH 7 buffer solutions by emptying the complete sachet contents into beakers and dissolving in the stated volume of distilled water. Pre-mixed and certified buffers may also be used.

8.2 Calibrate pH meter according to manufacturer's instructions. Record calibration settings in instrument log.

## 9.0 QUALITY CONTROL

9.1 Duplicates. For preacceptance samples, at least one duplicate must be analyzed per sample set or 10% of total samples. For incoming load samples, at least one duplicate analysis set must be analyzed per shift.

9.2 The normality of the titrants are checked against a primary standard on a regular basis.

9.3 Control Charts

9.3.1. Precision control charts for monitoring of relative percent difference of duplicate analysis are maintained.

9.3.2. Accuracy control charts for the normality checks are maintained.

## 10.0 PROCEDURE

10.1 Use a 10 ml disposable beaker to measure 10 ml of a liquid sample into a 50 ml disposable beaker. If the sample is solid, add 1.0 gm to 10 ml of DIW.

10.2 Stir gently with the calibrated and rinsed pH probe. Record the initial pH for the pH part of the fingerprint analysis.

10.3 For samples with pH greater than 10.5 or less than 4.5 a titration with 3 N acid or base, respectively, titrant is done. Titrate slowly with continuous stirring until the pH reaches 7.0.

10.4 Read and record the volume of titrant used.

10.5 Samples of high normality can use a smaller sample aliquot as required.

## 11.0 CALCULATIONS

11.1 Normality of sample =

$$\frac{\text{Normality of Titrant} \times \text{Volume of Titrant (ml)}}{\text{Volume of Sample (ml or gm)}}$$

11.2 Duplicate calculation

$$\% \text{ Difference} = \frac{(D_1 - D_2) \times 200}{D_1 + D_2}$$

Where: $D_1$ = first sample value
$D_2$ = second sample value

## 12.0 DATA FLAGGING AND REMEDIAL ACTION

12.1 Data will be flagged by the analyst if data generated creates an "out-of-control" situation on the Precision or Accuracy Control Chart.

12.2 Remedial action

12.2.1 When data is flagged, the following areas are reviewed by the analyst and/or supervisor:

12.2.1.1 Calibration and standardization.

12.2.1.2 Analysis trends as indicated by control charts.

12.3 When a problem is located, sample analysis is repeated.

## 13.0 REFERENCES

Chemistry, 2nd Edition, Yoder, Claude H, Snydam, Fred H, Snavely, Harcourt Brace Jovanovich, Inc., 1980, 1975.

Standard Methods for the Examination of Water & Wastewater, 16th Edition, American Public Health Association, American Water Works Association, Water Pollution Control Federation, 1985.

# USPCI ANALYTICAL PROCEDURE 2

## (USPCI - 2)

## WATER REACTIVITY SCREEN

## USPCI ANALYTICAL PROCEDURE 2

## (USPCI - 2)

## WATER REACTIVITY

### 1.0 SCOPE AND APPLICATION

1.1 This method is used to screen materials for violent reactions with water.

### 2.0 SUMMARY OF METHOD

2.1 Sample is slowly added to water until a 50/50 volume/volume mixture is obtained. The mixture is observed to detect heating (more than a 15°C temperature rise) or turbulent gas evolution (more than 10% of the mixture volume).

### 3.0 SAFETY

3.1 Always add sample slowly to water, not water to sample.

3.2 Wear appropriate gloves and safety glasses.

3.3 Perform the mixing in a hood to prevent gases evolved from entering the laboratory.

### 4.0 PROCEDURE

4.1 Pour 25 ml of water into a disposable 50 ml beaker. Slowly add sample until the beaker reaches the 50 ml level.

4.2 If the mixture warms significantly, usg a thermometer to check temperature. If it is more than 15°C above the initial water sample temperature the sample is considered to be water reactive.

4.3 If bubbles or gas is formed causing turbulence, the sample is also considered to be water reactive due to gas evolution.

4.4 If sample is water reactive due to temperature rise and the sample has a large enough acid or base normality to account for temperature rise due to acid or base dilution, the sample is noted to be water reactive due to acid or base dilution.

4.5 If the reaction is questionable, the amount of sample is scaled up with 10 times the amount of water and re-tested.

## 5.0 QUALITY CONTROL

5.1 Duplicates. For preacceptance samples, at least one duplicate must be analyzed per sample set or 10% of total samples. For incoming load samples, at least one duplicate analysis set must be analyzed per shift.

5.2 Because this test yields a "yes" or "no" answer, regular quality control charts will not be kept. All discrepancies between duplicate samples must be explained and noted.

# USPCI ANALYTICAL PROCEDURE 3

## (USPCI - 3)

## SOLIDS SCREEN

# USPCI ANALYTICAL PROCEDURE 3

## (USPCI - 3)

## SOLIDS SCREEN

### 1.0 SCOPE AND APPLICATION

This is a rapid and accurate method for determining the total solids of liquids, sludges and solid sample that are dried to maximum weight loss at approximately 105°C.

### 2.0 SUMMARY OF METHOD

10 grams of sample are accurately weighed and dried on a moisture balance. Weights before and after drying are compared to calculate % solids.

### 3.0 INTERFERENCES

Underheating and/or inadequate drying time will not remove all components normally volatilized at 105°C. Adequate heater and timer settings are developed for each type of sample to prevent these interferences.

### 4.0 SAFETY

4.1 This method should not be used with explosives or ignition could result.

4.2 Appropriate gloves and safety glasses should be worn while handling samples.

4.3 This method should e performed in the hood to prevent volatile compounds from entering the laboratory atmosphere.

### 5.0 APPARATUS AND EQUIPMENT

Ohaus Moisture Determination Balance Model 6010 with aluminum sample pans.

### 6.0 QUALITY CONTROL

6.1 Duplicates. For preacceptance sample, at least one duplicate must be analyzed per sample set or 10% of total samples. For incoming load samples, at least one duplicate analysis set must be analyzed per shift.

6.2 Control Samples are run on a regular basis.

6.3 Qaulity Control Charts are kept for monitoring precision (duplicates), and accuracy (control samples).

## 7.0 PROCEDURE

7.1 Approximately 10 grams of well mixed sample are accurately weighed onto a tared aluminum sample pan on the moisture balance. This weight is the initial weight.

7.2 The temperature setting is checked weekly when the control sample is run. The setting will be recorded on the control chart log. This will be the setting used each week. Set the timer setting at 10 minutes. Check sample at end of time, if free liquids are still present heat an additional 5 minutes.

7.3 % total solids is calculated using the formula:

$$\% \text{ total solids} = \frac{\text{Final Weight} \times 100}{\text{Initial Weight}}$$

## 8.0 REFERENCES

Instructions for Ohaus Moisture Detention Balance Model 6010, 1982.

# USPCI ANALYTICAL PROCEDURE 4

## (USPCI - 4)

## REACTIVE CYANIDES AND REACTIVE SULFIDES SCREEN

# USPCI ANALYTICAL PROCEDURE 4

## (USPCI - 4)

## REACTIVE CYANIDES AND REACTIVE SULFIDES SCREEN

### 1.0 SCOPE AND APPLICATION

This method provides a rapid qualitative test to determine the potential for samples to generate HCN or $H_2S$ upon acidification.

### 2.0 SUMMARY OF METHOD

A small amount of sample is acidified to pH $\leq$ 2 using nitric acid and the atmosphere above the sample is tested using Drager detector tubes for hydrogen cyanide and hydrogen sulfide.

### 3.0 SAFETY

3.1 Wear appropriate glove and safety glasses.

3.2 This test must be performed in a hood to prevent poisonous HCN and/or $H_2S$ from escaping into the lab atmosphere.

### 4.0 INTERFERENCES

According to the Drager tube handbook there are no interferences that prevent sensing of HCN, however it can be found from time to time that unknown substances will cause the tube to turn an orange rather than the tell-tale blood red. Also, the white from part of the tube will turn black ln the presence of $H_2S$. Sulphur dioxide may increase the measured concentration value of $H_2S$, but will not prevent $H_2S$ from being detected.

### 5.0 PROCEDURE

5.1 Approximately 25 ml of sample is placed in a disposable 50 ml beaker and acidified with 3 normal nitric acid until the pH is 2.0. Samples with initial pH values at or below 2.0 need not be acidified further.

5.2 While the ample is being acidified, the atmosphere directly above the sample is tested using a Drager gas detector. Sample tube Hydrogen Cyanide 2/a is used for HCN detection and sample tube Hydrogen Sulfide 100/a is used for $H_2S$ detection. The CN tube needs five pumps; the Sulfide tube one.

5.3 A reading of 125 on the HCN Drager tube will require qualitative analysis using EPA Method SW-846-9010. A reading of 250 on the $H_2S$ Drager tube will require qualitative analysis using EPA Method SW-846-9030. The values of 125 and 250 are one half (12) the values of cyanide and sulfide, respectively before additional treatment or alternate handling methods may be required.

## 6.0 QUALITY CONTROL

6.1 Duplicate samples are run at least once every set of 10 pre-acceptance samples.

6.2 Discrepancies (positive vs. negative results) between duplicate samples must be explained.

# USPCI ANALYTICAL PROCEDURE 5

## (USPCI - 5)

## EXPLOSIVITY METER VAPOR TEST (TLV SNIFF)

# USPCI ANALYTICAL PROCEDURE 5

# (USPCI - 5)

# EXPLOSIVITY METER VAPOR TEST (TLV SNIFF)

## 1.0 SCOPE AND APPLICATION

The TLV Sniffer is an extremely sensitive combustible gas and vapor sensing instrument; equipped with an audible alarm that can be set to sound at any desired level of gas concentration. The TLV is also useful for locating gas leaks. Another function is continuous self monitoring.

## 2.0 SUMMARY OF METHOD

To detect and measure concentrations of combustible gas in the air, the TLV Sniffer catalytically oxidizes gas in a pumped in sample of air by means of a catalyst-coated resistance element. The resistance of this element changes with changes in heat that are proportional to the amount of oxidized gas, thereby altering the electrical balance of the catalytic element as compared to the resistance of a reference element. Both the catalyst-coated ("active") element and the reference element are incorporated in a Wheatstone Bridge circuit in guch a way as to produce an electrical output proportional to their differences in resistance. Since any changes in air sample temperature and humidity affect both active and reference elements equally the electrical signal output is proportional to the concentrations of combustible gas or vapor in the sample of air (expressed in volumetric teams as ppm). However, sudden changes in humidity may affect the zero reading on the X 1 range. The instrument, therefore, should be zeroed at the same R.H. prevailing during use.

## 3.0 INTERFERENCES

3.1 Improper calibration of instrument or setting meter zero in the presence of impure air will cause inaccurate readings.

3.2 Wisps of cigarette smoke, fumes from autos, and subtle air contaminations from other sources may affect zero setting.

## 4.0 SAFETY

If high volumes of gas are detected or suspected, a respirator should be worn. No flames or sparks should ever be present.

## 5.0 APPARATUS AND EQUIPMENT

5.1 TLV Sniffer - Bacharach by United Technologies, or equivalent.

5.2 Gas Calibration Kit - Bacharach, Code 51-7199, or equivalent.

## 6.0 REAGENTS

None

## 7.0 SAMPLE HANDLING AND PRESERVATION

Keep sample container tightly sealed. **DO NOT** open until starting analysis. If highly volatile, refrigerate sample at 4 degrees Celsius.

## 8.0 CALIBRATION AND STANDARDIZATION

8.1 Battery test:

Turn MODE SELECTOR knob from OFF position to BATT TEST position. Meter pointer should come to rest in BATTERY GOOD range of meter scale. (Both a meter reading below BATTERY GOOD range and an audible signal warn of batteries too weak to sustain normal operation).

8.2 Setting meter pointer to zero:

8.2.1 Attach air sampling probe connector to instrument intake on left side of case by pulling back spring collar of connector, pressing connector over intake, and releasing spring collar.

8.2.2 Place TLV Sniffer in position in which meter indications will be read (usually in meter up position).

NOTE: Heat distribution from active and reference filaments of the detector sensor changes from vertical to horizontal position. The resulting change in electrical balance between elements causes a shift in pointer zero from one position to the other.

8.2.3 Set MODE SELECTOR switch to ppm x 100 and operate instrument for 10 minutes to allow circuits to stabilize.

8.2.4 In fresh air, set ZERO ADJUST knob at midpoint (five full turns from either extreme position). If fresh air is not available, use Bacharach Kit 51-7199 to apply known pure air to the Sniffer intake (instructions in kit).

8.2.5 Turn coarse adjustment screw, located under ZERO ADJUST knob, to move meter pointer to zero on the meter scale.

8.2.6 Turn MODE SELECTOR to ppm x 10 position and turn ZERO ADJUST knob to set pointer to zero.

8.2.7 Turn MODE SELECTOR to ppm x 1 position and turn ZERO ADJUST knob to set pointer to zero.

NOTE: The TLV Sniffer is extremely sensitive in the ppm x 1 range. $CO_2$ from breath too close to the intake, cigarette smoke, auto fumes, etc., can interfere with accurate setting of the pointer to meter zero.

8.3 Setting meter pointer deflection (gain calibration).

To insure proper operation and to check calibration, it is necessary to periodically check the instrument against a known standard blend of calibration gas.

The Bacharach Code 51-7199 gas calibration kit and optionally available Code 51-1120 rectified gas cylinder containing 500 ppm hexane in air are readily available to meet this requirement.

Connect the gas transfer assembly, making certain all connections are air tight. Use the retaining clips (2 each) to mount Flowmeter (06-6163) to its mounting bracket (51-1201). Make certain to connect rubber tubing at the base inlet connection on the flowmeter, then to the barbed fitting on the regulator and to the quick connect fitting previously installed on the TLV sample in (inlet fitting). Turn regulator valve (03-4318) fully counterclockwise (closed position) before attempting to screw regulator into calibration gas tank. This test is to be performed in a clean, fresh air (combustible free) environment. If this is not possible, substitute Code 51-7131 zero calibration gas for the Code 51-1120 cylinder of hexane-air mixture.

Connect the gas transfer assembly at the TLV sample in (inlet) fitting.

Open the regulator valve (clockwise) and adjust for flowmeter indication of (1) cfh to ensure adequate pump flow.

Remove Code 51-7131 zero calibration gas and substitute the Code 51-1120 cylinder of hexane-air mixture before proceeding with Step 6.

To calibrate the instrument in fresh air (combustible free) environment, proceed as follows:

8.3.1 Remove case cover for access to internal adjustments and temporarily break gas transfer assembly connection at the TLV Sample-In (inlet) fitting.

8.3.2 Turn FINE ZERO ADJUST (pot) full clockwise and then five turns counterclockwise to mid-range. Then turn COARSE ADJUST (pot) full clockwise and ten turns counterclockwise to mid-range.

8.3.3 Turn MODE SELECTOR to BAST TEST position. The meter pointer must indicate within BATTERY GOOD range, if not recharge.

Connect a Voltmeter between TP-3 (+) and ground (-), check for 6 VDC. If not, adjust for 6 VDC ± 0.01 VDC.

8.3.4 After allowing for five minute warm up, turn MODE SELECTOR switch to ppm x 100 position and adjust R-13 for meter pointer indication of scale zero.

8.3.5 Turn MODE SELECTOR switch to ppm x 10 position and adjust COARSE ADJUST or meter pointer indication of scale zero. Readjust per steps 4 and 5 until meter pointer indicates a relatively constant scale zero when MODE SELECTOR is switched between ppm x 100 range.

8.3.6 Turn MODE SETFCTOR switch to ppm x 10 position. Reconnect gas transfer assembly to TLV sample in (inlet) fitting. Open regulator valve (clockwise) and adjust for flowmeter indications of (1) cfh to ensure adequate pump flow. Allow one minute for meter pointer to achieve maximum indication, adjust R-3 the x 10 span adjuster until meter pointer indicates mid-scale (50) or 500 ppm. Remove gas, close regulator valve (fully CCW) and allow about two minutes for meter pointer to return to zero.

8.3.7 Turn MODE SELECT switch to ppm x 10 position. Then turn the FINE ZERO ADJUST until meter pointer indicates full scale 1000 ppm. Turn MODE SELECT switch to ppm x 100 position and adjust R-4 the x 100 span adjuster until meter pointer indicates scale zero.

8.3.8 Turn MODE SELECT switch to ppm x 10 position, then turn FINE ZERO ADJUST until meter pointer indicates 10 on the scale or 100 ppm.

8.3.9 Turn MODE SELECT switch to ppm x 1 position and adjust the x 1 span adjuster until meter pointer indicates 100 (full scale) or 100 ppm.

8.3.10 Turn FINE ZERO ADJUST until meter pointer indicates scale zero, the TLV is now calibrated and ready for use on the low range 0-100 ppm as a gas leak detector.

8.4 Resetting alarm response. If factory set alarm response at midpoint of the meter scale is not suitable, reset alarm response level as follows:

8.4.1 Turn meter zero coarse adustmet screw (located under ZERO ADJUST control knob at lower left on instrument panel) to set meter pointer to desired alarm point on meter scale.

8.4.2 Turn ALARM potentiometer adjustment screw until audible alarm sounds.

8.4.3 Turn meter zero coarse adjustment screw to return pointer to zero on meter scale.

8.5 Setting recording level. If recorder (range: 0-100 mv; impedance: 10,000 ohms or greater) is to be used, attach accessory recorder ack to RECORDER plug in right side of instrument case and set recording level as follows:

8.5.1 Set MODE SELECTOR knob to ppm x 100 or ppm x 10 as desired and apply combustible gas to instrument intake.

8.5.2 Turn RECORDER potentiometer adjustment screw until accessory recorder response corresponds with meter readings as desired.

## 9.0 QUALITY CONTROL

Calibration of the unit should be verified each day.

9.5.3 Duplicate samples are tested in each pre-acceptance sample batch or every 10 samples, whichever is more frequent. A quality control chart is kept on the duplicate sample values.

## 10.0 MONITORING TOXICITY

10.1 Monitor combustible gas and vapor to determine concentrations with respect to Threshold Limit Values as follows:

10.1.1 Turn MODE SELECTOR control knob to BATT TEST position and read condition of battery on meter dial. Install new recharged batteries, if necessary.

10.1.2 Turn MODE SELECTOR control to desired operating range, selected in accordance with the Threshold Limit Value for the toxic gas to be monitored (ppm x 1 for TLV from 0 to 100 ppm; ppm x 10 for TLV from 0 to 1,000 ppm; ppm x 100 TLV from 0 to 10,000 ppm).

10.1.3 Allow ten minute warm-up period with instrument in same position as it is to be used in service (meter facing up or meter facing to the side).

10.1.4 In fresh air before entering monitoring area, turn ZERO ADJUST control knob until meter pointer resets on zero.

10.1.5 For monitoring in noisy areas, insert jack of accessory earphone in plug on right side of instrument case.

10.1.6 Enter monitoring area and read ppm gas concentrations on meter. Audible warning sounds if gas concentration causes readings at midpoint of scale or above, or if toxic Threshold Limit Value has been exceeded, provided the alarm has been set for this response.

10.1.7 For readings above 10,000 ppm: Replace probe assembly 0023-7243 with dilution probe 0023-7355 and slide dilution probe O-ring to expose dilution holes of probe (extends range 10 x to read up to 100,000 ppm). Add in line filter and trap assembly, if sampling in dust or moisture laden areas.

10.2 Converting Hexane-calibrated meter ppm readings to ppm readings for other gases. Hexane gas is commonly used for facotry calibration and subsequent in service recalibrations of the TLV Sniffer. To determine ppm concentrations of gases other than hexane with instruments calibrated for hexane, multiply the ppm meter reading by the factor for the gas detected.

10.3 Converting ppm readings to percent level of lower explosive limit (% L.E.L.). To determine gas concentration levels in terms of percent of lower explosive limit from direct ppm readings for hexane or from calculated ppm concentration levels for other gasses:

10.3.1 Read ppm on TLV Sniffer indicating meter.

10.3.2 On 0-to-10,000 "ppm concentration in sample" horizontal scale at bottom of % L.E.L. Conversion chart (attached), locate position left to right representing ppm reading.

10.3.3 On slanted chart line representing kind of gas detected, find the point in vertical alignment over ppm reading point on horizontal scale.

10.3.4 On vertical scale at left labeled % L.E.L. Equivalent, read the percent of lower limit equivalent found in horizontal alignment with the point located on the slanted line.

10.4 Locating gas leak sources. To utilize the TLV sniffer is searching for gas leaks in tanks, pipes, hoses, containers, etc.:

10.4.1 Set MODE SELECTOR control knob to ppm x 1 position.

10.4.2 Search for exact location of leak with probe. Meter reading will increase as leak is approached and decrease as probe moves away from leak.

10.5 TLV sniff test procedure for sample fingerprint analysis.

10.5.1 The TLV sniffer probe is held over the surface (within 0.5 cm) of the sample. A positive reading indicates the possibility of volatile organics in the sample.

10.5.2 A reading over 200 ppm indicates the possibility of flammability and a flash point analysis is performed to test for flammability.

10.5.3 Quality Control

10.5.3.a Duplicate samples are tested in each sample batch or every 10 samples, whichever is more frequent.

10.5.3.b A quality control chart is kept on the duplicate sample values.

## 11.0 CALCULATIONS

TLV = ppm reading x scale

## 12.0 DATA FLAGGING AND REMEDIAL ACTION

12.1 Data will be flagged if:

12.1.1 Data generated creates an "out of control" situation on the precision control chart.

12.2 Remedial Action

12.2.1 When the data is flagged, the following areas are reviewed by the analyst and supervisor:

12.2.1.1 Analysis trends as indicated by control charts.

12.3 When a problem is located sample analysis is repeated.

## 13.0 REFERENCES

Instruction Manual TLV Sniffer, United Technologies Bacharach, Instruction 23-9613, Rev. No. 1, September, 1982.

# USPCI ANALYTICAL PROCEDURE 6

## (USPCI - 6)

## OXIDIZER SCREEN

## USPCI ANALYTICAL PROCEDURE 6

## (USPCI - 6)

## OXIDIZER SCREEN

### 1.0 SCOPE AND APPLICATION

This method is a rapid qualitative method for determining the presence of oxidizing materials in liquid and sludge samples.

### 2.0 SAFETY

2.1 Wear appropriate gloves and safety glasses when handling hazardous samples.

2.2 Perform analysis in the hood to prevent contact with sample vapors.

### 3.0 PROCEDURE

Wet a strip of KI - starch paper in HCl. Dip the wetted strip into the sample. Note the color that develops. Anywhere from light brown to dark purple or black indicates that oxidizing material is likely present. Light brown is generated on contact with nitric acid and deep purple forms on contact with hydrogen peroxide.

### 4.0 QUALITY CONTROL

At least one duplicate must be analyzed per sample set or for every 10 samples, which ever gives the greater frequency.

# USPCI ANALYTICAL PROCEDURE 7

## (USPCI - 7)

## RADIOACTIVITY SCREEN

# USPCI ANALYTICAL PROCEDURE 7

# (USPCI - 7)

# RADIOACTIVITY SCREEN

## 1.0 SCOPE AND APPLICATION

This method is to detect the presence of any radioactive material in a representative sample of waste.

## 2.0 SUMMARY OF METHOD

To detect and measure the presence of radioactivity in a sample it will be placed within six inches of a scintillation detector. A scintillation detector is capable of measuring low-level gamma radiation in micro R/hr. No waste with a reading 40 micro R/hr. above background will be accepted without authorization acceptance from the Utah Bureau of Radiation Control.

## 3.0 INTERFERENCE

No known interferences.

## 4.0 SAFETY

Treat all samples as if hazardous. Wear appropriate gloves, safety glasses, and lab coat. The sample container does not have to be opened to perform the test.

## 5.0 APPARATUS AND EQUIPMENT

Ludlum Model 19 Micro R Meter, or equivalent.

## 6.0 REAGENTS

None required.

## 7.0 SAMPLE HANDLING AND PRESERVATION

No preservation is needed. Keep sample tightly sealed. Place entire sample within six inches of the detector.

## 8.0 CALIBRATION AND STANDARDIZATION

The meter is to be recalibrated annually by the manufacturer.

## 9.0 QUALITY CONTROL

None.

## 10.0 PROCEDURE

10.1 Prior to turn-on, place the response switch in the S (slow) position and place audio switch in the off position.

10.2 Turn-on the meter by placing meter on the 0 to 50 micro R/hr scale.

10.3 Depress the BATT Test Button. If the meter pointer is below the check line replace the meter's batteries.

10.4 Depress the R (reset) Button. Check to see if meter pointer returns to Zero.

10.5 The meter is ready for use. Allow the meter to return to background activity approximately 10 to 20 micro R/hr Response time should be 10 to 15 seconds.

10.6 Place sample within six inches of the detector located in the front of the meter. Allow 10 to 15 seconds for meter response. If reading is less than 40 micro R/hr above background the test is negative. Any readings which are greater, the General Manager or Lab Manager will be notified.

## 11.0 CALCULATIONS

The meter is a direct readout. Ensure meter is set on the proper scale.

## 12.0 PRECISION AND ACCURACY

No historical data is available at this time.

## 13.0 DATA FLAGGING AND REMEDIAL ACTION

13.1 Data will be flagged by the analyst if readings exceed 40 micro R/hr above background.

## 14.0 REFERENCES

Instruction Manual for Ludlum Model 19 MICRO R Meter, Ludlum Measurement Inc., Sweetwater, Texas.

# USPCI ANALYTICAL PROCEDURE 8

## (USPCI - 8)

## FIXATION REQUIREMENT
## (RECIPE)

# USPCI ANALYTICAL PROCEDURE 8

## (USPCI - 8)

## FIXATION REQUIREMENT (RECIPE)

### 1.0 SCOPE AND APPLICATION

This test is for determining the amount of reagents (fly ash, cement kiln dust, lime, cement, silicate based reagents, activated carbon, water, etc...) that must be added to waste streams containing free liquids to stabilize the waste stream or to pass treatment standards.

### 2.0 SUMMARY OF METHOD:

A weighed amount of sample is mixed while slowly adding reagent(s) until no free liquids can be seen. The mixture is then weighed and the ratio of sample to reagent(s) is recorded. The mixture is then subjected to the Paint Filter Liquids Test (Method 9095), more reagent(s) is (are) added until the test shows no free liquids. The final ratio of reagent to sample is the one used for waste stream stabilization prior to landfilling. For determining if the mixture meets the treatment standard, the appropriate standard must be subjected to its appropriate test procedure.

### 3.0 SAFETY

Wear appropriate gloves and safety glasses when handling samples.

### 4.0 PROCEDURE:

4.1 Weigh approximately 25 grams of sample into a 50 ml disposable beaker.

4.2 Gradually add reagent(s) and mix until no free liquids are seen. Weigh mixture.

4.3 Subject mixture to Paint Filter Liquids Test (Method 9095) or to TCLP if stabilization is to meet CCWE treatment standards.

4.4 Add more reagent(s) if free liquids are found with method 9095.

4.5 Determine final ratio of reagent(s) to sample for adequate fixation of free liquids or to meet treatment standards.

## 5.0 QUALITY CONTROL

5.1 Duplicate samples are run for 1 sample in 10.

5.2 Quality control charts are kept to indicate the method provision on duplicate samples.

## 6.0 REFERENCES

SW-846 Method 9095 40 CFR Part 268

# USPCI ANALYTICAL PROCEDURE 9

(USPCI - 9)

CONDUCTIVITY

[RESERVED]

# USPCI ANALYTICAL PROCEDURE 10

## (USPCI - 10)

## REDUCER SCREEN

# USPCI ANALYTICAL PROCEDURE 10

# (USPCI - 10)

# REDUCER SCREEN

## 1.0 SCOPE AND APPLICATION

This method provides a spot test for the presence of reducing agents in a sample. This is a screening method value rather than a quantified result using a calorimetric method. It provides a POS/NEG result.

## 2.0 SUMMARY OF METHOD

As an indicator for a pH < 8. A small amount of sample is placed in a test tube containing $I_2$ starch solution. If a reducer is present, the blue color of the indicator will fade.

## 3.0 SAFETY

3.1 Wear gloves/glasses.

3.2 Perform test in a load to vent any possible fumes from sample and/or acetic acid.

3.3 Use caution using glacial acetic acid.

## 4.0 INTERFERENCES

No interferences are anticipated. However, strong bases will cause false positives. It is very important to adjust pH.

## 5.0 REAGENTS

5.1 Glacial Acetic Acid - ACS Grade for adjusting pH.

5.2 Reducing Agent Indicator.

5.2.1 Place 500 ml Deionized Water in a 1000 ml beaker and heat just short of boiling. Remove from heat.

5.2.2 Add 5 gm soluble starch and stir until all the starch is dissolved.

5.2.3 Dilute starch solution to 1:1 with Deionized Water. Allow to cool to room temperature.

5.2.4 Add 1 gm of elemental Iodine to 50 ml of Ethyl Alcohol. Stir until all the Iodine is dissolved.

5.2.5 After starch is cool, add 10 ml of Iodine solution to starch. Place this mixture in a dark bottle and store in a dark place.

## 6.0 PROCEDURE

6.1 A Starch solution produces a deep blue color in the presence of elemental Iodine. A reducing agent present in a sample will donate an electron to the Iodine and clear the solution.

6.1.1 Transfer 1 gm of sample to a 55 ml disposal beaker.

6.1.2 Add 10 ml Deionized Water.

6.1.3 Adjust the pH to < 8 with 1:1 acetic acid.

6.1.4 Add 20 ml indicator to another 55 ml beaker.

6.1.5 Add 10 drops of the pH adjusted sample solution to the beaker of indicator.

6.2 The blue color will fade if a reducing agent is present.

## 7.0 QUALITY CONTROL

7.1 Samples should be tested in duplicate at a frequency of not less than 10%.

7.2 A positive can be found using a solution of Sodium Thiosulfate.

## 8.0 REFERENCES

8.1 <u>Standard Methods for Examination of Water and Wastewater</u>, 16th Ed.

8.2 <u>Analytical Chemistry</u>, 4th Ed., Gary Christian.

# USPCI ANALYTICAL PROCEDURE 11

## (USPCI - 11)

## EXTRACTION FOR SOLIDS

# USPCI ANALYTICAL PROCEDURE 11

# (USPCI - 11)

# EXTRACTION FOR SOLIDS

## 1.0 SCOPE AND APPLICATION

1.1 This method is used in the screening of waste samples for high levels of organic halides. This method is primarily concerned with solvents, although some semi-volatiles (bp > 200°C) will be detected. Heavier PCBs do not extract well with polar solvents.

1.2 Hexane is the current solvent and yields good (> 90%) matrix spike recoveries. Other solvents can be used provided they yield adequate spike recoveries.

## 2.0 METHOD SUMMARY

A nominal 1 gram sample of waste is extracted with methanol using physical agitation.

## 3.0 INTERFERENCES

The TOX analyzer is relatively interferent free.

## 4.0 SAFETY PRECAUTIONS

Waste samples can contain extreme levels of hazardous compounds. The analyst should be acquainted with the waste stream and take precaution consummate with the potential risk.

## 5.0 APPARATUS AND EQUIPMENT

5.1 25 ml Septum capped vial, or equivalent.

5.2 Sample Agitator (optional).

## 6.0 REAGENTS

Hexane, interferent free, or equivalent.

## 7.0 SAMPLE HANDLING AND PRESERVATION

7.1 Samples should be extracted in a timely manner. No holding time exists for samples of this type. No preservative is required.

7.2 It is recommended that extracts not to be analyzed within 7 days be stored at 4°C $\pm 2^{C}$. Extracts should not be stored more than 30 days.

## 8.0 CALIBRATION AND STANDARDIZATION

For details of instrument calibration, see the applicable parts of the QA Plan (Instrument Calibration).

## 9.0 PROCEDURE

9.1 Obtain the tare weight of a 25 ml vial. (NOTE: All weights in this method are to the nearest 0.01 gram, unless otherwise noted.)

9.2 Add a nominal 1 gram of sample and record the gross weight.

9.3 Add 10 ml of MeOH to the sample and agitate for 1 minute.

## 10.0 SAMPLE CALCULATIONS

Quantity of TOX is expressed as mg/kg TOX (as chloride). Sample concentration is:

$$\text{mg/kg TOX} = \frac{(\mu\text{g TOX}) \times (\text{ml Extract}) \times 1000}{(\mu\text{l injected}) \times (\text{gm sample})}$$

## 11.0 QUALITY CONTROL

11.1 Method Blank - A method blank should be analyzed at a minimum frequency of 10% or 1 per extraction batch.

11.2 Matrix Spike/Matrix Spike Duplicate (MS/MSD) - an MS/MSD will be run at a minimum frequency of 10%.

11.3 2,4,6-Trichlorophenol will be used as the spiking compound. A solution independent of the calibration standard will be used.

## 12.0 METHOD PERFORMANCE

12.1 Method spike recoveries should be between 75% - 125%.

12.2 Method duplicate precision should be < 30% RPD.

## 13.0 REFERENCES

Waste dilution, Method 3580, SW-846

Sonification extraction, Method 3550, SW-846

# USPCI ANALYTICAL PROCEDURE 12

## (USPCI - 12)

## EXPLOSIVITY TEST

# TEST PLAN TO DETERMINE REACTIVITY OF EXPLOSIVELY CONTAMINATED SOIL AND SEDIMENT*

## I. PURPOSE

The purpose of this plan is to delineate the necessary tests, associated methods, and interpretation of results to determine whether a contaminated soil of sediment is classified as reactive due to its explosive properties. Such information is necessary to determine what (if any) processing is required prior to final disposal of such soil or sediment.

## II. REFERENCE

a. Title 40 Code of Federal Regulations, Part 261, Para 261.23, Characteristic of Reactivity.

b. EPA Publication SW 846, Test Method for Evaluating Solid Waste, Subsection) 6-2; Definition of Explosive Materials (attached as Incl. 1).

c. Army Technical Bulletin 700-2, Chapter 3: Minimum Test Criteria for Bulk Explosive Compositions and Solid Propellant Compositions.

d. FONECONS with Dr. H. Matsuguma, Chief, Chemistry Branch, Energetic Materials Division, Large Caliber Weapons System Laboratory, U.S. Army Armament Research and Development Command, SAB.

## III. BACKGROUND

Due to explosives production, employment, and disposal operations performed through the years at various military installations across the country, the Army owns property which contains potentially explosively contaminated soils and sediments. Efforts are underway to begin decontamination and close-out of such sites in compliance with Federal Environmental Regulations. Explosives, however, are governed by Ref 2a, which restricts reactive materials from begin landfilled, including placement in a hazardous waste landfill. By regulation, then, every Army site which contains explosive residues, which range from low parts per million up to fifty percent in the worst cases, would require treatment prior to final disposal. Since many sites with low levels of contamination are not expected to exhibit any explosive properties, identifying such sites would remove from them the requirement to treat the residues as reactive wastes. Therefore, tests are provided in this plan which are suitable for determining whether a contaminated soil or sediment is reactive due to explosivity according to Environmental Protection Agency definitions.

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*Three sediment samples are obtained from each lagoon and analyzed for explosive concentration. One sample is taken near the waste water influent point, another near the effluent point, and the third sample is taken from the middle of the lagoon.

a. Of the eight characteristics defining a reactive waste in Ref. 2a, the characteristics pertaining to explosive wastes are:

1. Capable of detonation or explosive reaction if subjected to a strong initiating source or if heated under confinement.

2. Capable of detonation or explosive decomposition or reaction at standard temperature or pressure.

3. Is a forbidden explosive as defined in 49 CFR 173.51 or a Class A or Class B explosive as defined in 49 CFR 173-53 and 88.

b. Ref. 2b defined tests for explosives which address the above definitions of reactivity as follows:

1. A Stability Test is performed by heating the residue to 75°C for 48 hours. This test defines a forbidden explosive according to 49 CFR 173.51.

2. A Detonation Test is performed by inserting a blasting cap into a sample and observing the detonation. Reaction of the sample to a strong initiating source and Class A explosives as defined in 49 CFR 173.53 are tested in this manner.

3. A Spark Test is performed by inserting a time fuze or an electric squit into a sample and observing for deflagration or detonation. This tests for explosives as defined in 49 CFR 173.53 (initiating explosives) and 49 CFR 173.88 (propellants).

4. An Impact Test is performed on the Bureau of Explosives Impact Apparatus to define Class A explosives according to 49 CFR 173.53.

c. In Ref. 2d, the above tests were discussed with Dr. H. Matsuguma of the Army's Primary Explosives Research Laboratory. Pertinent comment regarding the above tests were as follows:

1. The tests are adequate for a go/no-go evaluation of reactivity, excep that the results of the Impact Tests will be misleading at low explosive concentrations. Since impact testing is designed to be a severe test use for ranking reactions of various explosives. It is possible to eke out positive results even from minute quantities of explosives Supplementing Impact Test results with a Card Gap Test, as outlined i Ref. 2c, will better define the ability of a contaminated sample to propagate detonation.

2. Soil and sediment samples should be analyzed to determine the explosive constituents and their concentrations. However, the chemical analysis methods are not germane to the actual reactivity tests and are no addressed in this plan.

3. The Stability, Detonation, Impact, and Card Gap Tests are performed in a standard manner, although some minor modifications are required to accommodate preparation of environmental samples. The Spark Test is not a standard test for the Army, but with modification, can be performed in the same manner as the Detonation Test.

## V. TEST EQUIPMENT AND MATERIALS

The following items are required to perform the necessary explosives tests on one field sample. Ancillary laboratory equipment is not included.

a. One Bureau of Explosives Impact Apparatus.

b. One ventilated explosion-proof oven equipped to continuously record temperature.

c. One blasting machine or equivalent.

d. Electric firing wire.

e. Electric blasting caps.

  1. Five No. 8 electric blasting caps (contains 2 gms of 80/20 mixture mercury fulminate/potassium chlorate).

  2. Three engineer special electric blasting caps.

f. Five electric match head igniters.

g. Two inch diameter by 1 inch long pressed pentolite pellet, National Stock No. 1375-00-991-8891, as required.

h. Solid lead cylinders 1-1/2 inch diameter by 4 inches high as required.

f. One piece of mild steel plate SAE 1010 to 1030, 1/2 inch thick by 12 inches square.

j. Mild steel plates (SAE 1010 to 1030) 6 x 6 inches x 3/8 inch as required.

k. Tubing, steel cold drawn seamless, mechanical, composition 1015, 1-7/8 inch 00, 0.219 inch wall thickness by 5-1/2 inch long, as required.

## VI. SAMPLE PREPARATION:

A complete set of tests will require a five pound sample from each field sampling point. Samples will generally be received wet and possibly split into multiple containers for shipping purposes. The field sample will be prepared and split into laboratory samples as follows:

a. Recombine samples if necessary. Samples may be mixed while wet to achieve uniformity. Large chunks should be broken up, in a ball mill or mortar and pestle, using an operational shield if necessary.

b. The wet samples must be dried to the appropriate moisture content for testing.

1. The apropriate moisture content will be determined by taking a soil sample from a depth of four feet at a point close to the lagoon being sampled. That sample will then be weighed, dried in an oven until constant weight is achieved, and then reweighed. From this, the moisture content will be calculated.

2. For each soil or sediment sample to be tested for explosivity, a sub-sample will be drawn, weighed, oven dried, and then reweighed to determine its original moisture content. The amount of water weight which must be removed from each sample to reach the moisture content in 6b(1) above will then be calculated.

3. Each soil or sediment sample will then be weighed, spread in a thin layer on a tray, and then dried in an oven at 60°C until the amount of water weight calculated in 6b(2) above has been removed. The sample must be monitored and reweighed until the desired weight is reached.

c. Laboratory test samples will be prepared from the prepared field sample as follows:

1. For the Thermal Stability, Detonation, and Spark Tests, prepare a total of eleven samples by filling a four-ounce paper cup approximately 2/3 full. The sample should not contain large chunks and should be tamped as it is filled to insure continuity of the sample.

2. For the Impact Sensitivity Test, withdraw an approximate 200 mg sample from the field sample. This sample must then be carefully crushed so that 10 mg of uniformly fine consistency can be drawn from it. Description of the individual samples is incorporated under the Impact Sensitivity Test.

3. For the Card Gap Test, prepare three samples by filling the tubes listed in para. 5k with the material to be tested. Insure that the sample is continuous by tamping. If the consistency of the sample is such that it will not consolidate, a piece of light cellophane tape may be placed across the lower end to retain the sample.

## VII. TEST METHODS

Although it could be argued that some of these tests are not applicable to certain pure explosives, it is possible to encounter residues from mixed explosives or environmentally altered residues which will not behave in the classic manner. Therefore, the complete series of tests will be performed on each sample. Test results will be recorded on a data sheet similar to Fig. 1. Tests may be run in any convenient order.

a. Thermal Stability. Place one sample from para. 6d(1) in a constant temperature explosion-proof oven. Raise temperature of the oven to 75°C and maintain at 75°C for 48 consecutive hours. Temperatures will be continuously recorded. Constant observation is not required. Record results on data sheet.

b. Spark Test. Place a lead block (para. 5h) on the steel plate (para. 5i). Using a sample from para. 6d(1), make a small depression in the sample with stick or pencil, insert an electric match head igniter (para. 5f) into the depression and secure (tape) the igniter wires to the cup for stability. Place the cup on the lead block, connect the firing circuit, and remotely initiate. Deflagration will be evidenced by the energetic burning of the sample. Detonation of the sample will cause mushrooming of the lead block. Repeat the test five times or until evidence of a deflagration or detonation occurs, whichever is less. Record results on data sheet.

c. Detonation Test. Place a lead block (para. 5h) on the steel plate (para. 5i). Using a sample from para. 6d(1), press a hole about half the length of the blasting cap into the center of the sample using a pencil, then insert a No. 8 blasting cap into the sample. A wood block with a hole drilled in it similar to Fig. 2 may be used to support the blasting cap. Place the sample onto the lead block, connect the firing circuit, and remotely initiate. Detonation of the sample will cause mushrooming of the lead block. Repeat the test five times or until detonation occurs, whichever is less. Record results on a data sheet.

d. Impact Sensitivity Test: Conduct ten individual tests using one sample (para. 6d(2)) per test in the Bureau of Explosives Impact Apparatus. Place a 10 mg sample in the cup assembly. Drop the weight from the maximum height of the machine. Observe the result and record on the data sheet. Conduct tests at an ambient temperature of 25°C 5°C. Insure cup and anvil are thoroughly cleaned and dried between test runs.

e. Gap Test

1. Assemble the following items for each test to be conducted:

   a. One sample prepared according to para. 6d(3).

   b. Two pentolite pellets (para. 5g).

   c. One engineer's special electric blasting cap, J2 (para. 5e(2)).

   d. Blasting machine and firing wire (para 5c. and d).

   e. One 5 x 6 inch steel plate (para. 5j).

   f. Plastic material, 1/16 inch thick cut into 1/2 inch squares.

2. Arrange the materials as shown in Fig. 2. The witness plate is supported on two edges, about 6 inches above ground surface. The small plastic squares are placed on the plate to support the pipe and maintain a 1/16 inch air gap. The squares should be under the edge of the pipe, rather than under the explosive. The pentolite boosters are then placed on top of the sample as shown in Fig. 2, except that the gap cards and the cardboard tube are not used. The blasting cap is then placed on top of the pentolite (with a wood support ring) and remotely detonated. Detonation of the sample is indicated when a clean hole is cut in the witness plate. This test is performed three times or until a detonation occurs, whichever is less. Results are recorded on the data sheet.

## VIII. INTERPRETATION OF RESULTS

For the purpose of reactivity evaluation, a positive result from any single test will indicate that a given field sample is reactive, except that results from the Impact and Gap Tests should be considered together.

a. A sample is considered reactive due to instability if it detonates, deflagrates, or decomposes exothermically (as evidenced by a rise in temperature on the recorder) during the Thermal Stability Test.

b. The field sample is considered to be reactive if one lab sample detonates, deflagrates, or burns in a sustained flame during the Spark Test. Localized smoldering does not indicate reactivity.

c. The field sample is reactive if one sample detonates during the detonation test.

d. The Impact Sensitivity Test may be considered positive if detonation (explosion, flame, noise) occurs in at least 50 percent of the ten tests. Conversely, if detonation does not occur in at least 50 percent of the tests, the sample is non-reactive. However, the results of the Impact Sensitivity Test are the most difficult to interpret, since samples can exhibit partial response under such harsh treatment. In such cases, the Gap Test should be used as a discriminator, defining a material as reactive if it detonates once out of three tests.

## IX. CONCLUSION

The tests conducted under this plan exceed minimum requirements for determining reactivity due to explosive properties as specified in Ref. 2b. The Thermal Stability, Spark, and Detonation Tests are performed as specified. The Impact Sensitivity Test is performed in a manner more stringent than specified to insure that results are safe-sided Since Impact Sensitivity Tests may present results which are difficult to interpret, the Shock or Gap Test has been added as a discriminator to determine whether a questionable material is detonable under worst-case conditions. It can be stated with certainty that a sample which does not respond positively to these tests is not reactive due to explosive properties as defined in Ref. 2a.

# USPCI ANALYTICAL PROCEDURE 13

## (USPCI - 13)

## CYANIDE - PEROXIDE AMENABILITY
(RESERVED)

# USPCI ANALYTICAL PROCEDURE 14

## (USPCI - 14)

## CYANIDE - CHLORINATION AMENABILITY
(RESERVED)

# USPCI ANALYTICAL PROCEDURE 15

(USPCI - 15)

# CYANIDE - CONVERSION AMENABILITY (RESERVED)

# USPCI ANALYTICAL PROCEDURE 16

## (USPCI - 16)

## LIQUID WASTE COMPATIBILITY
(RESERVED)

# USPCI ANALYTICAL PROCEDURE 17

(USPCI - 17)

SOLUBLE SULFIDES
(RESERVED)

# USPCI ANALYTICAL PROCEDURE 18

## (USPCI - 18)

## SULFATE SCREEN
(RESERVED)

# USPCI ANALYTICAL PROCEDURE 19

## (USPCI - 19)

## SULFIDE - PEROXIDE AMENABILITY (RESERVED)

# USPCI ANALYTICAL PROCEDURE 20

(USPCI - 20)

## DISTILLATION
(RESERVED)

# USPCI ANALYTICAL PROCEDURE 21

# (USPCI - 21)

# RADIANT HEAT IGNITION TEST PROCEDURE

# (IGNITABLE SOLIDS SCREEN)

# USPCI ANALYTICAL PROCEDURE 21

## (USPCI - 21)

# RADIANT HEAT IGNITION TEST PROCEDURE

## (IGNITABLE SOLIDS SCREEN)

## 1.0 SUMMARY OF METHOD

A sample is placed 6 cm beneath a preheated radiant heat source and the time to ignition is detected by a thermocouple sensor and recorded on a strip chart recorder.

## 2.0 APPARATUS AND REAGENTS

2.1 Test chamber - as shown in Figure 1.

2.2 Sample container - aluminum weighing pan, 6 cm diameter x 1.7 cm deep.

2.3 Controller/sensor - as shown in figure 1.

2.4 Recorder - strip chart, variable speed and input impedance.

2.5 Safety Equipment

2.5.1 Flameproof gloves (Lab Safety Supply 1915M or equivalent)

2.5.2 Tongs - 53 cm (Fisher, 15-207 or equivalent)

2.5.3 Respirator (Fisher, 13-995-11 or equivalent)

2.6 Balance (minimum accuracy to nearest 100 mg)

## 3.0 TEST PROCEDURE

3.1 Locate the test chamber in a fireproof fume hood with the exhaust fan turned on.

3.2 If the hood is large enough, locate the controller/sensor and the recorder outside the hood but in proximity to the test chamber.

3.3 With an empty sample container on the sample platform, (Figure 1; part no. 5) position the sample platform to provide a heating element (Figure 1; part no. 1) to sample container (top rim) distance of 6 cm.

3.4 Position the thermocouple (Figure 1; part no 7) 1 cm above the top rim and centered over the sample container.

3.5 Connect the thermocouple to position 33 (Figure 1) on the controller/sensor using the cable supplied.

3.6 Connect the heater/solenoid cable (Figure 1; part no. 16, 17) to position 34 (Figure 1) on the controller/sensor.

3.7 Connect the power cord on the controller/sensor to a 110 VAC power source.

3.8 Set the variable transformer to 0.

3.9 Position a dummy sample container (blackened with carbon soot) directly under heat source. Close the chamber door.

3.10 Turn on the controller/sensor.

3.11 Turn on the heater. Set the variable transformer to the setting determined during the temperature adjustment procedure.

3.12 Preheat the radiant heat source for 30 minutes.

3.13 Transfer sample to the sample container to a depth of approximately 1 cm, making sure that the sample surface is level and smoothed.

**WARNING!!**

Extreme care must be exercised in testing materials known or suspected of being extremely flammable. Preliminary tests using greatly diminished sample sizes should be conducted prior to performing the actual test to insure the safety of the analyst. A reduced test sample depth should be used in cases where sample ignition is extremely rapid and/or violent.

3.14 Place the filled sample container outside, but in proximity to, the test chamber.

3.15 Start the recorder at a chart speed of 0.5 in/min and a full-scale sensitivity of 1 volt.

3.16 Open the side door and remove the dummy sample container.

3.17 Using tongs and flameproof gloves, pick up the filled sample container and place it onto the metal trough. With the tongs, slide the sample container under the radiant heat source. Immediately, close the side door and activate the recorder zero knob to mark the chart.

CAUTION!!

The placement of the sample beneath the radiant heat source, the marking of the recorder chart and the closing of the chamber door must be executed as quickly as possible to optimize the precision of the test results.

3.18 Raise the chamber door and remove the ignited sample from beneath the radiant heat source and carefully place it in the bottom of the chamber.

WARNING!!

Raise the fume hood and test chamber doors just to a level that facilitates removal of the sample from beneath the radiant heat source. Flameproof gloves should be used in combination with tongs to protect the analyst from the burning sample.

3.19 Extinguish the fire by smothering.

3.20 Prepare the chamber for the next sample by positioning the dummy sample container beneath the radiant heat source.

3.21 Lower the fume hood door to one-half the height of the chamber.

3.22 Measure the distance (cm) from the initial mark to ignition on the recorder. Calculate the time to ignition from the calibrated recorder speed.

3.23 Proceed with the analysis of subsequent samples by repeating steps 3.13 through 3.22

# USPCI ANALYTICAL PROCEDURE 21-A

# (USPCI - 21-A)

# IGNITABILITY TEST METHOD FOR SOLIDS

Revised December 12, 1991

## USPCI ANALYTICAL PROCEDURE 21-A

## (USPCI - 21-A)

## IGNITABILITY TEST METHOD FOR SOLIDS

### 1. Scope.

This test method is for the determination of flash point by Pensky-Martens closed cup tester of bulk solids from the solidification tanks prior to landfilling. It is to be used to verify compliance with the conditions outlined in Section 7.3 of the Waste Analysis Plan.

### 2. Definitions.

a. flash point --the lowest temperature at which application of a test flame causes the vapor of the specimen to ignite under the conditions specified under the test.
b. the sample is deemed to have flashed when a large flame appears and instantaneously propagates itself over the surface of the sample.
c. Occasionally, particularly near the actual flash point, the application of the test flame will cause a blue halo or an enlarged flame; this is not a flash and should be ignored.

### 3. Summary of Method.

The sample is heated at a slow, constant rate. A small flame is directed into the cup at regular intervals. The flash point is the lowest temperature at which application of the test flame causes the vapor above the sample to ignite.

### 4. Apparatus.

a. *Pensky-Martens Closed Flash Tester*, as described in Annex A1 (ASTM Method D 93).
b. *Thermometers* -- ASTM 9F (9C) Pensky-Martens low range Thermometer having a range from 20 to 230°F (-5 to +110°C) and conforming to the requirements of Specification E 1 (ASTM Method D 93) shall be used. Equally acceptable is IP thermometer 15F (15C), with specifications as shown in Annexes A2 and A3 (ASTM Method D 93).

## 5. Safety Precautions.

The operator must exercise and take appropriate safety precautions during the initial application of the test flame, since samples containing low flash material may give an abnormally strong flash when the test flame is first applied.

## 6. Sampling.

a. Erroneously high flash points may be obtained if precautions are not taken to avoid the loss of volatile material. Do not open containers unnecessarily and make a transfer unless the sample temperature is at least the equivalent of 18°F (8°C) below the expected flash point. Do not use samples from leaky containers for this test method.

b. This method must be run within one hour of the collection of the sample.

c. The sample used for this method may not be the same material as that used for a free liquids test or any other test.

## 7. Preparation of Apparatus.

Support the tester on a level, steady table. Unless the tests are made in a draft-free room or compartment, surround the tester on three sides with a shield, each section of which is about 18 inches wide and 24 inches high.

## 8. Procedure.

a. Thoroughly clean all parts of the cup and its accessories before starting the test, being sure to remove any solvent which had been used to clean the apparatus. Fill the cup with sample to be tested to the level indicated by the filling mark. Make sure the material is firmly packed in the cup. Place the lid on the cup and set the latter in the stove. Be sure to have the locating or looking device properly engaged. Insert the thermometer. Bring the material to be tested and the tester to a temperature of 60 ± 10°F (15 ± 5°C) or 20°F (11°C) lower than the estimated flash point, whichever is lower. Light the test flame and adjust it to 5/32 inch in diameter.

b. Supply the heat to raise the temperature 20°F. Hold at that temperature for one minute before flashing and record the result. Repeat this step until a temperature of 140°F is reached.

c. Repeat the test two more times using fresh sample and record the results.

## 9. Calculation and Report.

a. If the results of any of the three tests are at or below 140°F, the final result shall be recorded as <140°F.

b. If the results of all of the three tests are above 140°F, the final result shall be recorded as >140°F.

## 10. Calibration of Tester.

Follow Section 14.3 of ASTM Method D 93 for calibrating the tester.

# APPENDIX I

## EXCERPTS FROM

## "SAMPLERS AND SAMPLING PROCEDURES
## FOR
## HAZARDOUS WASTE STREAMS"

# SECTION 2

# CONCLUSIONS

The present study was designed to develop simple but effective sampling equipment for collecting represenative samples of hazardous wastes. In addition, recommended procedures for sample collection, handling, storage, and recording were to be developed. These primary objectives have been met, and the resulting sampling equipment and procedures are presented here.

The sampling equipment and procedures were designed to insure the widest possible applicability in the sampling of various types of hazardous wastes. The methods, however, are not intended to cover all possible sampling situations. Professional judgment on applicability must be exercised.

# SECTION 4

# SAMPLERS

Sampling of hazardous wastes requires different types of samplers. Some of these samplers are commercially available, but the others have to be fabricated. This section lists and describes suitable samplers. Their uses and commercial availability as well as directions for their use are reported. Directions for fabricating the commercially unavailable samplers are also outlined.

## COMPOSITE LIQUID WASTE SAMPLER (COLIWASA)

The Coliwasa is the single most important hazardous waste sampler discussed in this report. It was chosen from a number of other liquid samplers, based on laboratory and field tests, as the most practical. It permits the representative sampling of multiphase wastes of a wide range of viscosity, corrosivity, volatility, and solids content. Its simple design makes it easy to use and allow the rapid collection of samples, thus minimizing the exposure of the sample collector to potential hazards from the wastes. The sampler is not commercially available, but it is relatively easy and inexpensive to fabricate. The cost of fabrication is low enough that the contaminated parts may be discarded after a single use when they cannot be easily cleaned.

The recommended model of the Coliwasa is shown in Figure .. The history and development of this sampler is discussed in detail in Appendix A. The main parts of the Coliwasa consist of the sampling tube, the closure-locking mechanism, and the closure system.

The sampling tube consists of a 1.52-m(5-ft.) by 4.13-cm(1 5/8-in.) I.D. translucent plastic pipe, usually polyvinyl chloride (PVC) or borosilicate glass plumbing tube. The closure-locking mechanism consists of a short-length, channeled aluminum bar attached to the sampler's stopper rod by an adjustable swivel. The aluminum bar serves both as a T-handle and lock for the sampler's closure system. When the sampler is in the open position, the handle is place in the T-position and pushed down against the locking block. This manipulation pushes out the neoprene stopper and opens the sampling tube. In the close position, the handle is rotated until one leg of the T is squarely perpendicular against the locking block. This tightly seats the neoprene stopper against the bottom opening of the sampling tube and positively locks the sampler in the close position. The closure tension can be adjusted by shortening or lengthening the stopper rod by screwing it in or out

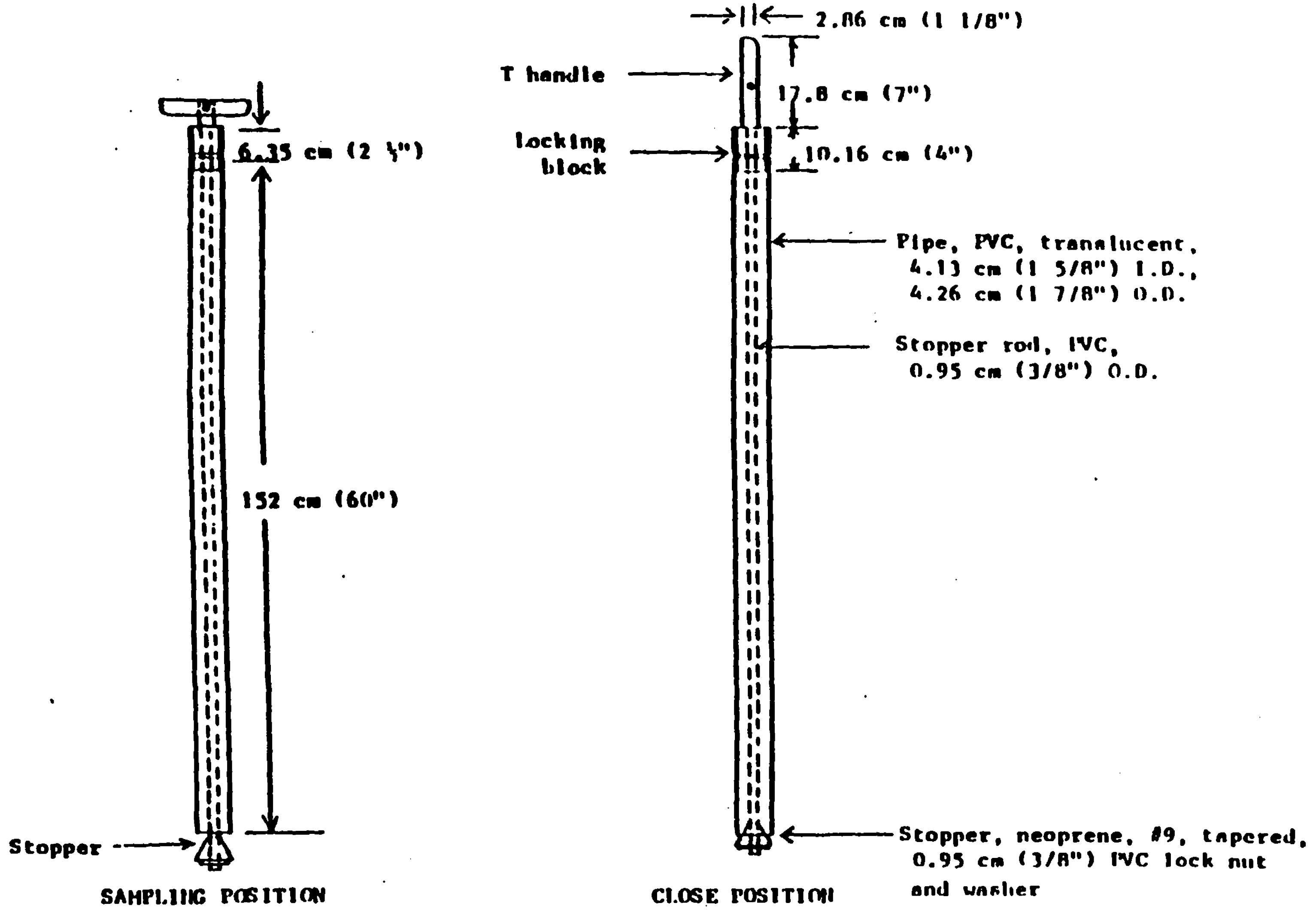


Figure 1. Composite liquid waste sampler (Coliwasa)

of the T-handle swivel. The closure system of the sampler consists of a sharply taped neoprene stopper attached to a 0.95-cm (3/8-in.) O.D. rod, usually PVC. The upper end of the stopper rod is connected to the swivel of the aluminum T-handle. The sharply tapered neoprene stopper can be fabricated according to specifications by plastic products manufacturers at an extremely high price, or it can be made in-house by grinding down the inexpensive stopper with a shop grinder as described in Note 1 of Appendix B.

Two types of Colivasa samplers are made, namely plastic or glass. The plastic type consists of translucent plastic (usually PVC) sampling tube. The glass Colivasa uses borosilicate glass plumbing pipe as the sampling tube and Teflon plastic stopper rod.

The complete list of parts for constructing the two types of Colivasa samplers is given in Appendix B. The suppliers and approximate costs of the parts as well as the directions for fabricating the commercially unavailable parts are also given.

The sampler is assembled as shown in Figure 1 and as follows:

1. Attach the swivel to the T-handle with the 3.18 cm(1¼ in.) long bolt and secure with the 0.48 cm(3/16 in.) National Coarse(NC) washer and lock nut.

2. Attach the neoprene stopper to one end of the stopper rod and secure with the 0.95 cm(3/8 in.) washer and lock nut.

3. Install the stopper and stopper rod assembly in the sampling tube.

4. Secure the locking block sleeve on the block with glue or screws. This block can also be fashioned by shaping a solid plastic rod on a lathe to the required dimensions.

5. Position the locking block on top of the sampling tube such that the sleeveless portion of the block fits inside the tube, the sleeve sits against the top end of the tube, and the upper end of the stopper rod slips through the center hole of the block.

6. Attach the upper end of the stopper rod to the swivel of the T-handle.

7. Place the sampler in the close position and adjust the tension on the stopper by screwing the T-handle in or out.

## Uses

The plastic Colivasa is used to sample most containerized liquid wastes except wastes that contain ketones, nitrobenzene, dimethylforamide, mesityl oxide, and tetrahydrofuran.[3,4]

The glass Coliwasa is used to sample all other containerized liquid wastes that cannot be sampled with the plastic Coliwasa except strong alkali and hydrofluoric acid solutions.

Procedure for Use

1. Choose the plastic or glass Coliwasa for the liquid waste to be sampled and assemble the sampler as shown in Figure 1.

2. Make sure that the sampler is clean (see Section 5).

3. Check to make sure the sampler is functioning properly. Adjust the locking mechanism if necessary to make sure the neoprene rubber stopper provides a tight closure.

4. Wear necessary protective clothing and gear and observe required sampling precautions (see Section 6).

5. Put the sampler in the open position by placing the stopper rod handle in the T-position and pushing the rod down until the handle sits against the sampler's locking block.

6. Slowly lower the sampler into the liquid waste. (Lower the sampler at a rate that permits the levels of the liquid inside and outside the sampler tube to be about the same. If the level of the liquid in the sampler tube is lower than that outside the sampler, the sampling rate is too fast and will result in a nonrepresentative sample).

7. When the sampler stopper hits the bottom of the waste container, push the sampler tube downward against the stopper to close the sampler. Lock the sampler in the close position by turning the T handle until it is upright and one end rests tightly on the locking block.

8. Slowly withdraw the sampler from the waste container with one hand while wiping the sampler tube with a disposable cloth or rag with the other hand.

9. Carefully discharge the sample into a suitable sample container (see Section 6) by slowly opening the sampler. This is done by slowly pulling the lower end of the T handle away from the locking block while the lower end of the sampler is positioned in a sample container.

10. Cap the sample container; attach label and seal; record in field log book; and complete sample analysis request sheet and chain of custody record.

11. Unscrew the T handle of the sampler and disengage the locking block. Clean sampler on site (see Section 5) or store the contaminated parts of the sampler in a plastic storage tube for subsequent cleaning. Store used rags in plastic bags for subsequent disposal.

12. Deliver the sample to the laboratory for analysis (see Section 6).

## SOLID WASTE SAMPLERS

A number of tools are available for sampling solid substances. The most suitable of these for sampling hazardous solid wastes are the grain sampler, sampling trier, and the trowel or scoop.

### Grain Sampler

The grain sampler (Figure 2) consists of two slotted telescoping tubes, usually made of brass or stainless steel. The outer tube has a conical, pointed tip on one end that permits the sampler to penetrate the material being sampled. The sampler is opened and closed by rotating the inner tube. Grain samplers are generally 61 to 100 cm (24 to 40 in.) long by 1.27 to 2.54 cm (½ to 1 in.) in diameter, and they are commercially available at laboratory supply houses.

Uses--

The grain sampler is used for sampling powdered or granular wastes or materials in bags, fiberdrums, sacks or similar containers. This sampler is most useful when the solids are no greater than 0.6 cm (¼ in.) in diameter.

Procedure for Use--

1. While the sampler is in the close position, insert it into the granular or powdered material or waste being sampled from a point near a top edge or corner, through the center, and to a point diagonally opposite the point of entry.[5]

2. Rotate the inner tube of the sampler into the open position.

3. Wiggle the sampler a few times to allow materials to enter the open slots.

4. Place the sampler in the close position and withdraw from the material being sampled.

5. Place the sampler in a horizontal position with the slots facing upward.

6. Rotate and slide out the outer tube from the inner tube.

7. Transfer the collected sample in the inner tube into a suitable sample container (see Section 6).

8. Collect two or more core samples at different points (see Section 6), and combine the samples in the same container.

9. Cap the sample container; attach label and seal; record in field log book; and complete sample analysis request sheet and chain of custody record.

10. Clean (see Section 5) or store the sampler in plastic bag for subsequent cleaning.

11. Deliver the sample to the laboratory for analysis (see Section 6).

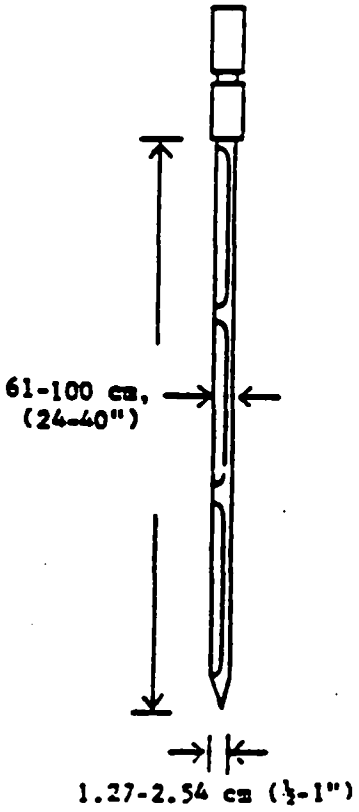


Figure 2. Grain sampler.

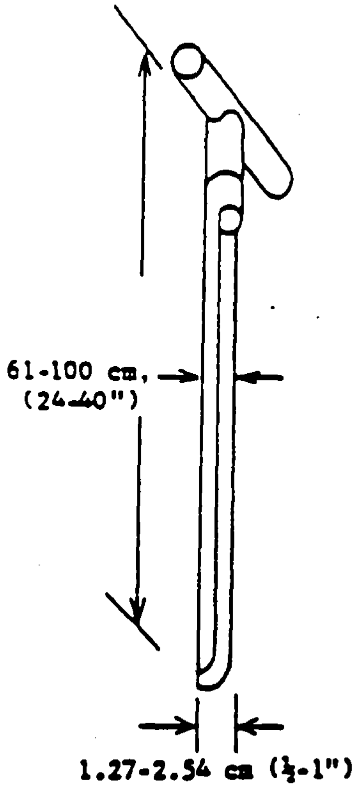


Figure 3. Sampling trier.

Sampling trier

A typical sampling trier (Figure 3) is a long tube with a slot that extends almost its entire length. The tip and edges of the tube slot are sharpened to allow the trier to cut a core of the material to be sampled when rotated after insertion into the material. Sampling triers are usually made of stainless steel with wooden handles. They are about 61 to 100 cm (24 to 40 in.) long and 1.27 to 2.54 cm (½ to 1 in.) in diameter. They can be purchased readily from laboratory supply houses.

Uses--

The use of the trier is similar to that of the grain sampler discussed above. It is preferred over the grain sampler when the powdered or granular material to be sampled is moist or sticky.

In addition, the sampling trier can be used to obtain soft or loosened soil samples up to a depth of 61 cm(24 in.) as outlined below.

Procedure for Use--

1. Insert the trier into the waste material at a 0 to 45° angle from horizontal. This orientation minimizes the spillage of sample from the sampler. Extraction of samples might require tilting of the containers.
2. Rotate the trier once or twice to cut a core of material.
3. Slowly withdraw the trier, making sure that the slot is facing upward.
4. Transfer the sample into a suitable container (see Section 6) with the aid of a spatula and/or brush.
5. Repeat the sampling at different points (see Section 6). Two or more times and combine the samples in the same sample container.
6. Cap the sample container; attach the label and seal; record in field log book; and complete sample analysis request sheet and chain of custody record.
7. Wipe the sampler clean, or store it in a plastic bag for subsequent cleaning.
8. Deliver the sample to the laboratory for analysis (see Section 6).

Trowel or Scoop

A garden-variety trowel looks like a small shovel (Figure 4). The blade is usually about 7 by 13 cm(3 by 5 in.) with a sharp tip. A laboratory scoop is similar to the trowel, but the blade is usually more curved and has a closed upper end to permit the containment of material. Scoops come in different sizes and makes. Stainless steel or polypropylene scoops with 7 by 15-cm(2 3/4 by 6-in.) blades are preferred. A trowel can be bought from hardware stores; the scoop can be bought from laboratory supply houses.

Uses--

An ordinary zinc-plated garden trowel can be used in some cases for sampling dry granular or powdered materials in bins or other shallow containers. The laboratory scoop, however, is a superior choice. It is usually made of materials less subject to corrosion or chemical reactions, thus lessening the probability of sample contamination.

The trowel or scoop can also be used in collecting top surface soil samples.

Procedure for Use--

1. At regular intervals (see Section 6), take small, equal portions of sample from the surface or near the surface of the material to be sampled.

2. Combine the samples in a suitable container (see Section 6).

3. Cap the container; attach the label and seal; record in field log book; and complete sample analysis request sheet and chain of custody record.

4. Deliver the sample to the laboratory for analysis (see Section 6).

## SOIL SAMPLERS

There is a variety of soil samplers used. For taking soil core samples, the scoop, sample trier, soil auger, and Veihmeyer sampler can be used. These samplers are commercially available and relatively inexpensive.

### Scoop or Trowel

See the preceding section on solid waste samplers for the description of a scoop or trowel (Figure 4).

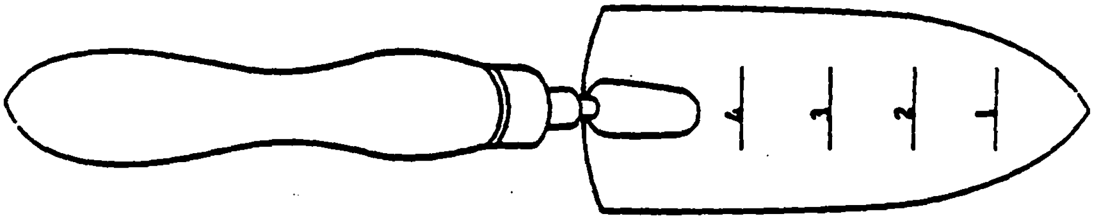


Figure 4. Trowel or scoop with calibrations.

Uses--

The scoop is used to collect soil samples up to 8 cm(3 in.) deep. It is simple to use, but identical mass sample units for a composite sample are difficult to collect with this sampler. The procedure for use of the scoop is outlined in the preceding section on solid waste samplers.

### Sampling Trier

See the preceding section on solid waste samplers for the description of a sampling trier (Figure 3).

Uses--

This sampler can be used to collect soil samples at a depth greater than 8 cm(3 in.). The sampling depth is determined by the hardness and types of soil being sampled. This sampler can be difficult to use in stony, dry, very heavy, or sandy soil. The collected sample tends to be slightly compacted, but this method permits observation of the core sample before removal.[6]

Procedure for Use--

Procedure for use of the sampling trier can be found in the section on solid waste samplers.

### Soil Auger

This tool consists of a hard metal central shaft and sharpened spiral blades (Figure 5). When the tool is rotated clockwise by its wooden T handle, it cuts the soil as it moves forward and discharges most of the loose soil upward. The cutting diameter is about 5 cm(2 in.). The length is about 1 m(40 in.), with graduations every 15.2 cm(6 in.). The length can be increased up to 2 m(80 in.). This tool can be bought from stores and, in some cases, from laboratory supply houses.

Uses--

The auger is particularly useful in collecting soil samples at depths greater than 8 cm(3 in.). This sampler destroys the structure of cohesive soil and does not distinguish between samples collected near the surface or toward the bottom. It is not recommended, therefore, when an undisturbed soil sample is desired.

Procedure for Use--

1. Select the sampling point (see Section 6) and remove unnecessary rocks, twigs, and other non-soil materials.

2. Install the sampler's wooden T handle in its socket.

3. Bore a hole through the middle of an aluminum pie pan large enough to allow the blades of the auger to pass through. The pan will be used to catch the sample brought to the surface by the auger.

4. Spot the pan against the selected sampling point.

5. Start augering through the hole in the pan until the desired sampling depth is reached.

6. Back off the auger and transfer the sample collected in the catch pan and the sample adhering to the auger to a suitable container (see Section 6). Spoon out the rest of the loosened sample with a sampling trier.

7. Repeat the sampling at different sampling points (see Section 6), and combine the samples in the same container as in step 6.

8. Cap the sample container; attach label and seal; record in field log book; and complete sample analysis request sheet and chain of custody record.

9. Brush off and wipe the sampler clean, or store it in a plastic bag for subsequent cleaning.

10. Deliver the sample to the laboratory for analysis (see Section 6).

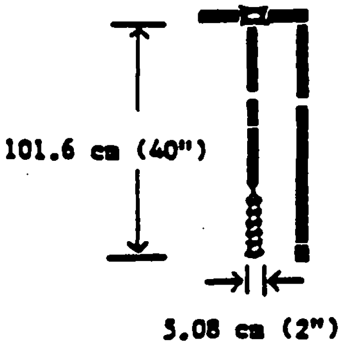


Figure 5. Soil auger.

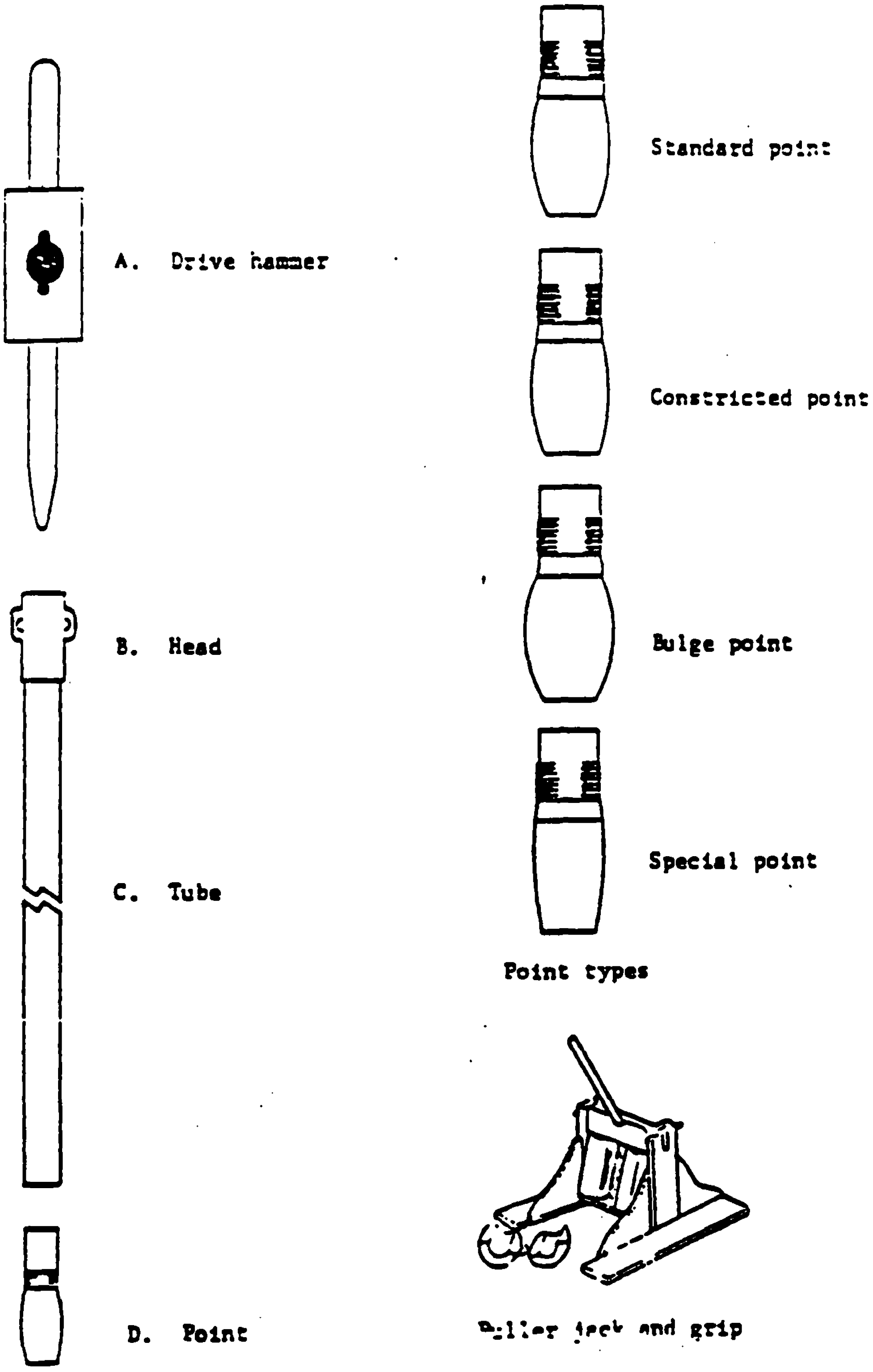


Figure 6. Veihmeyer sampler

### Veihmeyer Soil Sampler

This sampler was developed by Professor F.J. Veihmeyer of the University of California in Davis.[7] The parts of a basic sampler and the corresponding costs are given in Table 1, and the basic sampler is shown in Figure 6.

TABLE 1. BASIC PARTS AND COSTS OF A VEIHMEYER SOIL SAMPLER

| Part[a] | | Cost[b] |
|---|---|---|
| Tube, 1.5 m (5 ft.) | $ | 50.40 |
| Tube, 3 m (10 ft.) | | 84.75 |
| Tip, type A, general use | | 25.80 |
| Drive head | | 29.05 |
| Drop hammer, 6.8 kg (15 lb.) | | 71.85 |
| Puller jack and grip[c] | | 161.90 |
| Total | $ | 433.75 |

[a] Only one of each part is needed. They are manufactured by Hansen Machine Works, 334 N. 12th Street, Sacramento, CA 95815.

[b] Based on August 1, 1977, price list.

[c] Recommended for deep soil sampling.

The tube is chromium-molybdenum steel and comes in various standard lengths from 0.91 to 4.9 m(3 to 16 ft.) and calibrated every 30.48 cm(12 in.). Longer tubes can be obtained on special order. Different points (Figure 6) are also available for different types of soil and sampling. Each point is shaped to penetrate specific types of soil without pushing the soil ahead of it, thus preventing the core from compacting in the tube. The standard point is adequate for most general sampling purposes. The inside taper of each point is designed to keep the sample from being sucked out of the tube as it is pulled from the ground. The drive head protects the top of the tube from deforming when the tube is driven into the ground with the drive hammer. The hammer doubles as a drive weight and handle when pulling the sampler from the ground. When the sampler tube cannot be pulled easily from the ground, a special puller jack and grip are also available. Specifications for the various parts of the Veihmeyer sampler are given as follows:

| | |
|---|---|
| Points . . . . . | Chrome-molly steel, heat-treated. Includes a standard point for general use, a constricted point for deep sampling in heavy clay (keeps core from being sucked out of the tube), a bulge point for shallow sampling in heavy clay, and a special point for dry sand. (See Figure 6D). |
| Drive hammer . . | Standard weight is 6.8 kg (15 lb.). (See Figure 6A) |
| Tubes . . | Chrome-molly steel. Maximum length is 4.9 m (16 ft.). (See Figure 6C). |
| Head . . | Chrome-molly steel, heat-treated. (See Figure 6B). |
| Puller jack . . | Cast aluminum frame with steel roller assembly and handle. |
| Grip . . | Chrome-molly steel, heat-treated. |

Uses—

The Veihmeyer sampler is recommended for core sampling of most types of soil. It may not be applicable to sampling stony, rocky, or very wet soil.

Procedure for Use—

1. Assemble the sampler by screwing in the tip and the drive head on the sampling tube.

2. Insert the tapered handle (drive guide) of the drive hammer through the drive head.

3. Place the sampler in a perpendicular position on the soil to be sampled.

4. With the left hand holding the tube, drive the sampler into the ground to the desired sampling depth by pounding the drive head with the drive hammer. Do not drive the tube further than the tip of the hammer's drive guide.

5. Record the length of the tube that penetrated the ground.

6. Remove the drive hammer and fit the keyhole-like opening on the flat side of the hammer onto the drive head. In this position, the hammer serves as a handle for the sampler.

7. Rotate the sampler at least two revolutions to shear off the sample at the bottom.

8. Lower the sampler handle (hammer) until it just clears the two ear-like protrusions on the drive head and rotate about 90°.

9. Withdraw the sampler from the ground by pulling the handle (hammer) upwards. When the sampler cannot be withdrawn by hand, as in deep soil sampling, use the puller jack and grip.

10. Dislodge the hammer from the sampler; turn the sampler tube upside down; tap the head gently against the hammer; and carefully recover the sample from the tube. The sample should slip out easily.

11. Store the core sample, preferably, in a rigid, transparent, or translucent plastic tube when observation of soil layers is to be made. The use of the tube will keep the sample relatively undisturbed. In other cases, use a 1000-or 2000-ml (1-qt. or ½-gal) sample container (see Section 6) to store the sample.

12. Collect additional core samples at different points (see Section 6).

13. Label the samples; affix the seals; record in the field log book; complete analysis request sheet and chain of custody record; and deliver the samples to the laboratory for analysis (see Section 6).

## Waste Pile Sampler

A waste pile sampler (Figure 7) is essentially a large sampling trier. It is commercially available, but it can be easily fabricated from sheet metal plastic pipe. A polyvinyl chloride plumbing pipe 1.52 m(5 ft ) long by 3.2 cm(1¼ in.) I.D. by 0.32 cm(1/8 in.) wall thickness is adequate. The pipe is sawed lengthwise (about 60/40 split) until the last 10 cm(4 in.) The narrower piece is sawn off and hence forms a slot in the pipe. The edges of the slot and the tip of the pipe are sharpened to permit the sampler to cut into the waste material being sampled. The unsplit length of the pipe serves as the handle. The plastic pipe can be purchased from hardware stores.

### Uses--

The waste pile sampler is used for sampling wastes in large heaps with cross-sectional diameters greater than 1 m(39.4 in.). It can also be used for sampling granular or powdered wastes or materials in large bins, barges, or silos where the grain sampler or sampling trier is not long enough. This sampler does not collect representative samples when the diameters of the solid particles are greater than half the diameter of the tube.

### Procedure for Use--

1. Insert the sampler into the waste material being sampled at 0 to 45° from horizontal.

2. Rotate the sampler two or three times in order to cut a core of the material.

3. Slowly withdraw the sampler, making sure that the slot is facing upward.

4. Transfer the sample into a suitable container (see Section 6) with the aid of a spatula and/or brush.

5. Repeat the sampling at different sampling points (see Section 6) two or more times and combine the samples in the same sample container in step 4.

6. Cap the container; attach label and seal; record in field log book; and complete sample analysis request sheet and chain of custody record.

7. Wipe the sampler clean or store it in a plastic bag for subsequent cleaning.

8. Deliver the sample to the laboratory for analysis (see Section 6).

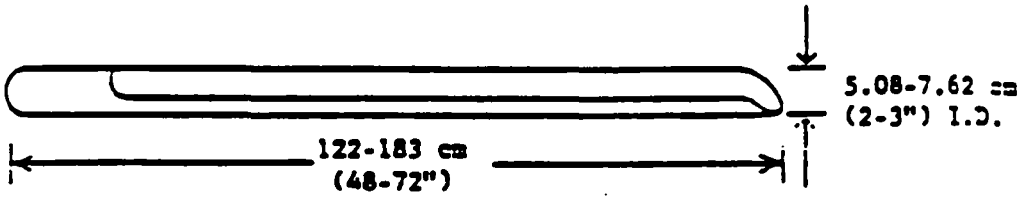


**Figure 7.** Waste pile sampler.

Pond Sampler

The pond sampler (Figure 8) consists of an adjustable clamp attached to the end of a two or three piece telescoping aluminum tube that serves as the handle. The clamp is used to secure a sampling beaker. The sampler is not commercially available, but it is easily and inexpensively fabricated. The tubes can be readily purchased from most hardware or swimming pool supply stores. The adjustable clamp and sampling beaker can be obtained from most laboratory supply houses. The materials required to fabricate the sampler are given in Table 2.

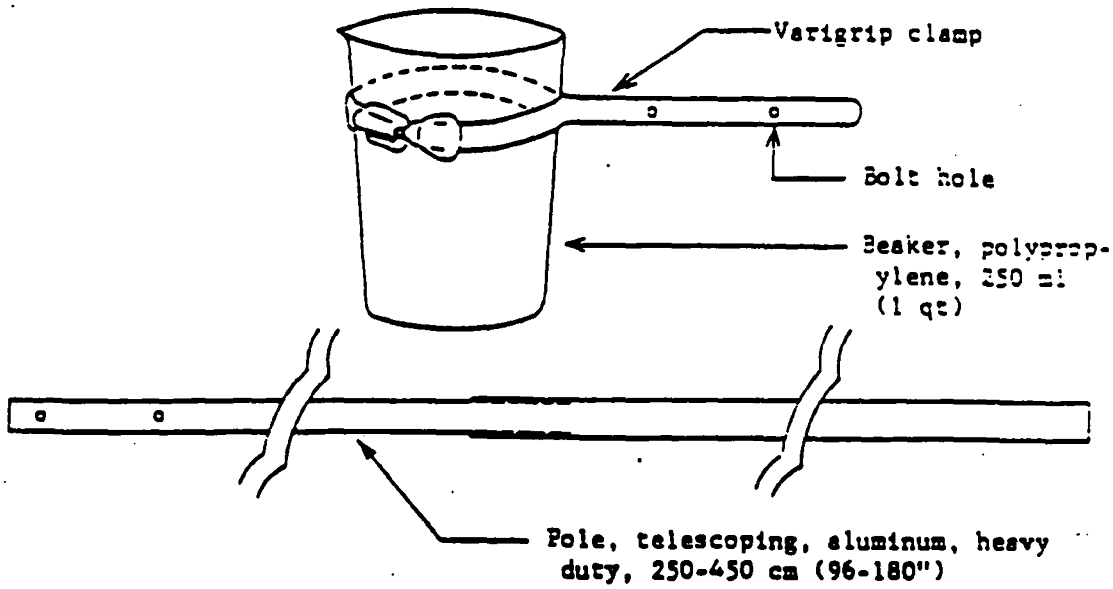


Figure 8. Pond sampler.

TABLE 2. BASIC PARTS AND APPROXIMATE COSTS OF A POND SAMPLER

| Quantity | Item | Supplier | Approximate cost |
|---|---|---|---|
| 1 | Clamp, adjustable, 6.4 to 8.9 cm(2½ to 3½ in.) for 250-to 600-ml(½ to 1¼-pt.) beakers | Laboratory supply houses | $ 7.00 |
| 1 | Tube, aluminum, heavy duty, telescoping extends 2.5 to 4.5 m(8 to 15 ft.) with joint cam locking mechanism. Pole diameters 2.54 cm(1 in.) I.D. and 3.18 cm(1¼ in.) I.D. | Olympic Swimming Pool Co. 807 Buena Vista Street, Alameda, Calif. 94501 or other general swimming pool supply houses. | 16.24 |
| 1 | Beaker, polypropylene, 250-ml(½ pt.) | Laboratory supply houses. | 1.00 |
| 4 | Bolts, 6.35 by 0.64 cm(2½ by ¼ in.) NC | Hardware stores | .20 |
| 4 | Nuts, 0.64 cm(¼ in.) NC | Hardware stores | .20 |
|  | Total |  | $24.64 |

Uses--

The pond sampler is used to collect liquid waste samples from disposal ponds, pits, lagoons, and similar reservoirs. Grab samples can be obtained at distances as far as 3.5 m (11½ ft) from the edge of the ponds. The tubular aluminum handle may bow when sampling very viscous liquids if sampling is not done slowly.

Procedure for Use--

1. Assemble the pond sampler. Make sure that the sampling beaker and the bolts and nuts that secure the clamp to the pole are tightened properly.
2. With proper protective garment and gear (see Section 6), take grab samples from the pond at different distances and depths (see Section 6).
3. Combine the samples in one suitable container (see Section 6).
4. Cap the container; label and affix the seal; record in field log book; and complete sample analysis request sheet and chain of custody record.
5. Dismantle the sampler; wipe the parts with terry towels or rags and store them in plastic bags for subsequent cleaning. Store used towels or rags in garbage bags for subsequent disposal.
6. Deliver the sample to the laboratory for analysis (see Section 6).

## Weighted Bottle Sampler

This sampler (Figure 9) consists of a bottle, usually glass, a weight sinker, a bottle stopper, and a line that is used to open the bottle and to lower and raise the sampler during sampling. There are a few variations of this sampler, as illustrated in the ASTM Methods D 270[8] and E 300[9]. The ASTM sampler, which uses a metallic bottle basket that also serves as weight sinker, is preferred. The weighted bottle sampler can either be fabricated or purchased.

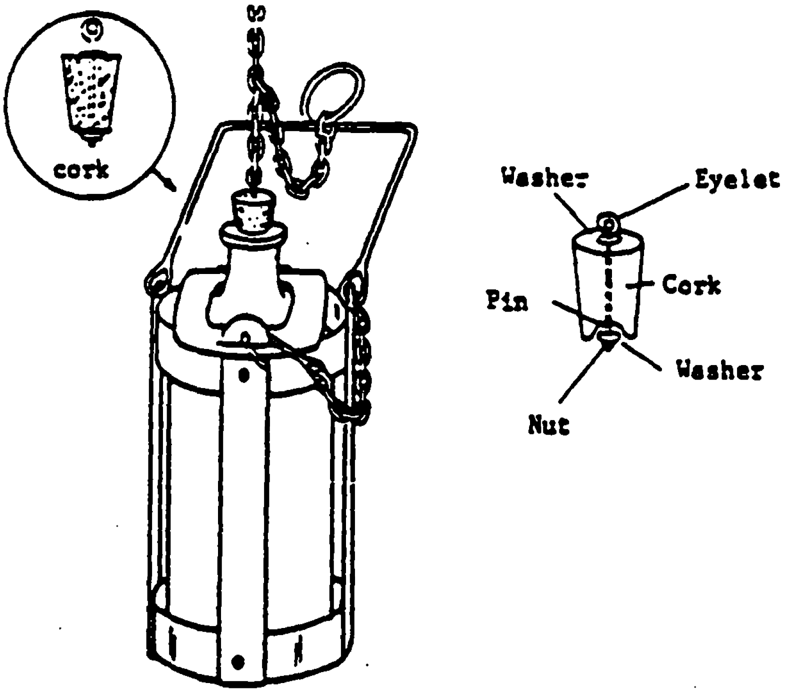
Uses--

The weighted bottle sampler can be used to sample liquids in storage tanks, wells, sumps, or other containers that cannot be adequately sampled with a Coliwasa. The sampler cannot be used to collect liquids that are incompatible or that react chemically with the weight sinker and line.

Procedure for use--

Assemble the weighted bottle sampler as shown in Figure 9.

2. Using protective sampling equipment, in turn, lower the sampler to proper depths to collect the following samples:
   - a) upper sample - middle of upper third of tank contents.
   - b) middle sample - middle of tank contents.
   - c) lower sample - near bottom of tank contents.
3. Pull out the bottle stopper with a sharp jerk of the sampler line.
4. Allow the bottle to fill completely, as evidence by the cessation of air bubbles.
5. Raise the sampler and retrieve and cap the bottle. Wipe off the outside of the bottle with a terry towel or rag. The bottle can serve as the sample container.
6. Label each of the three samples collected; affix seal; fill out sample analysis request sheet and chain of custody record; record in the field log book.
7. Clean onsite or store contaminated sampler in a plastic bag for subsequent cleaning.
8. Deliver the sample to the laboratory for analysis (see Section 6). Instruct the laboratory to perform analysis on each sample or a composite of the samples.



1000-ml (1-quart) weighted
bottle catcher

Figure 9. Weighted bottle sampler.

# APPENDIX II

# GLOSSARY OF TERMS

Accuracy -- Difference between individual analytical measurements and the true value, corresponding to the sum of systematic and random errors (Section 4.8.3).

Analyte — Compound or element of interest.

Analytical Method -- Set of written instructions completely defining the procedure to be adopted by the analyst in order to obtain an analytical result.

Audit -- Systematic check to determine the quality of operation of some function or activity.

Chain of Custody — An accurate written record which can be used to trace the possession and handling of a sample from the moment of collection through analysis.

Coefficient of Variation -- Same as Percent Relative Standard Deviation.

Confidence Limit -- The statistically derived upper and lower bounds of a particular quality control measurement. These bounds describe the range of acceptability for this measurement. Typically, the range is ±3 standard deviations ( 99% confidence interval ). The lower bound is never less than zero.

Control Samples -- Samples introduced into the sample stream to be used as monitors on the performance of the analytical method (Section 7.2).

Correlation coefficient — A number between -1 and 1 that indicates the degree of linear relationship between two sets of numbers. It is represented by an "r". It is calculated with the formula:

$$r_{xy} = \frac{n\Sigma XY - \Sigma X \Sigma Y}{\{[n\Sigma X^2 - (\Sigma X)^2][n\Sigma Y^2 - (\Sigma Y)^2]\}^{1/2}}$$

Where:

$X$ = The independent variable, such as concentration.

Correlation coefficient - con't

Y = The dependant variable, such as instrument response.

n = the total number of points in the data set.

An r value of either -1 and 1 means perfect linear agreement. $r = 0$ means no relationship at all.

For more information see Linear Regression and r test.

Data Quality -- Totality of features and characteristics of a data set that bears on its ability to satisfy a given purpose.

Holding Time -- The maximum time allowable between laboratory receipt of a sample and extraction and/or analysis.

Linear Regression -- The function that indicates the linear relationship between two variables (usually denoted X and Y). The average value of Y can be estimated with the formula:

$$\hat{Y} = mX + b$$

Where: $\hat{Y}$ = Y estimate ( called Y hat )
m = slope
b = Y intercept

$$m = \frac{n\Sigma XY - \Sigma X \Sigma Y}{n\Sigma X^2 - (\Sigma X)^2}$$

$$b = \frac{\Sigma X^2 \Sigma Y - \Sigma X \Sigma XY}{n\Sigma X^2 - (\Sigma X)^2}$$

X = The independent variable, such as concentration.

Y = The dependant variable, such as instrument response.

n = the total number of points in the data set.

Matrix Spike/Matrix Spike Duplicate - See Spike Sample.

Mean -- A measure of central tendency or location which is the sum of the observations divided by the number of observations.

$$\bar{X} = \Sigma X_i \div n$$

Where:

X = The independent variable, such as concentration.

n = the total number of points in the data set.

This is also called the Arithmetic Mean or Average.

Method -- An assemblage of measurement techniques and the order in which they are performed.

Method Blank -- Standard matrix sample to which no analyte of interest has been added that is processed in the same manner as a sample.

Outlier -- An extreme observation that is shown to have a low probability of belonging to a data population.

Precision -- Degree of mutual agreement among individual measurements made under prescribed conditions with a single test procedure.

Quality Assurance (QA) -- The total integrated program for assuring and documenting the reliability of monitoring and measurement data and for integrating quality planning, quality assessment, and quality improvement efforts to meet user requirements.

Quality Control (QC) -- The routine application of procedures for obtaining prescribed standards of performance in the monitoring and measurement process.

<u>r test</u> -- r test is the value used to evaluate the correlation coefficient used in linear regression. It is calculated as follows:

$$(r_{test})^2 = \frac{t^2}{t^2 + df}$$

$t$ = t value for df from a table of Students t Distribution

df = degrees of freedom

<u>Recovery</u> -- Difference between a known and observed value (the analytical results before and after spiking) divided by known amount (of spiking compound) and multiplied by 100 to convert to percentage.

$$Rec = 100 * observed + known$$

or

$$Rec = 100 * \frac{(observed - background)}{Spike}$$

<u>Relative Percent Difference</u> -- The absolute value of the difference between two numbers divided by the average of the two numbers:

$$RPD = \frac{|X - Y| * 200}{(X + Y)}$$

The RPD value is always a positive number.

Where:

$X$ = First observed result

$Y$ = Second observed result

<u>Relative Percent Standard Deviation</u> -- A measure of dispersion that is the standard deviation divided by the mean and multiplied by 100 to yield a percentage value.

$$\%\ RSD = \frac{100\sigma}{mean}$$

$\sigma$ = population standard deviation

<u>Replicate Sample</u> -- A sample prepared by sub-dividing a sample into two or more separate aliquots. Duplicate samples are considered to be two replicates.

<u>Response Factor</u> -- The change in size of peaks of standards that are run under the same conditions. The areas and retention time of the standards should not vary.

$$\text{RRF} = \frac{A_x}{A_{is}} \text{ times } \frac{C_{is}}{C_x}$$

Where:

$A_x$ - Area of analyte

$C_x$ - Concentration of analyte

$A_{is}$ - Area of internal Standard

$C_{is}$ - Concentration of internal standard

<u>Significant Figures</u> -- The number of digits used to express a result in scientific notation. All digits are expected to be known definitely, except the last digit, which may be in doubt.

<u>Spike Sample</u> -- A replicate sample to which predetermined quantities of target analytes are added prior to any preparation/analysis steps. Percent recoveries of the added analytes are calculated to assess method accuracy. Duplicate spike samples are analyzed to assess method precision. These samples are also called <u>Matrix Spike/Matrix Spike Duplicate</u> (MS/MSD) samples.

<u>Standard Deviation</u> -- A measure of variability of observations.

$$s^2 = [\ [1/(n-1)]\Sigma(X_i-\overline{X})^2\ ]^{1/2}$$

$$\sigma^2 = [\ [1/n]\Sigma(X_i-X)^2\ ]^{1/2}$$

$$s = \frac{\sigma}{[(n-1)/n]^{1/2}}$$

Standard Deviation - con't

Where:

$X_i$ = A data point.

$\bar{X}$ = The mean of the points.

n = the total number of points in the data set.

As n gets larger, $s \approx \sigma$ .

Standard Operating Procedure (SOP) -- A written document which details an operation, analysis or action whose mechanisms are thoroughly prescribed and which is commonly accepted as the method for performing certain routine or repetitive tasks.

Trip Blank -- Standard matrix sample, to which no analyte of interest has been added, that is transported to the sampling site and back, to ensure that no contamination is introduced during shipment.

t test -- The test to see if a value is significantly different from a population mean:

$$t_n = \frac{|X - \mu|}{\sigma}$$

$\mu$ = population mean

$\sigma$ = population standard deviation

The value for $t_n$ is an unsigned number. This value is compared to a critical value from a Student's t distribution table for the number of degree of freedom in $\mu$. If the $t_n$ value exceeds the critical value, X is considered an outlier.

Traceability -- The ability to completely reconstruct all activities from the time of sampling to data reporting, including all sample handling as well as instrument maintenance, QC results, and calibration curves.

Validity -- Degree to which the reported results represent that which they are intended to represent.

# APPENDIX III

# LDR NOTIFICATION/CERTIFICATION FORMS

[RESERVED]

Revised September 11, 1989

Gundle

19103 Gundle Rd.
Houston, Texas 77073
U.S.A.

Phone: (713) 443-8564
Toll Free: (800) 435-2008
Telex: 4620281 GUNDLE HOU
Fax: (713) 875-6010

April 27, 1989

Mr. Steve Mason
U.S. Pollution Control, Inc.
2000 Classen Center, Suite 320 South
Oklahoma City, OK 73106

Dear Mr. Mason:

In response to your request for specific guidelines of Gundline HD chemical resistance, please refer to the following listing of chemical types:

| | Gundline HD Resistance |
|---|---|
| Alcohols - | resistant to 100% concentration |
| * Aldehydes - | resistant to 100% concentration |
| Amines - | resistant to 100% concentration |
| * Aromatic and Aliphatic Hydrocarbons without Functional Groups - | resistant to 100% concentration |
| Detergents and Other Cleaning Products - | resistant to 100% concentration |
| Esters - | resistant to 100% concentration |
| * Ethers - | resistant to 100% concentration |
| * Halogenated Hydrocarbons - | resistant to 100% concentration |
| - | aromatic halogenation resistant to 1% concentration |
| Inorganic Acids - | nitric acid resistant to 10% concentration |
| - | chlorosulphonic acid resistant to 5% concentration |
| - | other acids resistant to 95% |
| Inorganic Bases - | resistant to 100% concentration |
| Inorganic Salts - | resistant to 100% concentration |
| * Ketones - | resistant to 100% concentration |
| Natural Fats and Oils - | resistant to 100% concentration |
| Organic Acids - | resistant to 100% concentration |

* These materials are significantly absorbed by the liner causing some "softening" but no degradation. Concentrations in liquid next to the liner should be prudently limited to 1500 ppm for long term containment. Concentrations in soils may, however, be higher.

Note 1: This information is for containment at 23 ± 5°C. Higher temperatures would increase reactivity, especially of the inorganic acids.

Note 2: This data does not apply to the mixture of certain chemicals, e.g., a strong acid and base would react releasing much heat and possible rupture of the liner at "hot spots".

Actual report data can be obtained from Gundle by requesting the chemical resistance reports index. Other specific data is available from independent laboratory testing and from polyethylene manufacturers. Please call us if you have further questions.

Very truly yours,

Mark Cadwallader

Mark Cadwallader
Director of Research
and Technical Development

MC:jg

cc: Deborah Wilkinson
Robert Johnson

analyses at the frequency described below.[11]

### 3.4.1 SUMMARY OF HOC TESTING[12]

All hazardous wastes which are received by (or generated by) the facility and are destined for land disposal, can be grouped into one of the following categories:

1. Wastes which are subject to sampling and analytical requirements under this Plan, in conjunction with the RCRA Waste Analysis Plan or,

2. Waste streams which are not amenable to the sampling and analytical requirements of this Plan, in conjunction with the RCRA Waste Analysis Plan, and thus exempt.

3. SW 846 Method 8150 shall be performed, in addition to the GC/ECD analyses, on each waste load when USPCI has knowledge or the generator indicates that D016, D017, K042, K043, or any phenoxyacetic acid herbicides are contained in such waste stream, or the waste stream shall be rejected.

All hazardous waste pre-acceptance samples, to the extent provided herein, will be tested for HOC's, using the referenced HOC Screening Analysis Procedure (except as provided in footnote 11). From the results of the pre-acceptance analytical data, the Permittee shall classify all wastes streams (see footnote 17) that are accepted or generated by the facility and destined for land disposal at the facility as HHOC waste streams if the HOC level is greater than 650 mg/l[13] for liquids or 650 mg/kg for solids.

Where Method 8150 is performed, the results from such test may be added to the 650 ppm rejection level to arrive at a higher rejection level of 900 ppm.

---

[11] The Permittee may substitute total waste analyses in place of HOC screen at any time. The total waste analysis must employ SW-846 methods in order to accurately determine the concentration level of the halogenated organic compounds (HOCs) listed in Appendix III of 40 CFR Part 268. Whenever the Permittee substitutes total waste analyses, the total waste analysis data obtained will supercede any HOC screening results or requirements for that waste stream.

[12] The details of the HOC testing program (HOC SCREENING PROTOCOL) can be found in Section 3.4.3.

[13] Hereafter, citations to mg/l for liquids shall also include mg/kg for solids.

### 3.4.2 FREQUENCY OF HOC TESTING

A total Halogenated Organic Compound screening analysis shall be performed for all waste streams not otherwise restricted or already being managed or presented as California List HOC waste(s) as part of the pre-acceptance procedures, unless the generator or generator's agent, or intermediate TSDF submits information specifically addressing the California List waste issue[14] indicating the material is not so regulated.

HOC TESTING FREQUENCY FOR HHOC WASTE STREAMS

The Permittee shall sample and analyze for HOC's at least 10% of all incoming loads of the HHOC waste streams identified through the Pre-acceptance Procedure to assist in the determining that the Appendix III HOC level is not above 1,000 mg/kg (Note: the specific Appendix III HOC constituents of concern will be previously identified during the pre-acceptance process by the GC/MS analysis).

Where only the HOC Screening Procedure (i.e. GC/ECD) has been performed on a hazardous waste stream, all loads of such hazardous waste streams destined for land disposal at the Grassy Mountain Facility will be analyzed for total HOC concentration[15].

If the material contains less than 1,000 mg/kg of HOC's, the material will be considered acceptable. If the HOC test indicates greater than 1,000 mg/kg is present, the material will be rejected for direct land disposal or the GC/MS analysis will be run for confirmatory acceptance[16].

### 3.4.3 HOC SCREENING PROTOCOL

SAMPLE PREPARATION FOR SOLID, LIQUID, OR SLUDGE PHASE WASTES

Refer to Section VII of the Standard Operating Procedure (SOP) Number GM-053.02 (HOC Screening Analysis) for various sample matrix preparation procedures.

---

[14] Information of this nature shall include additional certifications, additional analysis, submittal of waste profile sheets expressly addressing and California Waste issue, etc...

[15] See footnote #11.

[16] See footnote #11

### 3.4.2 FREQUENCY OF HOC TESTING

A total Halogenated Organic Compound screening analysis shall be performed for all waste streams not otherwise restricted or already being managed or presented as California List HOC waste(s) as part of the pre-acceptance procedures, unless the generator or generator's agent, or intermediate TSDF submits information specifically addressing the California List waste issue[14] indicating the material is not so regulated.

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[15] See footnote #11.

[16] See footnote #11

## 3.4 HOC SCREENING TEST

Land Disposal Restrictions regulations impose substantial new responsibilities on those facilities which manage hazardous waste. These new responsibilities include but are not limited to the requirements of 40 CFR 268.7(c)(2).

Paragraph 268.32(j)(2)[10] states:

> The initial generator of either a liquid hazardous waste containing polychlorinated biphenyls (PCBs) or a liquid or non liquid hazardous waste containing halogenated organic compounds (HOCs) must test his waste (not an extract or filtrate), or use knowledge of the waste, to determine whether the concentration levels in the waste equal or exceed the prohibition levels specified in this section.

The Permittee must screen wastes in such a way as to prevent wastes whose halogenated organic constituents concentration exceed the statutory prohibition levels. The Permittee shall use the "HOC Screening Analysis" (Standard Operating Procedure No. GM-053.02) as the screening procedure. The Permittee shall perform the HOC

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[10] This Federal Register citation pertains to California List wastes. The California List Waste requirements do not apply if the wastes meet the conditions listed in 40 CFR 268.32(g)(3) and 268.32(h). These paragraphs are given below:

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| 268.32(g)(3) | The wastes meet the applicable standards specified in Subpart D of this part or, where treatment standards are not specified, the wastes are in compliance with the applicable prohibition set forth in this section or RCRA section 3004(d). |
| 268.32(h) | The prohibitions are effective dates specified in paragraphs (a)(3), (d), and (e) of this section do not apply where the waste is subject to a Part 268 Subpart C prohibition and effective date for a specified HOC (such as hazardous waste chlorinated solvent, see e.g., paragraph 268.30(a)). |

### 3.4.4 PRE-ACCEPTANCE PROCEDURE

The Permittee shall perform HOC analyses using SOP Procedure Number GM-053.02 (and as subsequently revised by an approved permit modification) for all hazardous waste streams that are destined for land disposal at the facility, as part of the pre-acceptance procedure[17].

Liquid hazardous waste streams whose representative samples are found to contain HOC's in excess of 650 mg/l, and non-liquid hazardous waste streams whose representative extract samples are found to contain HOC's in excess of 650 mg/kg. during the pre-acceptance HOC screening analysis, are HHOC waste streams for the purposes of this Waste Analysis Plan.

### 3.4.5 COMPREHENSIVE ANALYSES

The Permittee may analyze the waste or have it analyzed to determine the concentration of each constituent listed on Appendix III of 40 CFR part 268 that may exist in the waste. If this laboratory uses EPA approved techniques and demonstrates that the sum of California List HOC's in the waste does not exceed 1,000 mg/l or mg/kg, as appropriate, the Permittee may land dispose this waste stream after recording these data in the operating record.

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[17] Each waste stream which has not been analyzed for HOC (under the Pre-acceptance Procedure) is a HHOC waste stream, unless conditions of footnote 10 are met.