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TECHNICAL ASSISTANCE DOCUMENT FOR COMPLYING WITH THE TC RULE AND IMPLEMENTING THE TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)

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Document Introduction

The goal of this document is to assist the regulated community to make proper utilization of the Toxicity Characteristic Leaching Procedure (TCLP) to demonstrate compliance with the Toxicity Characteristic (TC) and Land Ban Regulations. The following issues will be discussed:

- What is TCLP?
- When must TCLP be performed?
- Which analyte lists should be used to demonstrate compliance with TC and Land Ban regulations?
- How should a sampling strategy be developed?
- How much QA/QC and analytical deliverables are appropriate?
- How should one use the USEPA Region 2 TCLP data validation criteria?
- How should sampling plans be developed for multi-phase and oily materials?

The following topics were added to the 1994 version of this document:

- Obtaining CAMU variances from the Land Ban regulations.
- Inappropriateness of TCLP method for risk assessments.
- Characterizing heterogeneous solid wastes.
- Characterizing building demolition debris containing lead based paint.
- Implications of classifying non-hazardous wastes as hazardous.
- Region 2 State TCLP policy guidances.

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List of Abbreviations and Acronyms

AC	Alternating Current
ARARs	Applicable or Relevant and Appropriate Requirements
ASTM	American Society of Testing and Materials
BDAT	Best Demonstrated Available Technology
BNA	Base, Neutral and Acid Extractable Organics
CAMU	Corrective Action Management Unit
CBEC	Concentration Based Exemption Criteria
CCWE	Constituent Concentrations in Waste Extracts
CERCLA	Comprehensive Environmental Response Compensation and Liability Act of 1980
CESQG	Conditionally Exempt Small Quantity Generator
CFR	Code of Federal Regulations
CLP	Contract Laboratory Program
COC	Chain of Custody
CRDL	Contract Required Detection Limits
CTRL	Chronic Toxicity Reference Levels
CVAA	Cold Vapor Atomic Adsorbtion
CWA	Clean Water Act
DAF	Dilution Attenuation Factor
DC	Direct Current
DQO	Data Quality Objectives
DQOPP	Data Quality Objectives Planning Process
FAA	Flame Atomic Adsorbtion
FIFRA	Federal Insecticide, Fungicide, and Rodenticide Act
EP Tox	Extraction Procedure Toxicity
EQL	Estimated Quantitation Limit
GFAA	Graphite Furnace Atomic Absorption
HAZWRAP	Hazardous Waste Remedial Action Program
HSWA	Hazardous Solid Waste Amendments
HWN	Hazardous Waste Numbers P designates Acute Toxicity U designates Toxic Waste K designates Process Waste F designates Generic Source Waste D designates Characteristic Waste
ICP	Inductively Coupled Plasma
LDR	Land Disposal Restrictions
LQG	Large Quantity Generator
MCL	Maximum Contaminant Level
MCLG	Maximum Contaminant Level Goals
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NIPDWS	National Interim Primary Drinking Water Standards
NPDES	National Pollutant Discharge Elimination System
PCB	Polychlorinated Biphenyls
POHC	Principal Organic Hazardous Constituent
POTWs	Publicly Owned Treatment Works
PPIC	Pollution Prevention Information Clearinghouse
PPIES	Pollution Prevention Information Exchange System
PQL	Practical Quantitation Limit
QA	Quality Assurance
QAMS	Quality Assurance Management Staff
QC	Quality Control
RA	Regional Administrator

List of Abbreviations and Acronyms (Continued)

RCRA	Resource Conservation and Recovery Act
RFI	RCRA Facility Investigation
RSD	Risk Specific Doses
RfD	Referenced Doses
SDWA	Safe Drinking Water Act
SQG	Small Quantity Generator
SW-846	Solid Waste Procedures
TC	Toxicity Characteristic
TCLP	Toxicity Characteristic Leaching Procedure
TIC	Tentatively Identified Compound
TSCA	Toxic Substances Control Act
TSDF	Treatment Storage and Disposal Facility
UIC	Underground Injection Control
USDW	Underground Sources of Drinking Water
UST	Underground Storage Tank
VOA	Volatile Organic Analysis
ZHE	Zero Headspace Extraction

Chapter 1

Chapter 1

COMPLYING WITH THE TC RULE

Introduction

RCRA Overview

The Toxicity Characteristic Rule

TC Rule's Effect on Individual RCRA Regulations

Impact on Other RCRA Programs

Pollution Prevention

Conclusions

1.0 COMPLYING WITH THE TOXICITY CHARACTERISTIC RULE

The purpose of this chapter is to provide an understanding of the Toxicity Characteristic (TC) Rule, as it relates to hazardous waste management under the Resource Conservation and Recovery Act (RCRA). The development of hazardous waste issues in the United States is discussed first, giving the example of Love Canal, which is a case study of an uncontrolled hazardous waste site. Then an overview of RCRA is presented, with a comparison of the Extraction Procedure Toxicity (EP Tox) Test and the TC Rule, including the Toxicity Characteristic Leaching Procedure (TCLP) is presented. Finally, the impact of the TC Rule on RCRA and non-RCRA regulations is discussed.

1.1 Introduction

- Hazardous waste growth in America.
- RCRA system to control hazardous waste disposal.
- Determination of hazardous waste by testing.
- Changes to the testing procedure for hazardous waste. TC Rule replaces the EP Tox Test.
- Purpose of the manual.

- In America, about 500,000 companies generate approximately 170,000 metric tons of hazardous waste annually.
- In 1976, the Resource Conservation and Recovery Act (RCRA) was passed for proper management of hazardous waste to protect human health and the environment.
- The extraction procedure toxicity (EP Tox) test was one of the analytical methods used to ascertain if a waste was hazardous.
- The Toxicity Characteristic Leaching Procedure (TCLP) replaced the EP Tox test when determining if a solid waste is hazardous because it exhibits the toxicity characteristic.

1.2 RCRA OVERVIEW

1.2.1 Growth of Hazardous Waste in America: The Case of Love Canal

- America in the 1890s.
- The invention of electricity.
- New chemicals.
- Clustering of industries around power sources.
- Building of Love Canal to connect the lower and upper parts of Niagara Falls.
- DC power versus AC power.

- America in the 1890s was undergoing industrialization. With the invention of electric power, new chemicals were developed. A widely used chemical process was the electrolysis of sodium chloride (salt) to yield sodium hydroxide (lye), chlorine and hydrogen. Lye was mixed with waste animal fat from slaughter houses to produce soap. Ivory soap is still made this way. Chlorine, originally a useless byproduct, eventually was utilized as a raw material in the production of chlorinated solvents and pesticides. These chemicals had toxic effects which were not then understood.

- Direct current (DC) electricity was used for this electrolysis process. Industries in the 1890s which used significant quantities of electricity had to cluster around their DC power sources because DC electricity does not travel efficiently.

- In Niagara Falls, a hydroelectric dam generated low cost electrical energy. Industries were clustered in the area to obtain inexpensive DC power. Construction commenced on the Love Canal industrial transportation network to connect the lower and upper parts of the Niagara Falls area.

- However, when alternating current (AC) electricity was invented, electricity travelled much further over power lines. Thus, it was no longer necessary to cluster industries around the Niagara Falls area, and the Love Canal project was abandoned. Love Canal was subsequently used by industry as a hazardous waste dump, and leached toxic contaminants into the surrounding ground water and soil. Even after Love Canal was capped and closed, toxic chemicals continued to leach out.

- Most of the discarded hazardous chemicals were contained in the clay-lined Love Canal until school and highway construction ruptured the walls. This fracturing of the walls caused chemicals to contaminate local homes and the school built directly over the dump site. People living in the area started to experience health problems, including miscarriages, stillbirths, and chromosome damage.

- In 1980, President Jimmy Carter evacuated approximately 700 families out of the Love Canal area to protect their health.

- The purpose of RCRA is to prevent this type of inadequate hazardous waste management.

1.2.2 RCRA Cradle to Grave Concept

- RCRA tracks hazardous waste from cradle to grave:
  - the manifest system.
- Components:
  - generator
  - transporter
  - treatment storage and disposal facility (TSDF).
- Anyone generating hazardous waste must notify EPA:
  - generator definition.

- RCRA is considered a "cradle to grave" system because it regulates the handling of hazardous waste from creation to disposal. This ensures that hazardous waste is handled properly, and does not contaminate the environment.
- There are three hazardous waste handler classifications: the generator, who creates the hazardous waste; the transporter, who transports the hazardous waste to the ultimate disposal site; and the ultimate disposal site, which is called a treatment, storage and disposal facility (TSDF). Final disposition of hazardous waste often occurs after interim storage/treatment/recycling operations at several sites.
- Facilities that generate solid waste must determine if their solid waste is a hazardous waste.
- Facilities that generate hazardous waste must notify EPA or the authorized State agency, and obtain an EPA facility identification number (EPA ID number).
- The EPA or an authorized state agency issues an EPA ID number to make unique identification of each TSDF which handles hazardous waste.
- Hazardous waste generators must manage hazardous waste according to RCRA regulations. The generator must dispose of hazardous waste properly, and must complete a manifest enumerating the contents of the waste.
- The generator is responsible for using authorized transporters and disposal facilities.
- The generator can usually store hazardous waste on site for up to 90 days without a storage permit.

- Generator responsibilities:
  - Determination of hazardous waste
  - Notify EPA
  - Obtain EPA ID number
  - Prepare manifest
  - Dispose of waste using an authorized transporter and an authorized TSDF
  - Accumulation time
  - Annual reports
  - Contingency plans/Training requirements
- Generator categories - CESQG/SQG/LQG
- RCRA enforcement:
  - State versus Federal

- The generator must file bi-annual reports detailing the amount of waste disposed.
- EPA generator categories (many states have different generator categories):
  - LQG - Large quantity generators (LQG) generate more than 1000 kilograms (kg) of hazardous waste/month or more than 1 kg of acutely hazardous waste/month. LQGs are fully regulated and must comply with all generator requirements indicated above.
  - SQG - Small quantity generators (SQG) are generators which:
    - Generate between 100 and 1,000 kg/month of hazardous waste.
    - Accumulate no more than 6,000 kg of hazardous waste on site at any one time.
    - Accumulate hazardous waste on site for up to 180 days or 270 days if the disposal site for the waste is over 200 miles away.
    - Provide notification of hazardous waste activities, use manifests to dispose of hazardous waste, and dispose of hazardous waste at TSDFs, but do not file annual reports.

CESQG - Conditionally exempt small quantity generators (CESQG) are generators which:

- Generate less than 100 kg/month non-acute hazardous waste per calendar month or;
- Generate less than 1 kg/month acutely hazardous waste (P-waste code). There are also several acutely hazardous F-listed wastes.
- May never accumulate more than 1,000 kg of hazardous waste or greater than 1 kg of acutely hazardous waste at any time. If they do, they will be regulated as a SQG.
- Are subject to reduced requirements. SQGs do not need to notify EPA or State agencies, use manifests, or dispose of their hazardous waste in a TSDF (they may use a municipal or industrial landfill).

- RCRA enforcement:

  - EPA has delegated RCRA enforcement authority to many states. In those states, the hazardous waste regulations may not be less strict than EPA's regulations. Some states have regulations which are more stringent than EPA's.
  - EPA however, retains overall jurisdiction over all state RCRA programs.

1.2.3 Definitions of Hazardous Waste

- Only wastes classified as "solid wastes" may be characterized as "hazardous wastes".
- The definition of hazardous waste has four parts:
  - "Statutory definition"
  - "Listed waste" - Four lists: F, K, U, P
  - "Mixture rule" - Defines hazardous waste as being a mixture of a hazardous waste and a non-hazardous waste.
  - "Characteristic waste"
    - Ignitable
    - Corrosive
    - Reactive
    - Toxic - (TC Rule)

- Only wastes classified as "solid wastes" may be characterized as "hazardous wastes." Definition of solid waste:

  "The term solid waste means any garbage, refuse, or sludge, from a waste treatment plant, water supply treatment plant, or air pollution control facility; and other discarded material, including solid, liquid, semisolid, or contained gaseous material resulting from industrial, commercial, mining, and agricultural operations, and from community activities, but does not include solid or dissolved materials in irrigation return flows or industrial discharges which are point sources subject to permits under Section 402 of the Federal Water Pollution Control Act, as amended Statute 880; or source, special nuclear, or byproduct material as defined by the Atomic Energy Act of 1954, as amended (68 Statute 923)."[1]

- Definition of hazardous waste:

  "The term 'hazardous waste' means a solid waste, or combination of solid wastes, which because of its quantity, concentration, or physical, chemical or infectious characteristics may:

  (A) cause, or significantly contribute to an increase in mortality or an increase in serious irreversible, or incapacitating reversible, illness; or

  (B) pose a substantial present or potential hazard to human health or the environment when improperly treated, stored, transported, or disposed of, or otherwise managed."[2]

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[1] 42 United States Code (USC) 6903, Section 1004(27)

[2] - Ibid, (5)

Regulatory Definitions:

- Listed hazardous waste - Wastes classified as hazardous because of how they were produced. There are four lists of hazardous waste: F, K, U, and P.

  Four types of listed waste:

  - F Waste - List of waste from non-specific sources
  - K Waste - List of waste from specific sources
  - U Waste - List of discarded chemical products
  - P Waste - List of acutely toxic discarded chemical products

- Mixture rule - Any mixture of a listed hazardous waste and a non-hazardous waste is considered hazardous.

- Derived from rule - See 40 CFR 261.3c.

- Characteristic waste - Four characteristics are utilized to determine if a solid waste, which is not a listed hazardous waste, is classified as a hazardous waste.

- Characteristic of ignitability:
  - A flashpoint of < 140°F
  - For non-liquids - if the waste, when ignited, can burn spontaneously
  - An ignitable compressed gas
  - An oxidizer as defined in 49 CFR 173.151

- Characteristic of corrosivity:
  - The waste is aqueous and pH $\leq$ 2 or $\geq$ 12.5.
  - The waste corrodes steel at a rate of $\geq$ 6.35 millimeters/year.

- Characteristic of reactivity:
  - The waste is unstable and undergoes violent reaction.
  - The waste reacts violently with water.
  - The waste, when heated, is explosive.
  - The waste, when mixed with water, releases toxic gases.
  - The waste contains cyanide or a sulfide, and releases toxic gases when exposed to pH conditions between 2 and 12.5.
  - The waste can explode if shocked or heated.
  - The waste is defined as an explosive by U.S. Department of Transportation regulations.

- Characteristic of toxicity - A waste exhibits the characteristic of toxicity if the concentration of one or more of the 39 toxicity characteristic analytes in the TCLP aqueous extract exceeds regulatory action levels. This is known as the TC Rule. This replaces the EP Tox Test, which contained 8 inorganic and 6 organic constituents.

  Note: If wastes are listed solely because they exhibit a characteristic, and the resulting mixture no longer exhibits a characteristic, the material is no longer a hazardous waste.

1.2.4 Making a Hazardous Waste Determination

- Does the waste meet the definition of solid and hazardous waste?
- Is the waste excluded?
- Is the waste listed?
- Does the waste exhibit a characteristic of hazardous waste?

Hazardous Waste Determination

- Is the waste a solid waste?
- Is the solid waste excluded from the RCRA regulations?
- If the solid waste is not excluded from the hazardous waste regulations, is it a listed waste? By definition, listed wastes are hazardous wastes.
- If neither excluded nor listed, does this solid waste exhibit any of the characteristics of hazardous waste? If so, it is a hazardous waste.

  Note: Solid waste is determined to be hazardous waste by:

  - The generator's reasonable knowledge of the characteristics of the waste, or
  - The generator's testing of the waste.

Listed hazardous waste is hazardous regardless of analyte concentrations in TCLP extract.

1.3 THE TOXICITY CHARACTERISTIC RULE

1.3.1 EP Tox Test

- What is the Extraction Procedure Toxicity (EP Tox) Test?
- What is the EP Tox Test based on?
  - Landfill leaching,
  - 14 metals and organics form the basis of the EP Tox Test.
- Contaminant levels of the EP Tox Test
  - Relationship to drinking water standards,
  - Dilution attenuation factor (DAF).

- EP Tox Test was utilized prior to TCLP to ascertain if a waste exhibited the characteristic of toxicity. The EP Tox Test analyzed waste extracts for 14 specified chemical constituents.
- EP Tox Test was based on the assumption that chemicals placed in a landfill will leach at a uniform rate into the ground water.
- EP Tox regulatory action levels are based upon drinking water standards:
  - (allowable drinking water level) X 100 = EP Tox regulatory level (assumes that toxic chemicals leaching out of landfill will be diluted by a factor of 100);
  - The factor of 100 is termed a dilution attenuation factor (DAF).
  - The 14 metals and organic chemicals regulated by the drinking water program were assigned EP Tox regulatory levels.

- 8 metals	Arsenic Barium Cadmium Chromium Lead Mercury Selenium Silver
- 4 insecticides	Endrin Lindane Methoxychlor Toxaphene
- 2 herbicides	2,4 - D 2,4,5 - TP (silvex)

1.3.2 TCLP Test

- Why replace the EP Tox Test with the TC Rule?
- How is TC different than EP Tox?
- How regulatory limits are determined for the TC Rule.
  - Regulatory level = CTRL X DAF

- The EP Tox Test was replaced by the TC Rule because of a Congressional mandate for aggressive regulation of additional toxic constituents.
- The original EP Tox Test was not capable of leaching volatile organic compounds without unacceptable losses during the leach test.
- Major changes:
  - 25 additional organic chemicals
  - different leaching medium
  - procedural modifications for leaching volatile compounds
- The regulatory limit for the TCLP constituents was determined by multiplying the Chronic Toxicity Reference Level (CTRL) times the Dilution Attenuation Factor (DAF):

  Regulatory limit = CTRL X DAF

  The CTRL is a level below which health effects are not expected to occur. The CTRL is based on: drinking water standards maximum contaminant level (MCL); or for carcinogens, the risk specific dose (RSD); or for non-carcinogens, the reference dose (RfD).

TABLE 1-1 - TC RULE CONSTITUENTS

EPA WASTE NUMBER	HAZARDOUS CONSTITUENT[1]	Level (mg/l)	EPA WASTE NUMBER	HAZARDOUS CONSTITUENT[2]	Level (mg/l)	EPA WASTE NUMBER	HAZARDOUS CONSTITUENT[2]	Level (mg/l)
D004	Arsenic	5.0	D018	Benzene	0.5	D031	Heptachlor (& epoxide)	0.008
D005	Barium	100.0	D019	Carbon tetrachloride	0.5	D032	Hexachlorobenzene	0.13
D006	Cadmium	1.0	D020	Chlordane	0.03	D033	Hexachloro-1,3-butadiene	0.5
D007	Chromium	5.0	D021	Chlorobenzene	100.0	D034	Hexachloroethane	3.0
D008	Lead	5.0	D022	Chloroform	6.0	D035	Methyl ethyl ketone	200.0
D009	Mercury	0.2	D023	o-Cresol	200.0	D036	Nitrobenzene	2.0
D010	Selenium	1.0	D024	m-Cresol	200.0	D037	Pentachlorophenol	100.0
D011	Silver	5.0	D025	p-Cresol	200.0	D038	Pyridine	5.0
D012	Endrin	0.02	D026	Cresol (total)	200.0	D039	Tetrachloroethylene	0.7
D013	Lindane	0.4	D027	1,4-Dichlorobenzene	7.5	D040	Trichloroethylene	0.5
D014	Methoxychlor	10.0	D028	1,2-Dichloroethane	0.5	D041	2,3,5-Trichlorophenol	400.0
D015	Toxaphene	0.5	D029	1,1-Dichloroethylene	0.7	D042	2,4,6-Trichlorophenol	2.0
D016	2,4-D	10.0	D030	2,4-Dinitrotoluene	0.13	D043	Vinyl chloride	0.2
D017	2,4,5-TP (Silvex)	1.0						

[1] Original EP Tox constituents.
[2] Chemical constituent added by TC Rule (shaded areas).

1.3.3 TC Rule's Effect Upon Generators and TSDFs

- Generators
  - Notification
  - Manifests
  - Annual report
  - LDR
- In addition to the above requirements, TSDFs must
  - Obtain a new permit
  - Modify an existing permit
  - Close prior to obtaining a permit
  - Obtain interim status
  - Make changes to interim status
  - Meet minimum technology requirements for pretreatment

- Generators which produce newly regulated TC hazardous waste not previously regulated, must submit notification, use manifests, etc., as required by their generator status. Generator responsibilities are discussed in Section 1.2.2. If the waste was previously regulated under EP Tox, no additional requirements are applicable.

- Options for TSDFs

  Obtain a new permit

  - Land disposal facilities newly regulated by the TC Rule are required to comply with minimum technology requirements when new units are added, existing units are replaced, or existing units are laterally expanded.

  - New permit requirements are enumerated in 40 CFR 270.

  Modify an existing permit (see Table 1-2)

  - The three classes of permit modifications are based on the significance of the modification. This three-tiered process, which replaces the two-tiered major/minor process, is used by the permittee to initiate a permit change. In contrast, EPA uses the old major/minor process if it initiates a permit change.

  - Class 1 - modifications for routine changes;

  - Class 2 - modifications for changes of moderate complexity that allow the facility to respond to changing conditions; and

  - Class 3 - modifications for substantial facility alterations.

- If waste at a permitted Subtitle C facility exhibits the TC for constituents that were previously identified as EP toxic, the facility continues to comply with its permit. No permit modification is needed.

- Permitted Subtitle C facilities/units handling newly regulated TC wastes must submit permit modifications to incorporate:

    - new TC Rule wastes
    - new regulated units managing TC Rule wastes

TSDF Options

- Close the facility prior to obtaining a permit.

- Obtain interim status by submitting a Part A application as an interim permitted TSDF.

- Changes to interim status

    - Change the conditions of the Part A interim status permit application to reflect the new hazardous waste.
    - EPA's new procedures for interim status are listed in 40 CFR 270.72 (a)(1)
    - No prior approval is required for adding newly regulated units to Part A permit applications if:
        - Units were managing new wastes (e.g., TC wastes) on or before effective date.
        - Amended Part A was submitted by effective date.
        - Prior to March 7, 1989, new units received approval from EPA.
        - These new units are not subject to the reconstruction limit, which restricts cumulative interim status facility changes to less than 50% of the capital costs of a comparable new facility.

Changes to interim status - Facilities with Surface Impoundments

- Implementation of the TC Rule may cause some facilities to alter their management practices to avoid regulation of certain units under Subtitle C of RCRA.
- Retrofitting surface impoundments to accept TC wastes entails adding liners and leachate collection systems not installed when the impoundment was constructed.

Changes to interim status - Land Disposal Units

- New land disposal units should have submitted certification of compliance with ground water monitoring and financial responsibility requirements by September 25, 1991.

- Land Ban Requirements

  - Land disposal restrictions refer to restrictions on the land disposal of hazardous wastes. Restricted wastes must be treated as specified in the LDR regulations, otherwise they are banned from disposal on land.

  - Any TC Rule wastes regulated by the LDR regulations would be prohibited from land disposal.

- Minimum Technology Requirements - Surface Impoundments

- Surface impoundments which were newly regulated as a result of the TC Rule were required to meet minimum technology standards by March 29, 1994.

TABLE 1-2 - PERMIT MODIFICATIONS

Type of Unit Involved	Permit Change Needed	Modification Class
Tank or Container	Addition of **waste codes** or **units** that will not require additional or different management practices than specified in the permit.	2
	Addition of **waste codes** or **units** that will require additional or different management practices than specified in the permit.	3
Surface Impoundment, Landfill, Waste Pile, or Land Treatment	Addition of **waste codes** or **units** that will **not** require additional or different management practices than specified in the permit.	2
	Addition of **waste codes** or **units** that will require additional or different management practices than specified in the permit.	3
	Addition of **units.**	3
Incinerator	If waste does not contain a principal organic hazardous constituent (POHC) that is more difficult to incinerate and no additional performance standards are needed.	2
	If waste contains POHC that is more difficult to incinerate.	3
	If different performance standards are needed in the permit.	3

1.4 TC RULE'S EFFECT ON INDIVIDUAL RCRA REGULATIONS

1.4.1 General

- The TC Rule has potential impact on other parts of the RCRA program.
  - Regulations not impacted
  - Regulations which are impacted

- The TC Rule will not affect wastes already considered hazardous. The following RCRA regulations are not affected by the TC Rule:
  - Listed hazardous wastes (i.e., the F, K, P, and U lists)
  - Wastes that are hazardous by the "Mixture" and "Derived From" rules
  - Wastes already excluded from regulation

- The implementation of the TC Rule increases the categories and volume of solid waste classified as hazardous waste. The expanded definition causes additional solid waste to be classified as hazardous wastes. The following RCRA regulations are affected by the TC Rule:
  - Corrective action and closure
  - Land disposal restriction (LDR) regulations
  - Minimum technology requirements for surface impoundments and landfills
  - Mixture rule exemptions
  - Previously delisted wastes
  - Special waste exclusions

1.4.2 Corrective Action and Closure

- TC increased the universe of regulated facilities.

  Number of Subtitle C permitted and interim status facilities subject to corrective action increased.
  - Number of regulated units within permitted or interim status facilities undergoing closure increased.

- Excavated material from corrective action and closure that exhibits the TC must be managed as hazardous waste.

- TC levels are *not* used to set clean-up levels for corrective actions or clean closures.

- The TC Rule added more wastes of concern and brought more facilities under the RCRA program as hazardous waste management facilities. Therefore, additional facilities are newly subject to the Subtitle C corrective action and closure requirements.

  - Previously unregulated TSDFs managing TC Rule wastes were subject to RCRA requirements if they did not close or change management practices (e.g., exempt tanks) before the TC Rule became effective.

  - Existing RCRA facilities may have to amend their closure plans to reflect newly regulated units as the TC Rule expands the number of regulated units.

- Excavated materials, which did not previously exhibit the toxicity characteristic, may now have to be managed as hazardous waste because of the addition of newly added constituents to the regulated list.

- The Subtitle C corrective action program addresses remediation of hazardous waste releases from facilities subject to RCRA permitting. The TC Rule levels are neither action levels nor cleanup standards, both of which are developed from site-specific information gathered during the investigatory and evaluation phases of the remediation process.

1.4.3 Land Disposal Restrictions (LDR)

- LDR standards will continue to affect the 14 wastes previously regulated under the EP Tox Test.
- No LDR standards are currently promulgated for the 25 new TC constituents.
- LDR treatment standards are based on the best demonstrated available technology (BDAT) standards. The characteristic (regulatory) levels were developed using a risk-based approach.
- For some constituents, LDR treatment standards are set at the regulatory level.

- HSWA requires EPA to make an LDR determination for all newly listed wastes within six months of publication in the *Federal Register*, or by the effective date of the TC Rule ruling. Newly listed or identified wastes were not automatically prohibited from land disposal under LDRs if EPA failed to make this determination within six months (i.e., no "hammer" provisions).
  - EPA set LDR standards for the 14 original EP characteristic constituents, which EPA does not consider newly identified. These 14 constituents had to meet LDR treatment standards before land disposal on the effective date of the TC Rule.
  - The 25 additional organics identified by the TC Rule are considered newly identified, and as such have not yet been affected by LDR regulations.
- EPA is reviewing the treatability of each TC Rule constituent independently to determine LDR treatment standards for TC Rule wastes. These standards may differ from standards set for spent solvent wastes (F001-F005) based on differences in treatability.
- LDR treatment standards are based entirely on technology-based standards expressed as BDAT. While TC Rule levels are based upon health-based allowable concentration levels and dilution/attenuation factors, they are not the same as LDR treatment standards. However, for many TC wastes, EPA has set the LDR treatment standards at the regulatory level.
- This issue is being litigated. Therefore, the LDR treatment standards for TC Rule wastes may change.

1.4.4 Minimum Technology Requirements for Landfills and Surface Impoundments

- Landfills and surface impoundments newly regulated under RCRA because of the TC need to comply with minimum technology requirements
- HSWA requires that:
  - Interim status waste piles, landfills, and surface impoundments must meet certain minimum technology requirements
  - Surface impoundments must be retrofitted to meet minimum technology requirements

- Existing land disposal units, except surface impoundments, that already contained TC Rule wastes will not require retrofitting unless they are expanding, replacing units or continuing to place TC Rule wastes in these units.
- The minimum technology requirements (liners and leachate collection systems) for interim status surface impoundments are found in 40 CFR 265.221.
- Surface impoundments that become regulated under Subtitle C because of the TC Rule must have met the minimum technology requirements by March 29, 1994. This extension applied to those impoundments that contain the newly identified or characteristic wastes.

1.4.5 Exemption for Tanks (Minimum Technology Requirements)

- TC wastes treated in wastewater treatment tanks are exempt from hazardous waste management standards under 40 CFR 264.1(g) and 265.1(c).
- Generators that manage TC wastewaters in on-site surface impoundments may switch to exempt tanks in order to avoid Subtitle C requirements.
- However, generators and handlers should have converted their surface impoundments to tanks *prior* to effective date of the final rule to maintain the exemption.
- Facilities managing TC wastes after the effective date, even unintentionally, are subject to interim status requirements.

- 40 CFR 264.1(g) and 265.1(c) exempt wastewater treatment units containing hazardous waste from Subtitle C regulation.
- Generators that continue managing wastewaters in on-site surface impoundments or non-wastewaters on site will require either interim status or a RCRA permit modification/change during interim status, depending on whether the facility is currently a Subtitle C TSDF.

1.4.6 Mixture Rule Exemption

- The mixture rule exemption was not modified by the TC rule; mixtures of wastewaters and certain listed spent solvents are exempt from Subtitle C regulations unless the wastewaters:
  - Exhibit hazardous waste characteristic; or
  - Contain listed hazardous wastes not specified in the exemption.
- TC Rule may regulate currently exempted wastewaters under Subtitle C.

- The mixture rule under 40 CFR 261.3(a)(2)(iv) provides an exemption from RCRA Subtitle C requirements for mixtures of wastewaters and certain listed spent solvents in low concentrations.
- The mixture rule exemption only addresses hazardous waste listings. Therefore, the mixture rule exemption does not affect the TC Rule.
- The mixture rule exemption precludes mixtures of wastewaters and specific listed spent solvents from hazardous waste regulations, unless they exhibit a characteristic of hazardous waste.
- EPA proposed modifying the mixture rule exemption to make it more consistent with current risk information.
- The TC Rule regulatory levels are based on state-of-the-art toxicological data and risk assessment methodologies. In contrast, the mixture rule exemption levels are based upon less current risk information.

1.4.7 Previously Delisted Wastes

- A waste previously "excluded" under Subtitle C regulation may no longer be delisted if it exhibits a hazardous characteristic (e.g., the characteristic of toxicity).
- TC rule applies to already delisted wastes that now exhibit TC characteristics.
  - These wastes are no longer considered "not hazardous"
  - These wastes must now be managed under Subtitle C
- Because delisting levels are generally more stringent than the final TC levels, the impact of TC rule on previously delisted wastes is expected to be minimal.

- Wastes "excluded" from Subtitle C regulation under the delisting program may nevertheless be hazardous if they exhibit a hazardous characteristic (see 40 CFR 260.22). Hazardous waste characteristic levels are those above which a waste is hazardous due to a particular property; delisting levels are those below which a waste is not hazardous for any reason. Thus, it is reasonable that these two levels do not coincide.

- Although the TC Rule applies to delisted wastes, EPA does not, in general, expect that such wastes will become hazardous because of application of the revised TC Rule. However, if a previously delisted waste exhibits the TC Rule, it will again be subject to Subtitle C requirements, and the facility will have to notify EPA of its activity.

1.4.8 Special Waste Exemptions

- RCRA defines four special waste categories exempted from Subtitle C regulation:
  - Mining wastes
  - Mineral processing wastes
  - Oil and gas wastes
  - Domestic sewage
- Subtitle C regulations may apply to these special wastes on a case-by-case basis.
- Special waste exclusions are being reevaluated as mandated by Congress.

- If EPA determines that any special waste should be regulated under RCRA Subtitle C, the Agency will determine the applicability of the TC Rule to such wastes.
- After completing the studies required by RCRA Section 8002, EPA may determine that one or more special wastes should be regulated under RCRA Subtitle C. Such wastes would then be listed or the generators required to determine whether the wastes exhibit a hazardous characteristic, including those specified in the TC Rule.
- The TC Rule will have no direct effect on the following types of wastes:
  - Listed hazardous waste.
  - Wastes classified as hazardous by the "mixture" and "derived from" rules.
  - Wastes already excluded from regulation under 40 CFR 261.4

1.4.9 Listed Hazardous Waste

- TC rule has no effect on listings of hazardous waste
  - Wastes already *listed* as hazardous are always considered hazardous, unless they are delisted.

- Hazardous waste listings will continue to supplement the revised TC Rule. The TC Rule revisions do not eliminate any hazardous waste listings.

- Listed hazardous wastes continue to be hazardous even if they contain TC Rule constituents in concentrations below TC regulatory levels.

- TC Rule regulatory levels are not designed to identify the full range of wastes that may be toxic to human beings. Instead, the characteristic levels were established to protect human health.

- Listed wastes that do not exhibit the toxicity characteristic may nevertheless be hazardous because:
  - They contain listed hazardous waste constituents; or
  - They contain hazardous constituents that are not covered by the TC Rule.

- Listed wastes frequently contain hazardous constituents other than the ones cited in Appendix VII of 40 CFR Part 261. These additional hazardous constituents present in a waste may not be TC Rule constituents. Removing wastes from a hazardous waste listing without evaluating additional constituents would be inconsistent with the intent of RCRA §3001(f).

1.4.10 "Mixture" and "Derived From" Rules

- TC has no effect on the regulatory status of waste "mixtures" or "derived from" wastes:
  - Mixtures of listed wastes and solid wastes, and residues derived from listed wastes, are still hazardous until delisted.
- TC alone is not adequate to regulate mixtures and treatment residues.
- Problems may result by applying "mixture" and "derived from" rules.

- The "mixture" rule (40 CFR 261.2(a)(2)(iv)) states that any mixture of a listed hazardous waste and a solid waste is a RCRA hazardous waste.
- The "derived from" rule (40 CFR 261.3(c)) states that any waste derived from the treatment, storage, or disposal of a listed hazardous waste is hazardous.
- The "mixture" and "derived from" rules creates inequities in the classification of certain dilute wastes. For example, very low constituent concentrations in listed wastes may still be considered hazardous even after treatment.

1.4.11 Excluded Wastes

- TC Rule does not apply to wastes that are already excluded from Subtitle C regulations under 40 CFR 261.4:

  For example, household hazardous waste is excluded from Subtitle C; it remains excluded after the TC effective date.

- TC Rule does not add any exclusions to the applicability of previously promulgated hazardous waste characteristics.

- Wastes described in 40 CFR 261.4(b) that are already excluded from Subtitle C regulations will continue to be exempt from regulation as hazardous wastes, even if they exhibit the TC Rule.

- EPA does not at this time intend to expand the list of exemptions under 40 CFR 261.4(b) to include creosote- and pentachlorophenol-treated wood.

- Other wastes that are excluded from Subtitle C in 40 CFR 261.4(b) include:

  - Household hazardous wastes
  - Certain mining wastes
  - Certain solid wastes generated from farming or raising animals
  - Certain wastes generated from the combustion of coal or other fossil fuels
  - Wastes associated with the production of crude oil and natural gas
  - Some chromium containing wastes
  - Solid waste from extraction and processing of ores and minerals
  - Cement kiln dust wastes
  - Certain wood products

1.5 IMPACT OF TC RULE ON OTHER EPA PROGRAMS

1.5.1 Underground Storage Tanks (USTs)

- Two programs currently regulate underground storage tanks (USTs).
  - Subtitle C (tanks containing hazardous wastes) or Subtitle D (tanks containing non-hazardous solid wastes),
  - Subtitle I (tanks containing petroleum product or hazardous substance products).
- The TC Rule may increase the number of tanks regulated under Subtitle C.
- Product (petroleum, hazardous substance) that leaks may become a hazardous waste and may also exhibit TC.

- Petroleum contains several TC constituents. Therefore, it is likely that some petroleum-contaminated media will exhibit the TC.
- The management of any petroleum-contaminated media exhibiting TC would normally be subject to Subtitle C requirements for hazardous waste management. However, EPA believes further study of these impacts is necessary before imposing TC requirements on media and debris contaminated solely by petroleum from USTs.
- EPA has insufficient information on the impact of the TC Rule on UST cleanups; therefore, EPA has deferred a final decision on the application of the TC Rule to media and debris contaminated with petroleum from USTs exhibiting the D018-D043 waste characteristics that are subject to the 40 CFR Part 280 requirements.
- EPA believes deferral of a final decision concerning the application of the TC Rule to UST cleanups is necessary. Imposition of the Subtitle C requirements is likely to significantly delay cleanups and severely discourage the self-monitoring and voluntary reporting essential to implementing the UST program.

- Subtitle I and Subtitle C potentially overlap if a substance exhibits the TC characteristic and the origin of substance is not known.
- The contents of a tank determines which regulatory program (i.e., Subtitle C or Subtitle I) applies:
  - Subtitle C regulates hazardous wastes
  - Subtitle I regulates hazardous products and petroleum

- Hazardous product that leaks may become hazardous waste.
- Petroleum and hazardous product may exhibit the TC but may not be regulated under Subtitle C.
- Corrective action under Subtitle I addresses releases of product.
- Old releases of product not subject to Subtitle I may have occurred:
  - Via inactive tanks
  - In areas considered as RCRA solid waste management units
- If wastes exhibit the TC for D004-D017, RCRA standards may apply to these old releases.
- TC Rule excludes D018-D043 wastes from RCRA regulation if they are covered under Subtitle I Corrective Action:
  - Petroleum-contaminated soil and ground water
  - Petroleum-contaminated debris (tanks)
- EPA is studying impacts of Subtitle C regulation on petroleum-contaminated areas.
- Note: Petroleum contaminated media from aboveground storage tanks are also excluded from TCLP testing requirements where a state has an adequate treatment mechanism in place.

1.5.2 Comprehensive Environmental Response and Liability Act (CERCLA)

- CERCLA response actions must comply with all applicable, or relevant and appropriate requirements (ARARs), including RCRA regulations.
  - TC will cause more Superfund wastes to be classified as RCRA hazardous wastes.
  - Thus, more Superfund cleanups will be subject to RCRA regulations.
- TC will not, however, affect CERCLA clean-up levels.

- Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA or Superfund) addresses remediation of inactive waste sites.
- ARARs are "applicable or relevant and appropriate requirements." CERCLA must meet other Federal or State environmental requirements whenever they are applicable or relevant and appropriate to CERCLA actions.
- The primary effect of TC Rule on Superfund will be to regulate many organic constituents found at Superfund sites as RCRA hazardous wastes.
  - A current problem at many Superfund sites is determining if an organic constituent is from a listed RCRA hazardous waste.
  - There is often little evidence about the source of contamination that exists at Superfund sites to prove a waste is a listed waste. Therefore, a waste may not be managed under RCRA (but it will be handled in a protective manner according to risk assessment).
  - Under the TC regulation, if tetrachloroethylene is found above the TC regulatory level, the waste is hazardous regardless of its origin.
- As in RCRA corrective actions, Superfund response personnel will not use the TC to determine whether to undertake a clean-up action. The TC will affect decisions concerning the management of wastes generated during cleanup activities (i.e., hazardous wastes generated during cleanup must be managed in accordance with Subtitle C). For a lower cost Superfund cleanup option, please read the CAMU Rule discussion in Chapter 2.

1.5.3 Clean Water Act (CWA)

- The CWA regulates discharges of pollutants to surface waters and to publicly owned treatment works (POTWs).
- Regulatory levels of TC are consistent with those of the CWA:
  - NPDES effluent guidelines
  - Pretreatment standards
- Treated wastewaters that exhibit the TC Rule.

- The Clean Water Act (CWA) regulates discharges of hazardous substances to surface waters through the National Pollutant Discharge Elimination System (NPDES) permit program, and pretreatment standards for POTWs.
- EPA believes TC levels and CWA standards are consistent.
- Thus, CWA discharges are exempt from RCRA regulation under 40 CFR Part 261.
- Treated wastewaters exhibiting TC are regulated under RCRA unless:
  - discharged under NPDES permit
  - treated in POTW

- Objectives of the Clean Water Act
- Impact of the Clean Water Act

- Objectives of the CWA are:
  - To restore and maintain the chemical, physical, and biological integrity of the nation's waters;
  - To eliminate the discharge of pollutants into surface waters;
  - To attain water quality that provides for the protection and propagation of fish, shellfish, and wildlife, and provides for recreation in and on the water.

- Impact of the Clean Water Act:
  - In lieu of retrofitting and obtaining permits for existing surface impoundments to meet RCRA requirements, hazardous waste management facilities may utilize tank treatment and storage of hazardous wastewater. NPDES treatment tanks are exempt from RCRA permitting requirements.
  - Wastewater treatment facilities using surface impoundments to treat TC waste may be subject to RCRA regulations.
    - The Agency expects many owner/operators to replace surface impoundments with wastewater treatment tanks.
    - Wastewater treatment tanks are exempt from Subtitle C regulation under 40 CFR 264.1(g)(6) and 265.1(c).
  - If a POTW sludge (from wastewater treatment) exhibits TC, the owner/operator must treat the sludge to remove the characteristic.
    - Treatment may be through a pre-treatment program (i.e., before POTW receives discharge); or
    - After sludge is produced.

1.5.4 Safe Drinking Water Act (SDWA)

- Safe Drinking Water Act (SDWA) establishes maximum levels of contaminants acceptable in public drinking water supplies.
  - Maximum Contaminant Levels (MCLs)
  - Maximum Contaminant Level Goals (MCLGs)
- TC fate and transport models assume ingesting contaminated drinking water, and uses MCLs as the basis for setting regulatory levels for many TC constituents.

- SDWA protects human health from contaminants in drinking water.
- The specific objectives are:
  - To assure that all people served by public water systems be provided with a high quality water;
  - To remove contaminants found in water supplies to protect human health; and
  - To establish programs to protect underground sources of drinking water from contamination.
- SDWA primary drinking water regulations include MCLs for specific contaminants.
- Many TC levels are based on SDWA MCLs.

- Underground injection control (UIC) program regulates injection of fluids to protect underground sources of drinking water (USDWs).
- Five classes of wells are regulated under the UIC program:
  - Class I - municipal or industrial waste discharged beneath USDWs
  - Class II - oil and gas production
  - Class III - mineral recovery
  - Class IV - hazardous or radioactive waste into or above USDWs
  - Class V - all other wells used for injection of fluids, including septic tanks and sumps.

- Class I wells are often used by generators of hazardous waste or owner/operators of hazardous waste management facilities to inject hazardous waste below USDWs. The largest user of hazardous waste Class I wells is the chemical industry. Class I is the smallest class of wells with 554 reported in 1989. There are no Class I hazardous wells in Region 2.
- Class IV wells are banned with the exception of wells used for remediation of aquifers contaminated with hazardous wastes (40 CFR 144.13).
- The largest group of injection wells is Class V, with approximately 180,000 wells. The second largest group of wells is the Class II group with approximately 150,000 wells, followed by Class III wells with about 20,000 wells.
- The Agency is enforcing the ban on shallow injection of hazardous wastes. The Agency is also developing guidance on best management practices to reduce the amount and toxicity of wastes injected into Class V wells (40 CFR 144.24).
- The TC Rule may increase the number of Class I wells accepting TC waste, and bring newly identified hazardous Class I wells into the Subtitle C program.
- Some Class V wells may be illegally accepting hazardous wastes; the number may increase as a result of the TC Rule.
- Class V wells that may receive TC wastes:
  - Agricultural drainage wells;
  - Industrial drainage wells;
  - Experimental technology wells;
  - Industrial process water and waste disposal wells;
  - Automobile service station wells; and
  - Aquifer remediation-related wells.

- Wastes injected into some Class V wells are exempted from regulation as a hazardous waste. For example: geothermal electric and direct heat re-injection wells, several of the domestic wastewater disposal wells, most of the mineral and fossil fuel recovery-related wells, and certain experimental technology wells.

- Many of the facilities that operate Class V wells (e.g., auto service stations) also generate listed hazardous waste, such as solvents. It is possible that some facilities are not managing their listed wastes properly, and that hazardous wastes are entering Class V wells. Of course, when hazardous wastes are injected into Class V wells, they become Class IV wells.

- It is unclear at this time what effects TC will have on the UIC program because the Class V program is very new.

1.5.5 Federal Insecticide, Fungicide, and Rodenticide Act (FIFRA)

- FIFRA regulates the sale, distribution, and use of all pesticide products.
- RCRA regulates listed wastes, including pesticide product wastes.
- TC Rule:
  - adds one pesticide to the list of TC constituents; and
  - regulates more "multiple active ingredient" formulations.
- Other TC constituents may be ingredients in pesticides.
- If pesticide wastes exhibit TC, they are subject to Subtitle C regulation unless they are exempt.

- The following pesticide users are exempt from RCRA requirements:
  - Household pesticide users.
  - Farmers who triple rinse their containers, dispose of the containers on their own farm, and follow the pesticide manufacturer's label instruction for disposal.
  - Small quantity generators following reduced requirements. Many pesticide users are small quantity generators.
  - Properly emptied containers may be exempted from further RCRA requirements under 40 CFR 261.7. Many pesticide containers, therefore, may not be subject to regulation as hazardous waste.
- There is no change in listed pesticide wastes that are either pure, technical grade, or sole active ingredient *product* wastes; they will continue to be regulated under Subtitle C (P and U listings).
  - The exemption for arsenic-treated wood was not expanded in the TC Rule.
  - This exemption may be reevaluated in the future.
- Multiple active ingredient *products* are usually not regulated as RCRA Subtitle C wastes, but are instead regulated under RCRA Subtitle D or FIFRA.
- TC increases the potential for multiple active ingredient product wastes to be hazardous.
- Adding new pesticide constituents to the TC Rule will primarily affect commercial applicators, such as aerial applicators and pest control operators. If they use large quantities of multiple active ingredient pesticide products that have not previously been regulated, such applicators may be newly subject to RCRA Subtitle C requirements.
- Wastes from multiple active ingredient products that do not exhibit a hazardous waste characteristic will still be regulated under applicable FIFRA and RCRA Subtitle D requirements.

1.5.6 Used Oil Recycling Act

- Some used oil exhibits TC or ignitability characteristic.
- TC will affect used oil that is
  - used for road oiling;
  - that is dumped;
  - disposed in solid waste landfills and incinerators.
- TC will *not* affect used oil that is:
  - Managed by *do-it-yourselfers* (exempt as household hazardous waste);
  - Recycled through energy recovery (40 CFR Part 279 regulates this activity);
  - Recycled in any other manner (regulated under 40 CFR 279).

- Used oil may exhibit the toxicity or ignitability characteristic.
- *Disposed* used oil which exhibits the toxicity characteristic is subject to full RCRA Subtitle C regulation (40 CFR 279.80).
- However, oil that is *burned* for energy recovery is not regulated as a hazardous waste.
  - Used oil generated by household do-it-yourselfers is exempt form RCRA under 40 CFR 279.20(a)(1).
  - Used oil that exhibits one or more of the characteristics of hazardous wastes but is recycled in some other manner than being burned for energy recovery is exempt under 261.6(a)(43).
- Significant quantities of used oil may exhibit EP toxicity for metals, but little used oil is currently recognized as EP toxic.
- Shifts in used oil management practices may result from the TC Rule. Management practices may shift away from road oiling, dumping, and disposal in solid waste facilities to burning as fuel, recycling, and disposal in Subtitle C facilities.
- Standards for regulating used oil that is recycled were promulgated in the Federal Register on September 10, 1992 (FR 41566).

1.5.7 Toxic Substances Control Act (TSCA)

- TSCA addresses manufacturing, processing, and distributing hazardous substances such as PCBs.
- If TSCA-regulated *products* become wastes and contain D004-D017, they become RCRA regulated.
- If Polychlorinated Biphenyls (PCBs) are fully regulated under TSCA, TC rule exempts those PCB-wastes containing D018-D043 from RCRA regulations; not all PCB wastes are fully regulated under TSCA (D004-D017).
- Exempt wastes include PCB-containing dielectric fluids removed from:
  - Electrical transformers; and
  - Capacitors.

- Toxic Substances Control Act (TSCA) regulates toxic substances and specifically addresses PCB management and disposal.
- Dielectric fluids from electrical transformers, capacitors and associated PCB-contaminated electrical equipment could exhibit the TC because they may contain chlorinated benzenes.
  - These wastes exhibiting TC are *exempted* from Subtitle C management standards if they exhibit waste codes D018-D043.
  - The exemption applies only to certain wastes noted above that are fully regulated under TSCA, not to all PCB wastes.
- PCB wastes exhibiting D004-D017 characteristics (i.e., those hazardous under EP toxicity) remain regulated under RCRA if they are a D004-D017 waste under TC (i.e., contain other constituents).

1.6 POLLUTION PREVENTION

- Defining pollution prevention.
- Pollution prevention as a national priority for managing hazardous waste.
- Priorities of pollution prevention:
  - Source Reduction
  - Source Reduction exclusions
- Implementation.

- The Pollution Prevention Act of 1990 encourages waste reduction at the source rather than the management of waste already produced.
- An integral component of EPA's RCRA program is pollution prevention through waste minimization.
- Therefore, EPA's encourages hazardous waste source reduction rather than "end-of-pipe" controls.
- EPA has produced industry-specific outreach materials to assist industry in their waste minimization efforts (source reduction, process modifications, recycling, and the use of less toxic materials).
- Pollution prevention allows industry to:
  - Reduce costs of raw materials, hazardous waste treatment and disposal;
  - Minimize regulatory burdens of compliance;
  - Minimize liability for environmental problems and occupational safety problems;
  - Enhance efficiency and product quality.
- EPA encourages industries affected by this ruling to consider achieving compliance through pollution prevention.

- Source reduction is:
  - A practice that reduces the amount of any hazardous waste entering a waste stream or the environment that occurs prior to recycling, treatment, or disposal;
  - A practice that reduces hazards to public health and the environment due to release of hazardous substances, pollutants, or contaminants.
- Source reduction includes:
  - Equipment/technology modifications;
  - Process/procedure modifications;
  - Reformulations/redesign of products;
  - Substitution of raw materials;
  - Improvements in housekeeping, maintenance, training.
- Source reduction does not include:
  - Practices that alter the physical, chemical, biological characteristics or volume of a hazardous substance, pollutant, or contaminant through process or activity that, itself, is not integral to and necessary for the production of the product or service.
- EPA is implementing the strategy by:
  - Creating incentives for industry;
  - Building pollution prevention into their decision-making processes;
  - Making technical information available to help firms reduce waste generation through the use of:
    - The Pollution Prevention Information Clearinghouse (PPIC), a nationwide network of people and resources with direct experience in waste reduction strategies in many industries (202-260-1023);
    - The Pollution Prevention Information Exchange System (PPIES), a computer electronic bulletin board (703-506-1025) which contains a database of bulletins, programs, contacts, and reports related to pollution prevention.
  - Supporting the development of state programs to assist generators in their waste reduction efforts;
  - Initiating specific new regulatory requirements for generators to:
    - Certify on their hazardous waste manifests and annual permit reports that are reducing the volume or toxicity of their hazardous wastes as much as possible;

- Describe on their RCRA biennial reports the efforts undertaken during the year to reduce the volume and toxicity of their hazardous waste and compare the efforts to those in previous years;

- To require waste minimization/pollution prevention in RCRA permits for TSDFs that generate hazardous waste.

- EPA recommends owners/operators implementing waste minimization programs at the plant level:

  - Conduct a waste minimization assessment by selecting a few processes or waste streams for source reduction or recycling. Accurate records on the rate of generation and the cost of management should be retained;

  - Identify waste minimization techniques;

  - Practice inventory management (substitute less toxic source materials);

  - Modify equipment (upgrading the performance of process equipment, reducing leaks and malfunctions, installing conditioning or recovery systems);

  - Initiate production process changes; and

  - Recycle and reuse.

- Contact the Pollution Prevention Office, U.S. EPA, 401 M Street, SW, Washington, D.C. 20460 to obtain information or to offer suggestions on how the Agency might facilitate waste reduction efforts.

- Role of the TC Rule in the Pollution Prevention strategy:

  - By subjecting a larger number of toxic compounds to the RCRA regulations, it increases the costs to generators of managing solid wastes.

  - In effect, the TC forces waste managers to rethink their solid waste management practices due to the high cost of compliance with RCRA Subtitle C requirements.

  - The TC will alter the management of previously disposed wastes that might leach toxic contaminants by restricting their management in land-based units (surface impoundments, waste piles, lagoons, etc.).

1.7 CONCLUSIONS

- The TC Rule expands the definition of hazardous waste.
- The TC Rule adds 25 additional organic analytes.
- The TC Rule should encourage facilities to utilize less hazardous chemicals, to avoid the increased disposal costs associated with the TC Rule.

- By expanding the number of toxicity characteristic constituents from 14 to 39, EPA has expanded the definition of hazardous waste, and brought more wastes under the RCRA jurisdiction. This will keep additional wastes within the RCRA "cradle to grave" system and prevent them from harming human health or the environment.
- By adding organics to the EP Tox inorganics, the TC Rule addresses the potential harm these substances could cause in the environment.
- By increasing the costs of disposal for facilities which are now handling hazardous wastes which were formerly not regulated, facilities are encouraged to find substitutes or otherwise avoid chemicals which are hazardous.

Chapter 2

Chapter 2

APPLICATIONS OF THE TCLP METHOD

What is TCLP?

When is the Use of TCLP Applicable?

When is the Use of TCLP Inappropriate?

Sampling and Analysis Design

2.0 APPLICATIONS OF THE TCLP METHOD

Discussions in this chapter include:

- What is TCLP?
- When is the use of TCLP applicable?

2.1 What is TCLP?

- An analytical method to simulate leaching through a landfill. The leachate is analyzed for appropriate analytes.
- TCLP is comprised of four fundamental procedures:
  - sample preparation for leaching
  - sample leaching
  - preparation of leachate for analysis
  - leachate analysis
- The Toxicity Characteristic does *not* equal TCLP.

The toxicity characteristic leaching procedure is located in:

- Test Methods for Evaluating Solid Wastes, SW-846 Method 1311, July 1992.

Appendix I of this document contains the July 1992 version of Method 1311. When regulations specify the use of TCLP, approval to deviate from the method must be obtained from the State or EPA Region.

The Toxicity Characteristic (TC) is utilized to determine whether a solid waste is classified as a hazardous waste because it exhibits the characteristic of toxicity. The TC of a waste material is established by determining the levels of 8 metals and 31 organic chemicals in the TCLP extract of the waste. TC utilizes the TCLP method to generate leachate under controlled conditions. The regulatory levels of TC constituents in the TCLP leachate are listed in Table 2-1.

Table 2-1

TOXICITY CHARACTERISTIC CONSTITUENTS -- ALPHABETICAL

EPA HW[1] Number	Constituent	CTRL Basis	CTRLs (mg/l)	x DAF of 100 =	Regulatory Level (mg/l)
D004*	Arsenic	MCL	0.05		5.0
D005*	Barium	MCL	1.0		100.0
D018	Benzene	MCL	0.005		0.5
D006*	Cadmium	MCL	0.01		1.0
D019	Carbon tetrachloride	MCL	0.005		0.5
D020	Chlordane	RSD	0.0003		0.03
D021	Chlorobenzene	RfD	1.0		100.0
D022	Chloroform	RSD	0.06		6.0
D007*	Chromium	MCL	0.05		5.0
D026	Cresol	RfD	2.0		200.0[2]
D023	o-Cresol	RfD	2.0		200.0[2]
D024	m-Cresol	RfD	2.0		200.0[2]
D025	p-Cresol	RfD	2.0		200.0[2]
D016*	2,4-D	MCL	0.1		10.0
D027	1,4-Dichlorobenzene	MCL	0.075		7.5
D028	1,2-Dichloroethane	MCL	0.005		0.5
D029	1,1-Dichloroethylene	MCL	0.007		0.7
D030	2,4-Dinitrotoluene	RSD	0.0005		0.1[3]
D012*	Endrin	MCL	0.0002		0.02
D031	Heptachlor (and its epoxide)	RSD	0.00008		0.008
D032	Hexachlorobenzene	RSD	0.0002		0.1[3]
D033	Hexachloro-1,3-butadiene	RSD	0.005		0.5
D034	Hexachloroethane	RSD	0.03		3.0
D008*	Lead	MCL	0.05		5.0
D013*	Lindane	MCL	0.004		0.4
D009*	Mercury	MCL	0.002		0.2
D014*	Methoxychlor	MCL	0.1		10.0
D035	Methyl ethyl ketone	RfD	2.0		200.0
D036	Nitrobenzene	RfD	0.02		2.0
D037	Pentachlorophenol	RfD	1.0		100.0
D038	Pyridine	RfD	0.04		5.0[3]
D010*	Selenium	MCL	0.01		1.0
D011*	Silver	MCL	0.05		5.0
D039	Tetrachloroethylene	RSD	0.007		0.7
D015*	Toxaphene	MCL	0.005		0.5
D040	Trichloroethylene	MCL	0.005		0.5
D041	2,3,5-Trichlorophenol	RfD	4.0		400.0
D042	2,4,6-Trichlorophenol	RSD	0.02		2.0
D017*	2,4,5-TP (Silvex)	MCL	0.01		1.0
D043	Vinyl Chloride	MCL	0.002		0.2

* 14 original constituents based on drinking water standards.

1 Hazardous waste number

2 If o-, m-, and p-Cresol concentrations cannot be differentiated, the total Cresol concentration is used. Total Cresol regulatory level: 200 mg/l.

3 If quantitation limit is greater than the calculated regulatory level, quantitation limit becomes regulatory level.

2.2 When is the Use of TCLP Applicable?

The most common reasons for performing the TCLP are:

- Determining if an unknown waste is hazardous according to 40 CFR 261.24.
- Determining what type of disposal (hazardous waste or solid waste) is appropriate. Solid wastes *are not necessarily* Hazardous.
- Demonstrating the effectiveness of treatment processes to comply with Land Disposal Restrictions (LDR) or "Land Ban" requirements.
- Fulfilling shipping or transportation requirements.

Determining if a Waste is Hazardous

The toxicity characteristic regulations require generators to determine whether a solid waste is a regulated hazardous waste. Generators of potentially hazardous waste can determine if a waste is hazardous by one of the following methods:

- If a waste is excluded from regulation (40 CFR 261.4), no further determination is necessary.
- If the waste is listed per 40 CFR 261.30-261.35. Listings may be industry and process-specific (K-wastes) or may encompass all wastes from non-specific processes (F-wastes). Listings also include commercial chemical and off-specification products (P and U wastes).
- If a waste is not excluded or not listed as a hazardous waste, the generator must ascertain whether the waste exhibits any hazardous waste characteristic: toxicity, ignitability, corrosivity, or reactivity.
- A solid waste is classified as a hazardous waste because of characteristics, knowledge, or testing.
- If the waste is not listed, and there is not enough information to determine whether the Toxic Characteristic constituents are present above regulatory action levels, the TCLP test must be performed.
- The waste generator must certify in writing that the waste is not hazardous and must maintain records to demonstrate exclusion from RCRA requirements by knowledge or testing results.

Characterizing Waste for Disposal

- The RCRA regulations specify how listed and characteristic hazardous waste must be treated or disposed.

- Hazardous waste disposal facilities are permitted to accept specific categories of hazardous waste. Hazardous waste disposal facilities cannot accept hazardous waste without a manifest which lists the constituents or characteristics of the hazardous waste. TCLP waste characterization may be required by some disposal facilities.

- Disposal facilities may require initial waste testing. After analytical data are collected from a waste, process knowledge may be used instead of testing.

The Land Disposal Restrictions (LDR) regulate hazardous waste treatment and subsequent disposal. The following is a synopsis of the LDR regulations and their CFR citations:

- **40 CFR Part 268 Subpart A**

  The highlights are:

  - Definitions of "waste water" (40 CFR 268.2).

  - Material otherwise prohibited from land disposal may be treated in a surface impoundment if the residues from that treatment comply with applicable standards.

  - Petitions to allow land disposal of 40 CFR Part 268 Subpart C prohibited wastes must include comprehensive waste and simulation model sampling and analysis. This typically includes TCLP and other analysis.

  - Generators of restricted waste must either analyze their waste or its TCLP extract or use process knowledge to ascertain if the waste complies with 40 CFR Part 268 Subpart D treatment standards for land disposal. The generators must submit copies of the restricted waste's chemical analysis to the hazardous waste storage or disposal facility.

  - Restricted wastes are subject to the treatment standards of 40 CFR Part 268 Subpart D. The generator must notify the disposal company of the restriction and must supply test data if available.

- **40 CFR Part 268 Subpart B**

  This section outlines a timetable for waste disposal prohibitions and establishment of treatment standards.

- **40 CFR Part 268 Subpart C**

This section outlines waste disposal prohibitions. The following are examples of hazardous wastes that must meet LDR requirements:

- Solvent waste codes F001-F005, including waste from Comprehensive Environmental Response, Compensation and Liability Act of 1980 (CERCLA) actions.

- Dioxin containing waste codes F020-F023 and F026-F028, including CERCLA waste.

- California List wastes, including hazardous waste that contain 1,000 mg/L (liquid) or 1,000 mg/kg (non-liquid) of certain halogenated organic compounds, liquid hazardous wastes that contain $\geq$ 50 ppm PCBs, and liquid hazardous wastes that contain $\geq$ 134 mg/L nickel or $\geq$ 130 mg/L thallium.

- SW-846 Method 9095 must be used to determine if a waste is liquid.

- The initial generator of a California List hazardous waste must test the waste (not an extract), or use knowledge of the waste, to determine if the concentration levels in the waste meet the regulatory levels for California listed waste.

- Prohibitions for wastes with D, K, P, and U hazardous waste codes are also listed with effective dates of prohibition.

- **40 CFR Part 268 Subpart D**

This section outlines LDR treatment standards. 40 CFR Subpart D 268.41 lists treatment standards expressed as concentrations in the waste extract.

- A restricted waste may be land disposed only if the TCLP extract of a waste or waste treatment residue, does not exceed the values shown in Constituent Concentrations in Waste Extracts (CCWE) of 268.41 for any TC constituent. Some wastes require total constituent analysis instead of TCLP (see Appendix II).

- The regulation specifies waste/waste residue TCLP extract concentrations which may not be exceeded. The following Hazardous Waste Codes are exceptions: D004, D008 (lead), D031, K084, P010, P011, P012, P036, and U136 (all arsenic), K101 (o-nitroaniline, arsenic, cadmium, lead, and mercury), and K102 (o-nitrophenol, arsenic, cadmium, lead, and mercury).

- The arsenic and lead regulatory levels are based on the EP Tox Test, not TCLP. If waste/waste residue does not pass the TCLP test, the EP Tox Test may be used for these contaminants.

- LDR treatment standards are based entirely on technology-based standards expressed as Best Demonstrated Available Technology (BDAT). TC levels are based upon health-based allowable concentration levels and Dilution Attenuation Factors (DAFs). Therefore, the TC regulatory action levels are NOT the same as the LDR treatment standards in all cases. For many characteristic wastes, EPA has set the LDR treatment standards at the characteristic level. Note that EPA has not yet established LDR standards for D018 through D043 TC wastes.

LDR Records

- 40 CFR Part 268.7 requires hazardous waste generators and receivers to maintain the following records of process knowledge or data regarding:
  - A determination that a restricted waste does *not* meet treatment standards.
  - A determination that a restricted waste can be land disposed without further treatment.
  - A determination that waste is exempt under 40 CFR Part 268.5, 268.6 or a nationwide capacity variance under Subpart C of 40 CFR Part 268.
  - A determination to manage a prohibited waste in tanks or containers during waste treatment.
- The generator must certify that the waste is not hazardous either by knowledge of testing or by knowledge of process generation. While records are only required for hazardous waste, it is prudent to maintain records of non-hazardous waste classification.
- The generator must keep records on site for five years from the date that the waste was last sent to on-site or off-site treatment, storage, or disposal. The record retention time is extended if an enforcement action occurs within five years of waste generation.
- In general, the types of information required in all of the aforementioned records include:
  - Hazardous waste numbers
  - Manifest numbers
  - Waste analysis data if applicable
  - Treatment standards (including codes for required treatment technologies.)
  - Certification statements signed by the generator
  - Any waste analysis plans

- **LDR Variances: CAMUs are Designed to Reduce the Cost of On-Site Remediation**

  - Corrective action management unit (CAMU) regulations are enumerated in 40CFR260.10 and 40CFR270.2. CAMU is an area within a facility designated by the Regional Administrator (RA) for implementing CERCLA or RCRA corrective action requirements. A CAMU may only be used for the management of remediation wastes pursuant to corrective action requirements at a facility.

  - Placement of hazardous remediation waste into a CAMU will not automatically trigger LDRs. This variance from the LDRs can result in substantial cost reductions. CAMU boundaries are not confined to where contamination exists at the site; CAMU boundaries are based on where remediation waste will be managed.

- **Limitations and Conditions Applicable to CAMU Designations**

  - The CAMU shall facilitate the implementation of reliable, effective, protective, and cost-effective remedies;
  - Waste management activities associated with the CAMU shall not create unacceptable risks to humans or to the environment resulting from exposure to hazardous wastes or hazardous constituents;
  - The CAMU may only include uncontaminated areas of the facility if the incorporated area is more protective than management of such wastes at contaminated areas of the facility;
  - Areas within the CAMU, where wastes remain in place after closure of the CAMU, shall be managed and contained so as to minimize future releases to the extent practicable;
  - The CAMU shall expedite the timing of remedial activity implementation, when appropriate and practicable;
  - The CAMU shall enable the use, when appropriate, of treatment technologies (including innovative technologies) to enhance the long-term effectiveness of remedial actions by reducing the toxicity, mobility, or volume of wastes that will remain in place after closure of the CAMU; and
  - The CAMU shall, to the extent practicable, minimize the land area of the facility upon which wastes will remain in place after closure of the CAMU.

2.3 When is the Use of TCLP Inappropriate?

- **Risk Assessments**

The TCLP model assesses risk to ground water when potentially hazardous TC waste is co-disposed with garbage into sanitary landfills. The TCLP model does not assess risk when potentially TC waste is disposed in any other matrix. If a waste is hazardous because it exhibits the toxicity characteristic for mercury, the hazardous waste generator could attempt stabilization by adding cement and water to the waste, in a tank, and the resultant concrete may become non-hazardous. In this example, the concentration of mercury in the concrete's TCLP extract demonstrates that the waste is not a risk to the ground water beneath a sanitary landfill. Therefore, according to Federal regulations, this non-hazardous concrete can now be emplaced at the disposal site. The resultant concrete may only be emplaced on the disposal site if it does not exhibit the toxicity characteristic for mercury, unless a CAMU is obtained. The TCLP model discloses no information about potential risks to groundwater at the factory site where the mercury immobilized in the concrete is emplaced. EPA is designing site specific risk assessment models, but these will not be promulgated for several years.

When determining whether to use the TCLP for risk assessment, it is important to remember that TCLP simulates worst case management of hazardous waste in a landfill. Much caution must be used before TCLP data are used in risk assessment because the TCLP conditions rarely reflect actual site conditions. EPA's Science Advisory Board Report outlines many limitations of using TCLP for risk assessment at industrial sites. The Board recommends developing leach tests which are appropriate to site conditions. There are several excellent discussions on the inadequacies of TCLP organic analysis of solidified waste in "Stabilization and Solidification of Hazardous, Radioactive, and Mixed Wastes, ASTM, 1992, edited by T. Gilliam and C. Wiles (Appendix X of this document).

EPA's 1991 Science Advisory Board report on Leachability Phenomena (Appendix VII of this document) concluded that:

1. Many of the proposed uses of the EP and TCLP test have been inappropriate because the waste management scenarios of concern were not within the range of conditions used in the development of the tests themselves. In most cases of inappropriate use of the EP or TCLP tests, the justification given was that it was necessary to cite "standard" or "approved" methods. Even if it is acknowledged that the tests cannot be applied without significant change in test protocol itself, the need to use a previously "approved" test has been cited (page 3).

2. A variety of contaminant release tests and test conditions which incorporate adequate understanding of the important parameters that affect leaching should be developed and used to assess the potential release of contaminants from sources of concern. In scientific terms, no "universal" test procedure is likely to be developed that will always produce credible and relevant data for input to all decision making exercises (pages 7-8).

3. Leach test conditions appropriate to the situations being evaluated should be used for assessing long-term contaminant release potential. The best way to estimate the extent of contaminant release from a waste matrix of interest is to have a test that reflects realistic field conditions (page 13).

4. To facilitate the evaluation of risk implications of environmental releases, the EPA should coordinate the development of leach tests and the development of models in which the release terms are used (page 17).

The TCLP test cannot predict the potential for toxic chemicals to leach from oily waste, through soil, to contaminate ground water. This applies to both sanitary landfills and industrial sites. EPA and the American Society of Testing and Materials (ASTM) have formed a work group to develop a site-specific risk assessment model for oily waste. At a minimum, the model will incorporate physical and chemical characteristics of the oily waste and the soil. However, this model is not expected to be approved by EPA for several years. Until EPA approves this site-specific model for oily waste risk assessments, oily waste site assessments should be based on total constituent analysis, not TCLP extract analysis.

A percent reduction in organic contaminant concentrations between the original waste and a TCLP extract of stabilized/solidified waste yields very limited information. In fact, organic TCLP extract data from stabilized/solidified waste is not realistic because stabilization/solidification is not an appropriate treatment for organic wastes. Appendix X lists several papers that describe the problems with measuring organics in stabilized/solidified wastes. VOA data from stabilized/solidified waste is particularly ineffectual because concrete curing and concrete particle size reduction (hammering concrete into small pebbles) both produce heat, which evaporates volatiles from stabilized/solidified waste. Therefore, all or most of the volatiles will evaporate before analysis of the stabilized/solidified waste TCLP extract. This stabilization/solidification treatment will therefore appear to be very effective because of analyte evaporation. The semivolatile organic data in the TCLP extract of stabilized/solidified waste is of very limited value because the organic compounds are not very soluble in the TCLP extraction fluid. This usually results in comparable TCLP extract analyte concentrations between the original waste and the stabilized/solidified waste.

If waste is hazardous because it exhibits the toxicity characteristic for both benzene and mercury, adding cement and water will bind the mercury, and evaporate the benzene. At the present time, the LDR regulations do not specify how toxicity characteristic waste must be treated.

Appendix XV discusses risk assessment for disposal of solidified/stabilized waste and contaminated soils.

- **Unnecessary Hazardous Waste Determinations:**
  - Generator's knowledge of waste (e.g. chocolate ice cream).
  - Exempt waste (e.g. household garbage).
  - Material is not a solid waste (e.g. clean sand, laundry detergent).
  - Generator's testing of waste (total constituent analysis).
  - The solid waste is a listed hazardous waste.

- **Unnecessary Land Ban Determinations:**
  - Some LDRs are for total constituents, not TCLP extract concentrations.
  - Generator's testing of waste (total constituent analysis).
  - Pure liquid waste samples (waste is TCLP extract; waste would fail paint filter test).

- **Determination of Corrective Action Clean-up Levels and Clean Closures**

  TC levels are not used to set clean up levels for corrective actions or clean closures. Clean up levels are developed from site-specific information.

2.4 Sampling and Analysis Design

The following issues discussed in this section are critical in sampling and analysis design:

- Total constituents versus TCLP
- Specifying sample collection procedures

Total constituents versus TCLP - EPA Memorandum

EPA has several memoranda which address issues such as Characterizing Heterogeneous Material and Total Analysis Versus TCLP. These are presented in Appendix VI. The Office of Solid Waste - Methods Section, Notes on RCRA Methods and QA Activities, Memorandum #36, January 12, 1993, is helpful in delineating sampling design related to the analysis of samples for total constituents versus leachable constituents. The consequential portions of Memorandum #36 and a discussion of their significance follows.

Office of Solid Waste Memorandum - Methods Section #36, January 12, 1993

Page 10 Characterizing Heterogeneous Materials

> *Characterization of a solid waste is essential for determining whether a waste is hazardous or for developing management and treatment standards for hazardous materials. Current EPA regulations for characterizing waste includes determining the average property of the "universe or whole." This task is difficult when applied to heterogeneous wastes because conventional sampling and compositing techniques are often inadequate in providing a "representative sample" of the waste. As a result, analytical results are often biased and imprecise, making compliance decisions difficult.*

The above paragraph means that all of the samples collected from a heterogeneous waste do not have to be below the regulatory action level for the waste to be considered non-hazardous. What percent of the samples are allowed to be above regulatory action levels without classifying the waste as hazardous? Chapter 9 of SW-846 recommends utilizing the student "t" test to determine an appropriate percentage of samples that may be above regulatory action levels without classifying a solid waste as "hazardous."

Please be aware that the above discussion is only for heterogenous wastes with no obvious "hot spots" of highly concentrated hazardous wastes. Whenever regulatory agencies collect samples to determine compliance with the TC regulations, only the most contaminated media will be sampled. For example, if there is a one-acre mercury waste pile surrounded by ninety-nine acres of clean sand, TC inspectors will not collect samples of the clean sand.

The RCRA definition of "average property" is very different than the definition of "average" which was taught in elementary school. For example, if the TCLP extract regulatory action level is 50 mg/L, and our sample results are 80 mg/L and 0 mg/L, we can not compute the numerical average of analytical results as 40 mg/L, and affirm that the waste is not hazardous. To make proper characterization of the above waste, we would declare the waste hazardous because 50% of the samples were above regulatory action levels. Appendix XI discusses characterization of heterogeneous waste.

Pages 19-21 Totals Analysis Versus TCLP

Over the past year, the Agency has received a number of questions concerning the issue of total constituent analysis with respect to the TCLP. Section 1.2 of the TCLP allows for a compositional (total) analysis in lieu of the TCLP when the constituent of concern is absent from the waste, or if present, is at such a low concentration that the appropriate regulatory level could not be exceeded. A number of persons have contacted the MICE Service and have requested clarification on this issue with respect to a number of waste testing scenarios.

Wastes that contain less than 0.5% dry solids do not require extraction. The waste, after filtration, is defined as the TCLP extract. The filtered extract is then analyzed and the resulting concentrations are compared directly to the appropriate regulatory concentration.

For wastes that are 100% solid as defined by the TCLP, the maximum theoretical leachate concentration can be calculated by dividing the total concentration of the constituent by 20. The dilution factor of 20 reflects the liquid to solid ratio employed in the extraction procedure. This value then can be compared to the appropriate regulatory concentration. If this value is below the regulatory concentration, the TCLP need not be performed. If the value is above the regulatory concentration, the waste may then be subjected to the TCLP to determine its regulatory status.

The same principal applies to wastes that are less than 100% solid (i.e., wastes that have filterable liquid). In this case however, both the liquid and solid portion of the waste are analyzed for total constituency and the results are combined to determine the maximum leachable concentration of the waste. The following equation may be used to calculate this value:

$$\frac{[A \times B + [C \times D]]}{B + [20 L/kg \times D]} = E$$

where: *A = concentration of the analyte in liquid portion of the sample (mg/L)*

B = Volume of the liquid portion of the sample (L)

C = Concentration of analyte in the solid portion of the sample (mg/kg)

D = Weight of the solid portion of the sample (kg)

E = Maximum theoretical concentration in leachate (mg/L)

To illustrate this point, the following example is provided:

An analyst wishes to determine if a lead processing sludge could fail the TC for lead. The sludge is reported to have a low concentration of lead, and the analyst decides to perform a compositional analysis of the waste instead of the TCLP. A preliminary percent solids determination as described in the TCLP is performed. The percent solids is found to be 75%. Thus, for each 100 grams of this waste filtered, 25 grams of liquid and 75 grams of solid are obtained. It is assumed for the purposes of this calculation that the density of the filterable liquid is equal to one. The liquid and solid portion of the sample are then analyzed for total lead. The following data are generated:

Percent solids = 75%
Concentration of lead in the liquid phase = 0.023 mg/L
Volume of filtered liquid = 0.025 L
Concentration of lead in the solid phase = 85 mg/kg (wet weight)
Weight of the solid phase = 0.075 kg.

The calculated concentration is as follows:

$$\frac{[0.023\ mg/L \times .025L] + [85\ mg/kg \times .075kg]}{.025\ L + [20\ L/kg \times .075kg]} = 4.18\ mg/L$$

In this case, the maximum leachable concentration is below the 5 mg/L regulatory concentration for lead, and the TCLP need not be performed.

Non-aqueous based wastes (i.e., oily waste) may be calculated in the same manner as described above, except the concentration of constituents from the liquid portion of the waste (A in the above formula) are expressed in mg/kg units. Volumes also would be converted to weight units (kg). The final leachate concentration is expressed in mg/kg unit.

This memorandum should significantly reduce the number of TCLP samples analyzed to demonstrate compliance with the TC regulations. The profound regulatory impact of Notes on RCRA Methods and QA Activities Memorandum #36, pages 19-21, are most easily comprehended with the following uncomplicated monophasic examples. These examples explain how the calculations are made and evaluated to determine whether to analyze the total constituents or perform the TCLP.

Example 1. The TCLP Extract Regulatory Action Level for cadmium is 1 mg/L. A soil (with no liquid phase) contains 10 mg/kg of cadmium. Is the TCLP required?

The TCLP test for a solid matrix leaches one part of waste with twenty parts of an acetic acid buffer. Therefore, even if all of the cadmium was leached from the soil to the solvent, the maximum concentration in the TCLP extract would be 0.5 mg/L. Consequently, TCLP is not required.

Example 2. The TCLP Extract Regulatory Action Level for cadmium is 1 mg/kg. A soil (with no liquid phase) contains 100 mg/kg of cadmium. Is the TCLP required?

The TCLP test for a solid matrix leaches one part of waste with twenty parts of acetic acid buffer. Therefore, if all of the cadmium was leached from the soil to the solvent, the maximum concentration in the TCLP extract would be 5 mg/L. Consequently, TCLP is required.

Example 3. The TCLP Extract Regulatory Action Level for cadmium is 1 mg/L. A liquid with no solid phase contains 0.5 mg/L cadmium. Is the TCLP required?

The TCLP test for a liquid with no solid phase consists of filtration. The filtrate is the TCLP extract. Therefore, even if all of the cadmium passed through the filter, the maximum concentration of cadmium in the TCLP extract would 0.5 mg/L. Consequently, TCLP extraction is not required.

Example 4. The TCLP Extract Regulatory Action Level for cadmium is 1 mg/L. A liquid (with no solid phase) contains 5 mg/L cadmium. Is the TCLP extraction required?

The TCLP test for a liquid with no solid phase consists of filtration. The filtrate is the TCLP extract. Therefore, if all of the cadmium passed through the filter, the maximum concentration of cadmium in the TCLP extract would be 5 mg/L. Since the filtrate equals the TCLP extract, the waste exceeds the TC level for cadmium and is a hazardous waste. Therefore, the TCLP extraction is not required.

The following abstract and introduction of a research paper on the relationship between total mercury and TCLP mercury at Oak Ridge National Lab illustrates how difficult it is to estimate the mercury concentration in the TCLP extract from the mercury concentration in the soil.

HAZARDOUS WASTE & HAZARDOUS MATERIALS
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Effect of Chemical Form of Mercury on the Performance of Dosed Soils in Standard Leaching Protocols: EP and TCLP

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ABSTRACT

Application of the EP (Extraction Procedure) and TCLP (Toxicity Characteristic Leaching Procedure) to a variety of mercury-contaminated soils and solid wastes from U.S. Department of Energy facilities in Oak Ridge, Tennessee, has not shown any consistent relationship between test results and total mercury concentrations. To determine the effects of the chemical form of mercury on leaching results, samples of uncontaminated soil were dosed with four different forms of mercury (Hg^0, HgS, HgO, Hg_2O) at three concentrations (100, 1000, 10000 μg/g) and then subjected to headspace mercury vapor analysis and application of the two leaching protocols. None of the leaching protocol results for soil dosed with Hg^0 or HgS exceeded the Resource Conservation and Recovery Act (RCRA) limit (200 μg/L) even at the highest dosing level (10000 μg/g). For both mercury oxide forms only the higher soil concentrations (1000 and 10000 μg/g) yielded leachate concentrations exceeding the RCRA limit. In general, the TCLP yielded higher leachate concentrations of mercury than the EP. This study verified that, where the chemical form of mercury is unknown, total mercury concentrations in the soil or waste provide no clue as to the performance of a soil in the leaching protocols. Results of sample headspace analysis, in addition to TCLP results and analysis for total mercury, are recommended to fully evaluate the hazards of mercury-contaminated waste and soil.

INTRODUCTION

There are few regulatory and/or advisory criteria for mercury in contaminated soils, sediment, and waste. Typically, action or clean-up guidelines are developed on a case-by-case basis. The most widely applied criterion for mercury in solid material is the performance of solid samples analyzed with the U. S. Environmental Protection Agency Extraction Procedure (commonly referred to as the EP, reference 1), or (since September 25 1990, reference 2) the Toxicity Characteristic Leaching Procedure (TCLP). These protocols have been used to classify wastes under the Resource Conservation and Recovery Act (RCRA). EP or TCLP results, expressed as the concentration of mercury in the laboratory-generated leachate, are compared to 200 μg/L. Results above this criterion are classified as having failed the "toxicity characteristic." The 200 μg/L limit represents 100 times the Drinking Water Standard and requires the sample to contain at least 4 μg/g. The latter value is derived by multiplying the established limit, 200 μg/L, by the solution-to-solid ratio in the test, 0.02 L/g. Theoretically, any sample containing total mercury less than 4 μg/g could not fail the EP or TCLP protocols and be accordingly classified as hazardous under RCRA. While there are some minor differences between the TCLP and the EP protocols, most notably the composition of the extraction fluid, the established limit is 200 μg/L for both protocols. Rarely, if ever, will all the mercury in a soil or waste be completely leachable in a protocol. Therefore, soil containing considerably higher concentrations of mercury than 4 μg/g could theoretically pass the EP or TCLP protocols.

Application of the EP to a variety of mercury-contaminated soils and solid wastes from U.S. Department of Energy (DOE) facilities in Oak Ridge, Tennessee, has not shown any consistent relationship between test results and total mercury concentrations (Figure 1). Low yields of leachable mercury (less than the RCRA Limit of 200 μg/L) have not been uncommon for samples containing several thousand parts per million of total mercury and even for samples exhibiting visible beads of mercury. Conversely, some soils with relatively low concentrations of total mercury have exhibited high leachability of mercury (>200 μg/L) and been classified as hazardous under RCRA.

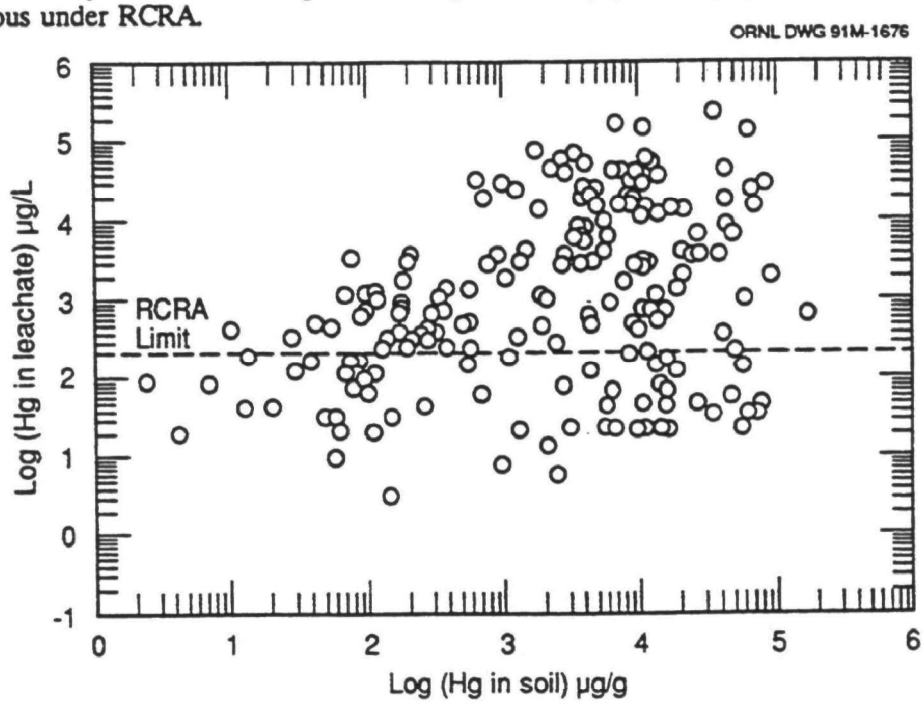


Figure 1 Relationship Between Total Mercury Concentration (μg/g) in Mercury-Contaminated Soils and Wastes and Mercury Concentration (μg/L) in Leachates Generated Using the EP Protocol.

Chapter 3

Chapter 3

TC AND TCLP PROJECT PLANNING

Data Quality Objectives

DQO Case Study:
Cadmium Contaminated Fly Ash Waste

Sampling and Analysis Design

Analytical Method Selection

3.0 TC AND TCLP PROJECT PLANNING

Planning a project prior to sampling and analysis promotes successful implementation. The conclusion of the planning process should result in an efficient sampling and analysis design which allows the collection of appropriate data. The data should promote making a correct decision on the storage, treatment or disposal of the waste. This chapter provides information in the following areas which are critical to project planning.

- Data Quality Objectives (DQOs)
- Sampling and analysis design
  - Sample containers, preservation, and storage
  - Sample volumes
  - Sample decontamination
  - Holding times
  - Field QC
  - Documentation

Barbara Metzger's 1992 speech on Environmental Data Use: "Meeting the Customer's Need", (Appendix XVI) - illustrates how site-specific Data Quality Objectives (DQOs) are utilized in environmental projects.

3.1 Data Quality Objectives

Environmental samples are often collected and analyzed without proper planning. The data collected may not allow a correct decision to be made.
In response to this problem, EPA has developed a planning process to facilitate clear definition of the decision to be made and the data required to make these decisions. This process is the Data Quality Objective (DQO) process. Prior to initiating sampling, the questions to be answered should be listed, prioritized and one primary question identified. These should be agreed upon by all parties, including the regulatory agencies and TSDFs, the site owners and operators/generators, and the technical staff.

The DQO Process should identify:

- What question will the data resolve?
- Why is a specific type, quantity and quality of data needed?
- How will the customer use the data to make a defensible decision?
- How much data are required?
- What resources are needed?

The information included in the DQO section describes the following information:

- DQO definition
- DQO Planning Process (DQO-PP) description
- Value of DQO-PP
- Example of DQO-PP implementation

The American Society of Testing and Materials (ASTM) Committee D34.02.10 is working with Quality Assurance Management Staff (QAMS) and the Office of Solid Waste to produce an ASTM Standard Practice which describes the DQO Planning Process (DQO-PP). Additional guidance is available from the Office of Emergency and Remedial Response, US EPA, Washington, D.C. 20460 in a document titled: "Data Quality Objectives Process for Superfund, Interim Final Guidance, EPA/540/G-93/071, Publication 9355.9-01, September 1993.

Many of the following flow charts, definitions and information are derived from these documents and from the draft ASTM document.

DQOs are defined by the ASTM document as:

Qualitative and quantitative statements derived from the DQO-PP describing the problems, decision rules, and the uncertainty of the decisions stated within the context of the problem.

The DQO Planning Process is:

> *A Total Quality Management tool developed by the US EPA to facilitate the planning of environmental data collection activities. The DQO Planning Process asks planners to focus their efforts by specifying the use of the data (the decision), the decision criteria, and their tolerance to accept an incorrect decision based on the data.*

Incorrect decisions can result from many causes. One cause of making incorrect decisions is insufficient or inadequate data to address the problem. The DQO-PP provides a method to allow the decision makers and technical specialists to assure that sufficient data of appropriate quality is collected at the proper time. In order to make the best decisions, the chance of making an incorrect decision must be understood. In order to examine the probability of making an incorrect decision, it must be understood that all measurements have error. Measurement error results from heterogeneity of the waste, errors in sampling methods and laboratory error. Measurement error is cumulative. The laboratory error is a small component in the overall measurement error. Decision makers must understand that there is a balance between resources and decision error. Decision makers must make informed decisions as to the cost versus the decision error and ultimately the measurement error.

The goal of this document is to assist the regulated community comply with the TC and TCLP Rules in a cost effective manner. Therefore, it is essential for the regulated community to understand the importance of obtaining the most cost effective sampling and analysis design which provides the appropriate decision error. If an appropriate sampling and analysis design is not utilized, the data may not allow a waste generator to accurately determine if a solid waste is a hazardous waste. The DQO-PP is essential for preparing a sampling and analysis design which balances the resources with the chance of making an incorrect decision.

What is the value of using the DQO-PP?

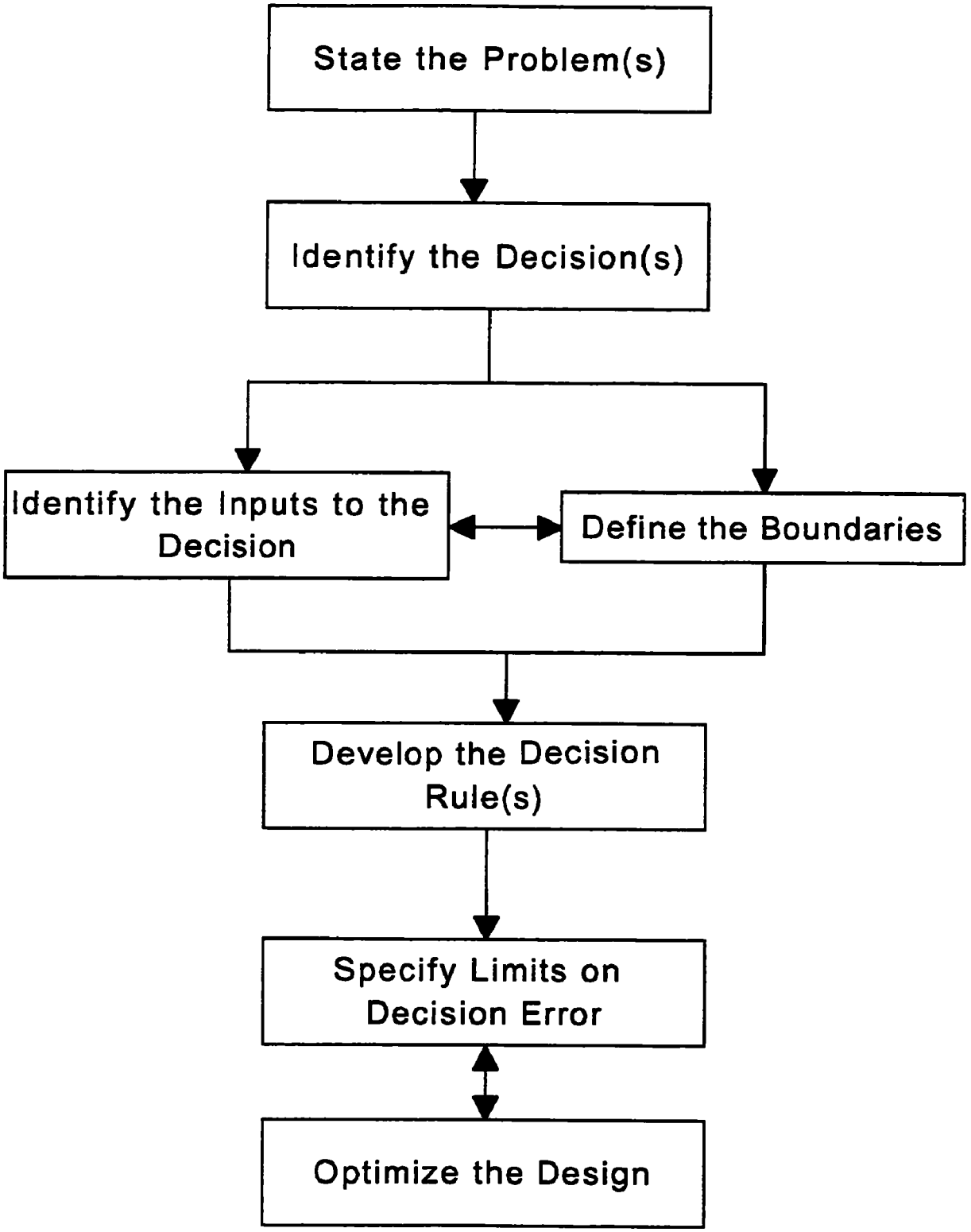
The DQO-PP:

- Helps users determine the amount, frequency, and quality of data needed.
- Saves resources by making data collection operations more efficient.
- Encourages communication between the data users, technical experts, and decision makers.
- Helps focus studies by clarifying vague objectives and narrowing the questions to the essential issues.
- Helps provide a logical process which facilitates documentation.

Additional critical information about the DQO-PP

- A statistical design, which may result from the DQO-PP, allows the uncertainty in the data and ultimately the decision uncertainty to be quantified. Chapter 9 of SW-846 outlines strategies for statistical design. The statistical design must be carefully applied to assure that the correct assumptions are made and that the assumptions address the question(s) related to the objectives.
- The DQO-PP is iterative. Projects should focus on essential questions and take a phased approach to answering these questions. This allows reevaluation of the DQOs as the data collection is completed. This iteration allows the resources to be efficiently used.
- The term "decision maker" used in this document may include owners and managers of facilities and regulators. Prior to undertaking large projects, the owners and managers may choose to involve the regulators to assure consensus is reached in the planning phase.

The following discussion presents summary information about each of the seven steps within the DQO Planning Process shown in Figure 3-1.



The DQO process is iterative. Changes in assumptions may be made as new information is obtained.

Figure 3-1 - The DQO Process

The DQO Planning Process has a logical problem solving structure which includes the following seven steps:

1. State the problem
2. Identify the decision(s)
3. Identify inputs to the decision(s)
4. Define the study boundaries
5. Develop decision rule(s)
6. Specify acceptable limits on decision error
7. Optimize the design

1. State the problem

The essential goal of this step is to focus the decision makers and the technical team on one or more problems. These problems should be as narrowly stated as possible. For waste generators, these problems may focus on whether a particular waste is hazardous.

2. Identify the decision

Clear and concise potential decision(s) should be agreed upon by the decision makers and the technical team. One or more decisions should be presented for each problem.

3. Identify the inputs to the decision

The goal is to list the data which are needed to make the decision. Questions such as whether metals or organic data must be generated should be addressed. Production or process data may be required if the task is related to a waste generator. Additionally, knowledge about the homogeneity of the material to be sampled may need to be determined. This data should also include any time factors, physical limitations, process data, and resources which may effect the sampling and analysis. The environmental characteristics such as analytes, method, and process knowledge required should also be listed.

4. Define the study boundaries

The boundaries are the limitations on the study. Examples include time and budget constraints, permit requirements, disposal requirements, and exposure levels. Any physical boundaries such as drum or container size is considered a spatial boundary. Sampling depth may be considered a boundary. Any changes in the waste concentration over time must be considered as a temporal boundary. This step is often performed simultaneously with the previous step.

5. Develop decision rule(s)

This is a statement which describes how the data will be summarized, collected, and compared to the decision. The statement should include actions which will be based on criteria and conclusions from the sampling and analysis. The statement should be an "if...then..." statement that incorporates the action limits. The statement should include the parameter to be measured, the mode of comparison (greater than, less than, average, etc.), action levels and actions to be taken. An example statement: If the average concentration in drum is greater than 1 mg/L of cadmium, the material will be disposed of as a hazardous waste.

Instead of statements, a decision logic diagram may be used to present the decisions and actions, and criteria. When complex decisions are required or when multiple criteria must be considered, the decision logic diagram is often more easily followed and understood.

6. Specify acceptable limits on decision error

The decision maker should understand that results of all studies have uncertainty and error. The goal is to quantitate the amount of uncertainty that the decision maker is willing to accept in making the decision. The key step is to move from a qualitative "feeling" of uncertainty to a quantitative level of uncertainty. The process for establishing this, in the case of a hazardous waste determination, includes the following:

i. Identify the consequences of incorrectly deciding the waste is not hazardous.

ii. Identify the consequences of incorrectly deciding the waste is hazardous.

iii. Rank these consequences by severity.

iv. Estimate the health risk and financial risk associated with an incorrect result.

v. Estimate how far below the regulatory limit one wants to be in order to decrease the consequence of an incorrect decision. Some statistical experience in assessing decision error and in assessing sampling and analysis design error is needed to make this assessment.

This information is incorporated into the decision rule. An example decision rule which incorporates the decision error is:

> Three samples per drum are collected and the concentrations from each drum are averaged. If the within drum average concentration of cadmium is greater than 0.7 mg/L of cadmium, then the material will be disposed of as a hazardous waste.

7. Optimize the design

In order to characterize a site or waste successfully, a sampling and analysis design must be established. The sampling and analysis design includes development of statistical and observational design alternatives, and specifies sampling, handling, and analysis methods. The design indicates the number and locations of samples based on the acceptable decision error which was agreed on during the DQO development. The most important input from the

DQO process is the degree of decision error which the decision maker will accept. When preparing a sampling and analysis design, the time and budgetary constraints should be evaluated to balance their importance versus the decision error.

The preliminary design may contain the following information:

- Spatial areas of interest
- Hot spots vérsus average values of contamination
- Particular contaminants of concern
- Desired levels of detection
- Which matrices will be investigated
- Patterns of contamination
- Stratification of the contaminants
- Contaminant degradation
- Temporal considerations (changes of concentration over time)
- Quality control samples designed to allow estimation of precision and accuracy, and background contamination
- Health and safety issues

Designs must be practical and achievable. There is no one correct design but rather an optimum design which balances resources with the data required to make a decision. Technical staff must work carefully to present several designs to decision makers along with the probability of decision error, resources, and benefits of each design.

Other factors used to select the appropriate measurement methods include:

- DQOs,
- required regulatory or risk assessment levels,
- method precision and accuracy, and
- contaminants of interest.

Improved accuracy, precision, and lower detection limits usually result in higher sampling and analytical costs because larger numbers of samples, improved instrumentation, and more field/analytical expertise may be required. The improved accuracy may result in decreased decision error. If matrix specific accuracy and precision data are not available, preliminary precision and accuracy studies must be performed. These studies should measure total error from sample heterogeneity, sampling, and analytical methods. Regulatory agencies may review these studies prior to method approval.

Prior to sampling design implementation, the decision makers must approve the design. Several designs may be presented to the regulators and decision makers. The level of uncertainty, advantages and disadvantages, budgetary and time constraints, and other relevant factors must be presented for each design. This approach allows the decision makers to properly assess options and to agree upon the best sampling design. Sampling designs are implemented after approval by the decision makers.

The technical team must continually evaluate the proposed designs with respect to the DQOs, health and safety criteria, budget and time constraints. If the proposed design does not meet the criteria, it may be altered. In extreme cases, the DQOs may be unattainable and must be altered. The DQOs should only be changed after consultation with the decision

makers and technical team members. The DQOs may need to be reevaluated if a decision cannot be made.

References for sampling design strategies are:

- U.S. EPA, Test Methods for Evaluating Solid Wastes, SW-846 Third Edition-Chapter 9. August 1993.

- Characterizing Heterogeneous Materials, July, 1992 (Appendix XII) .

Chapter 9 of SW 846 outlines several statistical design approaches. Many other design strategies exist and may be better suited for the situation at hand. It is wise to examine many statistical options and methods of evaluating the data during the planning phase.

An example of using the DQO-PP is presented in the following pages. This example demonstrates how to design a sampling program to determine whether fly ash from a municipal incinerator is a RCRA hazardous waste. This example is from a draft ASTM standard practice on the use of DQO-PP in waste management activities. The ASTM document was the product of a cooperative agreement between ASTM, US EPA Quality Assurance Management Staff and the Office of Solid Waste. The example was written by the ASTM D.34.02.10 committee.

3.2 DQO Case Study: Cadmium Contaminated Fly Ash Waste

Background

A municipal waste incineration facility located in the Midwest routinely removes "fly ash" from its flue gas scrubber system and disposes of it in a sanitary landfill. Previously, it was determined that the ash was "non-hazardous" under RCRA regulations. However, the incinerator has recently begun treating a new waste stream. As a result, a local environmental public interest group has asked that the ash be retested against RCRA standards before it is dumped. The group is primarily concerned that the ash could contain hazardous levels of cadmium from the new waste sources. The facility manager has agreed to test the ash and decides to employ the Data Quality Objectives process to help guide decision-making throughout the project.

The Code of Federal Regulations (CFR) Part 261 RCRA toxicity characteristic criteria for determining if a solid waste is hazardous requires collection of a "representative portion" of the waste and performance of Toxicity Characteristic Leaching Procedure (TCLP). During this process, the solid fly ash will be "extracted" or mixed in an acid solution for 18 hours. The extraction liquid will then be subjected to tests for specific metals.

Since the impact of the new waste stream is not known, a preliminary study was conducted to determine the variability of the concentration of the contaminants. Random samples were collected from the first 20 truck loads. Since process knowledge of the waste stream indicated that cadmium was the only toxicity characteristic (TC) constituent in the waste, these samples were analyzed individually for cadmium using the TCLP. The results were expressed as the average concentration along with the standard deviation.

DQO Development

The following is an example of the output from each step in the DQO process.

Assemble the Team -- The Plant Manager assembled a skeletal team consisting of himself and a representative of the current disposal facility staff. The two of them assembled the team with the responsibility to deal with this problem.

The decision makers evaluation team will include the incineration plant manager, a representative of the environmental public interest group, a representative of the community where the ash is currently being disposed of. The technical staff include a statistician, and a chemist with sampling experience.

1. State the Problem

The problem is to determine if any loads of fly ash are hazardous for cadmium under the RCRA TCLP. If so, those loads must be disposed of in a RCRA landfill.

2. Identify the Decision(s)

i. *Decision* -- Determine whether the concentration of cadmium in the waste fly ash exceeds the regulatory RCRA standards.

ii. *State the Actions That Could Result From the Decision* --

a) If the average concentration of cadmium is greater than the action level, then dispose of the waste fly ash in a RCRA landfill.

b) If the average concentration of cadmium is less than the action level, then dispose of the waste fly ash in a sanitary landfill.

3. Identify the Inputs Needed for the Decision

List the environmental variables or characteristics which are known from historical and regulatory information and information which must be obtained in order to make the decision.

Available Inputs

Preliminary Study Information -- Since the concern is with a new waste stream, the team ordered a pilot study of the fly ash to determine the variability in the concentration of cadmium between loads of fly-ash leaving the facility. They have determined that each load is fairly homogeneous. However, there is a high variability between loads due to the nature of the waste-stream. Most of the fly ash produced is not a RCRA hazardous waste and may be disposed of in a sanitary landfill. Because of this, the company has decided that testing each individual waste load before it leaves the facility would be the most economical. In that way, they could send loads of ash that exceeded the regulated cadmium concentrations to the higher cost RCRA landfills and continue to send the others to the sanitary landfill.

The study showed that the standard deviation of the cadmium concentration within a load was S_w = 0.4 mg/L and the standard deviation of the cadmium concentration between loads was S_b = 1.4 mg/L. Sample and quality control data indicates that a normal distribution can be assumed.

Identify Contaminants of Concern, Matrix, and Regulatory Limits -- The team identified the following factors critical to the problem:

- Contaminants of concern: cadmium soluble in the Toxic Characteristic Leaching Procedure (TCLP) extract.
- Sample Matrix: fly ash.
- Regulatory Threshold: 1 mg/L.

Specific Project Budget and Time Constraints -- The incinerator plant manager has requested that all stages of the operation be performed in a manner that minimizes the cost of sampling, chemical analysis and waste disposal. However, no formal cost constraints have been implemented.

The environmental public interest group has threatened to file a law suit for violation of environmental regulations if testing does not proceed within a "reasonable time-frame."

Contained in the trucks, the waste does not pose a threat to humans or the environment. Additionally, since the fly ash is not subject to change, disintegration or alteration, the chemical properties of the waste do not warrant any temporal constraints. However, in order to expedite decision making, the evaluation team has placed deadlines on sampling and reporting. The fly ash waste will be tested within 48 hours of being loaded on to waste hauling trailers. The analytical results from each sampling round should be completed and reported within 5 working days of sampling.

Identification of the testing methods -- In this case, 40 CFR Part 261, Appendix II specified the TCLP method SW 846 Method 1311. The leachate must be analyzed by an appropriate method. Potential methods of characterizing the leachate for cadmium include, but are not limited to, SW 846 methods 6010, 6020, 7130, or 7131.

Inputs To Be Determined

Method validation and QC -- The analytical method accuracy and precision and method detection limits in the fly ash matrix must be determined. The QC samples must be specified.

Identification of sampling procedure or devices -- The following must determined:

- Number of samples
- Sampling methods for composite or grab samples of ash
- QC requirements for sampling

4. Define the Boundaries of the Study

Define a detailed description of the spatial and temporal boundaries of the decision; characteristics that define the environmental media, objects or people of interest; and any practical considerations for the study.

i. *Specify the Characteristics that Define the Sample Matrix* -- The fly ash should not be mixed with any other constituents except the water used for dust control.

ii. *Identify Spatial Boundaries* -- The variability between loads was greater than within a load therefore, decisions will be made on each load. The waste fly ash will be tested after it has been deposited in the trailer used by the waste hauler. Separate decisions about the toxicity of the fly ash will be made for each load of ash leaving the incinerator facility. Each load of ash should fill the waste trailer at least 70%. In cases where the trailer is filled less than 70%, the trailer must wait on-site until more ash is produced and can fill the trailer to the appropriate capacity.

iii. *Identify Temporal Boundaries (The temporal boundaries of the study include the time frame over which the study should be conducted).* -- The study will be conducted until at least 30 data points are collected and the action limits, number and frequency of samples will be reevaluated after that time.

5. Develop a Decision Rule

The arithmetic mean of sample results will be compared to the action level.

Decision Rule:

a) If the average concentration of cadmium in a truck load is greater than the action level, then dispose of the waste fly ash in a RCRA landfill.

b) If the average concentration of cadmium in a truck load is less than the action level, then dispose of the waste fly ash in a sanitary landfill.

Note that the team has decided that the action level will be less than the regulatory level in order to decrease decision error at the regulatory level of 1 mg/L.

Develop Decision Error Constraints

The decision makers specify acceptable decision errors based on the consequences of making an incorrect decision. Both error rates have negative consequences.

The team must make a baseline assumption. Based on the initial pilot study each load will be assumed to be less than the regulatory level and the burden of proof will be to prove that the load is above the action level. In this example, there are two types of error that the evaluation team could make:

i. *false positive error* (declaring the load hazardous when it is not) -- If the true cadmium concentration is below 1 mg/L, but the average measured cadmium concentration is above the action level, the non-hazardous fly-ash waste will be sent to a RCRA landfill. The consequence of a false positive error is that the company will have to pay additional cost to dispose of the waste with a concentration between the action level and regulatory threshold at a RCRA facility as opposed to a less expensive method of disposal in a sanitary landfill.

ii. *false negative error* (declaring the load non-hazardous when it is hazardous) -- If the true cadmium concentration is equal to or greater than 1 mg/L, but the average measured cadmium concentration is below the action level, the hazardous fly-ash waste will be sent to a sanitary landfill. The consequence of a false negative error is that the fly-ash waste may be disposed in a manner that will be harmful to human health or the environment. Legal consequences and subsequent remedial costs are also possible consequences.

iii. *number of samples* -- The number of samples will depend on the uncertainty of estimating the true cadmium concentration for each load and the resources available to sample and to chemically analyze the samples.

The purpose of this stage of the process is to specify the probabilities of making an incorrect decision on either side of the "action level" that are acceptable to decision makers. The team must agree on which type of decision error is of greater concern, false positives or negatives and must target the level of false positives and negatives.

For this example, the project team is more concerned about false negatives because of the increased liability due to sending potentially hazardous waste to a sanitary landfill. The team set a target level of false negatives of 10% when the true concentration is 1 mg/L.

Decision Performance Curve

The Decision Performance Curve will be calculated to determine the action level and review the performance of the decision rule. To calculate the Decision Performance Curve, decision makers use the following steps:

Step 1: Number of samples are calculated with L = 0.2 mg/L, $\sigma = S_W = 0.4$ mg/L, and $\alpha = 0.05$ (or $Z_{\alpha/2} = 1.960$ for a 95% confidence level).

$$n = \left(\frac{1.960 \times 0.4}{0.2} \right)^2 \approx 16 .$$

where:

L = the limit of error on the average of 0.2 mg/L

S_W or standard deviation = 0.4 mg/L within a load

$Z_{\alpha/2} = 1.960$ for a 95% confidence level, the $\alpha/2$ percentile point of normal probability distribution e.g. $Z_{\alpha/2} = Z_{.0005}$. These are tabled values from a standard normal distribution at $Z_{.0005}$.

n = number of samples

Step 2: Calculate the action level (AL) from the specified false negative error of 10%. The probability calculations are based on an approximating normal probability distribution for the cadmium concentration measurements. This approximating normal probability has a mean = RT = 1.0 mg/L and a standard deviation = S_W = 0.4 mg/L. The 10% percentile point for the standardized normal probability distribution is $Z_{0.10} = 1.282$.

False Negative Error = Pr(Average < AL when the true concentration = RT) = 0.10.

or

$$\frac{AL - RT}{S_W / \sqrt{n}} = -Z_{0.10} ,$$

AL = Action Level
RT = Regulatory Threshold
$Z_{0.10}$ = Tabled Z-value from standard normal distribution at 0.10

$$AL = 1.0 mg/L - (1.282)(0.4 mg/L) / 4 = 1.0 mg/L - 0.13 mg/L ,$$

or

$$AL = 0.87\,mg/L\ .$$

Therefore, the decision rule is:

a) If (average concentration of cadmium) ≥ 0.87 mg/L, then dispose of the waste fly ash in a RCRA landfill.

b) If (average concentration of cadmium) < 0.87 mg/L, then dispose of the waste fly ash in a sanitary landfill.

The decision performance curve for this decision rule would have a probability of taking action (i.e., sending fly-ash waste to a RCRA landfill) of 0.90 at a possible true concentration value of RT = 1.0 mg/L.

Step 3: Calculate the true concentration (say, θ < RT) that corresponds to an action level of AL = 0.87 mg/L and a false positive error of 20%. The probability calculations are based on an approximating normal probability distribution for the cadmium concentration measurements. This approximating normal probability has a mean = θ mg/L and a standard deviation = S_w = 0.4 mg/L. The 20% percentile point for the standardized normal probability distribution is $Z_{0.20}$ = 0.848.

False Positive Error = $^{'}$Pr{ Average < AL when the true concentration = θ} = 0.20.

or

$$\frac{AL - \theta}{S_w / \sqrt{n}} = +Z_{0.20}\ ,$$

AL = Action Level

θ = True Value

$Z_{0.20}$ = Tabled Z-value from standard normal distribution at 0.10

$$\theta = 0.87\,mg/L - (0.848)(0.4\,mg/L) / 4 = 0.87\,mg/L - 0.08\,mg/L\ ,$$

or

$$\theta = 0.79\,mg/L\ .$$

The decision performance curve would have a probability of taking an action (i.e., sending fly-ash waste to a RCRA landfill) of 0.20 at a true cadmium concentration of θ = 0.79 mg/L. The possible true cadmium concentration values in the interval (0.79 mg/L, 1.0 mg/L) represents values that cause the decision rule to send fly-ash waste to a RCRA landfill even though the true concentration is below the regulatory threshold. This interval can be reduced by increasing the number of samples, by changing the false negative error or by changing the false positive error.

Step 4: Draw the decision performance curve by using the standardized normal probability distribution. The standardized normal probability distribution is defined as a normal probability distribution with mean = 0 and standard deviation = 1.0. There are many tables and computer programs that can be used to calculate probabilities for a standardized normal random variable, Z. A normal random variable, X, with mean = μ and standard deviation = σ can be transformed to a standardized normal random variable by $Z = (X - \mu)/\sigma$.

Prob(Action) = Pr(Average $\geq$ AL when the true concentration = θ).

$$Prob(Action) = 1.0 - Prob\left(Z \leq \frac{AL-\theta}{S_w / \sqrt{n}}\right),$$

$$Prob(Action) = 1.0 - Prob\left(Z \leq \frac{0.87-\theta}{0.1}\right).$$

Figure 3-2 plots the decision performance curve of Prob(Action) versus possible true concentration values θ.

7. OPTIMIZE THE DESIGN

The decision maker(s) will select the lowest cost sampling design that is expected to achieve the DQOs. The optimal design(s) for sampling the fly-ash waste will be generated by the statisticians on the evaluation team. The choice of sampling plan will be decided by consensus.

Figure 3-2 plots the probability of taking action (disposing of the waste in a RCRA landfill) versus different possible values for the true concentration in the TCLP extract. Note that various numbers of samples, n, are used to generate each curve. All of the curves meet the criteria of 10% false negatives at the regulatory threshold. The differences lie in the false positives versus the true concentration. If the true concentration value is equal to .87 mg/L (the action level) then the probability of taking action is .5 or 50% chance of taking action. The action level is below the regulatory threshold and does insure the agreed upon false negative rate of 10%. Note that if the regulatory level and the action level were equal, the likely chance of having a false negative would be 50% at 1 mg/L.

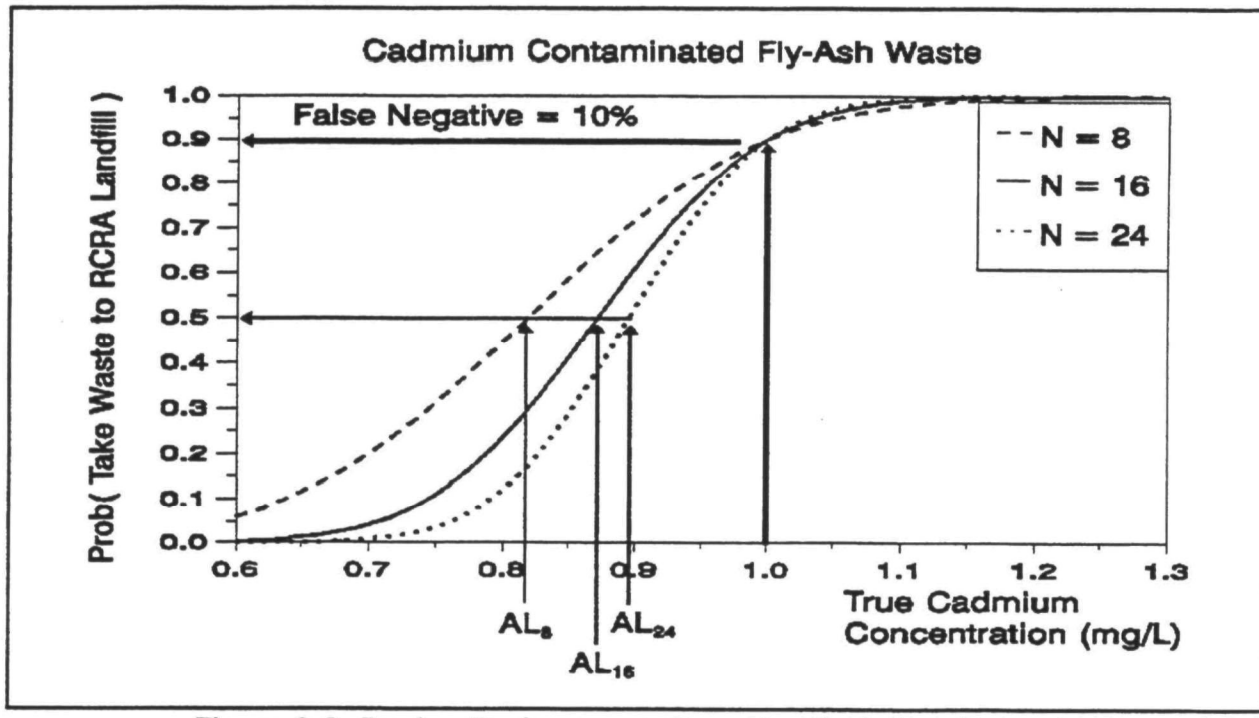


Figure 3-2. Design Performance Curve for Cadmium Example

Implementation -- After completion of the sampling and measurement process, the data assessment is performed. The concentration measurements from each load of fly-ash waste are averaged and compared to the action level. Any load with average concentrations less than the action level will be sent to a sanitary landfill, and those loads with average concentrations greater or equal to the action level will be sent to a RCRA landfill.

3.3 Sampling and Analysis Design

Specifying Sample Collection Procedures

- The methods and equipment used for sampling waste materials vary with the physical and chemical properties of the waste materials.
- 40 CFR Part 261, Appendix I lists several representative sampling methods. Unfortunately, for most matrices, selecting representative samples is an extremely difficult objective.
- The methods in the above reference are recommended. *No prior approval by EPA is required if alternate sampling methods are used.*
- All procedures for sampling should be documented and referenced.

Sample Containers, Preservation, and Storage

Prior to Extraction or Filtration

- No preservatives are added to the initial waste collected for TCLP filtration and extraction.
- Preservatives used after filtration and extraction are listed in Chapter 4 of this document.
- If organics are being analyzed, samples must be collected in glass containers with Teflon lid liners.
- Metals may be collected in polyethylene or glass containers.
- If practical, samples which will undergo Zero Headspace Extraction (ZHE) for volatiles should be collected in 40 mL glass Volatile Organic Analysis (VOA) vials with Teflon lids. Clay type soil samples, or other large particle size solid matrices which are difficult to put into narrow-mouth containers, should be collected in 250 mL wide mouth glass jars.
- Any sample which will undergo ZHE should be collected with minimal head space in the container.
- All samples should be stored at 4°C $\pm$ 2°C prior to extraction or filtration. Samples should be placed in coolers immediately after collection.

Sample Volumes

A discussion of required sample volume is presented in Chapter 4 which includes the following general points:

- A minimum of 100g of waste is needed to determine the percentage of solids, extraction fluid type, and particle size.

- A second aliquot of 100g is the minimum which must be extracted for non-volatiles. The amount of sample is dependent on the percent wet solids. The lower the percent wet solids, the greater the sample volume required for leaching.

- Another aliquot of at least 25g must be used for volatiles by ZHE. The amount of sample is dependent on the percent wet solids. The lower the percent wet solids, the more material which must be collected for leaching.

- If multiple phases are collected and the solids appear to be $\leq$0.5%, each phase may have to be analyzed individually. This is especially true of oily waste. If the oil will not pass through the filter, it will be considered a solid.

- Enough sample must be collected to allow for the matrix spike. Each matrix requires a spike. The same amount of material is required for the spike as for the sample.

- Extra sample volume may be needed if vessel leakage or breakage occurs. Multi-phasic samples require much larger sample volumes than monophasic samples.

Sampling Equipment Decontamination

Acceptable sampling equipment decontamination should be performed before sample collection. Decontamination may be required in the field if an adequate number of pieces of sampling equipment is not available to allow equipment to be dedicated to one sampling point.

Each EPA region and some states have special decontamination requirements. These decontamination requirements should be verified for compliance in a particular region or state. The RCRA decontamination procedures may differ from CERCLA decontamination procedures in some locations. For most TCLP sampling events, there are no sampling equipment decontamination criteria.

Paint or coatings on sampling equipment must be removed from any part of the equipment that may contact the sample.

The USEPA Region 2 decontamination procedure for CERCLA sampling and RCRA Facility Investigation (RFI) sampling is as follows:

a. wash and scrub with low phosphate detergent
b. tap water rinse
c. rinse with 10% HNO_3, ultra pure
d. tap water rinse
e. an acetone-only rinse or a methanol followed by hexane rinse (solvents must be pesticide grade or better)
f. thorough rinse with demonstrated analyte free water*
g. air dry, and
h. wrap in aluminum foil for transport

* The volume of water used during this rinse must be at least five times the volume of solvent used in Step e.

If metal samples are not being collected, the nitric acid rinse may be omitted. If organic samples are not being collected, the solvent rinse may be omitted.

Holding Times for TCLP

- TCLP has three sets of holding times (see Table 3-1). The first holding time commences with sample collection and ends with TCLP extraction. The second holding time is from TCLP extraction to preparative extraction. The final holding time is from preparative extraction to analysis.

Table 3-1 - TCLP Holding Times

Analysis Type	Days From Field Collection to TCLP Extraction	Days From TCLP Extraction to Preparative Extraction	Days From Preparative Extraction to Determinative Analysis
Volatiles	14	NA	14
Semivolatiles*	14	7	40
Metals, except Mercury	180	NA	180
Mercury	28	NA	28

* Pesticides and herbicides are deemed semivolatiles.

- Some regional and state agencies may alter these times. If Contract Laboratory Program (CLP) methods are used, the holding times from extraction to preparation and from preparation to determinative analysis will differ from the above times. However, to demonstrate compliance with the TC or Land Ban regulations, sample holding times may not exceed the holding times listed in the preceding table.

Field QC Samples

The following field QC samples may be collected during the sampling process:

- **Trip Blanks** are aliquots of analyte-free water brought to the field in sealed containers and transported back to the lab with the sample containers. Trip blanks, which are only analyzed for volatiles, are especially useful when aqueous volatiles are collected. Trip blanks allow one to assess contamination from transport and storage.

- **Equipment Blanks** are analyte-free water which is poured over the sampling equipment in the field after the final rinsing of equipment. Equipment blanks allow one to assess cross contamination and decontamination procedures.

  Several key issues must be understood when evaluating the need for equipment blanks. Equipment blanks are analyzed for total constituents while waste undergoes extraction. The TCLP leaching process uses 20 grams of leaching fluid per gram of

wet weight sample. Therefore, TCLP equipment blanks may not be cost effective for some types of TCLP sampling. However, if sampling is being done for legal purposes, such as enforcement actions or potential litigation, equipment blanks should be collected to assure that the data are legally defensible.

- **Field Duplicates** are samples collected from the same location and waste source at the same time. The goal is to determine variability in the waste or sample matrix. The frequency of these depends on the sampling design. Most agencies recommend at least 5%. Duplicates provide information about sampling and analysis precision.

- **Laboratory Duplicates** are samples which have sufficient volume to allow the laboratory to homogenize the sample, split the sample and prepare and analyze both aliquots as separate samples. The purpose is to assess precision between two laboratory analyses on the matrix.

Enough sample volume must be collected for the lab to generate matrix spikes and laboratory duplicates. The frequency of these samples varies, and depends on the different types of waste collected, the time over which they are collected, and the regulatory requirements.

Sampling Documentation

- All sample locations should be identified in a log book. Locations should be from a surveyed point when applicable. Both horizontal and vertical coordinates should be documented. Each sampling point should be assigned a unique number or other identifier.

- Unique sample numbers, collector, date and time of collection, container types, matrix and analysis required (including TCLP and the extract/filtrate analysis) should be documented in field log books, chain-of-custody (COC) forms, and on sample labels.

- In most cases, a COC should be used to document the collection and transport of samples to the laboratory. The chain of custody form should be signed and dated by individuals who collect, transport or receive the samples. Copies of COCs should be kept by sending and receiving parties.

- Any deviations from the sampling plan should be documented in the log books.

- The type of sampling equipment utilized must be documented in the log book.

- Ambient weather conditions at the sampling location(s) should be documented in the log book.

3.4 Analytical Method Selection

Strategy for Analytical Method Selection:

- Determine the analytes
- Determine the methods of analysis
- Specify detection limits and regulatory action levels
- Specify quality control samples and requirements

To specify appropriate analytes to demonstrate compliance with the TC regulations, the data requester needs to understand the basic groupings of analytes which are performed by each analytical method. The data requester also must understand the typical detection limits and the issues which revolve around not being able to achieve these limits. The following tables outline the TC constituent, the category of the analyte, and the potential methods of sample preparation and analysis. The current CLP contract required detection limits/quantitation limits and the SW-846 practical quantitation limits are also presented in this section.

The analytical method information should be used in conjunction with process knowledge and Table 3-2, which lists the TC constituents and regulatory levels, to plan the sampling and analysis.

TABLE 3-2 - TC ANALYTES AND THEIR REGULATORY LEVELS

Constituent[1]	Regulatory Level (mg/l)	Constituent[2]	Regulatory Level (mg/l)	Constituent[2]	Regulatory Level (mg/l)
Arsenic	5.0	Benzene	0.5	Heptachlor	0.008
Barium	100.0	Carbon tetrachloride	0.5	Hexachloro-benzene	0.13
Cadmium	1.0	Chlordane	0.03	Hexachloro-1,3-butadiene	0.5
Chromium	5.0	Chlorobenzene	100.0	Hexachloro-ethane	3.0
Lead	5.0	Chloroform	6.0	Methyl ethyl ketone	200.0
Mercury	0.2	o-Cresol	200.0	Nitro-benzene	2.0
Selenium	1.0	m-Cresol	200.0	Pentachloro-phenol	100.0
Silver	5.0	p-Cresol	200.0	Pyridine	5.0
Endrin	0.02	Cresol (total)	200.0	Tetrachloro-ethylene	0.7
Lindane	0.4	1,4-Dichloro-benzene	7.5	Trichloro-ethylene	0.5
Methoxy-chlor	10.0	1,2-Dichloroethane	0.5	2,3,5-Trichloro-phenol	400.0
Toxaphene	0.5	1,1-Dichloro-ethylene	0.7	2,4,6-Trichloro-phenol	2.0
2,4-D	10.0	2,4-Dinitrotoluene	0.13	Vinyl chloride	0.2
2,4,5-TP (Silvex)	1.0				

[1] Original EP Tox constituents.
[2] Chemical constituent added by TC Rule (shaded areas).

General Method Information

There are two categories of permissible methods used to analyze TCLP extracts:

1. Many state environmental agencies mandate the use of SW-846 methods for hazardous waste determinations. SW-846 methods are sometimes also required to demonstrate compliance with the RCRA regulations. Several different SW-846 methods may be used to analyze TC analytes. Appendix IX explains when SW-846 methods are mandatory.

2. Any appropriate EPA approved method may be used to demonstrate compliance with the TC regulations. However, if the data are going to be validated, CLP methods are recommended. Two semi-volatile TC analytes (m-cresol, pyridine) are not included in the CLP target compound list of analytes. Therefore, the method must be slightly modified to incorporate these two compounds.

Specifying detection limits and regulatory action levels

- The method or contract detection limits must be evaluated versus the regulatory TC limits. The method or contract limits must be lower than the regulatory limits.

- There are three compounds which have quantitation limits that exceed the regulatory limits: 2,4-dinitrotoluene, hexachlorobenzene, and pyridine. In these cases, the quantitation limit becomes the regulatory limit.

- The EPA Region 2 TCLP SAS Request (Appendix VIII), shows recommended TC detection limits which are increased in order to minimize matrix effects.

Metals Analysis Information

- Metals analysis can be performed by three methods: Inductively Coupled Plasma (ICP), Flame Atomic Absorption (FAA) and Graphite Furnace Atomic Absorption (GFAA).

    - Laboratories usually analyze metals, except mercury, in the TCLP extract by ICP.

    - Mercury is usually analyzed by Cold Vapor Atomic Absorption (CVAA).

    - The GFAA generates lower detection limits than ICP method.

- The CLP Contract Required Detection Limits (CRDLs) are the same for both ICP and GFAA.

Table 3-3 Metals Analysis Method By ICP

Analyte	SW 846 Preparation/ Analysis	SW 846 PQL, mg/L(1)	CLP CRDLs, mg/L (2)
Arsenic	3010/6010	.05	.01
Barium	3010/6010	.002	.2
Cadmium	3010/6010	.004	.005
Chromium	3010/6010	.007	.01
Lead	3010/6010	.04	.003
Selenium	3010/6010	.07	.005
Silver	7760 (prep only)/6010	.007	.01

(1) PQL = Practical Quantitation Limit = EQL = Estimated Quantitation Limit
(2) Contract Laboratory Program, Statement of Work for Inorganic Analysis, ILM03.0

Table 3-4 Metals Analysis Methods by GFAA and Mercury by CVAA

Analyte	SW 846 Preparation/ Analysis	SW 846 PQL, mg/L(1)	CLP CRDLs, mg/L (2)
Arsenic	7060/7060	.001	.01
Barium	3020/7080	.1	.2
Cadmium	3020/7131	.001	.005
Chromium	3020/7191	.001	.01
Lead	3020/7421	.001	.003
Selenium	7740/7740	.002	.005
Silver	7760 (prep only)/7760	.01	.01
Mercury	7470	.0002	.0002

(1) PQL = Practical Quantitation Limit = EQL = Estimated Quantitation Limit
(2) Contract Laboratory Program, Statement of Work for Inorganic Analysis, ILM03.0

Table 3-5 Pesticide and Herbicide Quantitation Limits by SW 846 and CLP

Analyte	SW 846 Preparation/ Analysis	SW 846 PQL, ug/L (1)	CLP CRQL, ug/L (2)
Pesticides			
endrin	3510 or 3520/ 8080B	0.06	0.10
lindane (gamma BHC)	3510 or 3520/ 8080B	0.04	0.05
methoxychlor	3510 or 3520/ 8080B	1.76	0.05
heptachlor	3510 or 3520/ 8080B	0.03	0.05
toxaphene	3510 or 3520/ 8080B	2.4	5.0
chlordane (4)	3510 or 3520/ 8080B	0.14	0.05
Herbicides			
2,4-D	8150A	12	(3)
2,4,5-TP (Silvex)	8150A	2.0	(3)

(1) PQL = Practical Quantitation Limit = EQL = Estimated Quantitation Limit
(2) Contract Laboratory Program, Statement of Work for Organic Analysis, OLM01.8
(3) No CLP methods exist for these compounds.
(4) SW-846 quantitation limits are for "technical" chlordane. CLP quantitation limits are for alpha and gamma chlordane.

Table 3-6 Quantitation Limits for Volatile TC Constituents

Volatiles	SW 846 Preparation/ Analysis	SW846 8240 PQL, ug/L (1)	CLP CRQL, ug/L (3)
benzene	8240B or 8020B	5	10
carbon tetrachloride	8240B or 8010B	5	10
chloroform	8240B or 8010B	5	10
chlorobenzene	8240B or 8010 or 8020B	5	10
1,2-dichloroethane	8240B or 8010B	5	10
1,1-dichloroethylene (1,1-dichloroethene)	8240B or 8010B	5	10
methyl ethyl ketone (2-butanone)	8240B (2) or 8015	100	10
tetrachloroethylene (tetrachloroethene)	8240B or 8010B	5	10
trichloroethylene (trichloroethene)	8240B or 8010B	5	10
vinyl chloride	8240B or 8010B	10	10

(1) PQL = Practical Quantitation Limit = EQL = Estimated Quantitation Limit
(2) Poor purging efficiency by this method produces a high detection limit.
(3) Contract Laboratory Program (CLP) Statement of Work for Organic Analysis, OLM01.8

Table 3-7 Quantitation Limits for Semivolatile TC Constituents

Semivolatiles (BNAs)	SW 846 Preparation/ Analysis	SW 846 PQL, ug/L (1)	CLP CRQL, ug/L (5)
o-cresol (2-methylphenol)	3510/8270B	10	10
m-cresol (3-methylphenol)	3510/8270B	10	(3)
p-cresol (4-methylphenol)	3510/8270B	10	10
1,4-dichlorobenzene	3510 or 3520/ 8270B	10	10
2,4-dinitrotoluene	3510 or 3520/ 8270B	10	10
hexachlorobutadiene (hexachloro-1,3-butadiene) (4)	3510 or 3520/ 8270B	10	10
hexachloroethane	3510 or 3520/ 8270B	10	10
hexachlorobenzene	3510 or 3520/ 8270B	10	10
nitrobenzene	3510 or 3520/ 8270B	10	10
pentachlorophenol	3510 or 3520/ 8270B	50	25
pyridine	3510 or 8270B	ND (2)	(3)
2,4,5-trichlorophenol	3510 or 3520/ 8270B	10	25
2,4,6-trichlorophenol	3510 or 3520/ 8270B	10	10

(1) PQL = Practical Quantitation Limit = EQL = Estimated Quantitation Limit
(2) ND = Not Determined. If these methods are used, the method detection limits must be determined.
(3) These analytes are not routinely part of the CLP method. If required for TC, these analytes must be specially requested. The CLP 2/88 extraction procedure must be used for the TC semivolatile analytes if these analytes are desired.
(4) Other methods quantitate this in the volatile fraction.
(5) Contract Laboratory Program, Statement of Work for Organic Analysis, OLM01.8

Chapter 4

Chapter 4

OVERVIEW OF THE TCLP METHOD

Preliminary Sample Preparation

Leaching Procedure for Non-Volatiles

Leaching Procedure for Volatiles

TCLP Method Quality Control

4.0 OVERVIEW OF THE TCLP METHOD

This chapter provides an overview of the TCLP method. Appendix I of this document includes a copy of the method and Appendix III provides worksheets which will be useful in understanding method calculations. The following topics are covered in this chapter:

- Preliminary sample preparation for leaching
- Leaching procedure for non-volatiles
- Leaching procedure for volatiles
- TCLP method QC

4.1 Preliminary Sample Preparation for Leaching

Prior to performing the leaching procedure, several preliminary determinations must be made. These include:

- Are there enough solids present for the leaching process?
- Is particle size reduction required?
- Are immiscible liquids present?
- Which leaching fluid should be used for non-volatile analytes?

Figure 4-1 provides a flow chart which delineates preliminary determinations. The first step is to take 100g of the waste, pass it through a 0.6 to 0.8 µm filter up to 50 psi and determine the percent solids. If the percent solids are greater than or equal to 0.5% on a dry weight basis, the solid must be leached. Any material which remains in the filtration apparatus is considered a solid. When liquids remain on the filtration apparatus because they are too viscous to pass through the filter, they are treated as solids. Any material which passes through the filter is the filtrate and considered a liquid. Therefore, viscous oils which do not pass through the filter are classified as solids. Oily waste will be discussed further at the end of this document. If the percent solids are less than 0.5%, the filtrate is the TCLP extract, and the laboratory analyzes the filtrate.

Particle Size

If the percent solids is > 0.5%, the laboratory analyst must determine whether particle size reduction will be required.

- The requirement is *not* to measure the size. However, the surface area and particle size must conform with one of the following criteria:
  - The solid must have a surface area per gram of material equal to or greater than 3.1 square centimeters.
  - The solid must be smaller than 1 cm in its narrowest dimension (i.e., pass through a 9.5 mm (0.375 inch) standard sieve).
- If the particle size is too large, cutting, grinding, or crushing may be utilized to decrease particle size.

Choosing the Leaching Solution

Figure 4-1 shows the determination of the type of leaching fluid for use. If the solid content is greater than or equal to 0.5%, and if the sample is being analyzed for metals or semivolatiles, the type of leaching solution must be determined. Note that the leaching solution determination step requires a smaller (1 mm) particle size than the analytical method because the leaching solution determination allows much less contact time between the leaching solution and the sample.

After weighing a 5.0 g subsample of the solid, adding 96.5 mL of reagent water, and stirring for 5 minutes, the pH is measured. If the pH is $\leq$ 5.0, fluid #1 is used. If the pH is > 5.0, 3.5 mL of 1N HCl is added. The mixture is heated to 50°C for 10 minutes and cooled. If the measured pH is less than 5.0, fluid # 1 is used. If the pH is greater than 5.0, extraction fluid #2 is used.

The heating cycle is a critical step. After the sample has been heated, it should be cooled to room temperature. The pH must be measured immediately after the sample has reached room temperature. If the solid waste does not remain in contact with the acidic solution under specified time and temperature conditions, an erroneous pH may be measured.

The leaching fluid for all volatiles is fluid #1. Fluid #1 is an acetic acid and sodium hydroxide buffer of pH 4.93 $\pm$ 0.05. Fluid #2 is an acetic acid solution of pH 2.88 $\pm$ 0.05.

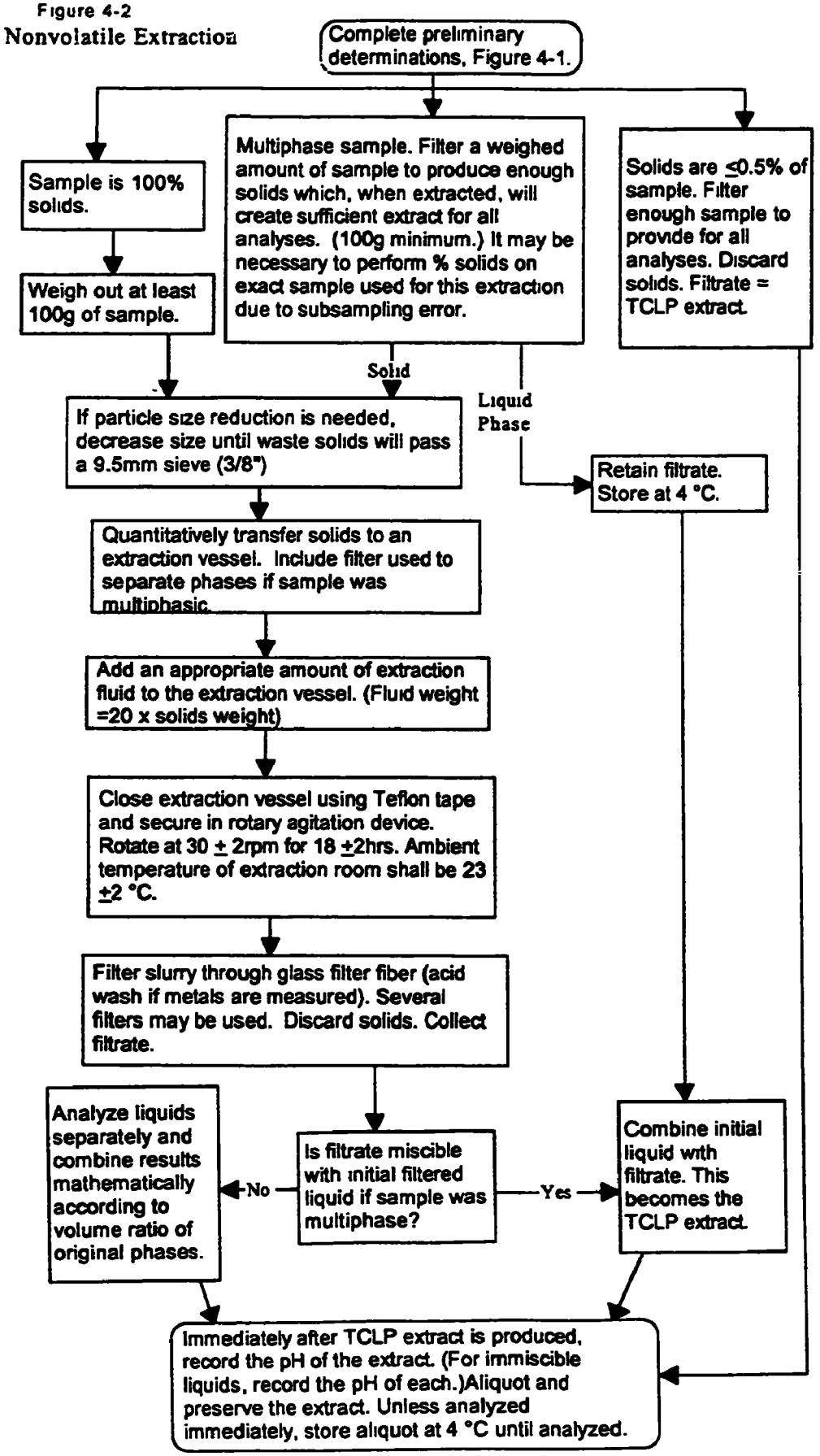
4.2 Leaching Procedure for Non-Volatiles

The non-volatiles include semivolatile organics, which are also called base, neutral, and acid extractables (BNAs), pesticides, herbicides, and metals. If the percent solids exceeds 0.5%, the solid is leached with the appropriate extraction fluid after any required particle size reduction. The following topics are discussed in this section:

- Determination of extraction fluid weight
- Sample and QC sample volumes
- Extract volumes required
- Issues when dealing with multi-phasic waste
- Initial filtrate versus TCLP leachate

The non-volatile extraction or leaching process is outlined in Figure 4-2. The extraction process includes placing the sample and appropriate fluid in the bottle extraction vessel and tumbling for 18 $\pm$ 2 hours and filtering the extract for subsequent analysis. The bottle extraction vessel is described in Section 4.2.2 of Method 1311, which is Appendix I of this document. In order to generate scientifically valid and legally defensible data, appropriate weights of environmental samples and leaching fluids must be used.

Figure 4-2
Nonvolatile Extraction



Determination of Extraction Fluid Weight

The following formula is used to compute the required weight of TCLP extraction fluid:

$$\text{Weight of Extraction Fluid} = 20 \times \frac{\%\text{Solids}}{100} \times \text{Weight of Waste Filtered}$$

The amount of extraction fluid required per extraction is 20 times the weight of wet filtered solids used in the extraction.

A minimum of 100g of waste material must be filtered to generate the solids utilized in the extraction. If the sample is 100% solids, a minimum of 100g must be used in the extraction. When aqueous environmental samples contain between one-half and ten percent solids, several kilograms of sample are required for analysis.

Sample and QC Sample Volumes

The generation of sufficient extract volume to perform all analysis is critical.

- The required volumes vary with the laboratory. *Check with the laboratory for actual volumes.*
- Depending on which metal analytes are selected, two or three digestions may be required.
- If matrix spikes or duplicates are performed, additional volume will be required. Labs may charge additional fees for these QC samples.

Table 4-1 Volume of Extract Required for One Nonvolatile Analysis

Analysis Type	Volume of TCLP Leachate Typically Required per Test
BNA	1 L
Chlorinated Pesticides	1 L
Herbicides	1 L
Metals	300 mL/digestion

The previous table outlines the "typical" volumes of extract or total leachate required for non-volatile analysis. *These volumes may vary with the laboratory. It is imperative that you check with the lab as to the amount of waste required for their analysis process.* The amount of waste varies with the percent solids. The lower the percent solids, the more waste will be needed for TCLP preliminary and final testing. If the waste sample is a filterable liquid with less than 0.5% solids, the volume listed in the previous table can be used as a guide for the minimum volume needed for the analysis.

Issues When Dealing with Multi-phasic Waste

- Subsampling stratified waste is difficult. Therefore, the analyst should consider calculating percent solids from the same sample container used for the TCLP extraction instead of compositing all the sample containers. This is the largest source of error in the TCLP leaching process. The laboratory must consider the amount of each phase present in each bottle and adjust the calculations accordingly.

- The particle size of multi-phasic material may be difficult to assess. The lab should identify procedures to classify multi-phasic samples which are not amenable to size measurement.

- Five grams of sample are usually used to determine the appropriate TCLP leaching fluid. If there is not enough volume of any individual phase, less material may be used.

- The pH of the filtrate should be recorded. This provides useful information when validating field or laboratory duplicates.

- The filtrate volume should also be measured. This information will be needed if the multiple phases must be mathematically combined.

Initial Filtrate Versus TCLP Leachate

Two liquids are generated when a multi-phasic waste is analyzed.

- Initial filtrate

- Leachate

- If the filtrate is miscible with the leachate, the two solutions are mixed prior to analysis.

- If the two solutions are not miscible, they are analyzed separately, and the results combined mathematically.

The mathematical calculations are performed via the following equation if the TCLP filtrate and extract are not miscible.

$$\text{Final analyte Concentration} = \frac{(V1)(C1) + (V2)(C2)}{V1 + V2}$$

where:

V1 = The volume of the first phase (L).
C1 = The concentration of the analyte of concern in the first phase (mg/L).
V2 = The volume of the second phase (L).
C2 = The concentration of the analyte of concern in the second phase (mg/L).

After generating the TCLP extract, the pH of the extract should be recorded. If the filtrate and TCLP extract are mixed, record the pH of the mixture as well as the original TCLP extract and filtrate. The TCLP extract or filtrate/extract mixture should be aliquoted for each analysis. Matrix spikes for all subsequent analyses must be added at this time.

The metals aliquot should be preserved to a pH<2 with nitric acid. Adjust the pH of a small portion of the TCLP extract or mixture prior to adjusting the entire metals aliquot. If a precipitate forms, do not adjust the pH of the sample extract. If nitric acid is not added, the sample should be analyzed as soon as possible after TCLP extraction. Metals analysis must include digestion prior to analysis. Aliquots for BNAs, herbicides and pesticides do not require chemical preservation. All aliquots must be stored at 4°C $\pm$ 2°C prior to analysis.

Example

The following example demonstrates how to calculate the weight of extraction fluid required to perform a TCLP extraction. In this example, the environmental sample contains 40% solids. Only metals will be analyzed since the waste is from a metals finishing shop.

In order to determine the total amount of waste required to generate 100g of solids, the following equation is used:

Amount of multi-phasic material = (10,000)/ (weight percent wet solids)

If 100g of original waste yields 40g of solid, the total amount of waste required to generate 100g of solid is 250g.

250g of total waste required = 10,000/40

Using the equation in Section 7.2.11 of Method 1311 (Appendix I):

$$\text{Weight of extraction fluid} = 20 \times \frac{\%\ \text{solids}}{100} \times \text{weight of waste material filtered}$$

$$20 \times \frac{40}{100} \times 250\text{g} = 2{,}000\text{g of extraction fluid}$$

Labs typically assume a density of 1 g/mL for the extraction fluid. Also, note that 40% is used, not 0.40, for the percent solids. Since 300 mL of extraction fluid is required for one complete metals digestion, using 250g of the multi-phasic waste will provide enough volume for the metals analysis. This allows enough volume to analyze one matrix spike. The use of matrix spikes will be discussed in the QC section of this chapter. The matrix spike sample size requirement is the same as for original environmental sample analysis.

If organic analysis were required, at least three times as much waste would have been used in the TCLP extraction. For matrix spikes analyses, a triple volume of TCLP leachate will be required. The terms analytical batch and waste type are not defined in the TCLP regulation. Most methods, including CLP and SW-846, indicate that a batch is the number of samples processed through preparation and analysis simultaneously, and should not exceed 20 samples of the same matrix. Most methods require that matrix spikes and matrix spike duplicates (MS/MSD) for organics be performed at 1 MS/MSD per processed batch, with a batch containing no more than 20 samples.

4.3 Leaching Procedure for Volatiles

Volatile organics are leached using different equipment than non-volatiles.

Figures 4-3 and 4-4 are the flow charts describing the volatile leaching procedure using the Zero Headspace Extractor (ZHE). The ZHE must be used when leaching volatiles. In order to minimize evaporation of volatiles, the volatile leaching procedure is performed on a separate aliquot of waste. Once the percent solids has been determined in the preliminary sample preparation phase, a second aliquot of the waste is used to generate the volatile analysis extract. In all cases, no more than 25g of solids should be placed in the ZHE because the total volume of the ZHE is 500-600 mLs. In order to prevent the loss of volatile compounds, heating or excessive sample manipulation must be kept to a minimum. The samples and equipment used in the process should be cooled to 4°C when possible to prevent loss of volatiles.

If the sample contains less than 0.5% dry solids, the filtrate is defined as the TCLP extract. The solid is discarded in this instance. The filtrate is collected in either a Tedlar bag or a glass syringe which is described in the equipment section of the procedures in Appendix I of this document. This filtrate becomes the TCLP extract.

Weight of Waste Charged to the ZHE

If the solids are > 0.5% dry solids, the material must be extracted.

If the solids are > 0.5% and < 5.0 %, a 500 g subsample of the waste is weighed and recorded.

If the solids are > 5.0%, the following formula is used to determine the amount of waste to place in the ZHE:

$$\text{Weight of Waste Charged in ZHE} = \frac{25 \times 100}{\text{Percent wet solids}}$$

If the solids are greater than 0.5% and the sample is multi-phasic, any solids must be examined for particle size prior to filtration. The sieve is *not* used to verify particle size for the volatile sample. Particles are measured with a ruler and should be less than 1 cm diameter. Any particle size reduction should be done with minimal exposure to air and without heat production. All apparatus used in this process should be cooled to 4°C.

Figure 4-3
Volatile by ZHE

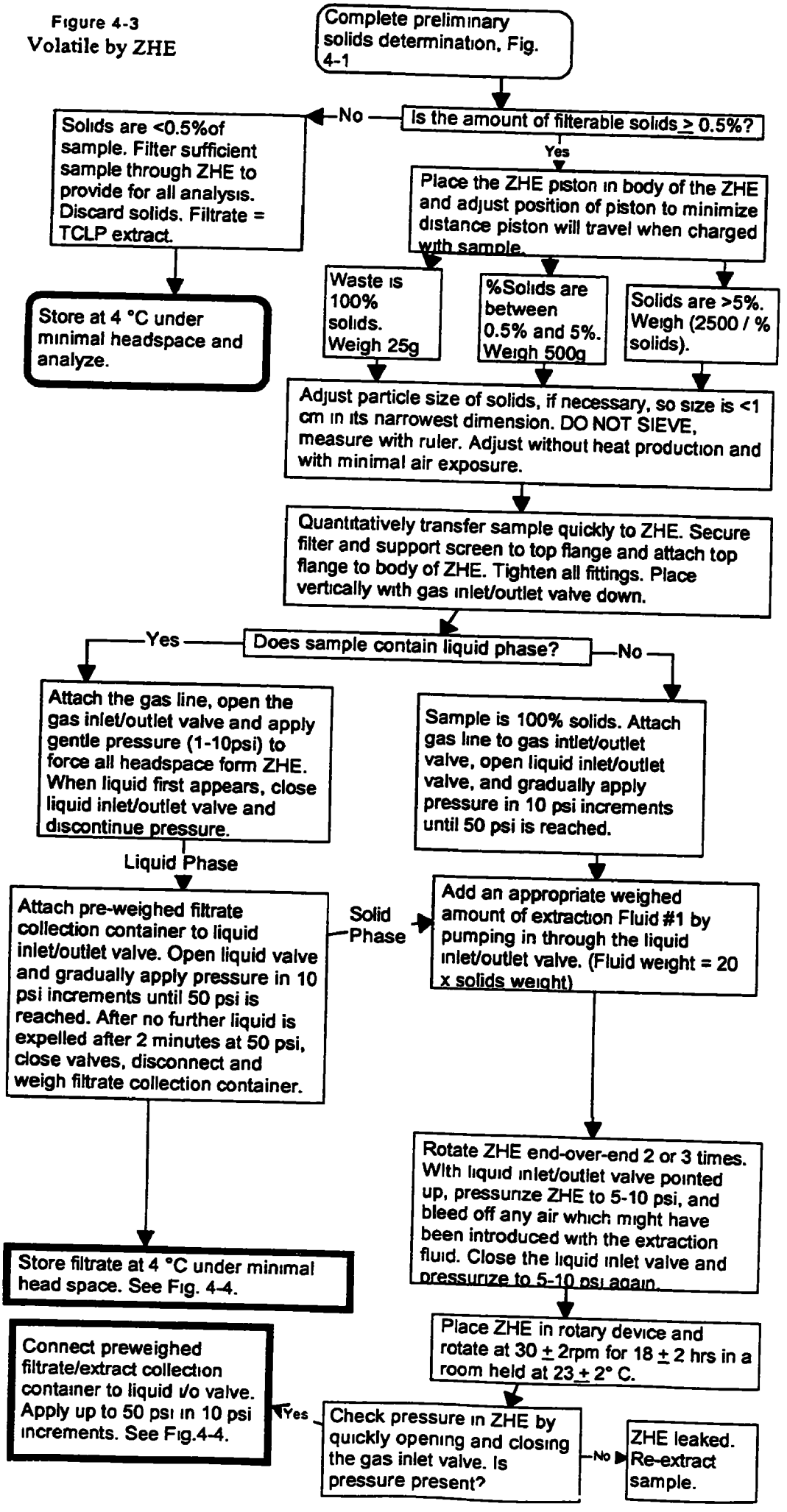
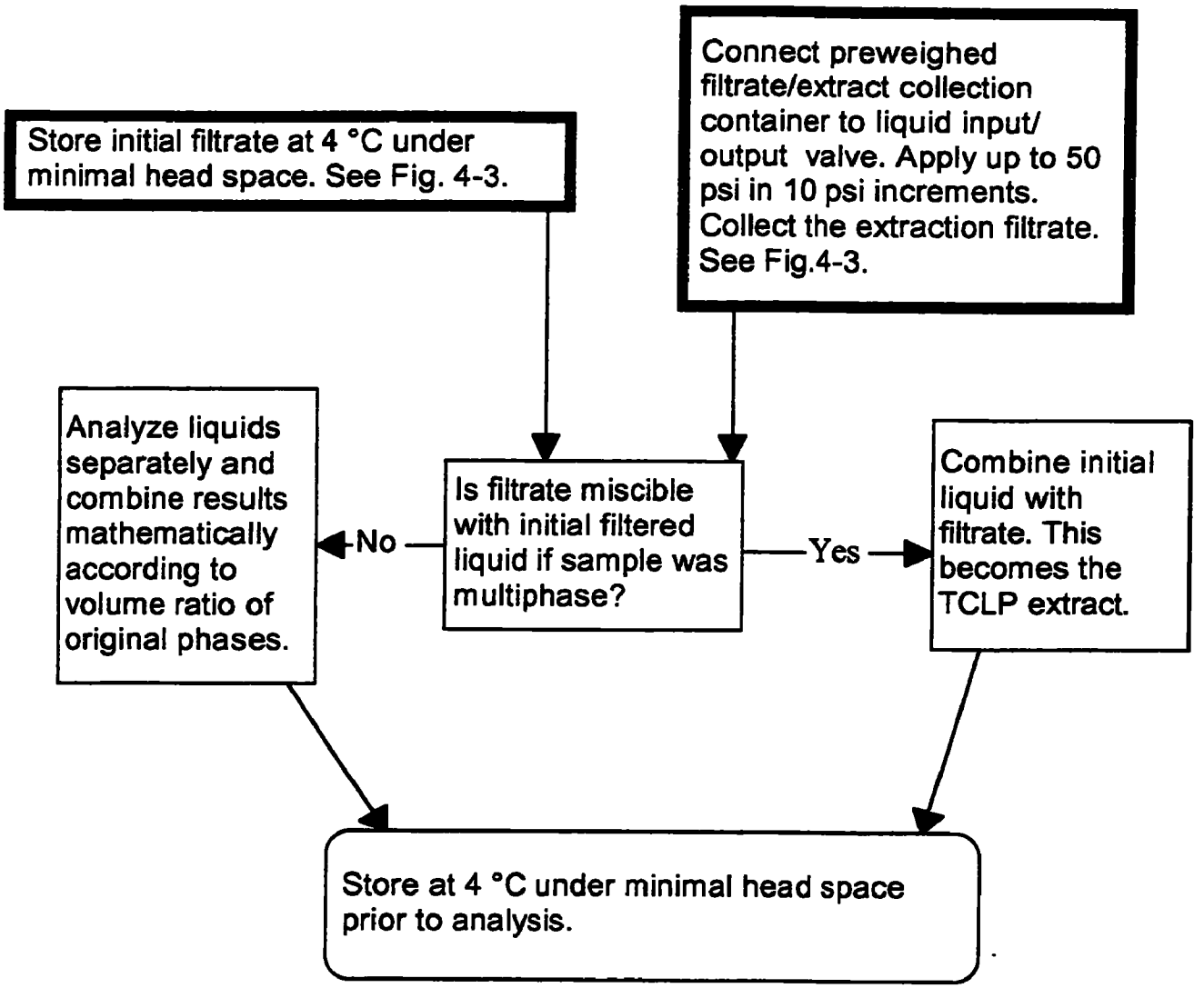


Figure 4-4
Volatiles by ZHE Continued



All waste which remains in the ZHE after reaching a pressure of 50 pounds per square inch (psi) is considered solid phase and undergoes leaching. Any liquid which is removed during filtration is considered liquid phase filtrate. The filtrate is captured in either a Tedlar bag or glass syringe. The solids are leached with fluid #1.

If the percent solids is 100%, a 25g sample of the solid is placed in the ZHE after any necessary particle size reduction is performed. The particle size reduction follows the same protocol requirements as volatile extraction of waste with solids content greater than 0.5% but less than 100%. The extraction is similar to the TCLP extraction of multi-phasic material.

When performing TCLP extraction for volatile analysis, extraction fluid #1 is always used. The quantity of extraction fluid is 20 times the solid waste weight used in the extraction.

The extraction is performed by placing the ZHE in the rotary agitator at 30 $\pm$ 2 rpm for 18 $\pm$2 hours. The ambient temperature is maintained at 23 $\pm$2°C during agitation. At the end of the agitation period, the ZHE piston pressure must be measured to verify that pressure was maintained during the extraction. If pressure was not maintained, the extraction must be repeated after the ZHE is examined for mechanical problems. If the pressure was maintained, the material in the ZHE is separated into solid and liquid phases by pressure filtration. A small amount of the liquid extract should be examined for miscibility with the previously captured filtrate. If these fluids are miscible, the liquid extract and the filtrate may be stored in the same container (Tedlar bag or syringe) with minimal or no headspace. This mixture becomes the TCLP extract for volatile analysis. If the two fluids are not miscible, they are stored in separate containers with minimal or no headspace. The volatile analysis are performed separately and combined mathematically using the same equation as for the non-volatile analysis. All extracts and fluids are kept at 4°C $\pm$2°C prior to analysis.

4.4 TCLP Method Quality Control

- TCLP extraction blank
- Method preparation blanks
- Calibration
- Matrix spikes

BIAS CORRECTION IS NO LONGER REQUIRED.

- Method of standard additions

TCLP Extraction Blanks

- A minimum of one TCLP extraction blank is generated for every 20 extractions processed in a given extraction vessel using the same fluid. Most labs have multiple extraction vessels. The common industry strategy is to generate one TCLP extraction blank for each group of samples processed simultaneously using the same batch of fluid.

Calibration

- Calibration should follow the respective method requirements. Typically a three to five point initial calibration followed by a single point continuing calibration is specified.

Method Preparation Blanks

- Preparation blanks performed for a specific analysis should follow the frequency and requirements of the method. Typical requirements are one per preparative batch from similar matrix for every 20 samples.

Matrix Spikes

- Matrix spikes are used to monitor the performance of the analytical methods on the matrix and to assess the presence of interferences.

- A matrix spike shall be performed for each waste type (waste water, soil, etc.) unless the result exceeds the regulatory level and the data are being used solely to indicate that the regulatory level is exceeded.

- A minimum of one sample from each "analytical batch" must be spiked. For spike samples, a double or triple volume of TCLP leachate will be required. The term analytical batch is not defined in the regulation. Most methods, such as CLP and SW846, indicate that a batch is no more than 20 samples of the same matrix

processed through preparation and analysis simultaneously. Based on this criteria, the minimal matrix spike frequency of analysis is one per 20 samples. However, many process batches may include from one to 19 samples and the frequency may increase with fewer samples processed. Some EPA Regions define each type of waste as a matrix, and require matrix spikes for each matrix.

- Matrix spikes (MS) are to be added after leaching and filtration but prior to preservation. Spikes are NOT to be added prior to the TCLP leaching.

- The spike should be added to the same nominal volume of TCLP extract as the unspiked sample.

- The spike concentration "should" be added at the regulatory level. If the expected concentration in the sample is as low as half the regulatory level, the spike concentration can be decreased to half the regulatory level. In all cases, the spike must be greater than 5 times the method detection limits.

- Matrix Spike Recoveries are calculated by:

  %Recovery = 100 (Measured value for the spiked sample minus measured value of the unspiked sample)/ known value of the spike

- When the matrix spike recovery falls below the expected analytical performance, alternate methods of analysis may be required to measure analyte concentration in the TCLP extract. The matrix spike recovery limits from the Contract Laboratory Protocol methods are used when the method is used.

  If the matrix spike recoveries exceed limits, other analytical methods such as isotopic dilution may be used to deal with the matrix effects. Typically, the holding times will be exceeded or near the limits when this occurs. If possible, resampling of the waste may be required to assure that the appropriate method is used and holding times are met.

- *Bias correction is no longer required.*

Surrogate Spikes

Surrogates are compounds which are not expected to be in the samples but are chemically similar to those being determined. The concentrations and specific compounds are listed in the appropriate methods. The recovery of the compounds are monitored with specific criteria either being found in the method or determined by statistical quality control in the laboratory.

Method of Standard Addition

Four equal volume **pre-digestion** aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third

should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked standard should be the same.

In order to determine the concentration of analyte in the sample, the analytical value of each solution is determined and a plot or linear regression performed. On the vertical axis the analytical value is plotted versus the concentrations of the standards on the horizontal axis. An example plot is shown in Figure 4-5. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown.

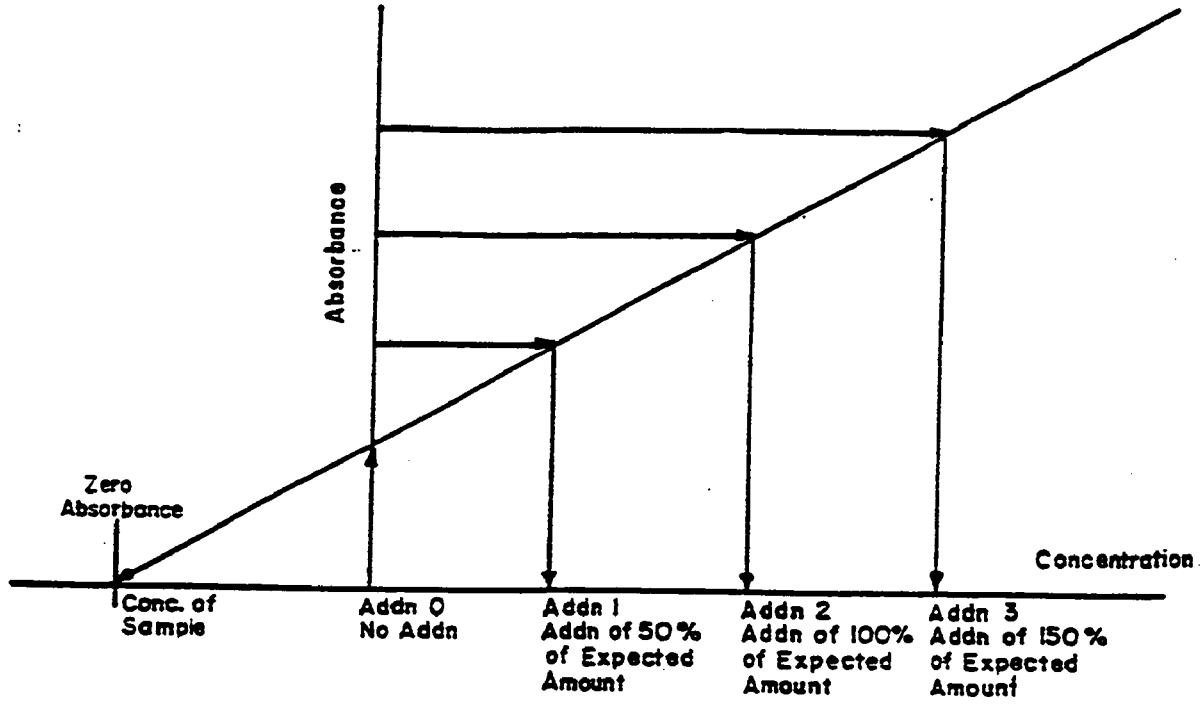


FIGURE4-5 STANDARD ADDITION PLOT

When must Standard Addition be used?

The method of standard additions is used for metallic contaminant determinations if both of the following criteria are met:

1. The matrix spike recovery from the TCLP extract is less than 50% and the unspiked sample concentration is less than the regulatory level.

2. The contaminant measured in the sample is within 20% of the regulatory level.

For the method of standard additions to be correctly applied, the following limitations must be taken into consideration:

- The plot of sample and standards must be linear over the concentration range of concern. For best results, the slope of the line should be similar to that of a plot of the aqueous standard.

- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

Holding Times

As previously discussed in Section 3, the holding times must be met. Sample data which exceed holding times are not acceptable for verifying that a waste does not exceed regulatory levels. However, if TCLP extract concentrations exceed regulatory action levels, and holding times are exceeded, the data are considered minimum values, and the data are considered valid.

Chapter 5

DATA VALIDATION AND DATA DELIVERABLES

5.0 DATA VALIDATION AND DELIVERABLES

This chapter addresses the following questions:

- What is data validation?
- When must TCLP data be validated in EPA Region 2?
- Which analytical deliverables are needed to validate TCLP data when utilizing the USEPA Region 2 Organic, Inorganic, and TCLP Data Validation Protocols?
- Which analytical deliverables are recommended for TCLP data which will not be validated?
- How should these deliverables be utilized to assess data quality and usability?

5.1 Data Validation

What is Data Validation?

"Data validation is a systematic process for reviewing a body of data against a set of criteria to provide assurance that the data are adequate for their intended use. Data validation consists of data editing, screening, checking, auditing, verifying, certifying and reviewing." (EPA Region 2 CERCLA QA Manual)

The most important criteria which the data reviewer evaluates are:

1. Holding times
2. Instrument tuning
3. Calibration and retention time windows
4. Blank contaminants
5. Surrogates (a measure of extraction efficiency)
6. Chromatographic performance (baseline, interference, retention time shift and peak resolution)
7. Emission interferences or spectral interference from other elements when reviewing metals data
8. Calculations
9. Transcription of numerical values to the required forms in the data package
10. Matrix effect errors; interference from the sample itself
11. Degradation of compounds during analysis

There is a substantial amount of uncertainty in all chemical data. In addition to lab error, there are field sampling errors, such as improper decontamination of field equipment, air bubbles in VOA vials, loss of samples, and failure to ship samples in a timely manner after collection. Different analytes have varying degrees of uncertainty.

TCLP data are expected to have significantly more inherent error than routine chemical analysis because additional procedures are performed by the laboratory analyst.

What is data qualification?

Qualifying data is a method of notifying the data user that some data have additional uncertainty. The Region 2 TCLP data validation protocol qualifies analytical data with the following flags:

- R Rejected (unusable)
- J Estimated
- UJ Estimated detection limit
- N Presumptively present (cannot positively identify an analyte)
- JN Presumptively present at an estimated concentration

The above qualification "flags" provide QC information to the data user. The Region 2 TCLP data validation protocol qualifies analytical data as unusable, estimated, presumptively present, or presumptively present at an estimated concentration.

Unusable data are rejected and qualified with an "R". When data are rejected, it doesn't mean that the analyte wasn't there - it means that either the test was not correctly performed or that the test was not appropriate for the matrix. Examples of reasons for rejecting data include: poor calibration, low surrogate recoveries, and air bubbles in volatile sample vials. If the data are needed, resampling and reanalysis must be performed. For example, the holding time for TCLP VOAs is 14 days from sampling until TCLP leaching, and then 14 days until analysis. If a sample is held for 30 days from collection until leaching, all non-detects and positive results below regulatory action levels will be rejected because analytes could have been present above regulatory action levels. Results above the regulatory action levels would be accepted. However, the site owner may still want resampling and reanalysis to assure that a false positive did not occur.

When data are qualified as estimated with a "J", it means that the data should be used with caution. The data are significantly imprecise, and the reported value given is little more than an estimate. Estimated means that the compound is present, but the exact concentration is uncertain.

When data are qualified with a "UJ", it means that the detection levels are uncertain. For example, this qualifier would be used when surrogate recoveries in organics are greater than 10% but not within the method criteria. The "UJ" notifies the data user that the detection limits are estimated.

When the analyte identity is uncertain, the qualifier "N" is used to indicate that it is presumptively present. This is used in data validation when a mass spectrum differs slightly from the required spectral criteria. Data validators use the qualifier "JN", presumptively present at an estimated concentration, much more often than the qualifier "N". The"JN" flag denotes both qualitative and quantitative uncertainty. This is typically used when tentatively identified compounds (TICs) from semivolatile gas chromatography/mass spectrometry analysis are presented. The concentration and identities of the TICs are uncertain and are flagged with "JN".

When Must TCLP Data Be Validated?

EPA Region 2 requires TCLP data for RCRA RFIs and many types of CERCLA sampling events to be validated. The RCRA program does not explicitly require the validation of routine TCLP analysis of waste materials to determine compliance with TC or LDR regulations.

Data validation reduces false negatives, false positives, and misquantitation in reported data. Misquantitation includes both laboratory arithmetic errors, and data qualified as estimated or presumptively present because of analytical problems. The costs for TCLP validation are quite variable, and depend specifically on which tasks the data user instructs the data validator to perform, and the quality of the laboratory analyzing the environmental samples. The cost of validating a single sample containing the 39 TC analytes is about $300-$500 per sample analyzed by a competent laboratory. In addition to the data validation cost, the laboratory will charge an additional fee, estimated at $200-$400 per sample, for generating analytical deliverables. The more a data user knows about a specific waste, the less useful data validation becomes. For example, if the data user knows which raw materials, final products, and by-products are in a waste, and has historical data that demonstrates that the TC analytes in the TCLP extract are far below regulatory action levels, TCLP validation would not be cost effective. Alternatively, when the data user has very limited knowledge of a waste's characteristics, decisions based on that data can result in significant disposal cost for management. Therefore, many businesses believe that it is prudent and cost effective to validate this type of TCLP data.

Some regulatory agencies, especially in the CERCLA program, do not allow laboratories to validate their own data. All laboratories review their own data for contractual compliance and analytical problems. Unfortunately, this assessment of contractual compliance may also be called data validation. Many laboratories now call their contractual compliance review "data review" to differentiate this review from data validation.

Contractual compliance is NOT the same as data validation. A lab can contractually fail and still produce technically valid data. An example of this occurrence is when contractual requirements for metals data indicated that results would be delivered to the client 40 days from receipt of the sample. If the laboratory did not deliver for 60 days, the laboratory failed the contract criteria, but the technical criteria were still met. Alternatively, a laboratory can contractually meet criteria but produce data which is not useable.

In order to validate TCLP data, the following must be ascertained:

- Are any specific data validation protocols required by a regulatory agency?
- What are the regulatory action levels?

The TC regulatory action levels are listed in Table 1. The Land Disposal Restrictions regulatory action levels are listed in Appendix II.

The EPA Region 2 TCLP, Inorganic and Organic Validation protocols are included in Appendix IV. If your region or state does not have a TCLP validation procedure, the Region 2 data validation protocol may be used. If the applicable sampling and analysis plan requires

regulatory approval, the data user, lab and regulator must agree on validation criteria prior to sample collection.

Phenols

When validating TCLP phenols, the TCLP extraction fluid may cause a matrix effect. This matrix effect may lower surrogate and matrix spike recoveries for phenols. As long as the matrix spike and surrogate recoveries are above 10%, the data should not be rejected. If a matrix effect precludes acceptable pyridene or phenols surrogate and matrix spike recoveries, a facility should request a meeting with the appropriate regulatory agency to discuss alternate methods for determining whether a solid waste exhibits TC.

After determining whether the project will require validation, the appropriate deliverables must be specified to allow the validation to occur. For example, if the validation requires calibration verification, raw instrument calibration data must be present for the validation to be performed.

5.2 Data Deliverables

General TCLP Data Deliverables

In order to validate and assess TCLP data, appropriate deliverables must be specified. Deliverables will differ depending on whether validation is required. These analytical deliverables must be specified before samples are collected. The following topics are discussed:

- Deliverables when no validation is required
- Additional deliverables which may assist in review
- USEPA Region 2 analytical deliverables
- Specifying data deliverables

Deliverables When No Validation is Required

When TCLP data are not validated, we recommend that the laboratory furnish the data user the following deliverables:

1. Sample description and sample identification numbers.
2. Analytes, concentrations, and units.
3. Level of contaminant(s) in method and TCLP blanks.
4. Matrix spike, QC check sample when applicable, and surrogate recoveries.
5. A description of matrix problems and analytical problems observed during analysis, and an assessment of how those problems will affect data usability.
6. A certification that samples were analyzed within method holding times (from the date of sample collection). This certification must include the sampling date, TCLP extraction dates, preparatory extraction dates, and analysis dates.

The laboratory staff are not always familiar with data validation protocols or with data usability on a project specific level. Therefore, in addition to information about the QC supplied by the laboratory, it may be beneficial to work with a specialist in this area. Some firms specialize in validating and assessing data quality.

Additional Deliverables Which May Assist in Review

While the data is the key factor, some information is not always captured in the analytical result. For example, if a procedure is modified due to a matrix problem, the procedure must be documented and the validator provided this information. The following information may facilitate validation.

- Chain of Custody records
- Analytical procedures used by the laboratory
- An example of a data calculation

The data user must specify the information (analytical deliverables) desired from the laboratory. If this is not done, only the final result, or the final result plus a QC summary, will be provided.

Region 2 Analytical Deliverables

The following TCLP analytical deliverables are required by the Region 2 TCLP data validation protocol:

1. The TCLP and preparative extraction dates and analysis dates.
2. Selection of extraction fluid data.
3. A physical description of the samples.
4. The sample weights and the extraction fluids weights.
5. The final volume of TCLP extract and the volume of extract analyzed.
6. The data used to compute percent dry solids and the weight of the liquid phase (if applicable).
7. Extraction logs for each sample, indicating the volume and pH of acid added. Were inorganic sample extracts properly preserved?
8. A description of the materials of construction for extraction vessels, filtration devices, and ZHE extraction devices (i.e. glass, Teflon, PVC, stainless steel, etc.).
9. The data used to compute TCLP extract concentrations for multi-phasic samples.
10. When VOA samples consist of oily waste that cannot be filtered, describe how the TCLP aqueous extract is separate from the oily waste.
11. A copy of the sampling log or trip report.*
12. Any evidence of leakage in the ZHE device.

*Item 11, which requires the presentation of the sampling log, may not be available from the laboratory. The sampling team may supply this information.

In order to facilitate analysis and validation, Associated Design and Manufacturing Corporation and Dr. Larry Jackson have developed work sheets (Appendix III) which may be used by the analytical laboratory to generate the above listed analytical deliverables. Additional descriptions of requirements and deliverables for modification of CLP analysis of leachate is presented in Appendix VIII.

Specifying Data Deliverables

EPA Region 2 *requires* TCLP data to be validated for RCRA RFIs and many types of CERCLA sampling events.

Specific regulations, as noted in Appendix IX, require SW-846. Many state environmental agencies require the use of SW-846 methods for hazardous waste determinations. SW-846 methods cannot be validated by CLP validation criteria because SW-846 methods do not specify analytical deliverables, and have different QC criteria than CLP methods. Therefore, validation protocols must be prepared for non-CLP methods such as those in SW-846.

For non-CLP methods, data validation criteria must include:

- Holding times for sample preparation and analysis
- Preparation logs
- Calibration
- Method and instrument blank data review
- Calculations
- Matrix spike data
- Duplicate results

For organic analysis, the following additional items must be included:

- Instrument tuning if GC/MS is used
- Surrogate recoveries
- Chromatographic performance (baseline, interference, shift and peak resolution)
- Mass spectral interpretation or compound identification

For metals analysis, the following additional items must be included:

- Whether method of standard addition or serial dilution were needed and performed correctly. The November 24, 1992 modification to the TCLP procedure mandates the use of method of standard additions under certain circumstances.

- Post digestion spike recoveries versus pre-digestion spike recoveries.

- The frequency of analysis of QC samples must be validated.

Chapter 6

Chapter 6

ANALYZING AND ASSESSING MULTI-PHASIC AND OILY WASTES

Definition of Oily Waste

Problems/Issues

Suggestions

Most Commonly Asked TCLP Question

Analytical Options

6.0 ANALYZING AND ASSESSING MULTI-PHASIC AND OILY WASTES

Analyzing and Assessing Multi-Phasic and Oily Wastes

This chapter provides strategies which may be beneficial in characterizing oily wastes. There is no single correct method to analyze these wastes.

- Definition of oily waste
- Problems/Issues
- Suggestions
- Most commonly asked TCLP question
- Analytical options

This chapter outlines the current issues and difficulties in performing TCLP on multiple phase and oily waste. This chapter is not meant to provide unequivocal answers, but to provide suggestions and strategies which may be successful. There is no single correct method in dealing with these materials. The initial discussion in this chapter provides references and information indicating that EPA understands the difficulties in applying the TCLP to multi-phasic and oily wastes. Subsequent discussions summarize possible strategies which may be used in leaching and analysis.

6.1 Definition of Oily Waste

Appendix VI contains several papers presented at the EPA's 1992 Waste Testing & Quality Assurance Symposium Work Shop on oily waste. The following is Clifford Narquis's of BP Research, definition of oily waste (see Appendix VII):

Although it is nearly impossible to precisely define the term "oily waste", the following analysis can provide a basis for further discussion:

a) An oil is generally an immiscible or relatively insoluble liquid, varying in composition but consisting of organic constituents. Petroleum oil principally consist of hydrocarbons; vegetable and animal oils are glycerides, and fatty acids; and essential oils are terpenes, alkaloids, etc.

b) An oily waste is an industrial process waste or residual bearing oil in a visual and/or measurable proportions.

c) Oil in oily wastes can occur in any matrix, including: sorbed to dry solids; in sludges or slurries; multi-phasic liquids or sludges/slurries with multi-phasic liquids, if water is present. Proper treatment and disposal of all such matrices is a concern of the petroleum industry.

d) Oily wastes possess a wide variety of compositions and physical and toxicological properties.

6.2 Problems/Issues

Problems with the TC Model

- The model does not differentiate between oily and aqueous liquids.
- The model assumes a person drinks 2 liters a day of well water for 70 years. This assumption is not applicable for oily wastes.
- The disposal scenario depicted by EPA is not an accurate description of today's practices. For example, liquids are no longer accepted in municipal landfills.
- The model does not correct for absorption of oily waste on soils. Oil may also adhere to other landfill matrices instead of mixing with the aqueous phases.

The difficulty in analyzing oily wastes by TCLP may be categorized as modeling, analytical and regulatory problems.

The Leachability Subcommittee of the EPA Science Advisory Board's Environmental Engineering Committee has published its recommendations. A copy of this report is in Appendix VII of this document. This document outlines the properties of an optimum leach test.

Issues with Oily and Multi-phasic Waste and TCLP

- It is difficult to separate the phases.
- Volatiles may evaporate during handling.
- The tumbling action of the two liter extraction vessel can form emulsions which are difficult to separate.
- The oily material may obstruct the filter. When this happens with the ZHE, the test must be repeated.
- Oily materials often yield oil and aqueous leachate which must be analyzed separately. This increases costs and time of analysis.
- The method requires determination of dry weight percent solids. When the "solid" is actually oil or organic, drying can be hazardous and inappropriate. It may be impossible to achieve a constant weight when performing a percent solids determination.

- When multiple phases and multiple bottles are used in sampling, each container will show different amounts of each phase.

- It may be impossible to separate solids from oil. If volatiles are analyzed, additional sample manipulation to remove solids will result in loss of volatiles.

6.3 Suggestions

- Suggestions for performing TCLP extractions on oily wastes include the following:
  - Planning
  - Regulatory approval
  - Separate phases for analysis
  - Documentation of phase type and volumes

As previously indicated, these are suggestions. There are no consistently and absolutely appropriate methods when performing TCLP extractions on oily wastes.

- Planning is more critical when oily or multi-phasic samples are collected and analyzed. Discuss the sample matrix with the laboratory before collecting samples.
- Once an approach is formulated, regulatory approval may be needed. This approval is of greater importance if deviations from the TCLP extraction method are required due to the matrix.
- If two liquid phases are present, each phase should be separated and analyzed individually.
- The SW-846 Methods specify several procedures for analyzing oily waste. BNA methods include 3580B for preparation followed by 8270B. The pesticides method includes 3580B using hexane as the extraction solvent followed by 8080B. The VOAs are analyzed by 8240B. Metals can be prepared by method 3040B and analyzed by appropriate analytical methods.
- The number, appearance, and volume of each phase should be documented before collection of the sample. The phase volume can be estimated by measuring the height of the phase in the container and the diameter of the container. This information can be used to estimate the amounts of material available for testing.
- Phase volume should be estimated after sample collection and prior to analysis.
- In multi-phasic liquid samples, the relative density of each phase should be documented.

- When multiple containers of multi-phasic waste are received, each container will have different amounts of each phase. If multiple sample containers are collected and each container is multi-phasic, the number, appearance, volume and relative density should be documented for each container.

- If regional and state regulators will allow, one container can be mixed and analyzed. By knowing the volumes in the other sample containers, the total composition can be mathematically calculated.

- If one phase is organic and contains < 0.5% solids, this may be directly characterized by the appropriate analytical method after filtration without TCLP extraction.

- Subsampling increases the possibility of sampling error.

- The percent solids should be determined in multi-phasic samples before filling the ZHE. This prevents overfilling the ZHE.

- The TCLP method requires drying the solids at 100°C $\pm$ 20°C to determine percent dry solids. This may not be achievable for organic multi-phasic material because of safety considerations. If this cannot be done, the reason should be documented. The percent wet solids is used to calculate the weight of extraction fluid. If this occurs, the lab should discuss this with the client prior to using the percent wet solids as the solids content. This will greatly effect the final analyte concentrations.

- Particle size reduction is difficult on oily material because the solids congeal. This is especially true if the material cannot be dried.

- Extreme caution should be taken when adding acid to organic waste. Heating the organic waste in the presence of acid to 50°C should be done with great caution. This may be required in order to determine which extraction fluid is used.

6.4 Most Commonly Asked TCLP Question

I have an oily waste, which flows through a filter. My detection limits are higher than the regulatory action levels. What should I do?

You have four options:

1. Recycle or burn.

2. Classify by prior knowledge as non-hazardous.

3. Treat the waste as hazardous.

4. If the oil passes through the filter, analyze the TCLP leachate.

1. If the oily waste can be classified as used oil, it can be burned or recycled and a TCLP analysis is not needed (40 CFR 266.40; 261.6(a)).

2. The waste can be treated as hazardous if no information is available to allow classification by prior knowledge.

3. By knowledge of the generation of the oily waste, the generator may be able to certify that the waste could not contain any of the TC analytes at concentrations above the regulatory action levels. (40 CFR 262.11c(2)). The waste may be a regulated hazardous waste under other EPA waste code classifications.

4. The liquid which passes through the filter can be analyzed to determine whether it contains TC analytes. If the SW 846 methods are not appropriate for TC analytes, any method which is sensitive enough to meet regulatory limits and has documented QC may be used.

The following pages includes correspondence on oily waste explaining EPA's strategy for classifying oily wastes as hazardous or non-hazardous.

OhioEPA
State of Ohio Environmental Protection Agency

P.O. Box 1049, 1800 WaterMark Dr
Columbus, Ohio 43266-0149
(614) 644-3020 Fax (614) 644-2329

Richard F Celeste
Governor

Ocrober 30, 1990

Gail Hansen
U.S. Environmental Protection Agency
Office of Solid Waste
Methods Section
Washington, D.C. 20460

Dear Ms. Hansen:

I receive many inquiries on SW-846 detection limits. One caller stated that he had samples analyzed under SW-846 protocol which totaled over $75,000, only to find that many of the constituents had detections limits above regulated values. Another caller had industrial waste (baghouse residue) tested under TCLP and noted that the detection limits of the constituents were all below regulated levels except for chlordane which in eight out of nine samples was 0.045 mg/L, versus the regulated value of 0.03 mg/L. I need suggestions on the appropriate response to these inquiries, specifically:

(1) Assuming a given laboratory has followed proper protocol, If detection limits of constituents in a waste sample are in excess of but close to regulated values, is the sample considered hazardous?

(2) Using the chlordane situation (above) as an example, what analytical procedures can a laboratory use, for example clean-up and dilution, outside of procedures specified under a given method (eg. TCLP), which are permissible by the U.S. EPA? Can Method 8250 (semi-volatiles), for example, be used to confirm or as a substitute for TCLP in analyzing chlordane?

(3) Is there an upcoming FR updating and clarifying analytical problems in the TCLP analytical section?

Your help will be appreciated in resolving the concerns outlined in this communication. If you need additional information, I may be contacted at (614) 644-2956.

Sincerely,

Arthur L. Coleman, Jr.

Art Coleman
Technical Assistance Section
Division of Solid and Hazardous Waste Management

ALC/pas

cc: Karl Bremer, USEPA, Region V
Steve McBride, DERR
Dr. Gary Davidson, Chief, Public Health Laboratories, ODH
David E. Vanderberg, Regional Manager, Kemron Environmental Services
Gerry G. Ioannides, Chief, Environmental Services, Ohio EPA

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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

NOV 09 1990

Toxic & Haz. Waste

NOV 8 1990

OFFICE OF
SOLID WASTE AND EMERGENCY RESPONSE

Art Coleman
Technical Assistance Section
Division of Solid and Hazardous Waste Management
Ohio EPA
P.O. Box 1049
1800 WaterMark Dr.
Columbus, OH 43266-0149

Dear Mr. Coleman:

I am writing in reponse to your letter of October 30,1990 concerning the questions you raised with Method 1311 (TCLP).

In answer to your first question, there are situations when a laboratory is asked to perform an inappropriate test. The TCLP was not intended to be applied to certain matrices, such as oils or neat solvents. In these instances, the waste usually goes through the filter and is, by definition, a liquid and its own extract. The analysis of this liquid extract for organics entails diluting it before injecting it into a GC or GC/MS. The dilution often results in detection limits being much higher than the regulatory thresholds. If this is the case, you **must assume your waste is hazardous** since the laboratory cannot demonstrate non-hazardousness with TCLP for these materials. We currently do not have the technology to address this issue.

In answer to your second question, a laboratory **must** use the TCLP if testing for hazardousness under the Toxicity Characteristic or if assessing effectiveness of waste treatment under the Land Disposal Restrictions Program. These two regulations actually contain the method as an appendix and it is, therefore, part of the law. However, the extract obtained from the TCLP may be analyzed by **any** method as long as that method has documented QC and the method is sensitive enough to meet the regulatory limit. In other words, the lab does not have to use SW-846 methods because these methods are intended to serve only as a guidance for the regulated community. SW-846 methods that are currently in draft form (e.g., 8250 for chlordane) may also be used to analyze the extract.

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In answer to your third question, there are no plans to prepare a clarifying FR update in the near future.

I hope these answers have sufficiently addressed your concerns. If you have any further questions, please give me a call at (202) 475-6722 or write me again at the above address.

Sincerely yours,

Gail Hansen

Gail Hansen
Health Scientist
Methods Section
(OS-331)

cc: Alec McBride
Jeanne Hankins
Hugh Davis, OWPE
Leon Lazarus, Region II

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OFFICE OF
SOLID WASTE AND EMERGENCY RESPONSE

RECEIVED

JUN 20 1991

Toxic & Haz Waste

Ms. Elaine McPherson
Technical Sales Representative
IT Corporation
17605 Fabrica Way
Cerritos, CA 90701

Dear Ms. McPherson:

I am writing in reference to your letter of April 11, 1991 concerning the handling of TCLP extractions as they apply to oily wastes.

We do not recommend performing the extract on the oily waste that passes through the filter as Margo Jackisch of SAIC suggested to you. First of all, the TCLP determines release potential in two steps, the first of which I will discuss here as it specifically applies to your situation. The initial filtration step separates the solid phase of a waste from its liquid phase. This liquid phase represents the primary waste leachate or the liquid fraction of a waste that is mobile and can be released from a landfill. In your case, the oil goes through the filter and, by definition, becomes its own leachate which is then analyzed directly.

If your waste is a used oil that is destined for recycling, there is no need to characterize the waste since it would be exempt under 40 CFR Section 261.6(a)(2)(iii) and (a)(3)(iii). It is the decision to dispose of the waste, in lieu of recycling, that triggers the waste characterization requirement. If your waste is a used oil that cannot be recycled and is destined for disposal, generators are required to make a hazard determination. If the generator chooses to test for the Toxicity Characteristic, the generator must use the TCLP or an approved alternative method, as described in 40 CFR 261.24. The extract obtained from the TCLP may be analyzed by any method, provided the method used has documented QC and is sensitive enough to meet the regulatory threshold for the constituents of concern.

In cases where the TCLP results on used oil or oily wastes are inconclusive, including cases where the detection limit for a constituent is higher than the regulatory threshold, generators may use their knowledge of the processes involved in the

generation of the waste to make a hazard determination or resort to an alternative analytical method to get an answer. This has been necessary with volatile organics. At this time, the Agency is conducting studies of an automated headspace analysis methodology coupled with isotope dilution mass spectrometry in order to achieve greater analytical sensitivity for all TC volatile analytes, including vinyl chloride. We suggest the use of this approach where needed. Currently, only a working draft method (copy enclosed) is available. Pending the outcome of Agency studies, the draft method will be revised and proposed for inclusion in SW-846.

For further assistance, please call the MICE (Methods Information Communications Exchange) at (703) 821-4789. Calls are recorded on an answering machine and, for the majority of questions, responses are provided within 24 hours. I hope this information has sufficiently addressed your questions.

Sincerely yours,

Gail Hansen

Gail Hansen
Environmental Health Scientist
Methods Section (OS-331)

cc: David Bussard
Alec McBride
Steve Cochran
Mike Petruska
John Austin
Leon Lazarus, Region II
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RCRA/Superfund Hotline
MICE Line

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OFFICE OF
SOLID WASTE AND EMERGENCY RESP

JUN 20 1991

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Appendix I

Appendix I
TCLP Methods From
40 CFR 261 Appendix II

SW 846 Method 1311
(Method Without Typographical Errors)

July 1992

METHOD 1311

TOXICITY CHARACTERISTIC LEACHING PROCEDURE

1.0 SCOPE AND APPLICATION

1.1 The TCLP is designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes.

1.2 If a total analysis of the waste demonstrates that individual analytes are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the TCLP need not be run.

1.3 If an analysis of any one of the liquid fractions of the TCLP extract indicates that a regulated compound is present at such high concentrations that, even after accounting for dilution from the other fractions of the extract, the concentration would be above the regulatory level for that compound, then the waste is hazardous and it is not necessary to analyze the remaining fractions of the extract.

1.4 If an analysis of extract obtained using a bottle extractor shows that the concentration of any regulated volatile analyte exceeds the regulatory level for that compound, then the waste is hazardous and extraction using the ZHE is not necessary. However, extract from a bottle extractor cannot be used to demonstrate that the concentration of volatile compounds is below the regulatory level.

2.0 SUMMARY OF METHOD

2.1 For liquid wastes (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 μm glass fiber filter, is defined as the TCLP extract.

2.2 For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. A special extractor vessel is used when testing for volatile analytes (see Table 1 for a list of volatile compounds). Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 μm glass fiber filter.

2.3 If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 INTERFERENCES

3.1 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

4.0 APPARATUS AND MATERIALS

4.1 Agitation apparatus: The agitation apparatus must be capable of rotating the extraction vessel in an end-over-end fashion (see Figure 1) at 30 ± 2 rpm. Suitable devices known to EPA are identified in Table 2.

4.2 Extraction Vessels

4.2.1 Zero-Headspace Extraction Vessel (ZHE). This device is for use only when the waste is being tested for the mobility of volatile analytes (i.e., those listed in Table 1). The ZHE (depicted in Figure 2) allows for liquid/solid separation within the device, and effectively precludes headspace. This type of vessel allows for initial liquid/solid separation, extraction, and final extract filtration without opening the vessel (see Section 4.3.1). The vessels shall have an internal volume of 500-600 mL, and be equipped to accommodate a 90-110 mm filter. The devices contain VITON®[1] O-rings which should be replaced frequently. Suitable ZHE devices known to EPA are identified in Table 3.

For the ZHE to be acceptable for use, the piston within the ZHE should be able to be moved with approximately 15 psi or less. If it takes more pressure to move the piston, the O-rings in the device should be replaced. If this does not solve the problem, the ZHE is unacceptable for TCLP analyses and the manufacturer should be contacted.

The ZHE should be checked for leaks after every extraction. If the device contains a built-in pressure gauge, pressurize the device to 50 psi, allow it to stand unattended for 1 hour, and recheck the pressure. If the device does not have a built-in pressure gauge, pressurize the device to 50 psi, submerge it in water, and check for the presence of air bubbles escaping from any of the fittings. If pressure is lost, check all fittings and inspect and replace O-rings, if necessary. Retest the device. If leakage problems cannot be solved, the manufacturer should be contacted.

Some ZHEs use gas pressure to actuate the ZHE piston, while others use mechanical pressure (see Table 3). Whereas the volatiles procedure (see Section 7.3) refers to pounds per square inch (psi), for the mechanically actuated piston, the pressure applied is measured in torque-inch-pounds. Refer to the manufacturer's instructions as to the proper conversion.

VITON® is a trademark of Du Pont.

4.2.2 Bottle Extraction Vessel. When the waste is being evaluated using the nonvolatile extraction, a jar with sufficient capacity to hold the sample and the extraction fluid is needed. Headspace is allowed in this vessel.

The extraction bottles may be constructed from various materials, depending on the analytes to be analyzed and the nature of the waste (see Section 4.3.3). It is recommended that borosilicate glass bottles be used instead of other types of glass, especially when inorganics are of concern. Plastic bottles, other than polytetrafluoroethylene, shall not be used if organics are to be investigated. Bottles are available from a number of laboratory suppliers. When this type of extraction vessel is used, the filtration device discussed in Section 4.3.2 is used for initial liquid/solid separation and final extract filtration.

4.3 Filtration Devices: It is recommended that all filtrations be performed in a hood.

4.3.1 Zero-Headspace Extractor Vessel (ZHE): When the waste is evaluated for volatiles, the zero-headspace extraction vessel described in Section 4.2.1 is used for filtration. The device shall be capable of supporting and keeping in place the glass fiber filter and be able to withstand the pressure needed to accomplish separation (50 psi).

NOTE: When it is suspected that the glass fiber filter has been ruptured, an in-line glass fiber filter may be used to filter the material within the ZHE.

4.3.2 Filter Holder: When the waste is evaluated for other than volatile analytes, any filter holder capable of supporting a glass fiber filter and able to withstand the pressure needed to accomplish separation may be used. Suitable filter holders range from simple vacuum units to relatively complex systems capable of exerting pressures of up to 50 psi or more. The type of filter holder used depends on the properties of the material to be filtered (see Section 4.3.3). These devices shall have a minimum internal volume of 300 mL and be equipped to accommodate a minimum filter size of 47 mm (filter holders having an internal capacity of 1.5 L or greater, and equipped to accommodate a 142 mm diameter filter, are recommended). Vacuum filtration can only be used for wastes with low solids content (<10%) and for highly granular, liquid-containing wastes. All other types of wastes should be filtered using positive pressure filtration. Suitable filter holders known to EPA are shown in Table 4.

4.3.3 Materials of Construction: Extraction vessels and filtration devices shall be made of inert materials which will not leach or absorb waste components. Glass, polytetrafluoroethylene (PTFE), or type 316 stainless steel equipment may be used when evaluating the mobility of both organic and inorganic components. Devices made of high density polyethylene (HDPE), polypropylene (PP), or polyvinyl chloride (PVC) may be used only when evaluating the mobility of metals. Borosilicate glass bottles are recommended for use over other types of glass bottles, especially when inorganics are analytes of concern.

4.4 Filters: Filters shall be made of borosilicate glass fiber, shall contain no binder materials, and shall have an effective pore size of 0.6 to 0.8 μm, or equivalent. Filters known to EPA which meet these specifications are identified in Table 5. Pre-filters must not be used. When evaluating the mobility of metals, filters shall be acid-washed prior to use by rinsing with 1N nitric acid followed by three consecutive rinses with deionized distilled water (a minimum of 1 L per rinse is recommended). Glass fiber filters are fragile and should be handled with care.

4.5 pH Meters: The meter should be accurate to $\pm$ 0.05 units at 25 °C.

4.6 ZHE Extract Collection Devices: TEDLAR®[2] bags or glass, stainless steel or PTFE gas-tight syringes are used to collect the initial liquid phase and the final extract of the waste when using the ZHE device. The devices listed are recommended for use under the following conditions:

4.6.1 If a waste contains an aqueous liquid phase or if a waste does not contain a significant amount of nonaqueous liquid (i.e., <1% of total waste), the TEDLAR® bag or a 600 mL syringe should be used to collect and combine the initial liquid and solid extract.

4.6.2 If a waste contains a significant amount of nonaqueous liquid in the initial liquid phase (i.e., >1% of total waste), the syringe or the TEDLAR® bag may be used for both the initial solid/liquid separation and the final extract filtration. However, analysts should use one or the other, not both.

4.6.3 If the waste contains no initial liquid phase (is 100% solid) or has no significant solid phase (is 100% liquid), either the TEDLAR® bag or the syringe may be used. If the syringe is used, discard the first 5 mL of liquid expressed from the device. The remaining aliquots are used for analysis.

4.7 ZHE Extraction Fluid Transfer Devices: Any device capable of transferring the extraction fluid into the ZHE without changing the nature of the extraction fluid is acceptable (e.g., a positive displacement or peristaltic pump, a gas tight syringe, pressure filtration unit (see Section 4.3.2), or other ZHE device).

4.8 Laboratory Balance: Any laboratory balance accurate to within $\pm$ 0.01 grams may be used (all weight measurements are to be within $\pm$ 0.1 grams).

4.9 Beaker or Erlenmeyer flask, glass, 500 mL.

4.10 Watchglass, appropriate diameter to cover beaker or Erlenmeyer flask.

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[2] TEDLAR® is a registered trademark of Du Pont.

4.11 Magnetic stirrer.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Reagent Water. Reagent water is defined as water in which an interferant is not observed at or above the method's detection limit of the analyte(s) of interest. For nonvolatile extractions, ASTM Type II water or equivalent meets the definition of reagent water. For volatile extractions, it is recommended that reagent water be generated by any of the following methods. Reagent water should be monitored periodically for impurities.

5.2.1 Reagent water for volatile extractions may be generated by passing tap water through a carbon filter bed containing about 500 grams of activated carbon (Calgon Corp., Filtrasorb-300 or equivalent).

5.2.2 A water purification system (Millipore Super-Q or equivalent) may also be used to generate reagent water for volatile extractions.

5.2.3 Reagent water for volatile extractions may also be prepared by boiling water for 15 minutes. Subsequently, while maintaining the water temperature at 90 $\pm$ 5 degrees C, bubble a contaminant-free inert gas (e.g. nitrogen) through the water for 1 hour. While still hot, transfer the water to a narrow mouth screw-cap bottle under zero-headspace and seal with a Teflon-lined septum and cap.

5.3 Hydrochloric acid (1N), HCl, made from ACS reagent grade.

5.4 Nitric acid (1N), HNO_3, made from ACS reagent grade.

5.5 Sodium hydroxide (1N), NaOH, made from ACS reagent grade.

5.6 Glacial acetic acid, CH_3CH_2OOH, ACS reagent grade.

5.7 Extraction fluid.

5.7.1 Extraction fluid # 1: Add 5.7 mL glacial CH_3CH_2OOH to 500 mL of reagent water (See Section 5.2), add 64.3 mL of 1N NaOH, and dilute to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 4.93 $\pm$ 0.05.

5.7.2 Extraction fluid # 2: Dilute 5.7 mL glacial CH_3CH_2OOH with reagent water (See Section 5.2) to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 2.88 $\pm$ 0.05.

NOTE: These extraction fluids should be monitored frequently for impurities. The pH should be checked prior to use to ensure that these fluids are made up accurately. If impurities are found or the pH is not within the above specifications, the fluid shall be discarded and fresh extraction fluid prepared.

5.8 Analytical standards shall be prepared according to the appropriate analytical method.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples shall be collected using an appropriate sampling plan.

6.2 The TCLP may place requirements on the minimal size of the field sample, depending upon the physical state or states of the waste and the analytes of concern. An aliquot is needed for preliminary evaluation of which extraction fluid is to be used for the nonvolatile analyte extraction procedure. Another aliquot may be needed to actually conduct the nonvolatile extraction (see Section 1.4 concerning the use of this extract for volatile organics). If volatile organics are of concern, another aliquot may be needed. Quality control measures may require additional aliquots. Further, it is always wise to collect more sample just in case something goes wrong with the initial attempt to conduct the test.

6.3 Preservatives shall not be added to samples before extraction.

6.4 Samples may be refrigerated unless refrigeration results in irreversible physical change to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.

6.5 When the waste is to be evaluated for volatile analytes, care shall be taken to minimize the loss of volatiles. Samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes (e.g., samples should be collected in Teflon-lined septum capped vials and stored at 4 °C. Samples should be opened only immediately prior to extraction).

6.6 TCLP extracts should be prepared for analysis and analyzed as soon as possible following extraction. Extracts or portions of extracts for metallic analyte determinations must be acidified with nitric acid to a pH < 2, unless precipitation occurs (see Section 7.2.14 if precipitation occurs). Extracts should be preserved for other analytes according to the guidance given in the individual analysis methods. Extracts or portions of extracts for organic analyte determinations shall not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent losses. See Section 8.0 (QA requirements) for acceptable sample and extract holding times.

7.0 PROCEDURE

7.1 Preliminary Evaluations

Perform preliminary TCLP evaluations on a minimum 100 gram aliquot of waste. This aliquot may not actually undergo TCLP extraction. These preliminary evaluations include: (1) determination of the percent solids (Section 7.1.1); (2) determination of whether the waste contains insignificant solids and is, therefore, its own extract after filtration (Section 7.1.2); (3) determination of whether the solid portion of the waste requires particle size reduction (Section 7.1.3); and (4) determination of which of the two extraction fluids are to be used for the nonvolatile TCLP extraction of the waste (Section 7.1.4).

7.1.1 Preliminary determination of percent solids: Percent solids is defined as that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure, as described below.

7.1.1.1 If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solids) proceed to Section 7.1.3.

7.1.1.2 If the sample is liquid or multiphasic, liquid/solid separation to make a preliminary determination of percent solids is required. This involves the filtration device described in Section 4.3.2 and is outlined in Sections 7.1.1.3 through 7.1.1.9.

7.1.1.3 Pre-weigh the filter and the container that will receive the filtrate.

7.1.1.4 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure.

7.1.1.5 Weigh out a subsample of the waste (100 gram minimum) and record the weight.

7.1.1.6 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Centrifugation is to be used only as an aid to filtration. If used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

7.1.1.7 Quantitatively transfer the waste sample to the filter holder (liquid and solid phases). Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4 °C reduces the amount of expressed liquid over what would be expressed at room temperature then allow the sample to warm up to room temperature in the device before filtering.

NOTE: If waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and

subtract it from the sample weight determined in Section 7.1.1.5 to determine the weight of the waste sample that will be filtered.

Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within any 2 minute period), stop the filtration.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.1.1.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying vacuum or pressure filtration, as outlined in Section 7.1.1.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

7.1.1.9 Determine the weight of the liquid phase by subtracting the weight of the filtrate container (see Section 7.1.1.3) from the total weight of the filtrate-filled container. Determine the weight of the solid phase of the waste sample by subtracting the weight of the liquid phase from the weight of the total waste sample, as determined in Section 7.1.1.5 or 7.1.1.7.

Record the weight of the liquid and solid phases. Calculate the percent solids as follows:

$$\text{Percent solids} = \frac{\text{Weight of solid (Section 7.1.1.9)}}{\text{Total weight of waste (Section 7.1.1.5 or 7.1.1.7)}} \times 100$$

7.1.2 If the percent solids determined in Section 7.1.1.9 is equal to or greater than 0.5%, then proceed either to Section 7.1.3 to determine whether the solid material requires particle size reduction or to Section 7.1.2.1 if it is noticed that a small amount of the filtrate is entrained in wetting of the filter. If the percent solids determined in Section 7.1.1.9 is less than 0.5%, then proceed to Section 7.2.9 if the

nonvolatile TCLP is to be performed and to Section 7.3 with a fresh portion of the waste if the volatile TCLP is to be performed.

7.1.2.1 Remove the solid phase and filter from the filtration apparatus.

7.1.2.2 Dry the filter and solid phase at 100 ± 20 °C until two successive weighing yield the same value within ± 1%. Record the final weight.

NOTE: Caution should be taken to ensure that the subject solid will not flash upon heating. It is recommended that the drying oven be vented to a hood or other appropriate device.

7.1.2.3 Calculate the percent dry solids as follows:

$$\text{Percent dry solids} = \frac{\text{(Wt. of dry waste + filter) - tared wt. of filter}}{\text{Initial wt. of waste (Section 7.1.1.5 or 7.1.1.7)}} \times 100$$

7.1.2.4 If the percent dry solids is less than 0.5%, then proceed to Section 7.2.9 if the nonvolatile TCLP is to be performed, and to Section 7.3 if the volatile TCLP is to be performed. If the percent dry solids is greater than or equal to 0.5%, and if the nonvolatile TCLP is to be performed, return to the beginning of this Section (7.1) and, with a fresh portion of waste, determine whether particle size reduction is necessary (Section 7.1.3) and determine the appropriate extraction fluid (Section 7.1.4). If only the volatile TCLP is to be performed, see the note in Section 7.1.4.

7.1.3 Determination of whether the waste requires particle size reduction (particle size is reduced during this step): Using the solid portion of the waste, evaluate the solid for particle size. Particle size reduction is required, unless the solid has a surface area per gram of material equal to or greater than 3.1 cm^2, or is smaller than 1 cm in its narrowest dimension (i.e., is capable of passing through a 9.5 mm (0.375 inch) standard sieve). If the surface area is smaller or the particle size larger than described above, prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described above. If the solids are prepared for organic volatiles extraction, special precautions must be taken (see Section 7.3.6).

NOTE: Surface area criteria are meant for filamentous (e.g., paper, cloth, and similar) waste materials. Actual measurement of surface area is not required, nor is it recommended. For materials that do not obviously meet the criteria, sample specific methods would need to be developed and employed to measure the surface area. Such methodology is currently not available.

7.1.4 Determination of appropriate extraction fluid: If the solid content of the waste is greater than or equal to 0.5% and if the sample will be extracted for nonvolatile constituents (Section 7.2), determine the appropriate fluid (Section 5.7) for the nonvolatiles extraction as follows:

NOTE: TCLP extraction for volatile constituents uses only extraction fluid #1 (Section 5.7.1). Therefore, if TCLP extraction for nonvolatiles is not required, proceed to Section 7.3.

7.1.4.1 Weigh out a small subsample of the solid phase of the waste, reduce the solid (if necessary) to a particle size of approximately 1 mm in diameter or less, and transfer 5.0 grams of the solid phase of the waste to a 500 mL beaker or Erlenmeyer flask.

7.1.4.2 Add 96.5 mL of reagent water to the beaker, cover with a watchglass, and stir vigorously for 5 minutes using a magnetic stirrer. Measure and record the pH. If the pH is <5.0, use extraction fluid #1. Proceed to Section 7.2.

7.1.4.3 If the pH from Section 7.1.4.2 is >5.0, add 3.5 mL 1N HCl, slurry briefly, cover with a watchglass, heat to 50 °C, and hold at 50 °C for 10 minutes.

7.1.4.4 Let the solution cool to room temperature and record the pH. If the pH is <5.0, use extraction fluid #1. If the pH is >5.0, use extraction fluid #2. Proceed to Section 7.2.

7.1.5 If the aliquot of the waste used for the preliminary evaluation (Sections 7.1.1 - 7.1.4) was determined to be 100% solid at Section 7.1.1.1, then it can be used for the Section 7.2 extraction (assuming at least 100 grams remain), and the Section 7.3 extraction (assuming at least 25 grams remain). If the aliquot was subjected to the procedure in Section 7.1.1.7, then another aliquot shall be used for the volatile extraction procedure in Section 7.3. The aliquot of the waste subjected to the procedure in Section 7.1.1.7 might be appropriate for use for the Section 7.2 extraction if an adequate amount of solid (as determined by Section 7.1.1.9) was obtained. The amount of solid necessary is dependent upon whether a sufficient amount of extract will be produced to support the analyses. If an adequate amount of solid remains, proceed to Section 7.2.10 of the nonvolatile TCLP extraction.

7.2 Procedure When Volatiles are not Involved

A minimum sample size of 100 grams (solid and liquid phases) is recommended. In some cases, a larger sample size may be appropriate, depending on the solids content of the waste sample (percent solids, See Section 7.1.1), whether the initial liquid phase of the waste will be miscible with the aqueous extract of the solid, and whether inorganics, semivolatile organics, pesticides, and herbicides are all analytes of concern. Enough solids should be generated for extraction such that the volume of TCLP extract will be sufficient to support all

of the analyses required. If the amount of extract generated by a single TCLP extraction will not be sufficient to perform all of the analyses, more than one extraction may be performed and the extracts from each combined and aliquoted for analysis.

7.2.1 If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solid, see Section 7.1.1), weigh out a subsample of the waste (100 gram minimum) and proceed to Section 7.2.9.

7.2.2 If the sample is liquid or multiphasic, liquid/solid separation is required. This involves the filtration device described in Section 4.3.2 and is outlined in Sections 7.2.3 to 7.2.8.

7.2.3 Pre-weigh the container that will receive the filtrate.

7.2.4 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure. Acid wash the filter if evaluating the mobility of metals (see Section 4.4).

NOTE: Acid washed filters may be used for all nonvolatile extractions even when metals are not of concern.

7.2.5 Weigh out a subsample of the waste (100 gram minimum) and record the weight. If the waste contains <0.5% dry solids (Section 7.1.2), the liquid portion of the waste, after filtration, is defined as the TCLP extract. Therefore, enough of the sample should be filtered so that the amount of filtered liquid will support all of the analyses required of the TCLP extract. For wastes containing >0.5% dry solids (Sections 7.1.1 or 7.1.2), use the percent solids information obtained in Section 7.1.1 to determine the optimum sample size (100 gram minimum) for filtration. Enough solids should be generated by filtration to support the analyses to be performed on the TCLP extract.

7.2.6 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Use centrifugation only as an aid to filtration. If the waste is centrifuged, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

7.2.7 Quantitatively transfer the waste sample (liquid and solid phases) to the filter holder (see Section 4.3.2). Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4 °C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering.

NOTE: If waste material (>1% of the original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and

subtract it from the sample weight determined in Section 7.2.5, to determine the weight of the waste sample that will be filtered.

Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when the liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within a 2 minute period), stop the filtration.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.2.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase. Weigh the filtrate. The liquid phase may now be either analyzed (See Section 7.2.12) or stored at 4 °C until time of analysis.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying vacuum or pressure filtration, as outlined in Section 7.2.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the extraction as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

7.2.9 If the waste contains <0.5% dry solids (see Section 7.1.2), proceed to Section 7.2.13. If the waste contains >0.5% dry solids (see Section 7.1.1 or 7.1.2), and if particle size reduction of the solid was needed in Section 7.1.3, proceed to Section 7.2.10. If the waste as received passes a 9.5 mm sieve, quantitatively transfer the solid material into the extractor bottle along with the filter used to separate the initial liquid from the solid phase, and proceed to Section 7.2.11.

7.2.10 Prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described in Section 7.1.3. When the surface area or particle size has been appropriately altered, quantitatively transfer the solid material into an extractor bottle. Include the filter used to separate the initial liquid from the solid phase.

NOTE: Sieving of the waste is not normally required. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended. If sieving is necessary, a Teflon coated sieve should be used to avoid contamination of the sample.

7.2.11 Determine the amount of extraction fluid to add to the extractor vessel as follows:

$$\text{Weight of extraction fluid} = \frac{20 \times \text{percent solids (Section 7.1.1)} \times \text{weight of waste filtered (Section 7.2.5 or 7.2.7)}}{100}$$

Slowly add this amount of appropriate extraction fluid (see Section 7.1.4) to the extractor vessel. Close the extractor bottle tightly (it is recommended that Teflon tape be used to ensure a tight seal), secure in rotary agitation device, and rotate at 30 ± 2 rpm for 18 ± 2 hours. Ambient temperature (i.e., temperature of room in which extraction takes place) shall be maintained at 23 ± 2 °C during the extraction period.

NOTE: As agitation continues, pressure may build up within the extractor bottle for some types of wastes (e.g., limed or calcium carbonate containing waste may evolve gases such as carbon dioxide). To relieve excess pressure, the extractor bottle may be periodically opened (e.g., after 15 minutes, 30 minutes, and 1 hour) and vented into a hood.

7.2.12 Following the 18 ± 2 hour extraction, separate the material in the extractor vessel into its component liquid and solid phases by filtering through a new glass fiber filter, as outlined in Section 7.2.7. For final filtration of the TCLP extract, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filter(s) shall be acid-washed (see Section 4.4) if evaluating the mobility of metals.

7.2.13 Prepare the TCLP extract as follows:

7.2.13.1 If the waste contained no initial liquid phase, the filtered liquid material obtained from Section 7.2.12 is defined as the TCLP extract. Proceed to Section 7.2.14.

7.2.13.2 If compatible (e.g., multiple phases will not result on combination), combine the filtered liquid resulting from Section 7.2.12 with the initial liquid phase of the waste obtained in Section 7.2.7. This combined liquid is defined as the TCLP extract. Proceed to Section 7.2.14.

7.2.13.3 If the initial liquid phase of the waste, as obtained from Section 7.2.7, is not or may not be compatible with the filtered liquid resulting from Section 7.2.12, do not combine these liquids. Analyze these liquids, collectively defined as the TCLP extract, and combine the results mathematically, as described in Section 7.2.14.

7.2.14 Following collection of the TCLP extract, the pH of the extract should be recorded. Immediately aliquot and preserve the extract for analysis. Metals aliquots must be acidified with nitric acid to

pH <2. If precipitation is observed upon addition of nitric acid to a small aliquot of the extract, then the remaining portion of the extract for metals analyses shall not be acidified and the extract shall be analyzed as soon as possible. All other aliquots must be stored under refrigeration (4 °C) until analyzed. The TCLP extract shall be prepared and analyzed according to appropriate analytical methods. TCLP extracts to be analyzed for metals shall be acid digested except in those instances where digestion causes loss of metallic analytes. If an analysis of the undigested extract shows that the concentration of any regulated metallic analyte exceeds the regulatory level, then the waste is hazardous and digestion of the extract is not necessary. However, data on undigested extracts alone cannot be used to demonstrate that the waste is not hazardous. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to ± 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)\ (C_1) + (V_2)\ (C_2)}{V_1 + V_2}$$

where:

V_1 = The volume of the first phase (L).
C_1 = The concentration of the analyte of concern in the first phase (mg/L).
V_2 = The volume of the second phase (L).
C_2 = The concentration of the analyte of concern in the second phase (mg/L).

7.2.15 Compare the analyte concentrations in the TCLP extract with the levels identified in the appropriate regulations. Refer to Section 8.0 for quality assurance requirements.

7.3 Procedure When Volatiles are Involved

Use the ZHE device to obtain TCLP extract for analysis of volatile compounds only. Extract resulting from the use of the ZHE shall not be used to evaluate the mobility of nonvolatile analytes (e.g., metals, pesticides, etc.).

The ZHE device has approximately a 500 mL internal capacity. The ZHE can thus accommodate a maximum of 25 grams of solid (defined as that fraction of a sample from which no additional liquid may be forced out by an applied pressure of 50 psi), due to the need to add an amount of extraction fluid equal to 20 times the weight of the solid phase.

Charge the ZHE with sample only once and do not open the device until the final extract (of the solid) has been collected. Repeated filling of the ZHE to obtain 25 grams of solid is not permitted.

Do not allow the waste, the initial liquid phase, or the extract to be exposed to the atmosphere for any more time than is absolutely necessary. Any

manipulation of these materials should be done when cold (4 °C) to minimize loss of volatiles.

7.3.1 Pre-weigh the (evacuated) filtrate collection container (See Section 4.6) and set aside. If using a TEDLAR® bag, express all liquid from the ZHE device into the bag, whether for the initial or final liquid/solid separation, and take an aliquot from the liquid in the bag for analysis. The containers listed in Section 4.6 are recommended for use under the conditions stated in Sections 4.6.1 - 4.6.3.

7.3.2 Place the ZHE piston within the body of the ZHE (it may be helpful first to moisten the piston O-rings slightly with extraction fluid). Adjust the piston within the ZHE body to a height that will minimize the distance the piston will have to move once the ZHE is charged with sample (based upon sample size requirements determined from Section 7.3, Section 7.1.1 and/or 7.1.2). Secure the gas inlet/outlet flange (bottom flange) onto the ZHE body in accordance with the manufacturer's instructions. Secure the glass fiber filter between the support screens and set aside. Set liquid inlet/outlet flange (top flange) aside.

7.3.3 If the waste is 100% solid (see Section 7.1.1), weigh out a subsample (25 gram maximum) of the waste, record weight, and proceed to Section 7.3.5.

7.3.4 If the waste contains < 0.5% dry solids (Section 7.1.2), the liquid portion of waste, after filtration, is defined as the TCLP extract. Filter enough of the sample so that the amount of filtered liquid will support all of the volatile analyses required. For wastes containing $\geq$ 0.5% dry solids (Sections 7.1.1 and/or 7.1.2), use the percent solids information obtained in Section 7.1.1 to determine the optimum sample size to charge into the ZHE. The recommended sample size is as follows:

7.3.4.1 For wastes containing < 5% solids (see Section 7.1.1), weigh out a 500 gram subsample of waste and record the weight.

7.3.4.2 For wastes containing $\geq$ 5% solids (see Section 7.1.1), determine the amount of waste to charge into the ZHE as follows:

$$\text{Weight of waste to charge ZHE} = \frac{25}{\text{percent solids (Section 7.1.1)}} \times 100$$

Weigh out a subsample of the waste of the appropriate size and record the weight.

7.3.5 If particle size reduction of the solid portion of the waste was required in Section 7.1.3, proceed to Section 7.3.6. If

particle size reduction was not required in Section 7.1.3, proceed to Section 7.3.7.

7.3.6 Prepare the waste for extraction by crushing, cutting, or grinding the solid portion of the waste to a surface area or particle size as described in Section 7.1.3. Wastes and appropriate reduction equipment should be refrigerated, if possible, to 4 °C prior to particle size reduction. The means used to effect particle size reduction must not generate heat in and of itself. If reduction of the solid phase of the waste is necessary, exposure of the waste to the atmosphere should be avoided to the extent possible.

NOTE: Sieving of the waste is not recommended due to the possibility that volatiles may be lost. The use of an appropriately graduated ruler is recommended as an acceptable alternative. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended.

When the surface area or particle size has been appropriately altered, proceed to Section 7.3.7.

7.3.7 Waste slurries need not be allowed to stand to permit the solid phase to settle. Do not centrifuge wastes prior to filtration.

7.3.8 Quantitatively transfer the entire sample (liquid and solid phases) quickly to the ZHE. Secure the filter and support screens onto the top flange of the device and secure the top flange to the ZHE body in accordance with the manufacturer's instructions. Tighten all ZHE fittings and place the device in the vertical position (gas inlet/outlet flange on the bottom). Do not attach the extract collection device to the top plate.

NOTE: If waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the ZHE, determine the weight of this residue and subtract it from the sample weight determined in Section 7.3.4 to determine the weight of the waste sample that will be filtered.

Attach a gas line to the gas inlet/outlet valve (bottom flange) and, with the liquid inlet/outlet valve (top flange) open, begin applying gentle pressure of 1-10 psi (or more if necessary) to force all headspace slowly out of the ZHE device into a hood. At the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure. If filtration of the waste at 4 °C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering. If the waste is 100% solid (see Section 7.1.1), slowly increase the pressure to a maximum of 50 psi to force most of the headspace out of the device and proceed to Section 7.3.12.

7.3.9 Attach the evacuated pre-weighed filtrate collection container to the liquid inlet/outlet valve and open the valve. Begin applying gentle pressure of 1-10 psi to force the liquid phase of the sample into the filtrate collection container. If no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When liquid flow has ceased such that continued pressure filtration at 50 psi does not result in any additional filtrate within a 2 minute period, stop the filtration. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect and weigh the filtrate collection container.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.3.10 The material in the ZHE is defined as the solid phase of the waste and the filtrate is defined as the liquid phase.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying pressure filtration, this material will not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the TCLP extraction as a solid.

If the original waste contained <0.5% dry solids (see Section 7.1.2), this filtrate is defined as the TCLP extract and is analyzed directly. Proceed to Section 7.3.15.

7.3.11 The liquid phase may now be either analyzed immediately (See Sections 7.3.13 through 7.3.15) or stored at 4 °C under minimal headspace conditions until time of analysis. Determine the weight of extraction fluid #1 to add to the ZHE as follows:

$$\text{Weight of extraction fluid} = \frac{20 \times \text{percent solids (Section 7.1.1)} \times \text{weight of waste filtered (Section 7.3.4 or 7.3.8)}}{100}$$

7.3.12 The following Sections detail how to add the appropriate amount of extraction fluid to the solid material within the ZHE and agitation of the ZHE vessel. Extraction fluid #1 is used in all cases (See Section 5.7).

7.3.12.1 With the ZHE in the vertical position, attach a line from the extraction fluid reservoir to the liquid inlet/outlet valve. The line used shall contain fresh extraction fluid and should be preflushed with fluid to eliminate any air pockets in the line. Release gas pressure on the ZHE piston (from the gas inlet/outlet valve), open the liquid inlet/outlet valve,

and begin transferring extraction fluid (by pumping or similar means) into the ZHE. Continue pumping extraction fluid into the ZHE until the appropriate amount of fluid has been introduced into the device.

7.3.12.2 After the extraction fluid has been added, immediately close the liquid inlet/outlet valve and disconnect the extraction fluid line. Check the ZHE to ensure that all valves are in their closed positions. Manually rotate the device in an end-over-end fashion 2 or 3 times. Reposition the ZHE in the vertical position with the liquid inlet/outlet valve on top. Pressurize the ZHE to 5-10 psi (if necessary) and slowly open the liquid inlet/outlet valve to bleed out any headspace (into a hood) that may have been introduced due to the addition of extraction fluid. This bleeding shall be done quickly and shall be stopped at the first appearance of liquid from the valve. Re-pressurize the ZHE with 5-10 psi and check all ZHE fittings to ensure that they are closed.

7.3.12.3 Place the ZHE in the rotary agitation apparatus (if it is not already there) and rotate at 30 ± 2 rpm for 18 ± 2 hours. Ambient temperature (i.e., temperature of room in which extraction occurs) shall be maintained at 23 ± 2 °C during agitation.

7.3.13 Following the 18 ± 2 hour agitation period, check the pressure behind the ZHE piston by quickly opening and closing the gas inlet/outlet valve and noting the escape of gas. If the pressure has not been maintained (i.e., no gas release observed), the device is leaking. Check the ZHE for leaking as specified in Section 4.2.1, and perform the extraction again with a new sample of waste. If the pressure within the device has been maintained, the material in the extractor vessel is once again separated into its component liquid and solid phases. If the waste contained an initial liquid phase, the liquid may be filtered directly into the same filtrate collection container (i.e., TEDLAR® bag) holding the initial liquid phase of the waste. A separate filtrate collection container must be used if combining would create multiple phases, or there is not enough volume left within the filtrate collection container. Filter through the glass fiber filter, using the ZHE device as discussed in Section 7.3.9. All extract shall be filtered and collected if the TEDLAR® bag is used, if the extract is multiphasic, or if the waste contained an initial liquid phase (see Sections 4.6 and 7.3.1).

NOTE: An in-line glass fiber filter may be used to filter the material within the ZHE if it is suspected that the glass fiber filter has been ruptured.

7.3.14 If the original waste contained no initial liquid phase, the filtered liquid material obtained from Section 7.3.13 is defined as the TCLP extract. If the waste contained an initial liquid phase, the

filtered liquid material obtained from Section 7.3.13 and the initial liquid phase (Section 7.3.9) are collectively defined as the TCLP extract.

7.3.15 Following collection of the TCLP extract, immediately prepare the extract for analysis and store with minimal headspace at 4 °C until analyzed. Analyze the TCLP extract according to the appropriate analytical methods. If the individual phases are to be analyzed separately (i.e., are not miscible), determine the volume of the individual phases (to 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)\ (C_1) + (V_2)\ (C_2)}{V_1 + V_2}$$

where:

V_1 = The volume of the first phases (L).
C_1 = The concentration of the analyte of concern in the first phase (mg/L).
V_2 = The volume of the second phase (L).
C_2 = The concentration of the analyte of concern in the second phase (mg/L).

7.3.16 Compare the analyte concentrations in the TCLP extract with the levels identified in the appropriate regulations. Refer to Section 8.0 for quality assurance requirements.

8.0 QUALITY ASSURANCE

8.1 A minimum of one blank (using the same extraction fluid as used for the samples) must be analyzed for every 20 extractions that have been conducted in an extraction vessel.

8.2 A matrix spike shall be performed for each waste type (e.g., wastewater treatment sludge, contaminated soil, etc.) unless the result exceeds the regulatory level and the data are being used solely to demonstrate that the waste property exceeds the regulatory level. A minimum of one matrix spike must be analyzed for each analytical batch. As a minimum, follow the matrix spike addition guidance provided in each analytical method.

8.2.1 Matrix spikes are to be added after filtration of the TCLP extract and before preservation. Matrix spikes should not be added prior to TCLP extraction of the sample.

8.2.2 In most cases, matrix spikes should be added at a concentration equivalent to the corresponding regulatory level. If the analyte concentration is less than one half the regulatory level, the spike concentration may be as low as one half of the analyte concentration, but may not be not less than five times the method detection limit. In order to avoid differences in matrix effects, the matrix spikes must be

added to the same nominal volume of TCLP extract as that which was analyzed for the unspiked sample.

8.2.3 The purpose of the matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist. Use of other internal calibration methods, modification of the analytical methods, or use of alternate analytical methods may be needed to accurately measure the analyte concentration in the TCLP extract when the recovery of the matrix spike is below the expected analytical method performance.

8.2.4 Matrix spike recoveries are calculated by the following formula:

$$\%R\ (\%\text{Recovery}) = 100\ (X_s - X_u)/K$$

where:
X_s = measured value for the spiked sample,
X_u = measured value for the unspiked sample, and
K = known value of the spike in the sample.

8.3 All quality control measures described in the appropriate analytical methods shall be followed.

8.4 The use of internal calibration quantitation methods shall be employed for a metallic contaminant if: (1) Recovery of the contaminant from the TCLP extract is not at least 50% and the concentration does not exceed the regulatory level, and (2) The concentration of the contaminant measured in the extract is within 20% of the appropriate regulatory level.

8.4.1. The method of standard additions shall be employed as the internal calibration quantitation method for each metallic contaminant.

8.4.2 The method of standard additions requires preparing calibration standards in the sample matrix rather than reagent water or blank solution. It requires taking four identical aliquots of the solution and adding known amounts of standard to three of these aliquots. The forth aliquot is the unknown. Preferably, the first addition should be prepared so that the resulting concentration is approximately 50% of the expected concentration of the sample. The second and third additions should be prepared so that the concentrations are approximately 100% and 150% of the expected concentration of the sample. All four aliquots are maintained at the same final volume by adding reagent water or a blank solution, and may need dilution adjustment to maintain the signals in the linear range of the instrument technique. All four aliquots are analyzed.

8.4.3 Prepare a plot, or subject data to linear regression, of instrument signals or external-calibration-derived concentrations as the dependant variable (y-axis) versus concentrations of the additions of standard as the independent variable (x-axis). Solve for the intercept of

the abscissa (the independent variable, x-axis) which is the concentration in the unknown.

8.4.4 Alternately, subtract the instrumental signal or external-calibration-derived concentration of the unknown (unspiked) sample from the instrumental signals or external-calibration-derived concentrations of the standard additions. Plot or subject to linear regression of the corrected instrument signals or external-calibration-derived concentrations as the dependant variable versus the independent variable. Derive concentrations for unknowns using the internal calibration curve as if it were an external calibration curve.

8.5 Samples must undergo TCLP extraction within the following time periods:

SAMPLE MAXIMUM HOLDING TIMES [Days]				
	From: Field collection To: TCLP extraction	From: TCLP extraction To: Preparative extraction	From: Preparative extraction To: Determinative analysis	Total elapsed time
Volatiles	14	NA	14	28
Semi-volatiles	14	7	40	61
Mercury	28	NA	28	56
Metals, except mercury	180	NA	180	360

NA = Not applicable

If sample holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding the holding time is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory level.

9.0 METHOD PERFORMANCE

9.1 Ruggedness. Two ruggedness studies have been performed to determine the effect of various perturbations on specific elements of the TCLP protocol. Ruggedness testing determines the sensitivity of small procedural variations which might be expected to occur during routine laboratory application.

9.1.1 Metals - The following conditions were used when leaching a waste for metals analysis:

Varying Conditions	
Liquid/Solid ratio	19:1 vs. 21:1
Extraction time	16 hours vs. 18 hours
Headspace	20% vs. 60%
Buffer #2 acidity	190 meq vs. 210 meq
Acid-washed filters	yes vs. no
Filter type	0.7 μm glass fiber vs. 0.45 μm vs. polycarbonate
Bottle type	borosilicate vs. flint glass

Of the seven method variations examined, acidity of the extraction fluid had the greatest impact on the results. Four of 13 metals from an API separator sludge/electroplating waste (API/EW) mixture and two of three metals from an ammonia lime still bottom waste were extracted at higher levels by the more acidic buffer. Because of the sensitivity to pH changes, the method requires that the extraction fluids be prepared so that the final pH is within ± 0.05 units as specified.

9.1.2 Volatile Organic Compounds - The following conditions were used when leaching a waste for VOC analysis:

Varying Conditions	
Liquid/Solid ratio	19:1 vs. 21:1
Headspace	0% vs. 5%
Buffer #1 acidity	60 meq vs. 80 meq
Method of storing extract	Syringe vs. Tedlar® bag
Aliquotting	yes vs. no
Pressure behind piston	0 psi vs. 20 psi

None of the parameters had a significant effect on the results of the ruggedness test.

9.2 Precision. Many TCLP precision (reproducibility) studies have been performed, and have shown that, in general, the precision of the TCLP is comparable to or exceeds that of the EP toxicity test and that method precision is adequate. One of the more significant contributions to poor precision appears to be related to sample homogeneity and inter-laboratory variation (due to the nature of waste materials).

9.2.1 Metals - The results of a multi-laboratory study are shown in Table 6, and indicate that a single analysis of a waste may not be adequate for waste characterization and identification requirements.

9.2.2 Semi-Volatile Organic Compounds - The results of two studies are shown in Tables 7 and 8. Single laboratory precision was excellent with greater than 90 percent of the results exhibiting an RSD less than 25 percent. Over 85 percent of all individual compounds in the multi-laboratory study fell in the RSD range of 20 - 120 percent. Both studies concluded that the TCLP provides adequate precision. It was also determined that the high acetate content of the extraction fluid did not present problems (i.e., column degradation of the gas chromatograph) for the analytical conditions used.

9.2.3 Volatile Organic Compounds - Eleven laboratories participated in a collaborative study of the use of the ZHE with two waste types which were fortified with a mixture of VOCs. The results of the collaborative study are shown in Table 9. Precision results for VOCs tend to occur over a considerable range. However, the range and mean RSD compared very closely to the same collaborative study metals results in Table 6. Blackburn and Show concluded that at the 95% level of significance: 1) recoveries among laboratories were statistically similar, 2) recoveries did not vary significantly between the two sample types, and 3) each laboratory showed the same pattern of recovery for each of the two samples.

10.0 REFERENCES

1. Blackburn, W.B. and Show, I. "Collaborative Study of the Toxicity Characteristics Leaching Procedure (TCLP)." Draft Final Report, Contract No. 68-03-1958, S-Cubed, November 1986.

2. Newcomer, L.R., Blackburn, W.B., Kimmell, T.A. "Performance of the Toxicity Characteristic Leaching Procedure." Wilson Laboratories, S-Cubed, U.S. EPA, December 1986.

3. Williams, L.R., Francis, C.W.; Maskarinec, M.P., Taylor D.R., and Rothman, N. "Single-Laboratory Evaluation of Mobility Procedure for Solid Waste." EMSL, ORNL, S-Cubed, ENSECO.

Table 1.
Volatile Analytes[1,2]

Compound	CAS No.
Acetone	67-64-1
Benzene	71-43-2
n-Butyl alcohol	71-36-3
Carbon disulfide	75-15-0
Carbon tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chloroform	67-66-3
1,2-Dichloroethane	107-06-2
1,1-Dichloroethylene	75-35-4
Ethyl acetate	141-78-6
Ethyl benzene	100-41-4
Ethyl ether	60-29-7
Isobutanol	78-83-1
Methanol	67-56-1
Methylene chloride	75-09-2
Methyl ethyl ketone	78-93-3
Methyl isobutyl ketone	108-10-1
Tetrachloroethylene	127-18-4
Toluene	108-88-3
1,1,1,-Trichloroethane	71-55-6
Trichloroethylene	79-01-6
Trichlorofluoromethane	75-69-4
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1
Vinyl chloride	75-01-4
Xylene	1330-20-7

[1] When testing for any or all of these analytes, the zero-headspace extractor vessel shall be used instead of the bottle extractor.

[2] Benzene, carbon tetrachloride, chlorobenzene, chloroform, 1,2-dichloroethane, 1,1-dichloroethylene, methyl ethyl ketone, tetrachloroethylene, and vinyl chloride are toxicity characteristic constituents.

Table 2.
Suitable Rotary Agitation Apparatus[1]

Company	Location	Model No.
Analytical Testing and Consulting Services, Inc.	Warrington, PA (215) 343-4490	4-vessel extractor (DC20S) 8-vessel extractor (DC20) 12-vessel extractor (DC20B) 24-vessel extractor (DC24C)
Associated Design and Manufacturing Company	Alexandria, VA (703) 549-5999	2-vessel (3740-2-BRE) 4-vessel (3740-4-BRE) 6-vessel (3740-6-BRE) 8-vessel (3740-8-BRE) 12-vessel (3740-12-BRE) 24-vessel (3740-24-BRE)
Environmental Machine and Design, Inc.	Lynchburg, VA (804) 845-6424	8-vessel (08-00-00) 4-vessel (04-00-00)
IRA Machine Shop and Laboratory	Santurce, PR (809) 752-4004	8-vessel (011001)
Lars Lande Manufacturing	Whitmore Lake, MI (313) 449-4116	10-vessel (10VRE) 5-vessel (5VRE) 6-vessel (6VRE)
Millipore Corp.	Bedford, MA (800) 225-3384	4-ZHE or 4 2-liter bottle extractor (YT310RAHW)

[1] Any device that rotates the extraction vessel in an end-over-end fashion at 30 ± 2 rpm is acceptable.

Table 3.
Suitable Zero-Headspace Extractor Vessels[1]

Company	Location	Model No.
Analytical Testing & Consulting Services, Inc.	Warrington, PA (215) 343-4490	C102, Mechanical Pressure Device
Associated Design and Manufacturing Company	Alexandria, VA (703) 549-5999	3745-ZHE, Gas Pressure Device
Lars Lande Manufacturing[2]	Whitmore Lake, MI (313) 449-4116	ZHE-11, Gas Pressure Device
Millipore Corporation	Bedford, MA (800) 225-3384	YT30090HW, Gas Pressure Device
Environmental Machine and Design, Inc.	Lynchburg, VA (804) 845-6424	VOLA-TOX1, Gas Pressure Device
Gelman Science	Ann Arbor, MI (800) 521-1520	15400 Gas Pressure Device

[1] Any device that meets the specifications listed in Section 4.2.1 of the method is suitable.

[2] This device uses a 110 mm filter.

Table 4.
Suitable Filter Holders[1]

Company	Location	Model/ Catalogue No.	Size
Nucleopore Corporation	Pleasanton, CA (800) 882-7711	425910 410400	142 mm 47 mm
Micro Filtration Systems	Dublin, CA (800) 334-7132 (415) 828-6010	302400 311400	142 mm 47 mm
Millipore Corporation	Bedford, MA (800) 225-3384	YT30142HW XX1004700	142 mm 47 mm

[1] Any device capable of separating the liquid from the solid phase of the waste is suitable, providing that it is chemically compatible with the waste and the constituents to be analyzed. Plastic devices (not listed above) may be used when only inorganic analytes are of concern. The 142 mm size filter holder is recommended.

Table 5.
Suitable Filter Media[1]

Company	Location	Model	Pore Size (μm)
Millipore Corporation	Bedford, MA (800) 225-3384	AP40	0.7
Nucleopore Corporation	Pleasanton, CA (415) 463-2530	211625	0.7
Whatman Laboratory Products, Inc.	Clifton, NJ (201) 773-5800	GFF	0.7
Micro Filtration Systems	Dublin, CA (800) 334-7132 (415) 828-6010	GF75	0.7
Gelman Science	Ann Arbor, MI (800) 521-1520	66256 (90mm) 66257 (142mm)	0.7

[1] Any filter that meets the specifications in Section 4.4 of the Method is suitable.

Table 6. Multi-Laboratory TCLP Metals, Precision

Waste	Extraction Fluid	Metal	$\bar{X}$	S	%RSD
Ammonia Lime Still Bottoms	#1	Cadmium	0.053	0.031	60
	#2		0.023	0.017	76
	#1	Chromium	0.015	0.0014	93
	#2		0.0032	0.0037	118
	#1	Lead	0.0030	0.0027	90
	#2		0.0032	0.0028	87
API/EW Mixture	#1	Cadmium	0.0046	0.0028	61
	#2		0.0005	0.0004	77
	#1	Chromium	0.0561	0.0227	40
	#2		0.105	0.018	17
	#1	Lead	0.0031	0.0031	100
	#2		0.0124	0.0136	110
Fossil Fuel Fly Ash	#1	Cadmium	0.080	0.069	86
	#2		0.093	0.067	72
	#1	Chromium	0.017	0.014	85
	#2		0.070	0.040	57
	#1	Lead	0.0087	0.0074	85
	#2		0.0457	0.0083	18
				%RSD Range = 17 - 118	
				Mean %RSD = 74	

NOTE: $\bar{X}$ = Mean results from 6 - 12 different laboratories
Units = mg/L
Extraction Fluid #1 = pH 4.9
#2 = pH 2.9

Table 7. Single-Laboratory Semi-Volatiles, Precision

Waste	Compound	Extraction Fluid	$\bar{X}$	S	%RSD
Ammonia Lime Still Bottoms	Phenol	#1	19000	2230	11.6
		#2	19400	929	4.8
	2-Methylphenol	#1	2000	297	14.9
		#2	1860	52.9	2.8
	4-Methylphenol	#1	7940	1380	17.4
		#2	7490	200	2.7
	2,4-Dimethylphenol	#1	321	46.8	14.6
		#2	307	45.8	14.9
	Naphthalene	#1	3920	413	10.5
		#2	3827	176	4.6
	2-Methylnaphthalene	#1	290	44.8	15.5
		#2	273	19.3	7.1
	Dibenzofuran	#1	187	22.7	12.1
		#2	187	7.2	3.9
	Acenaphthylene	#1	703	89.2	12.7
		#2	663	20.1	3.0
	Fluorene	#1	151	17.6	11.7
		#2	156	2.1	1.3
	Phenanthrene	#1	241	22.7	9.4
		#2	243	7.9	3.3
	Anthracene	#1	33.2	6.19	18.6
		#2	34.6	1.55	4.5
	Fluoranthrene	#1	25.3	1.8	7.1
		#2	26.0	1.8	7.1
API/EW Mixture	Phenol	#1	40.7	13.5	33.0
		#2	19.0	1.76	9.3
	2,4-Dimethylphenol	#1	33.0	9.35	28.3
		#2	43.3	8.61	19.9
	Naphthalene	#1	185	29.4	15.8
		#2	165	24.8	15.0
	2-Methylnaphthalene	#1	265	61.2	23.1
		#2	200	18.9	9.5
					%RSD Range = 1 - 33
					Mean %RSD = 12

NOTE: Units = µg/L
Extractions were performed in triplicate
All results were at least 2x the detection limit
Extraction Fluid #1 = pH 4.9
#2 = pH 2.9

Table 8. Multi-Laboratory Semi-Volatiles, Precision

Waste	Compound	Extraction Fluid	$\bar{X}$	S	%RSD
Ammonia Lime	BNAs	#1	10043	7680	76.5
Still Bottoms (A)		#2	10376	6552	63.1
API/EW	BNAs	#1	1624	675	41.6
Mixture (B)		#2	2074	1463	70.5
Fossil Fuel	BNAs	#1	750	175	23.4
Fly Ash (C)		#2	739	342	46.3
					Mean %RSD = 54

NOTE: Units = µg/L
$\bar{X}$ = Mean results from 3 - 10 labs
Extraction Fluid #1 = pH 4.9
#2 = pH 2.9

%RSD Range for Individual Compounds

A, #1	0 - 113
A, #2	28 - 108
B, #1	20 - 156
B, #2	49 - 128
C, #1	36 - 143
C, #2	61 - 164

Table 9. Multi-Laboratory (11 Labs) VOCs, Precision

Waste	Compound	$\bar{X}$	S	%RSD
Mine Tailings	Vinyl chloride	6.36	6.36	100
	Methylene chloride	12.1	11.8	98
	Carbon disulfide	5.57	2.83	51
	1,1-Dichloroethene	21.9	27.7	127
	1,1-Dichloroethane	31.4	25.4	81
	Chloroform	46.6	29.2	63
	1,2-Dichloroethane	47.8	33.6	70
	2-Butanone	43.5	36.9	85
	1,1,1-Trichloroethane	20.9	20.9	100
	Carbon tetrachloride	12.0	8.2	68
	Trichloroethene	24.7	21.2	86
	1,1,2-Trichloroethene	19.6	10.9	56
	Benzene	37.9	28.7	76
	1,1,2,2-Tetrachloroethane	34.9	25.6	73
	Toluene	29.3	11.2	38
	Chlorobenzene	35.6	19.3	54
	Ethylbenzene	4.27	2.80	66
	Trichlorofluoromethane	3.82	4.40	115
	Acrylonitrile	76.7	110.8	144
Ammonia Lime Still Bottoms	Vinyl chloride	5.00	4.71	94
	Methylene chloride	14.3	13.1	92
	Carbon disulfide	3.37	2.07	61
	1,1-Dichloroethene	52.1	38.8	75
	1,1-Dichloroethane	52.8	25.6	49
	Chloroform	64.7	28.4	44
	1,2-Dichloroethane	43.1	31.5	73
	2-Butanone	59.0	39.6	67
	1,1,1-Trichloroethane	53.6	40.9	76
	Carbon tetrachloride	7.10	6.1	86
	Trichloroethene	57.3	34.2	60
	1,1,2-Trichloroethene	6.7	4.7	70
	Benzene	61.3	26.8	44
	1,1,2,2-Tetrachloroethane	3.16	2.1	66
	Toluene	69.0	18.5	27
	Chlorobenzene	71.8	12.0	17
	Ethylbenzene	3.70	2.2	58
	Trichlorofluoromethane	4.05	4.8	119
	Acrylonitrile	29.4	34.8	118
				%RSD Range = 17 - 144
				Mean %RSD = 75

NOTE: Units = μg/L

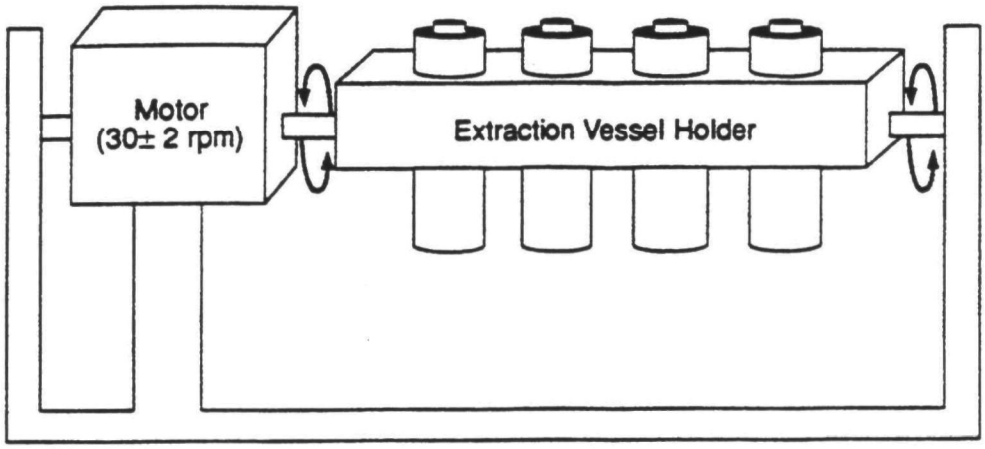


Figure 1. Rotary Agitation Apparatus

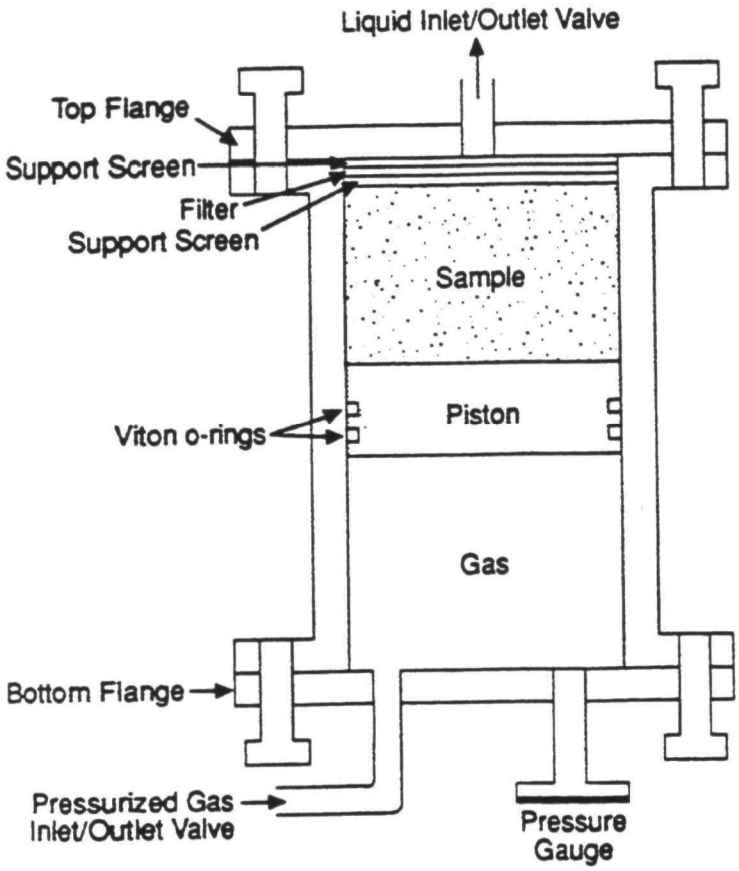
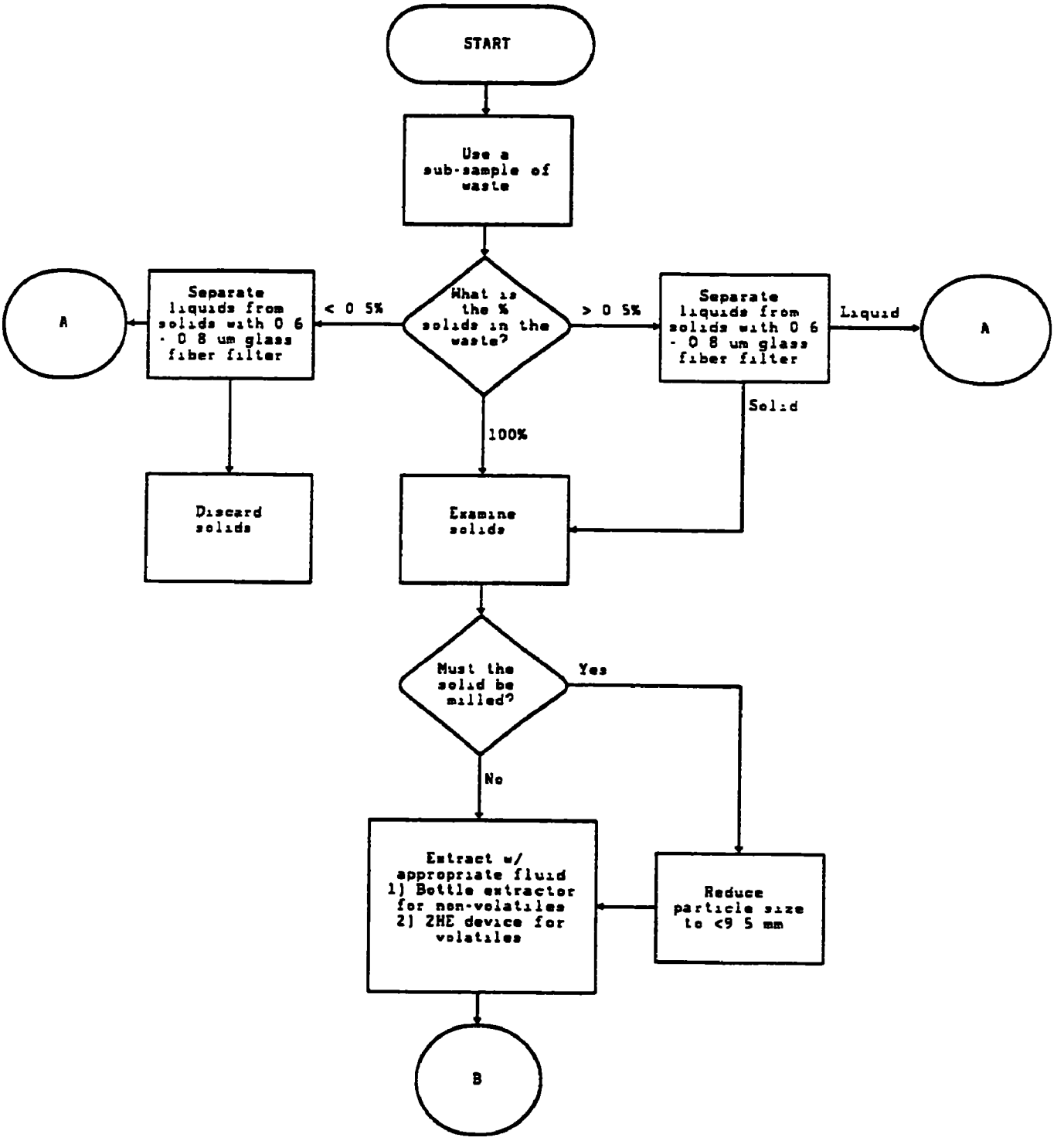
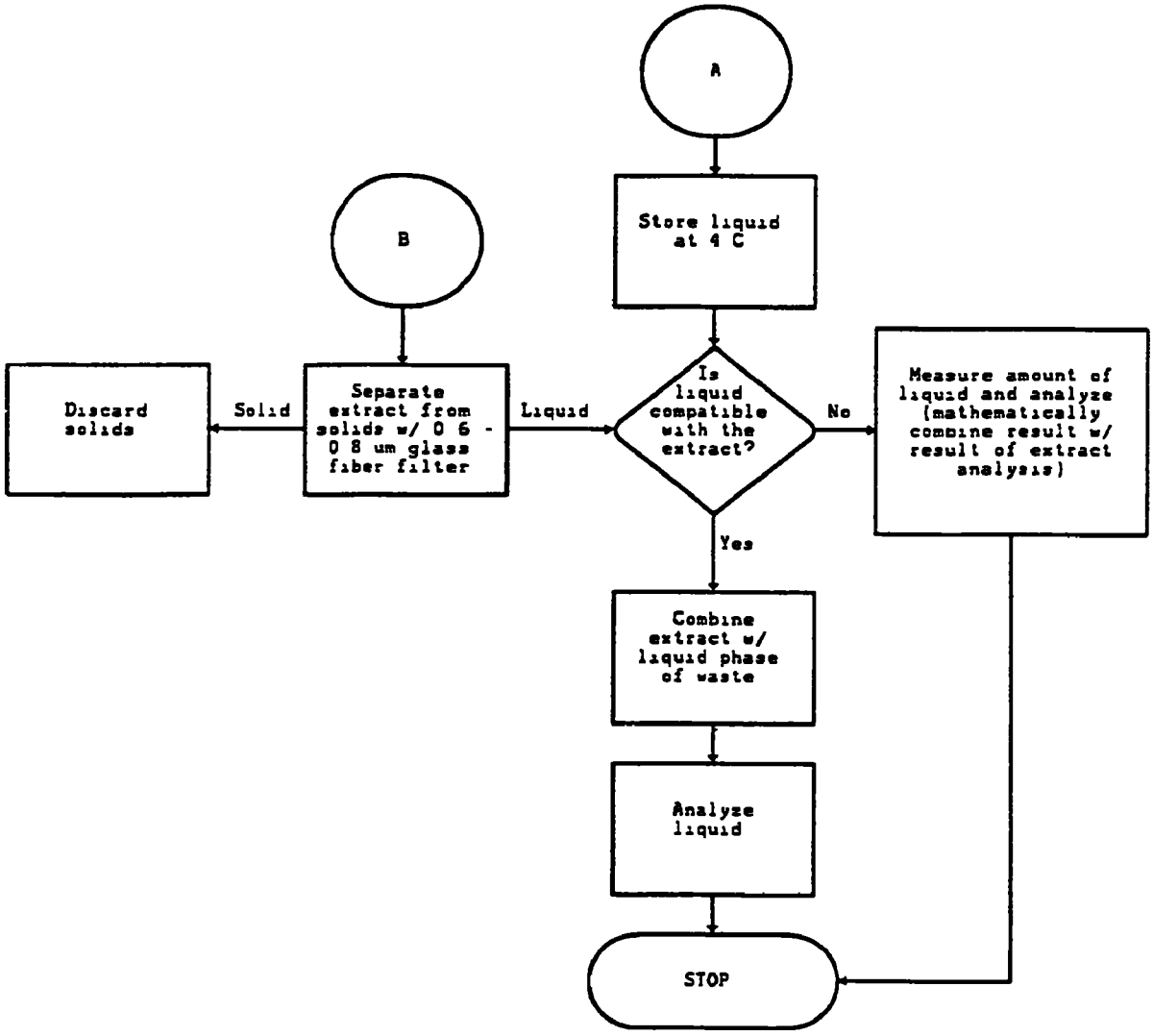


Figure 2. Zero-Headspace Extractor (ZHE)

TOXICITY CHARACTERISTIC LEACHATE PROCEDURE



TOXICITY CHARACTERISTIC LEACHATE PROCEDURE



Appendix II

Appendix II
40 CFR 268 Subpart D Land Ban Treatment Standards

(1) The wastes meet the applicable standards specified in subpart D of this part;

(2) Persons have been granted an exemption from a prohibition pursuant to a petition under §268.6, with respect to those wastes and units covered by the petition;

(3) The wastes meet the applicable alternate standards established pursuant to a petition granted under §268.44;

(4) Persons have been granted an extension to the effective date of a prohibition pursuant to §268.5, with respect to the wastes covered by the extension.

(i) To determine whether a hazardous waste identified in this section exceeds the applicable treatment standards specified in §§268.41 and 268.43, the initial generator must test a representative sample of the waste extract or the entire waste, depending on whether the treatment standards are expressed as concentrations in the waste extract or the waste, or the generator may use knowledge of the waste. If the waste contains constituents in excess of the applicable levels in subpart D of this part, the waste is prohibited from land disposal, and all requirements of part 268 are applicable, except as otherwise specified.

[57 FR 37271, Aug. 18, 1992]

§268.37 Waste specific prohibitions—ignitable and corrosive characteristic wastes whose treatment standards were vacated.

(a) Effective August 9, 1993, the wastes specified in 40 CFR 261.21 as D001 (and is not in the High TOC Ignitable Liquids Subcategory), and specified in §261.22 as D002, that are manged in systems other than those hose discharge is regulated under the lean Water Act (CWA), or that inject n Class I deep wells regulated under he Safe Drinking Water Act (SDWA), r that are zero dischargers that enage in CWA-equivalent treatment bere ultimate land disposal, are prohibed from land disposal. CWA-equivant treatment means biological treatent for organics, alkaline lorination or ferrous sulfate precipiction for cyanide, precipitation/sedientation for metals, reduction of xavalent chromium, or other treatent technology that can be demonstrated to perform equally or greater than these technologies.

(b) Effective February 10, 1994, the wastes specified in 40 CFR 261.21 as D001 (and is not in the High TOC Ignitable Liquids Subcategory), and specified in §261.22 as D002, that are managed in systems defined in 40 CFR 144.6(e) and 146.6(e) as Class V injection wells, that do not engage in CWA-equivalent treatment before injection, are prohibited from land disposal.

[58 FR 29885, May 24, 1993]

Subpart D—Treatment Standards

§268.40 Applicability of treatment standards.

(a) A restricted waste identified in §268.41 may be land disposed only if an extract of the waste or of the treatment residue of the waste developed using the test method in appendix II of part 261 does not exceed the value shown in Table CCWE of §268.41 for any hazardous constituent listed in Table CCWE for that waste, with the following exceptions: D004, D008, K031, K084, K101, K102, P010, P011, P012, P036, P038, and U136. These wastes may be land disposed only if an extract of the waste or of the treatment residue of the waste developed using either the test method in 40 CFR part 261, appendix II, or the test method in appendix IX of this part, does not exceed the concentrations shown in Table CCWE of §268.41 for any hazardous constituent listed in Table CCWE for that waste.

(b) A restricted waste for which a treatment technology is specified under §268.42(a), or hazardous debris for which a treatment technology is specified under §268.45, may be land disposed after it is treated using that specified technology or an equivalent treatment method approved by the Administrator under the procedures set forth in §268.42(b). For waste displaying the characteristic of ignitability (D001) and reactivity (D003), that are diluted to meet the deactivation treatment standard in §268.42(a) Tables 1 and 2 (DEACT), the treater must comply with the precautionary measures specified in 40 CFR 264.17(b) and 265.17(b) of this chapter.

(c) Except as otherwise specified in §268.43(c), a restricted waste identified

in §268.43 may be land disposed only if the constituent concentrations in the waste or treatment residue of the waste do not exceed the value shown in Table CCW of §268.43 for any hazardous constituents listed in Table CCW for that waste.

(d) If a treatment standard has been established in §§268.41 through 268.43 for a hazardous waste that is itself hazardous debris, the waste is subject to those standards rather than the standards for hazardous debris under §268.45.

[52 FR 25790, July 8, 1987, as amended at 55 FR 22689, June 1, 1990, 56 FR 3879, Jan. 31, 1991, 57 FR 37272, Aug. 18, 1992, 58 FR 29885, May 24, 1993]

§268.41 Treatment standards expressed as concentrations in waste extract.

(a) Table CCWE identifies the restricted wastes and the concentrations of their associated constituents which may not be exceeded in the extract of a waste or waste treatment residual extracted using the test method in appendix I of this part for the allowable land disposal of such wastes. Compliance with these concentrations is required based upon grab samples.

268.41 TABLE CCWE.—CONSTITUENT CONCENTRATIONS IN WASTE EXTRACT

Waste code	Commercial chemical name	See also	Regulated hazardous constituent	CAS No for regulated hazardous constituent	Wastewaters		Nonwastewaters	
					Concentration (mg/l)	Notes	Concentration (mg/l)	Notes
D004	NA	Table CCW in 268 43	Arsenic	7440–38–2	NA		5 0	(1)
D005	NA	Table CCW in 268.43.	Barium	7440–39–3	NA		100	
D006	NA	Table CCW in 268 43	Cadmium	7440–43–9	NA		1 0	
D007	NA	Table CCW in 268 43.	Chromium (Total)	7440–47–32	NA		5 0	
D008	NA	Table CCW in 268 43	Lead	7439–92–1	NA		5 0	(1)
D009 (Low Mercury Subcategory—less than 260 mg/kg Mercury)	NA	Table 2 in 268 42 and Table CCW in 268 43	Mercury	7439–97–6	NA		0.20	
D010	NA	Table CCW in 268 43	Selenium	7782–49–2	NA		5 7	
D011	NA	Table CCW in 268 43	Silver	7440–22–4	NA		5 0	
F001–F005 spent solvents	NA	Table CCW in 268 43	Carbon disulfide	75–15–0	NA		4 8	
			Cyclohexanone	108–94–1	NA		0 75	
			Methanol	67–56–1	NA		0 75	
F006	NA	Table CCW in 268 43	Cadmium	7440–43–9	NA		0 066	
			Chromium (Total)	7440–47–32	NA		5 2	
			Lead	7439–92–1	NA		0 51	
			Nickel	7440–02–0	NA		0 32	
			Silver	7440–22–4	NA		0 072	
F007	NA	Table CCW in 268 43	Cadmium	7440–43–9	NA		0 066	
			Chromium (Total)	7440–47–32	NA		5 2	
			Lead	7439–92–1	NA		0 51	
			Nickel	7440–02–0	NA		0 32	
			Silver	7440–22–4	NA		0 072	
F008	NA	Table CCW in 268 43	Cadmium	7440–43–9	NA		0 066	
			Chromium (Total)	7440–47–32	NA		5 2	
			Lead	7439–92–1	NA		0 51	
			Nickel	7440–02–0	NA		0 32	
			Silver	7440–22–4	NA		0 072	
F009	NA	Table CCW in 268 43	Cadmium	7440–43–9	NA		0 066	
			Chromium (Total)	7440–47–32	NA		5 2	
			Lead	7439–92–1	NA		0 51	
			Nickel	7440–02–0	NA		0 32	
			Silver	7440–22–4	NA		0 072	

F011	NA	Table CCW in 268.43.	Cadmium	7440–43–9	NA		0 066	
			Chromium (Total)	7440–47–32	NA		5.2	
			Lead	7439–92–1	NA		0 51	
			Nickel	7440–02–0	NA		0 32	
			Silver	7440–22–4	NA		0 072	
F012	NA	Table CCW in 268.43.	Cadmium	7440–43–9	NA		0 066	
			Chromium (Total)	7440–47–32	NA		5 2	
			Lead	7439–92–1	NA		0 51	
			Nickel	7440–02–0	NA		0 32	
			Silver	7440–22–4	NA		0 072	
F019	NA	Table CCW in 268.43.	Chromium (Total)	7440–47–32	NA		5 2	
F020–F023 and F026–F028 dioxin containing wastes[2]	NA	NA	HxCDD-All Hexachloro-dibenzo-p-dioxins		<1 ppb		<1 ppb	
			HxCDF-All Hexachloro-dibenzofurans					
			PeCDD-All Pentachloro-dibenzo-p-dioxins		<1 ppb		<1 ppb	
			PeCDF-All Pentachloro-dibenzofurans					
			TCDD-All Tetrachloro-dibenzo-p-dioxins		<1 ppb		<1 ppb	
			TCDF-All Tetrachloro-dibenzofurans					
			2,4,5-Trichlorophenol		<1 ppb		<1 ppb	
			2,4,6-Trichlorophenol					
			2,3,4,6-Tetrachlorophenol		<1 ppb		<1 ppb	
			Pentachlorophenol					
				95–95–4	<1 ppb		<1 ppb	
				88–06–2	<0 05 ppm		<0 05 ppm	
				58–90–2	<0.05 ppm		<0 05 ppm	
				87–86–5	<0 01 ppm		<0.01 ppm	
F024	NA	Table CCW in 268.43.	Chromium (Total)	7440–47–32	NA		0 073	
			Lead	7439–92–1	NA		[Reserved]	
			Nickel	7440–02–0	NA		0 088	
F037	NA	Table CCW in 268.43.	Chromium (Total)	7440–47–32	NA		1 7	
			Nickel	7440–02–0	NA		0 20	
F038	NA	Table CCW in 268 43.	Chromium (Total)	7440–47–32	NA	1.7		
			Nickel	7440–02–0	NA	0.20		
F039(and D001 and D002 wastes prohibited under § 268.37).	NA	Table 2 in 268.42 and Table CCW in 268.43.	Antimony	7440–36–0	NA		0.23	
			Arsenic	7440–38–2	NA		5 0	
			Barium	7440–39–3	NA		52	
			Cadmium	7440–43–9	NA		0 066	
			Chromium (Total)	7440–47–32	NA		5.2	
			Lead	7439–92–1	NA		0 51	
			Mercury	7439–97–6	NA		0 025	
			Nickel	7440–02–0	NA		0 32	
			Selenium	7782–49–2	NA		5 7	
			Silver	7440–22–4	NA		0 072	
K001	NA	Table CCW in 268.43.	Lead	7439–92–1	NA		0.51	
K002	NA	Table CCW in 268.43.	Chromium (Total)	7440–47–32	NA		0.094	
			Lead	7439–92–1	NA		0.37	

268 41 TABLE CCWE.—CONSTITUENT CONCENTRATIONS IN WASTE EXTRACT—Continued

Waste code	Commercial chemical name	See also	Regulated hazardous constituent	CAS No for regulated hazardous constituent	Wastewaters		Nonwastewaters	
					Concentration (mg/l)	Notes	Concentration (mg/l)	Notes
K003	NA	Table CCW in 268.43.	Chromium (Total)	7440-47-32	NA		0 094	
			Lead	7439-92-1	NA		0.37	
K004	NA	Table CCW in 268.43	Chromium (Total)	7440-47-32	NA		0 094	
			Lead	7439-92-1	NA		0 37	
K005	NA	Table CCW in 268 43	Chromium (Total)	7440-47-32	NA		0 094	
			Lead	7439-92-1	NA		0.37	
K006 (anhydrous)	NA	Table CCW in 268.43	Chromium (Total)	7440-47-32	NA		0 094	
			Lead	7439-92-1	NA		0 37	
K006 (hydrated)	NA	Table CCW in 268.43.	Chromium (Total)	7440-47-32	NA		5.2	
K007	NA	Table CCW in 268.43	Chromium (Total)	7440-47-32	NA		0 094	
			Lead	7439-92-1	NA		0 37	
K008	NA	Table CCW in 268.43	Chromium (Total)	7440-47-32	NA		0 094	
			Lead	7439-92-1	NA		0 37	
K015	NA	Table CCW in 268 43.	Chromium (Total)	7440-47-32	NA		1 7	
			Nickel	7440-02-0	NA		0 2	
K021	NA	Table CCW in 268 43	Antimony	7440-36-0	NA		0.23	
K022	NA	Table CCW in 268.43	Chromium (Total)	7440-47-32	NA		5.2	
			Nickel	7440-02-0	NA		0 32	
K028	NA	Table CCW in 268 43.	Chromium (Total)	7440-47-32	NA		0 073	
			Lead	7439-92-1	NA		0 021	
K031	NA	Table CCW in 268.43	Nickel	7440-02-0	NA		0 068	
			Arsenic	7440-38-2	NA		5 6	(1)
K046	NA	Table CCW in 268 43	Lead	7439-92-1	NA		0 18	
K048	NA	Table CCW in 268 43	Chromium (Total)	7440-47-32	NA		1 7	
			Nickel	7440-02-0	NA		0 20	
K049	NA	Table CCW in 268 43	Chromium (Total)	7440-47-32	NA		1 7	
			Nickel	7440-02-0	NA		0.20	
K050	NA	Table CCW in 268 43.	Chromium (Total)	7440-47-32	NA		1 7	
			Nickel	7440-02-0	NA		0.20	
K051	NA	Table CCW in 268 43	Chromium (Total)	7440-47-32	NA		1 7	
			Nickel	7440-02-0	NA		0 20	
K052	NA	Table CCW in 268 43	Chromium (Total)	7440-47-32	NA		1 7	
			Nickel	7440-02-0	NA		0 20	
K061	NA	Table CCW in 268 43	Chromium (Total)	7440-47-32	NA		1 7	
			Nickel	7440-02-0	NA		0.20	
			Antimony	7440-36-0	NA		2 1	
			Arsenic	7440-38-2	NA		0 055	
			Barium	7440-39-3	NA		7 6	
			Beryllium	7440-41-7	NA		0 014	
			Cadmium	7440-43-9	NA		0 19	
K069 (Calcium Sulfate Subcategory)	NA	Table 2 in 268 42 and Table CCW in 268 43.	Chromium (Total)	7440-47-32	NA		0 33	
			Lead	7439-92-1	NA		0.37	
			Mercury	7439-97-6	NA		0 009	
			Nickel	7440-02-0	NA		5	
			Selenium	7782-49-2	NA		0 16	
			Silver	7440-22-4	NA		0 3	
			Thallium		NA		0 078	
			Zinc	7440-66-6	NA		5.3	
K071	NA	Table CCW in 268 43	Cadmium	7440-43-9	NA		0 14	
			Lead	7439-92-1	NA		0.24	
K083	NA	Table CCW in 268 43	Mercury	7439-97-6	NA		0.025	
K084	NA	Table CCW in 268 43	Nickel	7440-02-2	NA		0 088	
			Arsenic	7440-38-2	NA		5 6	(1)
K085	NA	Table CCW in 268 43.	Chromium (Total)	7440-47-32	NA		0 094	
			Lead	7439-92-1	NA		037	
K087	NA	Table CCW in 268 43.	Lead	7439-92-1	NA		0.51	
K100	NA	Table CCW in 268 43.	Cadmium	7440-43-9	NA		0 066	
			Chromium (Total)	7440-47-32	NA		5.2	
			Lead	7439-92-1	NA		0.51	
K101	NA	Table CCW in 268.43.	Arsenic	7440-38-2	NA		5 6	(1)
K102	NA	Table CCW in 268.43.	Arsenic	7440-38-2	NA		5 6	(1)
K106 (Low Mercury Subcategory—less than 260 mg/kg Mercury—residues from RMERC).	NA	Table 2 in 268.42 and Table CCW in 268 43.	Mercury	7439-97-6	NA		0 020	
K106 (Low Mercury Subcategory—less than 260 mg/kg Mercury—that are not residues from RMERC)	NA	Table 2 in 268 42 and Table CCW in 268.43.	Mercury	7439-97-6	NA		0 025	
K115	NA	Table CCW in 268.43.	Nickel	7440-02-0	NA		0.32	
P010	Arsenic acid	Table CCW in 268.43.	Arsenic	7440-38-2	NA		5.6	(1)
P011	Arsenic pentoxide	Table CCW in 268.43	Arsenic	7440-38-2	NA		5.6	(1)
P012	Arsenic trioxide	Table CCW in 268.43.	Arsenic	7440-38-2	NA		5.6	(1)

268.41 TABLE CCWE.—CONSTITUENT CONCENTRATIONS IN WASTE EXTRACT—Continued

Waste code	Commercial chemical name	See also	Regulated hazardous constituent	CAS No. for regulated hazardous constituent	Wastewaters		Nonwastewaters	
					Concentration (mg/l)	Notes	Concentration (mg/l)	Notes
P013	Barium cyanide	Table CCW in 268.43	Barium	7440-39-3	NA		52	
P036	Dichlorophenylarsine	Table CCW in 268.43	Arsenic	7440-38-2	NA		5.6	(1)
P038	Diethylarsine	Table CCW in 268.43.	Arsenic	7440-38-2	NA		5.6	(1)
P065 (Low Mercury Subcategory—Less than 260 mg/kg Mercury—residues from RMERC).	Mercury fulminate	Table 2 in 268.42 and Table CCW in 268.43.	Mercury	7439-97-6	NA		0.20	
P065 (Low Mercury Subcategory—Less than 260 mg/kg Mercury—incinerator residues (and are not residues from RMERC))	Mercury fulminate	Table 2 in 268.42 and Table CCW in 268.43	Mercury	7439-97-6	NA		0.025	
P073	Nickel carbonyl	Table CCW in 268.43	Nickel	7440-02-0	NA		0.32	
P074	Nickel cyanide	Table CCW in 268.43.	Nickel	7440-02-0	NA		0.32	
P092 (Low Mercury Subcategory—Less than 260 mg/kg Mercury—residues from RMERC)	Phenyl mercury acetate	Table 2 in 268.42 and Table CCW in 268.43	Mercury	7439-97-6	NA		0.20	
P092 (Low Mercury Subcategory—Less than 260 mg/kg Mercury—incinerator residues (and are not residues from RMERC))	Phenyl mercury acetate	Table 2 in 268.42 and Table CCW in 268.43	Mercury	7439-97-6	NA		0.025	
P099	Potassium silver cyanide	Table CCW in 268.43.	Silver	7440-22-4	NA		0.072	
P103	Selenourea	Table CCW in 268.43	Selenium	7782-49-2	NA		5.7	

P104	Silver cyanide	Table CCW in 268.43	Silver	7440-22-4	NA		0.072	
P110	Tetraethyl lead	Table CCW in 268.43.	Lead	7439-92-1	NA		0.51	
P114	Thallium selenite	Table CCW in 268.43.	Selenium	7782-49-2	NA		5.7	
U032	Calcium chromate	Table CCW in 268.43	Chromium (Total)	7440-47-32	NA		0.094	
U051	Creosote	Table CCW in 268.43.	Lead	7439-92-1	NA		0.51	
U136	Cacodylic acid	Table CCW in 268.43	Arsenic	7440-38-2	NA		5.6	(1)
U144	Lead acetate	Table CCW in 268.43.	Lead	7439-92-1	NA		0.51	
U145	Lead phosphate	Table CCW in 268.43.	Lead	7439-92-1	NA		0.51	
U146	Lead subacetate	Table CCW in 268.43.	Lead	7439-92-1	NA		0.51	
U151 (Low Mercury Subcategory—Less than 260 mg/kg Mercury—residues from RMERC)	Mercury	Table CCW in 268.43 and Table 2 in 268.42.	Mercury	7439-97-6	NA		0.20	
U151 (Low Mercury Subcategory—Less than 260 mg/kg Mercury—that are not residues from RMERC	Mercury	Table CCW in 268.43 and Table 2 in 268.42.	Mercury	7439-97-6	NA		0.025	
U204	Selenium dioxide	Table CCW in 268.43	Selenium	7782-49-2	NA		5.7	
U205	Selenium sulfide	Table CCW in 268.43.	Selenium	7782-49-2	NA		5.7	

[1] These treatment standards have been based on EP Leachate analysis but this does not preclude the use of TCLP analysis.
[2] These waste codes are not subcategorized into wastewaters and nonwastewaters.
Note. NA means Not Applicable.

(b) When wastes with differing treatment standards for a constituent of concern are combined for purposes of treatment, the treatment residue must meet the lowest treatment standard for the constituent of concern, except that mixtures of high and low zinc nonwastewater K061 are subject to the treatment standard for high zinc K061.

(c) The treatment standards for the constituents in F001–F005 which are listed in Table CCWE only apply to wastes which contain one, two, or all three of these constituents. If the waste contains any of these three constituents along with any of the other 26 constituents found in F001–F005, then only the treatment standards in §268.43 Table CCW are required.

[51 FR 10642, Nov 7, 1986; 52 FR 21017, June 4, 1987, as amended at 55 FR 22689, June 1, 1990, 56 FR 3879, Jan. 31, 1991; 56 FR 41177, Aug 19, 1991, 57 FR 8089, Mar. 6, 1992; 57 FR 37272, Aug. 18, 1992; 58 FR 29885, May 24, 1993]

§268.42 Treatment standards expressed as specified technologies.

(a) The following wastes in paragraphs (a)(1) and (a)(2) of this section and in Table 2 and Table 3 of this section must be treated using the technology or technologies specified in paragraphs (a)(1) and (a)(2) and Table 1 of this section.

(1) Liquid hazardous wastes containing polychlorinated biphenyls (PCBs) at concentrations greater than or equal to 50 ppm but less than 500 ppm must be incinerated in accordance with the technical requirements of 40 CFR 761.70 or burned in high efficiency boilers in accordance with the technical requirements of 40 CFR 761.60. Liquid hazardous wastes containing polychlorinated biphenyls (PCBs) at concentrations greater than or equal to 500 ppm must be incinerated in accordance with the technical requirements of 40 CFR 761.70. Thermal treatment under this section must also be in compliance with applicable regulations in parts 264, 265, and 266.

(2) Nonliquid hazardous wastes containing halogenated organic compounds (HOCs) in total concentration greater than or equal to 1,000 mg/kg and liquid HOC-containing wastes that are prohibited under §268.32(e)(1) of this part must be incinerated in accordance with the requirements of 40 CFR part 264, subpart O, or 40 CFR part 265, subpart O. These treatment standards do not apply where the waste is subject to a part 268, subpart D, treatment standard for a specific HOC (such as a hazardous waste chlorinated solvent for which a treatment standard is established under §268.41(a)).

(3) A mixture consisting of wastewater, the discharge of which is subject to regulation under either section 402 or section 307(b) of the Clean Water Act, and *de minimis* losses of materials from manufacturing operations in which these materials are used as raw materials or are produced as products in the manufacturing process, and that meet the criteria of the D001 ignitable liquids containing greater than 10% total organic constituents (TOC) subcategory, is subject to the DEACT treatment standard described in Table 1 of this section. For purposes of this paragraph, *de minimis* losses include those from normal material handling operations (e.g., spills from the unloading or transfer of materials from bins or other containers, leaks from pipes, valves or other devices used to transfer materials); minor leaks from process equipment, storage tanks, or containers; leaks from well-maintained pump packings and seals; sample purgings; and relief device discharges.

TABLE 1 —TECHNOLOGY CODES AND DESCRIPTION OF TECHNOLOGY-BASED STANDARDS

Technology code	Description of technology-based standards
ADGAS	Venting of compressed gases into an absorbing or reacting media (i.e., solid or liquid)—venting can be accomplished through physical release utilizing valves/piping, physical penetration of the container; and/or penetration through detonation
AMLGM	Amalgamation of liquid, elemental mercury contaminated with radioactive materials utilizing inorganic reagents such as copper, zinc, nickel, gold, and sulfur that result in a nonliquid, semi-solid amalgam and thereby reducing potential emissions of elemental mercury vapors to the air.

TABLE 1 —TECHNOLOGY CODES AND DESCRIPTION OF TECHNOLOGY-BASED STANDARDS—Continued

Technology code	Description of technology-based standards
BIODG	Biodegradation of organics or non-metallic inorganics (i.e., degradable inorganics that contain the elements of phosphorus, nitrogen, and sulfur) in units operated under either aerobic or anaerobic conditions such that a surrogate compound or indicator parameter has been substantially reduced in concentration in the residuals (e.g., Total Organic Carbon can often be used as an indicator parameter for the biodegradation of many organic constituents that cannot be directly analyzed in wastewater residues).
CARBN	Carbon adsorption (granulated or powdered) of non-metallic inorganics, organo-metallics, and/or organic constituents, operated such that a surrogate compound or indicator parameter has not undergone breakthrough (e.g., Total Organic Carbon can often be used as an indicator parameter for the adsorption of many organic constituents that cannot be directly analyzed in wastewater residues). Breakthrough occurs when the carbon has become saturated with the constituent (or indicator parameter) and substantial change in adsorption rate associated with that constituent occurs
CHOXD	Chemical or electrolytic oxidation utilizing the following oxidation reagents (or waste reagents) or combinations of reagents: (1) Hypochlorite (e.g., bleach), (2) chlorine, (3) chlorine dioxide, (4) ozone or UV (ultraviolet light) assisted ozone, (5) peroxides, (6) persulfates, (7) perchlorates, (8) permanganates, and/or (9) other oxidizing reagents of equivalent efficiency, performed in units operated such that a surrogate compound or indicator parameter has been substantially reduced in concentration in the residuals (e.g., Total Organic Carbon can often be used as an indicator parameter for the oxidation of many organic constituents that cannot be directly analyzed in wastewater residues). Chemical oxidation specifically includes what is commonly referred to as alkaline chlorination.
CHRED.	Chemical reduction utilizing the following reducing reagents (or waste reagents) or combinations of reagents: (1) Sulfur dioxide; (2) sodium, potassium, or alkali salts or sulfites, bisulfites, metabisulfites, and polyethylene glycols (e.g., NaPEG and KPEG); (3) sodium hydrosulfide, (4) ferrous salts; and/or (5) other reducing reagents of equivalent efficiency, performed in units operated such that a surrogate compound or indicator parameter has been substantially reduced in concentration in the residuals (e.g., Total Organic Halogens can often be used as an indicator parameter for the reduction of many halogenated organic constituents that cannot be directly analyzed in wastewater residues). Chemical reduction is commonly used for the reduction of hexavalent chromium to the trivalent state.
DEACT	Deactivation to remove the hazardous characteristics of a waste due to its ignitability, corrosivity, and/or reactivity.
FSUBS	Fuel substitution in units operated in accordance with applicable technical operating requirements
HLVIT	Vitrification of high level mixed radioactive wastes in units in compliance with all applicable radioactive protection requirements under control of the Nuclear Regulatory Commission.
IMERC.	Incineration of wastes containing organics and mercury in units operated in accordance with the technical operating requirements of 40 CFR part 264 subpart O and part 265 subpart O. All wastewater and nonwastewater residues derived from this process must then comply with the corresponding treatment standards per waste code with consideration of any applicable subcategories (e.g., High or Low Mercury Subcategories).
INCIN.	Incineration in units operated in accordance with the technical operating requirements of 40 CFR part 264 subpart O and part 265 subpart O.
LLEXT	Liquid-liquid extraction (often referred to as solvent extraction) of organics from liquid wastes into an immiscible solvent for which the hazardous constituents have a greater solvent affinity, resulting in an extract high in organics that must undergo either incineration, reuse as a fuel, or other recovery/reuse and a raffinate (extracted liquid waste) proportionately low in organics that must undergo further treatment as specified in the standard.
MACRO	Macroencapsulation with surface coating materials such as polymeric organics (e.g., resins and plastics) or with a jacket of inert inorganic materials to substantially reduce surface exposure to potential leaching media. Macroencapsulation specifically does not include any material that would be classified as a tank or container according to 40 CFR 260.10.
NEUTR	Neutralization with the following reagents (or waste reagents) or combinations of reagents: (1) Acids; (2) bases; or (3) water (including wastewaters) resulting in a pH greater than 2 but less than 12.5 as measured in the aqueous residuals.
NLDBR.	No land disposal based on recycling.
PRECP	Chemical precipitation of metals and other inorganics as insoluble precipitates of oxides, hydroxides, carbonates, sulfides, sulfates, chlorides, flourides, or phosphates. The following reagents (or waste reagents) are typically used alone or in combination: (1) Lime (i.e., containing oxides and/or hydroxides of calcium and/or magnesium; (2) caustic (i.e., sodium and/or potassium hydroxides; (3) soda ash (i.e., sodium carbonate), (4) sodium sulfide; (5) ferric sulfate or ferric chloride, (6) alum, or (7) sodium sulfate. Additional flocculating, coagulation or similar reagents/processes that enhance sludge dewatering characteristics are not precluded from use.
RBERY	Thermal recovery of Beryllium
RCGAS	Recovery/reuse of compressed gases including techniques such as reprocessing of the gases for reuse/resale; filtering/adsorption of impurities, remixing for direct reuse or resale, and use of the gas as a fuel source.
RCORR	Recovery of acids or bases utilizing one or more of the following recovery technologies: (1) Distillation (i.e., thermal concentration), (2) ion exchange; (3) resin or solid adsorption, (4) reverse osmosis; and/or (5) incineration for the recovery of acid—Note, this does not preclude the use of other physical phase separation or concentration techniques such as decantation, filtration (including ultrafiltration), and centrifugation, when used in conjunction with the above listed recovery technologies
RLEAD.	Thermal recovery of lead in secondary lead smelters.

TABLE 1.—TECHNOLOGY CODES AND DESCRIPTION OF TECHNOLOGY-BASED STANDARDS—Continued

Technology code	Description of technology-based standards
RMERC	Retorting or roasting in a thermal processing unit capable of volatilizing mercury and subsequently condensing the volatilized mercury for recovery. The retorting or roasting unit (or facility) must be subject to one or more of the following: (a) a National Emissions Standard for Hazardous Air Pollutants (NESHAP) for mercury, (b) a Best Available Control Technology (BACT) or a Lowest Achievable Emission Rate (LAER) standard for mercury imposed pursuant to a Prevention of Significant Deterioration (PSD) permit, or (c) a state permit that establishes emission limitations (within meaning of section 302 of the Clean Air Act) for mercury. All wastewater and nonwastewater residues derived from this process must then comply with the corresponding treatment standards per waste code with consideration of any applicable subcategories (e.g., High or Low Mercury Subcategories).
RMETL	Recovery of metals or inorganics utilizing one or more of the following direct physical/removal technologies: (1) Ion exchange, (2) resin or solid (i.e., zeolites) adsorption; (3) reverse osmosis, (4) chelation/solvent extraction; (5) freeze crystalization; (6) ultrafiltration and/or (7) simple precipitation (i.e., crystalization)—*Note:* This does not preclude the use of other physical phase separation or concentration techniques such as decantation, filtration (including ultrafiltration), and centrifugation, when used in conjunction with the above listed recovery technologies.
RORGS	Recovery of organics utilizing one or more of the following technologies: (1) Distillation, (2) thin film evaporation, (3) steam stripping; (4) carbon adsorption; (5) critical fluid extraction, (6) liquid-liquid extraction, (7) precipitation/crystalization (including freeze crystallization); or (8) chemical phase separation techniques (i.e., addition of acids, bases, demulsifiers, or similar chemicals).—*Note:* this does not preclude the use of other physical phase separation techniques such as a decantation, filtration (including ultrafiltration), and centrifugation, when used in conjunction with the above listed recovery technologies.
RTHRM	Thermal recovery of metals or inorganics from nonwastewaters in units identified as industrial furnaces according to 40 CFR 260.10 (1), (6), (7), (11), and (12) under the definition of "industrial furnaces"
RZINC	Recmelting in high temperature metal recovery units for the purpose of recovery of zinc.
STABL	Stabilization with the following reagents (or waste reagents) or combinations of reagents. (1) Portland cement; or (2) lime/pozzolans (e.g., fly ash and cement kiln dust)—this does not preclude the addition of reagents (e.g., iron salts, silicates, and clays) designed to enhance the set/cure time and/or compressive strength, or to overall reduce the leachability of the metal or inorganic.
SSTRP	Steam stripping of organics from liquid wastes utilizing direct application of steam to the wastes operated such that liquid and vapor flow rates, as well as, temperature and pressure ranges have been optimized, monitored, and maintained. These operating parameters are dependent upon the design parameters of the unit such as, the number of separation stages and the internal column design. Thus, resulting in a concensed extract high in organics that must undergo either incineration, reuse as a fuel, or other recovery/reuse and an extracted wastewater that must undergo further treatment as specified in the standard.
WETOX	Wet air oxidation performed in units operated such that a surrogate compound or indicator parameter has been substantially reduced in concentration in the residuals (e.g., Total Organic Carbon can often be used as an indicator parameter for the oxidation of many organic constituents that cannot be directly analyzed in wastewater residues).
WTRRX	Controlled reaction with water for highly reactive inorganic or organic chemicals with precautionary controls for protection of workers from potential violent reactions as well as precautionary controls for potential emissions of toxic/ignitable levels of gases released during the reaction.

Note 1: When a combination of these technologies (i.e., a treatment train) is specified as a single treatment standard, the order of application is specified in §268.42, Table 2 by indicating the five letter technology code that must be applied first, then the designation "to," (an abbreviation for "followed by"), then the five letter technology code for the technology that must be applied next, and so on.

Note 2: When more than one technology (or treatment train) are specified as *alternative* treatment standards, the five letter technology codes (or the treatment trains) are separated by a semicolon (;) with the last technology preceded by the word "OR". This indicates that any one of these BDAT technologies or treatment trains can be used for compliance with the standard.

268.42 TABLE 2.—TECHNOLOGY-BASED STANDARDS BY RCRA WASTE CODE

Waste code	See also	Waste descriptions and/or treatment subcategory	CAS No. for regulated hazardous constituents	Technology code	
				Wastewaters	Nonwastewaters
D001	Table CCWE in 268.41 and Table CCW in 268.43	All descriptions based on 40 CFR 261.21, except for the §261.21(a)(1) High TOC subcategory, managed in non-CWA/non-CWA-equivalent/non-Class SDWA systems	NA	DEACT, and meet F039, or FSUBS, RORGS, or INCIN	DEACT, and meet F039; or FSUBS, RORGS, or INCIN

268.42 TABLE 2.—TECHNOLOGY-BASED STANDARDS BY RCRA WASTE CODE—Continued

Waste code	See also	Waste descriptions and/or treatment subcategory	CAS No. for regulated hazardous constituents	Technology code	
				Wastewaters	Nonwastewaters
D001	NA	All descriptions based on 40 CFR 261.21, except for the §261.21(a)(1) High TOC subcategory, managed in CWA, CWA-equivalent, or Class I SDWA systems.	NA	DEACT	DEACT.
D001	NA	All descriptions based on 40 CFR 261.21(a)(1)—High TOC Ignitable Liquids Subcategory—Greater than or equal to 10% total organic carbon	NA	NA	FSUBS, RORGS, or INCIN.
D002	Table CCWE in 268.41 and Table CCW in 268.43.	Acid, alkaline, and other subcategory based on 261.22 managed in non-CWA/non-CWA-equivalent/non-Class I SDWA systems.	NA	DEACT and meet F039	DEACT and meet F039
D002	NA	Acid, alkaline, and other subcategory based on 261.22 managed in CWA, CWA-equivalent, or Class I SDWA systems.	NA	DEACT	DEACT.
D003	NA	Reactive Sulfides based on 261.23(a)(5).	NA	DEACT but not including dilution as a substitute for adequate treatment.	DEACT but not including dilution as a substitute for adequate treatment.
D003	NA	Explosives based on 261.23(a)(6), (7), and (8).	NA	DEACT	DEACT.
D003	NA	Water reactives based on 261.23(a)(2), (3), and (4).	NA	NA	DEACT.
D003	NA	Other reactives based on 261.23(a)(1).	NA	DEACT	DEACT
D006	NA	Cadmium containing batteries.	7440-43-9	NA	RTHRM.
D008	NA	Lead acid batteries (Note: This standard only applies to lead acid batteries that are identified as RCRA hazardous wastes and that are not excluded elsewhere from regulation under the land disposal restrictions of 40 CFR 268 or exempted under other EPA regulations (see 40 CFR 266.80.).	7439-92-1	NA	RLEAD.
D009	Table CCWE in 268.41 and Table CCW in 268.43.	Mercury (High Mercury Subcategory—greater than or equal to 260 mg/kg total Mercury—contains mercury and organics (and are not incinerator residues)).	7439-97-6	NA	IMERC, or RMERC.

Waste code	See also	Waste descriptions and/or treatment subcategory	CAS No. for regulated hazardous constituents	Technology code	
				Wastewaters	Nonwastewaters
D009	Table CCWE in 268.41 and Table CCW in 268.43.	Mercury: (High Mercury Subcategory—greater than or equal to 260 mg/kg total Mercury—inorganics (including incinerator residues and residues from RMERC)).	7439-97-6	NA	RMERC.
D012	Table CCW in 268.43.	Endrin	72-20-8	BIODG; or INCIN	NA.
D013	Table CCW in 268.43.	Lindane	58-89-9	CARBN; or INCIN	NA.
D014	Table CCW in 268.43.	Methoxychlor	72-43-5	WETOX; or INCIN	NA.
D015	Table CCW in 268.43.	Toxaphene	8001-35-1	BIODG; or INCIN	NA.
D016	Table CCW in 268.43.	2,4-D	94-75-7	CHOXD; BIODG; or INCIN.	NA.
D017	Table CCW in 268.43.	2,4,5-TP	93-72-1	CHOXD; or INCIN	NA.
F005	Table CCWE in 268.41 and Table CCW in 268.43.	2-Nitropropane	79-46-9	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN
F005	Table CCWE in 268.41 and Table CCW in 268.43.	2-Ethoxyethanol	110-80-5	BIODG; or INCIN	INCIN.
F024	Table CCWE in 268.41 and Table CCW in 268.43.		NA	INCIN	INCIN.
K025	NA	Distillation bottoms from the production of nitrobenzene by the nitration of benzene.	NA	LLEXT fb SSTRP fb CARBN; or INCIN.	INCIN.
K026	NA	Stripping still tails from the production of methyl ethyl pyridines.	NA	INCIN	INCIN.
K027	NA	Centrifuge and distillation residues from toluene diisocyanate production.	NA	CARBN; or INCIN	FSUBS; or INCIN.
K039	NA	Filter cake from the filtration of diethylphosphorodithioic acid in the production of phorate.	NA	CARBN; or INCIN	FSUBS; or INCIN.
K044	NA	Wastewater treatment sludges from the manufacturing and processing of explosives.	NA	DEACT	DEACT.
K045	NA	Spent carbon from the treatment of wastewater containing explosives.	NA	DEACT	DEACT.
K047	NA	Pink/red water from TNT operations.	NA	DEACT	DEACT.
K069	Table CCWE in 268.41 and Table CCW in 268.43.	Emission control dust/sludge from secondary lead smelting: Non-Calcium Sulfate Subcategory.	NA	NA	RLEAD.

Waste code	See also	Waste descriptions and/or treatment subcategory	CAS No. for regulated hazardous constituents	Technology code	
				Wastewaters	Nonwastewaters
K106	Table CCWE in 268.41 and Table CCW in 268.43.	Wastewater treatment sludge from the mercury cell process in chlorine production: (High Mercury Subcategory—greater than or equal to 260 mg/kg total mercury).	NA	NA	RMERC.
K107		Column bottoms from product separation from the production of 1,1-dimethylhydrazine (UDMH) from carboxylic acid hydrazides.	NA	INCIN; or CHOXD fb, CARBN; or BIODG fb CARBN.	INCIN.
K108		Condensed column overheads from product separation and condensed reactor vent gases from the production of 1,1-dimethylhydrazine (UDMH) from carboxylic acid hydrazides.	NA	INCIN; or CHOXD fb, CARBN; or BIODG fb CARBN.	INCIN.
K109		Spent filter cartridges from product purification from the production of 1,1-dimethylhydrazine (UDMH) from carboxylic acid hydrazides.	NA	INCIN; or CHOXD fb, CARBN; or BIODG fb CARBN.	INCIN.
K110		Condensed column overheads from intermediate separation from the production of 1,1-dimethylhydrazine (UDMH) from carboxylic acid hydrazides.	NA	INCIN; or CHOXD fb, CARBN; or BIODG fb CARBN.	INCIN.
K112		Reaction by-product water from the drying column in the production of toluenediamine via hydrogenation of dinitrotoluene.	NA	INCIN; or CHOXD fb, CARBN; or BIODG fb CARBN.	INCIN.
K113	NA	Condensed liquid light ends from the purification of toluenediamine in the production of toluenediamine via hydrogenation of dinitrotoluene.	NA	CARBN; or INCIN	FSUBS; or INCIN.
K114	NA	Vicinals from the purification of toluenediamine in the production of toluenediamine via hydrogenation of dinitrotoluene.	NA	CARBN; or INCIN	FSUBS; or INCIN.
K115	NA	Heavy ends from the purification of toluenediamine in the production of toluenediamine via hydrogenation of dinitrotoluene.	NA	CARBN; or INCIN	FSUBS; or INCIN.

268.42 TABLE 2.—TECHNOLOGY-BASED STANDARDS BY RCRA WASTE CODE—Continued

Waste code	See also	Waste descriptions and/or treatment subcategory	CAS No. for regulated hazardous constituents	Technology code	
				Wastewaters	Nonwastewaters
K116	NA	Organic condensate from the solvent recovery column in the production of toluene diisocyanate via phosgenation of toluenediamine.	NA	CARBN, or INCIN	FSUBS, or INCIN
K123		Process wastewater (including supernates, filtrates, and washwaters) from the production of ethylenebisdithiocarbamic acid and its salts.	NA	INCIN, or CHOXD fb (BIODG or CARBN)	INCIN
K124		Reactor vent scrubber water from the production of ethylenebisdithiocarbamic acid and its salts.	NA	INCIN, or CHOXD fb (BIODG or CARBN)	INCIN
K125		Filtration, evaporation, and centrifugation solids from the production of ethylenebisdithiocarbamic acid and its salts.	NA	INCIN; or CHOXD fb (BIODG or CARBN).	INCIN
K126		Baghouse dust and floor sweepings in milling and packaging operations from the production or formulation of ethylene bisdithiocarbamic acid and its salts.	NA	INCIN, or CHOXD fb (BIODG or CARBN).	INCIN.
P001	NA	Warfarin (>0.3%)	81-81-2	(WETOX or CHOXD) fb CARBN; or INCIN.	FSUBS, or INCIN
P002	NA	1-Acetyl-2-thiourea	591-08-2	(WETOX or CHOXD) fb CARBN, or INCIN	INCIN.
P003	NA	Acrolein	107-02-8	NA	FSUBS, or INCIN.
P005	NA	Allyl alcohol	107-18-6	(WETOX or CHOXD) fb CARBN, or INCIN.	FSUBS; or INCIN
P006	NA	Aluminum phosphide	20859-73-8	CHOXD, CHRED, or INCIN	CHOXD; CHRED, or INCIN
P007	NA	5-Aminoethyl 3-Isoxazolol.	2763-96-4	(WETOX or CHOXD) fb CARBN, or INCIN	INCIN.
P008	NA	4-Aminopyridine	504-24-5	(WETOX or CHOXD) fb CARBN; or INCIN	INCIN.
P009	NA	Ammonium picrate	131-74-8	CHOXD, CHRED, CARBN; BIODG, or INCIN	FSUBS; CHOXD, CHRED, or INCIN
P014	NA	Thiophenol (Benzene thiol).	108-98-5	(WETOX or CHOXD) fb CARBN, or INCIN.	INCIN.
P015	NA	Beryllium dust	7440-41-7	RMETL, or RTHRM	RMETL, or RTHRM.
P016	NA	Bis(chloromethyl)ether	542-88-1	(WETOX or CHOXD) fb CARBN, or INCIN	INCIN
P017	NA	Bromoacetone	598-31-2	(WETOX or CHOXD) fb CARBN, or INCIN	INCIN.
P018	NA	Brucine	357-57-3	(WETOX or CHOXD) fb CARBN, or INCIN	INCIN.
P022	Table CCW in 268.43	Carbon disulfide	75-15-0	NA	INCIN
P023	NA	Chloroacetaldehyde	107-20-0	(WETOX or CHOXD) fb CARBN, or INCIN	INCIN
P026	NA	1-(o-Chlorophenyl) thiourea.	5344-82-1	(WETOX or CHOXD) fb CARBN, or INCIN	INCIN
P027	NA	3-Chloropropionitrile	542-76-7	(WETOX or CHOXD) fb CARBN, or INCIN	INCIN
P028	NA	Benzyl chloride	100-44-7	(WETOX or CHOXD) fb CARBN, or INCIN	INCIN
P031	NA	Cyanogen	460-19-5	CHOXD, WETOX or INCIN	CHOXD, WETOX, or INCIN

268.42 TABLE 2.—TECHNOLOGY-BASED STANDARDS BY RCRA WASTE CODE—Continued

Waste code	See also	Waste descriptions and/or treatment subcategory	CAS No. for regulated hazardous constituents	Technology code	
				Wastewaters	Nonwastewaters
P033	NA	Cyanogen chloride	506-77-4	CHOXD, WETOX or INCIN	CHOXD; WETOX, or INCIN.
P034	NA	2-Cyclohexyl-4,6-dinitrophenol.	131-89-5	(WETOX or CHOXD) fb CARBN, or INCIN.	INCIN.
P040	NA	O,O-Diethyl O-pyrazinyl phosphorothioate.	297-97-2	CARBN, or INCIN	FSUBS; or INCIN
P041	NA	Diethyl-p-nitrophenyl phosphate.	311-45-5	CARBN, or INCIN	FSUBS, or INCIN.
P042	NA	Epinephrine	51-43-4	(WETOX or CHOXD) fb CARBN; or INCIN	INCIN.
P043	NA	Diisopropyl fluorophosphate (DFP)	55-91-4	CARBN, or INCIN	FSUBS; or INCIN
P044	NA	Dimethoate	60-51-5	CARBN, or INCIN	FSUBS or INCIN
P045	NA	Thiofanox	39196-18-4	(WETOX or CHOXD) fb CARBN; or INCIN	INCIN.
P046	NA	alpha, alpha-Dimethylphenethylamine.	122-09-8	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
P047	NA	4,6-Dinitro-o-cresol salts	534-52-1	(WETOX or CHOXD) fb CARBN, or INCIN.	INCIN.
P049	NA	2,4-Dithiobiuret	541-53-7	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
P054	NA	Aziridine	151-56-4	(WETOX or CHOXD) fb CARBN, or INCIN	INCIN.
P056	Table CCW in 268.43	Fluorine	7782-41-4	NA	ADGAS fb NEUTR.
P057	NA	Fluoroacetamide	640-19-7	(WETOX or CHOXD) fb CARBN, or INCIN	INCIN.
P058	NA	Fluoroacetic acid, sodium salt.	62-74-8	(WETOX or CHOXD) fb CARBN, or INCIN.	INCIN.
P062	NA	Hexaethyltetraphosphate	757-58-4	CARBN; or INCIN	FSUBS; or INCIN.
P064	NA	Isocyanic acid, ethyl ester.	624-83-9	(WETOX or CHOXD) fb CARBN; or INCIN	INCIN
P065	Table CCWE in 268.41 and Table CCW in 268.43.	Mercury fulminate. (High Mercury Subcategory—greater than or equal to 260 mg/kg total Mercury—either incinerator residues or residues from RMERC).	628-86-4	NA	RMERC.
P065	Table CCWE in 268.41 and Table CCW in 268.43.	Mercury fulminate (All Nonwastewaters that are not incinerator residues or are not residues from RMERC, regardless of Mercury Content).	628-86-4	NA	IMERC.
P066	NA	Methomyl	16752-77-5	(WETOX or CHOXD) fb CARBN, or INCIN.	INCIN.
P067	NA	2-Methylaziridine	75-55-8	(WETOX or CHOXD) fb CARBN, or INCIN.	INCIN.
P068	NA	Methyl hydrazine	60-34-4	CHOXD; CHRED, CARBN, BIODG; or INCIN.	FSUBS; CHOXD, CHRED, or INCIN.
P069	NA	Methyllactonitrile	75-86-5	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
P070	NA	Aldicarb	116-06-3	(WETOX or CHOXD) fb CARBN, or INCIN	INCIN.
P072	NA	1-Naphthyl-2-thiourea	86-88-4	(WETOX or CHOXD) fb CARBN, or INCIN.	INCIN.
P075	NA	Nicotine and salts	154-11-5	(WETOX or CHOXD) fb CARBN, or INCIN.	INCIN
P076	NA	Nitric oxide	10102-43-9	ADGAS	ADGAS
P078	NA	Nitrogen dioxide	10102-44-0	ADGAS	ADGAS.
P081	NA	Nitroglycerin	55-63-0	CHOXD, CHRED, CARBN, BIODG; or INCIN	FSUBS, CHOXD, CHRED, or INCIN
P082	Table CCW in 268.43.	N-Nitrosodimethylamine	62-75-9	NA	INCIN

268.42 TABLE 2.—TECHNOLOGY-BASED STANDARDS BY RCRA WASTE CODE—Continued

Waste code	See also	Waste descriptions and/or treatment subcategory	CAS No. for regulated hazardous constituents	Technology code	
				Wastewaters	Nonwastewaters
P084	NA	N-Nitrosomethylvinylamine.	4549-40-0	(WETOX or CHOXD) fb CARBN; OR INCIN.	INCIN.
P085	NA	Octamethylpyrophosphoramide.	152-16-9	CARBN; or INCIN	FSUBS; or INCIN.
P087	NA	Osmium tetroxide	20816-12-0	RMETL; or RTHRM	RMETL; or RTHRM.
P088	NA	Endothall	145-73-3	(WETOX or CHOXD) fb CARBN; or INCIN.	FSUBS; or INCIN.
P092	Table CCWE in 268.41 and Table CCW in 268.43.	Phenyl mercury acetate: (High Mercury Subcategory—greater than or equal to 260 mg/kg total Mercury—either incinerator residues or residues from RMERC).	62-38-4	NA	RMERC.
P092	Table CCWE in 268.41 and Table CCW in 268.43.	Phenyl mercury acetate: (All nonwastewaters that are not incinerator residues and are not residues from RMERC; regardless of Mercury Content).	62-38-4	NA	IMERC; or RMERC.
P093	NA	N-Phenylthiourea	103-85-5	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
P095	NA	Phosgene	75-44-5	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
P096	NA	Phosphine	7803-51-2	CHOXD; CHRED; or INCIN.	CHOXD; CHRED; or INCIN.
P102	NA	Propargyl alcohol	107-19-7	(WETOX or CHOXD) fb CARBN; or INCIN.	FSUBS; or INCIN.
P105	NA	Sodium azide	26628-22-8	CHOXD; CHRED; CARBN; BIODG; or INCIN.	FSUBS, CHOXD; CHRED; or INCIN.
P108	NA	Strychnine and salts	157-24-9	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
P109	NA	Tetraethyldithiopyrophosphate.	3689-24-5	CARBN; or INCIN	FSUBS; or INCIN.
P112	NA	Tetranitromethane	509-14-8	CHOXD; CHRED; CARBN; BIODG; or INCIN.	FSUBS, CHOXD; CHRED; or INCIN.
P113	Table CCW in 268.43.	Thallic oxide	1314-32-5	NA	RTHRM; or STABL.
P115	Table CCW in 268.43.	Thallium (I) sulfate	7446-18-6	NA	RTHRM; or STABL.
P116	NA	Thiosemicarbazide	79-19-6	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
P118	NA	Trichloromethanethiol	75-70-7	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
P119	Table CCW in 268.43.	Ammonium vanadate	7803-55-6	NA	STABL.
P120	Table CCW in 268.43.	Vanadium pentoxide	1314-62-1	NA	STABL.
P122	NA	Zinc Phosphide (<10%)	1314-84-7	CHOXD; CHRED; or INCIN.	CHOXD; CHRED; or INCIN.
U001	NA	Acetaldehyde	75-07-0	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
U003	Table CCW in 268.43.	Acetonitrile	75-05-8	NA	INCIN.
U006	NA	Acetyl Chloride	75-36-5	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
U007	NA	Acrylamide	79-06-1	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
U008	NA	Acrylic acid	79-10-7	(WETOX or CHOXD) fb CARBN; or INCIN.	FSUBS; or INCIN.
U010	NA	Mitomycin C	50-07-7	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
U011	NA	Amitrole	61-82-5	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
U014	NA	Auramine	492-80-8	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.

268.42 TABLE 2.—TECHNOLOGY-BASED STANDARDS BY RCRA WASTE CODE—Continued

Waste code	See also	Waste descriptions and/or treatment subcategory	CAS No. for regulated hazardous constituents	Technology code	
				Wastewaters	Nonwastewaters
U015	NA	Azaserine	115-02-6	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
U016	NA	Benz(c)acridine	225-51-4	(WETOX or CHOXD) fb CARBN; or INCIN.	FSUBS; or INCIN.
U017	NA	Benzal chloride	98-87-3	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
U020	NA	Benzenesulfonyl chloride.	98-09-9	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
U021	NA	Benzidine	92-87-5	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
U023	NA	Benzotrichloride	98-07-7	CHOXD; CHRED; CARBN; BIODG; or INCIN.	FSUBS; CHOXD; CHRED; or INCIN.
U026	NA	Chlornaphazin	494-03-1	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
U033	NA	Carbonyl fluoride	353-50-4	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
U034	NA	Trichloroacetaldehyde (Chloral).	75-87-6	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
U035	NA	Chlorambucil	305-03-3	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
U038	Table CCW in 268.43.	Chlorobenzilate	510-15-6	NA	INCIN.
U041	NA	1-Chloro-2,3-epoxypropane (Epichlorohydrin).	106-89-8	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
U042	Table CCW in 268.43.	2-Chloroethyl vinyl ether	110-75-8	NA	INCIN.
U046	NA	Chloromethyl methyl ether.	107-30-2	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
U049	NA	4-Chloro-o-toluidine hydrochloride.	3165-93-3	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
U053	NA	Crotonaldehyde	4170-30-3	(WETOX or CHOXD) fb CARBN; or INCIN.	FSUBS; or INCIN.
U055	NA	Cumene	98-82-8	(WETOX or CHOXD) fb CARBN; or INCIN.	FSUBS; or INCIN.
U056	NA	Cyclohexane	110-82-7	(WETOX or CHOXD) fb CARBN; or INCIN.	FSUBS; or INCIN.
U057	Table CCW in 268.43.	Cyclohexanone	108-94-1	NA	FSUBS; or INCIN.
U058	NA	Cyclophosphamide	50-18-0	CARBN; or INCIN	FSUBS; or INCIN.
U059	NA	Daunomycin	20830-81-3	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
U062	NA	Diallate	2303-16-4	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
U064	NA	1,2,7,8-Dibenzopyrene	189-55-9	(WETOX or CHOXD) fb CARBN or INCIN.	FSUBS; or INCIN.
U073	NA	3,3-Dichlorobenzidine	91-94-1	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
U074	NA	cis-1,4-Dichloro-2-butylene.	1476-11-5	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
		trans-1,4-Dichloro-2-butylene		WETOX or CHOXD) fb CARBN; or INCIN	
U085	NA	1,2:3,4-Diepoxybutane	1464-53-5	(WETOX or CHOXD) fb CARBN; or INCIN.	FSUBS; or INCIN.
U086	NA	N,N-Diethylhydrazine	1615-80-1	CHOXD; CHRED; CARBN; BIODG; or INCIN.	FSUBS; CHOXD; CHRED; or INCIN.
U087	NA	0,0-Diethyl S-methyldithiophosphate.	3288-58-2	CARBN; or INCIN	FSUBS; or INCIN.
U089	NA	Diethyl stilbestrol	56-53-1	(WETOX or CHOXD) fb CARBN; or INCIN.	FSUBS; or INCIN.
U090	NA	Dihydrosafrole	94-58-6	(WETOX or CHOXD) fb CARBN; or INCIN.	FSUBS; or INCIN.
U091	NA	3,3-Dimethoxybenzidine.	119-90-4	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
U092	NA	Dimethylamine	124-40-3	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
U093	Table CCW in 268.43.	p-Dimethylaminoazobenzene.	621-90-9	NA	INCIN.

268.42 TABLE 2.—TECHNOLOGY-BASED STANDARDS BY RCRA WASTE CODE—Continued

Waste code	See also	Waste descriptions and/or treatment subcategory	CAS No. for regulated hazardous constituents	Technology code	
				Wastewaters	Nonwastewaters
U094	NA	7,12-Dimethyl benz(a)anthracene	57-97-6	(WETOX or CHOXD) fb CARBN; or INCIN	FSUBS, or INCIN
U095	NA	3,3'-Dimethylbenzidine	119-93-7	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
U096	NA	a,a-Dimethyl benzyl hydroperoxide	80-15-9	CHOXD, CHRED, CARBN, BIODG, or INCIN	FSUBS, CHOXD, CHRED, or INCIN
U097	NA	Dimethylcarbomyl chloride	79-44-7	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
U098	NA	1,1-Dimethylhydrazine	57-14-7	CHOXD, CHRED, CARBN, BIODG; or INCIN.	FSUBS, CHOXD, CHRED, or INCIN
U099	NA	1,2-Dimethylhydrazine	540-73-8	CHOXD, CHRED, CARBN; BIODG, or INCIN	FSUBS, CHOXD, CHRED, or INCIN
U103	NA	Dimethyl sulfate	77-78-1	CHOXD, CHRED, CARBN, BIODG, or INCIN.	FSUBS, CHOXD, CHRED; or INCIN.
U109	NA	1,2-Diphenylhydrazine	122-66-7	CHOXD, CHRED; CARBN, BIODG, or INCIN.	FSUBS, CHOXD; CHRED, or INCIN
U110	NA	Dipropylamine	142-84-7	(WETOX or CHOXD) fb CARBN, or INCIN.	INCIN.
U113	NA	Ethyl acrylate	140-88-5	(WETOX or CHOXD) fb CARBN; or INCIN.	FSUBS, or INCIN
U114	NA	Ethylene bisdithiocarbamic acid.	111-54-6	(WETOX or CHOXD) fb CARBN; or INCIN	INCIN
U115	NA	Ethylene oxide	75-21-8	(WETOX or CHOXD) fb CARBN, or INCIN.	CHOXD, or INCIN
U116	NA	Ethylene thiourea	96-45-7	(WETOX or CHOXD) fb CARBN, or INCIN.	INCIN
U119	NA	Ethyl methane sulfonate	62-50-0	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN
U122	NA	Formaldehyde	50-00-0	(WETOX or CHOXD) fb CARBN; or INCIN.	FSUBS, or INCIN
U123	NA	Formic acid	64-18-6	(WETOX or CHOXD) fb CARBN, or INCIN.	FSUBS, or INCIN.
U124	NA	Furan	110-00-9	(WETOX or CHOXD) fb CARBN; or INCIN.	FSUBS, or INCIN
U125	NA	Furfural	98-01-1	(WETOX or CHOXD) fb CARBN; or INCIN	FSUBS, or INCIN
U126	NA	Glycidaldehyde	765-34-4	(WETOX or CHOXD) fb CARBN, or INCIN	FSUBS, or INCIN.
U132	NA	Hexachlorophene	70-30-4	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN
U133	NA	Hydrazine	302-01-2	CHOXD; CHRED, CARBN, BIODG, or INCIN.	FSUBS; CHOXD, CHRED; or INCIN.
U134	Table CCW in 268.43	Hydrogen Flouride	7664-39-3	NA	ADGAS fb NEUTR, or NEUTR
U135	NA	Hydrogen Sulfide	7783-06-4	CHOXD, CHRED, or INCIN.	CHOXD, CHRED, or INCIN
U143	NA	Lasiocarpine	303-34-4	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
U147	NA	Maleic anhydride	108-31-6	(WETOX or CHOXD) fb CARBN, or INCIN	FSUBS, or INCIN
U148	NA	Maleic hydrazide	123-33-1	(WETOX or CHOXD) fb CARBN, or INCIN	INCIN
U149	NA	Malononitrile	109-77-3	(WETOX or CHOXD) fb CARBN, or INCIN	INCIN.
U150	NA	Melphalan	148-82-3	(WETOX or CHOXD) fb CARBN, or INCIN	INCIN
U151	Table CCWE in 268.41 and Table CCW in 268.43	Mercury (High Mercury Subcategory—greater than or equal to 260 mg/kg total Mercury)	7439-97-6	NA	RMERC
U153	NA	Methane thiol	74-93-1	(WETOX or CHOXD) fb CARBN, or INCIN.	INCIN

268.42 TABLE 2.—TECHNOLOGY-BASED STANDARDS BY RCRA WASTE CODE—Continued

Waste code	See also	Waste descriptions and/or treatment subcategory	CAS No. for regulated hazardous constituents	Technology code	
				Wastewaters	Nonwastewaters
U154	NA	Methanol	67-56-1	(WETOX or CHOXD) fb CARBN; or INCIN	FSUBS, or INCIN
U156	NA	Methyl chlorocarbonate	79-22-1	(WETOX or CHOXD) fb CARBN, or INCIN	INCIN
U160	NA	Methyl ethyl ketone peroxide	1338-23-4	CHOXD, CHRED, CARBN, BIODG, or INCIN.	FSUBS, CHOXD, CHRED, or INCIN.
U163	NA	N-Methyl N'-nitro N-Nitrosoguanidine.	70-25-7	(WETOX or CHOXD) fb CARBN, or INCIN.	INCIN.
U164	NA	Methylthiouracil	56-04-2	(WETOX or CHOXD) fb CARBN, or INCIN	INCIN.
U166	NA	1,4-Naphthoquinone	130-15-4	(WETOX or CHOXD) fb CARBN, or INCIN.	FSUBS; or INCIN.
U167	NA	1-Naphthylamine	134-32-7	(WETOX or CHOXD) fb CARBN, or INCIN.	INCIN.
U168	Table CCW in 268.43.	2-Naphthylamine	91-59-8	NA	INCIN.
U171	NA	2-Nitropropane	79-46-9	(WETOX or CHOXD) fb CARBN, or INCIN.	INCIN.
U173	NA	N-Nitroso-di-n-ethanolamine	1116-54-7	(WETOX or CHOXD) fb CARBN, or INCIN.	INCIN
U176	NA	N-Nitroso-N-ethylurea	759-73-9	(WETOX or CHOXD) fb CARBN; or INCIN	INCIN
U177	NA	N-Nitroso-N-methylurea	684-93-5	(WETOX or CHOXD) fb CARBN; or INCIN	INCIN.
U178	NA	N-Nitroso-N-methylurethane.	615-53-2	(WETOX or CHOXD) fb CARBN, or INCIN.	INCIN
U182	NA	Paraldehyde	123-63-7	(WETOX or CHOXD) fb CARBN; or INCIN	FSUBS; or INCIN
U184	NA	Pentachloroethane	76-01-7	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN.
U186	NA	1,3-Pentadiene	504-60-9	(WETOX or CHOXD) fb CARBN; or INCIN	FSUBS; or INCIN.
U189	NA	Phosphorus sulfide	1314-80-3	CHOXD, CHRED, or INCIN.	CHOXD; CHRED, or INCIN.
U191	NA	2-Picoline	109-06-8	(WETOX or CHOXD) fb CARBN, or INCIN.	INCIN
U193	NA	1,3-Propane sultone	1120-71-4	(WETOX or CHOXD) fb CARBN; or INCIN	INCIN.
U194	NA	n-Propylamine	107-10-8	(WETOX or CHOXD) fb CARBN, or INCIN	INCIN.
U197	NA	p-Benzoquinone	106-51-4	(WETOX or CHOXD) fb CARBN, or INCIN.	FSUBS; or INCIN
U200	NA	Reserpine	50-55-5	(WETOX or CHOXD) fb CARBN; or INCIN	INCIN.
U201	NA	Resorcinol	108-46-3	(WETOX or CHOXD) fb CARBN, or INCIN	FSUBS; or INCIN.
U202	NA	Saccharin and salts	81-07-2	(WETOX or CHOXD) fb CARBN, or INCIN.	INCIN
U206	NA	Streptozotocin	18883-66-4	(WETOX or CHOXD) fb CARBN; or INCIN.	INCIN
U213	NA	Tetrahydrofuran	109-99-9	(WETOX or CHOXD) fb CARBN, or INCIN.	FSUBS, or INCIN
U214	Table CCW in 268.43.	Thallium (I) acetate	563-68-8	NA	RTHRM, or STABL.
U215	Table CCW in 268.43	Thallium (I) carbonate	6533-73-9	NA	RTHRM, or STABL.
U216	Table CCW in 268.43	Thallium (I) chloride	7791-12-0	NA	RTHRM, or STABL.
U217	Table CCW in 268.43	Thallium (I) nitrate	10102-45-1	NA	RTHRM or STABL.
U218	NA	Thioacetamide	62-55-5	(WETOX or CHOXD) fb CARBN, or INCIN.	INCIN
U219	NA	Thiourea	62-56-6	(WETOX or CHOXD) fb CARBN, or INCIN.	INCIN.
U221	NA	Toluenediamine	25376-45-8	CARBN, or INCIN	FSUBS, or INCIN.
U222	NA	o-Toluidine hydrochloride	636-21-5	(WETOX or CHOXD) fb CARBN, or INCIN	INCIN
U223	NA	Toluene diisocyanate	26471-62-5	CARBN, or INCIN	FSUBS, or INCIN

268.42 TABLE 2 —TECHNOLOGY-BASED STANDARDS BY RCRA WASTE CODE—Continued

Waste code	See also	Waste descriptions and/or treatment subcategory	CAS No for regulated hazardous constituents	Technology code	
				Wastewaters	Nonwastewaters
U234	NA	sym-Trinitrobenzene	99-35-4	(WETOX or CHOXD) fb CARBN, or INCIN	INCIN
U236	NA	Trypan Blue	72-57-1	(WETOX or CHOXD) fb CARBN, or INCIN	INCIN
U237	NA	Uracil mustard	66-75-1	(WETOX or CHOXD) fb CARBN, or INCIN	INCIN
U238	NA	Ethyl carbamate	51-79-6	(WETOX or CHOXD) fb CARBN, or INCIN	INCIN
U240	NA	2 4-Dichlorophenoxy-acetic (salts and esters)	[1] 94-75-7	(WETOX or CHOXD) fb CARBN, or INCIN	INCIN
U244	NA	Thiram	137-26-8	(WETOX or CHOXD) fb CARBN, or INCIN	INCIN
U246	NA	Cyanogen bromide	506-68-3	CHOXD, WETOX; or INCIN	CHOXD, WETOX, or INCIN
U248	NA	Warfarin (3%) or less	81-81-2	(WETOX or CHOXD) fb CARBN; or INCIN	FSUBS, or INCIN
U249	NA	Zinc Phosphide (<10%)	1314-84-7	CHOXD, CHRED, or INCIN.	CHOXD, CHRED, or INCIN
U328		o-toluidine	95-53-4	INCIN, or CHOXD fb, (BIODG or CARBN), or BIODG fb CARBN.	INCIN; or Thermal Destruction
U353		p-toluidine	106-49-0	INCIN, or CHOXD fb, (BIODG or CARBN), or BIODG fb CARBN	INCIN, or Thermal Destruction
U359		2-ethoxy-ethanol	110-80-5	INCIN, or CHOXD fb, (BIODG or CARBN), or BIODG fb CARBN	INCIN, or FSUBS

[1] CAS Number given for parent compound only
[2] This waste code exists in gaseous form and is not categorized as wastewater or nonwastewater forms.
Note. NA means Not Applicable

268.42 TABLE 3.—TECHNOLOGY-BASED STANDARDS FOR SPECIFIC RADIOACTIVE HAZARDOUS MIXED WASTE

Waste code	Waste descriptions and/or treatment category	CAS No	Technology code	
			Wastewaters	Nonwastewaters
D002	Radioactive high level wastes generated during the reprocessing of fuel rods subcategory	NA	NA	HLVIT
D004	Radioactive high level wastes generated during the reprocessing of fuel rods subcategory	NA	NA	HLVIT
D005	Radioactive high level wastes generated during the reprocessing of fuel rods subcategory	NA	NA	HLVIT
D006	Radioactive high level wastes generated during the reprocessing of fuel rods subcategory	NA	NA	HLVIT
D007	Radioactive high level wastes generated during the reprocessing of fuel rods subcategory	NA	NA	HLVIT
D008	Radioactive lead solids subcategory (Note. these lead solids include, but are not limited to, all forms of lead shielding, and other elemental forms of lead. These lead solids do not include treatment residuals such as hydroxide sludges, other wastewater treatment residuals, or incinerator ashes that can undergo conventional pozzolanic stabilization, nor do they include organolead materials that can be incinerated and stabilized as ash.)	7439-92-1	NA	MACRO
D008	Radioactive high level wastes generated during the reprocessing of fuel rods subcategory	NA	NA	HLVIT
D009	Elemental mercury contaminated with radioactive materials	7439-97-6	NA	AMLGM
D009	Hydraulic oil contaminated with mercury, radioactive materials subcategory	7439-97-6	NA	IMERC
D009	Radioactive high level wastes generated during the reprocessing of fuel rods subcategory	NA	NA	HLVIT
D010	Radioactive high level wastes generated during the reprocessing of fuel rods subcategory	NA	NA	HLVIT
D011	Radioactive high level wastes generated during the reprocessing of fuel rods subcategory	NA	NA	HLVIT
U151	Mercury. Elemental mercury contaminated with radioactive materials	7439-97-6	NA	AMLGM

Note NA means Not Applicable

(b) Any person may submit an application to the Administrator demonstrating that an alternative treatment method can achieve a measure of performance equivalent to that achieved by methods specified in paragraphs (a), (c), and (d) of this section for wastes or specified in Table 1 of § 268.45 for hazardous debris. The applicant must submit information demonstrating that his treatment method is in compliance with federal, state, and local requirements and is protective of human health and the environment. On the basis of such information and any other available information, the Administrator may approve the use of the alternative treatment method if he finds that the alternative treatment method provides a measure of performance equivalent to that achieved by methods specified in paragraphs (a), (c), and (d) of this section for wastes or in Table 1 of § 268.45 for hazardous debris. Any approval must be stated in writing and may contain such provisions and conditions as the Administrator deems appropriate. The person to whom such approval is issued must comply with all limitations contained in such a determination.

(c) As an alternative to the otherwise applicable subpart D treatment standards, lab packs are eligible for land disposal provided the following requirements are met:

(1) The lab packs comply with the applicable provisions of 40 CFR 264.316 and 40 CFR 265.316;

(2) All hazardous wastes contained in such lab packs are specified in appendix IV or appendix V to part 268;

(3) The lab packs are incinerated in accordance with the requirements of 40 CFR part 264, subpart O or 40 CFR part 265, subpart O; and

(4) Any incinerator residues from lab packs containing D004, D005, D006, D007, D008, D010, and D011 are treated in compliance with the applicable treatment standards specified for such wastes in subpart D of this part.

(d) Radioactive hazardous mixed wastes with treatment standards specified in Table 3 of this section are not subject to any treatment standards specified in §§ 268.41 or 268.43, or Table 2 of this section. Radioactive hazardous mixed wastes not subject to treatment standards in Table 3 of this section remain subject to all applicable treatment standards specified in §§ 268.41, 268.43, and Table 2 of this section. Hazardous debris containing radioactive waste is not subject to the treatment standards specified in Table 3 of this section but is subject to the treatment standards specified in § 268.45.

[51 FR 40642, Nov. 7, 1986, as amended at 52 FR 25790, July 8, 1987; 55 FR 22692, June 1, 1990; 56 FR 3884, Jan. 31, 1991; 57 FR 8089, Mar. 6, 1992; 57 FR 37273, Aug. 18, 1992; 58 FR 29885, May 24, 1993]

§ 268.43 Treatment standards expressed as waste concentrations.

(a) Table CCW identifies the restricted wastes and the concentrations of their associated hazardous constituents which may not be exceeded by the waste or treatment residual (not an extract of such waste or residual) for the allowable land disposal of such waste or residual. Compliance with these concentrations is required based upon grab samples, unless otherwise noted in the following Table CCW.

268.43 TABLE CCW.—CONSTITUENT CONCENTRATIONS IN WASTES

Waste code	Commercial chemical name	See also	Regulated hazardous constituent	CAS number for regulated hazardous constituent	Wastewaters		Nonwastewaters	
					Concentration (mg/l)	Notes	Concentration (mg/kg)	Notes
D003 (Reactive Cyanides Subcategory based on 261.23(a)(5))	NA	NA	Cyanides (Total)	57-12-5	(1)		590	(2)
			Cyanides (Amenable)	57-12-5	0.86		30	
D004	NA	Table CCWE in 268.41	Arsenic	7440-38-2	5.0		NA	
D005	NA	Table CCWE in 268.41	Barium	7440-39-3	100		NA	
D006	NA	Table CCWE in 268.41	Cadmium	7440-43-9	1.0		NA	
D007	NA	Table CCWE in 268.41	Chromium (Total)	7440-47-32	5.0		NA	
D008	NA	Table CCWE in 268.41.	Lead	7439-92-1	5.0		NA	
D009	NA	Table CCWE in 268.41	Mercury	7439-97-6	0.20		NA	
D010	NA	Table CCWE in 268.41	Selenium	7782-49-2	1.0		NA	
D011	NA	Table CCWE in 268.41.	Silver	7440-22-4	5.0		NA	
D012	NA	Table 2 in 268.42	Endrin	72-20-8	NA		0.13	(3)
D013	NA	Table 2 in 268.42	Lindane	58-89-9	NA		0.066	(3)
D014	NA	Table 2 in 268.42	Methoxychlor	72-43-5	NA		0.18	(3)
D015	NA	Table 2 in 268.42	Toxaphene	8001-35-1	NA		1.3	(3)
D016	NA	Table 2 in 268.42	2,4-D	94-75-7	NA		10.0	(3)
D017	NA	Table 2 in 268.42	2,4,5-TP (Silvex)	93-76-5	NA		7.9	(3)(4)(5)(6)
F001—F005 spent solvents	NA		Acetone	67-64-1	0.28		160	
			Benzene	71-43-2	0.070		3.7	(3)
			n-Butyl alcohol	71-36-3	5.6		2.6	
			Carbon tetrachloride	56-23-5	0.057		5.6	
			Chlorobenzene	108-90-7	0.057		5.7	
			Cresol (m- and p-isomers)		0.77		3.2	
			o-cresol		0.11		5.6	
			o-Dichlorobenzene	95-50-1	0.088		6.2	
			Ethyl acetate	141-7-6	0.34		33	
			Ethyl benzene	100-41-4	0.057		6.0	
			Ethyl ether	60-29-7	0.12		160	
			Isobutyl alcohol	78-83-1	5.6		170	
			Methylene chloride	75-6-2	0.089		33	
			Methyl ethyl ketone	78-93-3	0.28		36	
			Methyl isobutyl ketone	108-10-1	0.14		33	
			Nitrobenzene	98-95-3	0.068		14	
			Pyridine	110-86-1	0.014		16	

268.43 TABLE CCW.—CONSTITUENT CONCENTRATIONS IN WASTES—Continued

Waste code	Commercial chemical name	See also	Regulated hazardous constituent	CAS number for regulated hazardous constituent	Wastewaters		Nonwastewaters	
					Concentration (mg/l)	Notes	Concentration (mg/kg)	Notes
			Tetrachloroethylene	127-18-4	0.056		5.6	
			Toluene	108-88-3	0.08		28	
			1,1,1-Trichloroethane	71-55-6	0.054		5.6	
			1,1,2-Trichloroethane	79-00-5	0.030		7.6	(1)
			Trichloroethylene	79-01-6	0.054		5.6	
			1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	0.057		28	
			Trichloromono-fluoromethane	75-69-4	0.02		33	
F001-F005 spent solvents (Pharmaceutical Industry-Wastewater Subcategory)	NA	NA	Xylenes (total)		0.32		28	
			Methylene chloride	75-09-2	0.44		NA	
F006	NA	Table CCWE in 268.41	Cyanides (Total)	57-12-5	1.2		590	
			Cyanides (Amenable)	57-12-5	0.86		30	
			Cadmium	7440-43-9	1.6		NA	
			Chromium	7440-47-32	0.32		NA	
			Lead	7439-92-1	0.040		NA	
			Nickel	7440-02-0	0.44		NA	
F007	NA	Table CCWE in 268.41.	Cyanides (Total)	57-12-5	1.9		590	
			Cyanides (Amenable)	57-12-5	0.1		30	
			Chromium (Total)	7440-47-32	0.32		NA	
			Lead	7439-92-1	0.04		NA	
			Nickel	7440-02-0	0.44		NA	
F008	NA	Table CCWE in 268.41.	Cyanides (Total)	57-12-5	1.9		590	
			Cyanides (Amenable)	57-12-5	0.1		30	
			Chromium	7440-47-32	0.32		NA	
			Lead	7439-92-1	0.04		NA	
			Nickel	7440-02-0	0.44		NA	
F009	NA	Table CCWE in 268.41.	Cyanides (Total)	57-12-5	1.9		590	
			Cyanides (Amenable)	57-12-5	0.1		30	
			Chromium	7440-47-32	0.32		NA	
			Lead	7439-92-1	0.04		NA	
			Nickel	7440-02-0	0.44		NA	
F010	NA	NA	Cyanides (Total)	57-12-5	1.9		1.5	
			Cyanides (Amenable)	57-12-5	0.1		NA	

F011	NA	Table CCWE in 268.41	Cyanides (Total)	57-12-5	1.9		110	
			Cyanides (Amenable)	57-12-5	0.1		9.1	
			Chromium (Total)	7440-47-32	0.32		NA	
			Lead	7439-92-1	0.04		NA	
			Nickel	7440-02-0	0.44		NA	
F012	NA	Table CCWE in 268.41	Cyanides (Total)	57-12-5	1.9		110	
			Cyanides (Amenable)	57-12-5	0.1		9.1	
			Chromium (Total)	7440-47-32	0.32		NA	
			Lead	7439-92-1	0.04		NA	
			Nickel	7440-02-0	0.44		NA	
F019	NA	Table CCWE in 268.41	Cyanides (Total)	57-12-5	1.2		590	(2)
			Cyanides (Amenable)	57-12-5	0.86		30	(2)
			Chromium (Total)	7440-47-32	0.32		NA	
F024	NA	Table CCWE in 268.41 and Table 2 in 268.42 (Note: F024 organic standards must be treated via incineration (INCIN)).	2-Chloro-1,3-butadiene	126-99-8	0.28	(1)	0.28	(1)
			3-Chloropropene					
			1,1-Dichloroethane	107-05-1	0.28	(1)	0.28	(1)
			1,2-Dichloroethane	75-34-3	0.014	(1)	0.014	(1)
			1,2-Dichloropropane					
			cis-1,3-Dichloropropene	107-06-2	0.014	(1)	0.014	(1)
			trans-1,3-Dichloropropene					
			Bis(2-ethylhexyl)- phthalate	78-87-5	0.014	(1)	0.014	(1)
			Hexachloroethane					
			Chromium (Total)					
			Nickel	10061-01-5	0.014	(1)	0.014	(1)
				10061-02-6	0.014	(1)	0.014	(1)
				117-81-7	0.036	(1)	1.8	(1)
				67-72-1	0.036	(1)	1.8	(1)
				7440-47-32	0.35		NA	
				7440-02-0	0.47		NA	

268.43 TABLE CCW.—CONSTITUENT CONCENTRATIONS IN WASTES—Continued

Waste code	Commercial chemical name	See also	Regulated hazardous constituent	CAS number for regulated hazardous constituent	Wastewaters		Nonwastewaters	
					Concentration (mg/l)	Notes	Concentration (mg/kg)	Notes
F025 (Light Ends Subcategory).	NA	NA	Chloroform	67-66-3	0 046	[2]	6.2	[2]
			1,2-Dichloroethane	107-06-2	0.21	[2]	6.2	[2]
			1,1-Dichloroethylene					
			Methylene chloride	75-35-4	0 025	[2]	6 2	[2]
			Carbon tetrachloride					
			1,1,2-Trichloroethane					
			Trichloroethylene	75-9-2	0.089	[2]	31	[2]
			Vinyl chloride					
				56-23-5	0 057	[2]	6.2	[2]
				79-00-5	0 054	[2]	6.2	[2]
				79-01-6	0 054	[2]	5 6	[2]
F025 (Spent Filters or Aids and Desicants Subcategory)	NA	NA		75-01-4	0 27	[2]	33	[2]
			Chloroform	67-66-3	0 046	[2]	6 2	[2]
			Methylene chloride	75-9-2	0 089	[2]	31	[2]
			Carbon tetrachloride					
			1,1,2-Trichloroethane	56- 23-5	0 057	[2]	6.2	[2]
			Trichloroethylene					
			Vinyl chloride	79-00-5	0 054	[2]	6.2	[2]
			Hexachlorobenzene					
			Hexachlorobutadiene					
			Hexachloroethane	79-01-6	0 054	[2]	5 6	[2]
				75-01-4	0 27	[2]	33	[2]
				118-74-1	0 055	[2]	37	[2]
				87-68-3	0 055	[2]	28	[2]
F037	NA	Table CCWE in 268 41		67-72-1	0 055	[2]	30	[2]
			Acenaphthene	208-96-8	0 059	[2]	NA	
			Anthracene	120-12-7	0 059	[2]	28	[2]
			Benzene	71-43-2	0 14	[2]	14	[2]
			Benzo(a)anthracene	50-32-8	0 059	[2]	20	[2]
			Benzo(a)pyrene	117-81-7	0 061	[2]	12	[2]
			Bis(2-ethylhexyl) phthalate	75-15-0	0.28	[2]	7.3	[2]
			Chrysene	218-01-9	0 059	[2]	15	[2]
			Di-n-butyl phthalate	105-67-9	0 057	[2]	3 6	[2]
			Ethylbenzene	100-41-4	0 057	[2]	14	[2]
			Fluorene	86-73-7	0 059	[2]	NA	
			Naphthalene	91-20-3	0.059	[2]	42	[2]
			Phenanthrene	85-01-8	0 059	[2]	34	[2]
			Phenol	108-85-2	0 039	[2]	3.6	[2]
			Pyrene	129-00-0	0.067	[2]	36	[2]
			Toluene	108-88-3	0 08	[2]	14	[2]
			Xylene(s)		0.32	[2]	22	[2]
			Cyanides (Total)	57-12-5	0.028	[2]	1.8	[2]
			Chromium (Total)	7440-47-32	0.2		NA	
			Lead	7439-92-1	0.037		NA	
F038	NA	Table CCWE in 268 41	Benzene	71-43-2	0 14	[2]	14	[2]
			Benzo(a)pyrene	50-32-8	0 061	[2]	12	[2]
			Bis(2-ethylhexyl) phthalate	117-81-7	0 28	[2]	7.3	[2]
			Chrysene	218-01-9	0.059	[2]	15	[2]
			Di-n-butyl phthalate	84-74-2	0 057	[2]	3.6	[2]
			Ethylbenzene	100-41-4	0 057	[2]	14	[2]
			Fluorene	86-73-7	0 059	[2]	NA	
			Naphthalene	91-20-3	0 059	[2]	42	[2]
			Phenanthrene	85-01-8	0 059	[2]	34	[2]
			Phenol	108-85-2	0.039	[2]	3.6	[2]
			Pyrene	129-00-0	0.067	[2]	36	[2]
			Toluene	108-88-3	0.080	[2]	14	[2]
			Xylene(s)		0.32	[2]	22	[2]
			Cyanides (Total)	57-12-5	0 028	[2]	1 8	[2]
			Chromium (Total)	7440-47-32	0.2		NA	
			Lead	7439-92-1	0.037		NA	
F039 (and D001 and D002 wastes prohibited under § 268 37)	NA	Table 2 in 268 42 and Table CCWE in 268 41	Acetone	67-64-1	0.28	[2]	160	[2]
			Acenaphthalene	208-96-8	0 059	[2]	3 4	[2]
			Acenaphthene	83-32-9	0 059	[2]	4 0	[2]
			Acetonitrile	75-05-8	0.17	[2]	NA	
			Acetophenone	96-86-2	0 010	[2]	9 7	[2]
			2-Acetylaminofluorene	53-96-3	0 059	[2]	140	[2]
			Acrolein					
			Acrylonitrile	107-02-8	0.29	[2]	NA	
			Aldrin	107-13-1	0.24	[2]	84	[2]
				309-00-2	0.021	[2]	0.066	[2]
			4-Aminobiphenyl	92-67-1	0 13	[2]	NA	
			Aniline	62-53-3	0.81	[2]	14	[2]
			Anthracene	120-12-7	0.059	[2]	4 0	[2]
			Aramite	140-57-8	0.36	[2]	NA	
			Aroclor 1016	12674-11-2	0.013	[2]	0 92	[2]
			Aroclor 1221	11104-28-2	0.014	[2]	0 92	[2]
			Aroclor 1232	11141-16-5	0.013	[2]	0 92	[2]
			Aroclor 1242	53469-21-9	0.017	[2]	0.92	[2]
			Aroclor 1248	12672-29-6	0.013	[2]	0 92	[2]
			Aroclor 1254	11097-69-1	0 014	[2]	1.8	[2]
			Aroclor 1260	11096-82-5	0.014	[2]	1.8	[2]
			alpha-BHC	319-84-6	0.00014	[2]	0 066	[2]
			beta-BHC	319-85-7	0.00014	[2]	0.066	[2]
			delta-BHC	319-86-8	0.023	[2]	0.066	[2]
			gamma-BHC	58-89-9	0.0017	[2]	0 066	[2]
			Benzene	71-43-2	0.14	[2]	35	[2]
			Benz(a)anthracene	56-55-3	0.059	[2]	8.2	[2]
			Benzo(b)- fluoranthene	205-99-2	0.055	[2]	3 4	[2]

268.43 TABLE CCW.—CONSTITUENT CONCENTRATIONS IN WASTES—Continued

Waste code	Commercial chemical name	See also	Regulated hazardous constituent	CAS number for regulated hazardous constituent	Wastewaters		Nonwastewaters	
					Concentration (mg/l)	Notes	Concentration (mg/kg)	Notes
			Benzo(k)- fluoranthene	207-08-9	0 059	[2]	3 4	[3]
			Benzo(g,h,i)- perylene	191-24-2	0 0055	[2]	1 5	[3]
			Benzo(a)pyrene	50-32-8	0 061	[2]	8.2	[3]
			Bromodichloromethane	75-27-4	0 35	[2]	15	[3]
			Bromoform (Tribromomethane)	75-25-2	0 63	[2]	15	[3]
			Bromomethane (methyl bromide)	74-83-9	0 11	[2]	15	[3]
			4-Bromophenyl phenyl ether	101-55-3	0 055	[2]	15	[3]
			n-Butyl alcohol	71-36-3	5 6	[2]	2 6	[3]
			Butyl benzyl phthalate	85-68-7	0 017	[2]	7 9	[3]
			2-sec-Butyl-4 6-dinitrophenol	88-85-7	0 066	[2]	2 5	[3]
			Carbon tetrachloride	56-23-5	0 057	[2]	5 6	[3]
			Carbon disulfide	75-15-0	0 014	[2]	NA	
			Chlordane	57-74-9	0 0033	[2]	0 13	[3]
			p-Chloroaniline	106-47-8	0 46	[2]	16	[3]
			Chlorobenzene	108-90-7	0 057	[2]	5 7	[3]
			Chlorobenzilate	510-15-6	0 10	[2]	NA	
			2-Chloro-1,3-butadiene	126-99-8	0 057	[2]	NA	
			Chlorodibromomethane	124-48-1	0 057	[2]	15	
			Chloroethane	75-00-3	0 27	[2]	6 0	
			bis(2-Chloroethoxy) methane	111-91-1	0 036	[2]	7.2	
			bis(2-Chloroethyl) ether	111-44-4	0 033	[2]	7 2	
			Chloroform	67-66-3	0 046	[2]	5 6	
			bis(2-Chloroisopropyl) ether	39638-32-9	0 055	[2]	7 2	
			p-Chloro-m-cresol	59-50-7	0 018	[2]	14	
			Chloromethane (Methyl chloride).	74-87-3	0 19	[2]	33	[3]
			2-Chloronaphthalene	91-8-7	0 055	[2]	5 6	[3]
			2-Chlorophenol	95-57-8	0 044	[2]	5 7	[3]
			3-Chloropropylene	107-05-1	0 036	[2]	28	[3]
			Chrysene	218-01-9	0 059	[2]	8 2	[3]
			o-Cresol	95-48-7	0 11	[2]	5 6	[3]
			Cresol (m- and p-isomers)		0 77	[2]	3 2	[3]
			Cyclohexanone	108-94-1	0.36	[2]	NA	
			1,2-Dibromo-3-chloropropane	96-12-8	0 11	[2]	15	[3]
			1,2-Dibromoethane (Ethylene dibromide)	106-93-4	0 028	[2]	15	[3]
			Dibromomethane	74-95-3	0 11	[2]	15	[3]
			2,4-Dichlorophenoxyacetic acid (2, 4-D)	94-75-7	0 72	[2]	10	[3]
			o,p -DDD	53-19-0	0 023	[2]	0 087	[3]
			p,p'-DDD	72-54-8	0 023	[2]	0 087	[3]
			o,p'-DDE	3424-82-6	0.031	[2]	0.087	[3]
			p,p'-DDE	72-55-9	0 031	[2]	0 087	[3]
			o,p'-DDT	789-02-6	0 0039	[2]	0.087	[3]
			p,p'-DDT	50-29-3	0.0039	[2]	0 087	[3]
			Dibenz(a,h) anthracene	53-70-3	0 055	[2]	8.2	[3]
			Dibenzo(a,e) pyrene	192-65-4	0 061	[2]	NA	
			m-Dichlorobenzene	541-73-1	0 036	[2]	6 2	[3]
			o-Dichlorobenzene	95-50-1	0 088	[2]	6 2	[3]
			p-Dichlorobenzene	106-46-7	0 090	[2]	6.2	[3]
			Dichlorodifluoromethane	75-71-8	0.23	[2]	7.2	[3]
			1,1-Dichloroethane	75-34-3	0 059	[2]	7.2	[3]
			1,2-Dichloroethane	107-06-2	0.21	[2]	7.2	[3]
			1,1-Dichloroethylene	75-35-4	0 025	[2]	33	[3]
			trans-1,2-Dichloroethylene		0 054	[2]	33	[3]
			2,4-Dichlorophenol	120-83-2	0 044	[2]	14	[3]
			2,6-Dichlorophenol	87-65-0	0 044	[2]	14	[3]
			1,2-Dichloropropane	78-87-5	0 85	[2]	18	[3]
			cis-1,3-Dichloropropene	10061-01-5	0 036	[2]	18	[3]
			trans-1,3-Dichloropropene	10061-02-6	0.036	[2]	18	[3]
			Dieldrin	60-57-1	0 017	[2]	0 13	[3]
			Diethyl phthalate	84-66-2	0.20	[2]	28	[3]
			2,4-Dimethyl phenol	105-67-9	0.036	[2]	14	[3]
			Dimethyl phthalate	131-11-3	0 047	[2]	28	[3]
			Di-n-butyl phthalate	84-74-2	0 057	[2]	28	[3]
			1,4-Dinitrobenzene	100-25-4	0 32	[2]	2.3	[3]
			4,6-Dinitro-o-cresol	534-52-1	0 28	[2]	160	[3]
			2 4-Dinitrophenol	51-28-5	0 12	[2]	160	[3]
			2 4-Dinitrotoluene	121-14-2	0 32	[2]	140	[3]
			2,6-Dinitrotoluene	606-20-2	0 55	[2]	28	[3]
			Di-n-octyl phthalate	117-84-0	0 017	[2]	28	[3]
			Di-n-propylnitrosoamine	621-64-7	0 40	[2]	14	[3]
			Diphenylamine	122-39-4	0 52	[2]	NA	
			1,2-Diphenyl hydrazine	122-66-7	0 087	[2]	NA	
			Diphenyl nitrosamine	621-64-7	0 40	[2]	NA	
			1, 4-Dioxane	123-91-1	0 12	[2]	170	[3]
			Disulfoton	298-04-4	0 017	[2]	6 2	[3]
			Endosulfan I	939-98-8	0.023	[2]	0.066	[3]
			Endosulfan II	33213-6-5	0 029	[2]	0 13	[3]
			Endosulfan sulfate	1031-07-8	0.029	[2]	0 13	[3]
			Endrin	72-20-8	0.0028	[2]	0 13	[3]
			Endrin aldehyde	7421-93-4	0.025	[2]	0 13	[3]
			Ethyl acetate	141-78-6	0.34	[2]	33	[3]
			Ethyl cyanide	107-12-0	0.24	[2]	360	[3]
			Ethyl benzene	100-41-4	0.057	[2]	6 0	[3]
			Ethyl ether	60-29-7	0.12	[2]	160	[3]
			bis(2-Ethylhexyl) phthalate	117-81-7	0.28	[2]	28	[3]
			Ethyl methacrylate	97-63-2	0.14	[2]	160	[3]
			Ethylene oxide	75-21-8	0.12	[2]	NA	
			Famphur	52-85-7	0.017	[2]	15	[3]
			Fluoranthene	206-44-0	0.068	[2]	8.2	[3]
			Fluorene	86-73-7	0.059	[2]	4 0	[3]

268.43 TABLE CCW.—CONSTITUENT CONCENTRATIONS IN WASTES—Continued

Waste code	Commercial chemical name	See also	Regulated hazardous constituent	CAS number for regulated hazardous constituent	Wastewaters		Nonwastewaters	
					Concentration (mg/l)	Notes	Concentration (mg/kg)	Notes
			Fluorotrichloromethane	75-69-4	0.020	[3]	33	[3]
			Heptachlor	76-44-8	0 0012	[3]	0 066	[3]
			Heptachlor epoxide	1024-57-3	0 016	[3]	0 066	[3]
			Hexachlorobenzene	118-74-1	0 055	[3]	37	[3]
			Hexachlorobutadiene	87-68-3	0 055	[3]	28	[3]
			Hexachlorocyclopentadiene	77-47-4	0 057	[3]	3 6	[3]
			Hexachlorodibenzofurans	---	0 000063	[3]	0.001	[3]
			Hexachlorodibenzo-p-dioxins	---	0 000063	[3]	0 001	[3]
			Hexachloroethane	67-72-1	0 055	[3]	28	[3]
			Hexachloropropene	1888-71-7	0 035	[3]	28	[3]
			Indeno(1,2,3-c,d)pyrene	193-39-5	0 0055	[3]	8.2	[3]
			Iodomethane	74-88-4	0 19	[3]	65	[3]
			Isobutanol	78-83-1	5 6	[3]	170	[3]
			Isodrin	465-73-6	0.021	[3]	0.066	[3]
			Isosafrole	120-58-1	0 081	[2]	2.6	[2]
			Kepone	143-50-8	0 0011	[2]	0 13	[2]
			Methacrylonitrile	126-98-7	0 24	[2]	84	[2]
			Methanol	67-56-1	5 6	[2]	NA	
			Methapyrilene	91-80-5	0 081	[2]	1 5	[2]
			Methoxychlor	72-43-5	0 25	[2]	0 18	[2]
			3-Methylcholanthrene	56-49-5	0 0055	[2]	15	[2]
			4,4-Methylene-bis-(2-chloroaniline)	101-14-4	0 50	[2]	35	[2]
			Methylene chloride	75-09-2	0 089	[2]	33	[2]
			Methyl ethyl ketone	78-93-3	0 28	[2]	36	[2]
			Methyl isobutyl ketone	108-10-1	0 14	[2]	33	[2]
			Methyl methacrylate	80-62-6	0 14	[2]	160	[2]
			Methyl methansulfonate	66-27-3	0 018	[2]	NA	
			Methyl parathion	298-00-0	0 014	[2]	4 6	[2]
			Naphthalene	91-20-3	0.059	[2]	3.1	[2]
			2-Naphthylamine	91-59-8	0 52	[2]	NA	
			p-Nitroaniline	100-01-6	0 028	[2]	28	[2]
			Nitrobenzene	98-95-3	0 068	[2]	14	[2]
			5-Nitro-o-toluidine	99-55-8	0.32	[2]	28	[2]
			4-Nitrophenol	100-02-7	0 12	[2]	29	[2]
			N-Nitrosodiethylamine	55-18-5	0 40	[2]	28	[2]
			N-Nitrosodimethylamine	62-75-9	0 40	[2]	NA	
			N-Nitroso-di-n-butylamine	924-16-3	0 40	[2]	17	[2]
			N-Nitrosomethyl-ethylamine	10595-95-6	0 40	[2]	2.3	[2]
			N-Nitrosomorpholine	59-89-2	0 40	[2]	2.3	[2]
			N-Nitrosopiperidine	100-75-4	0 013	[2]	35	[2]
			N-Nitrosopyrrolidine	930-55-2	0 013	[2]	35	[2]
			Parathion	56-38-2	0.014	[2]	4 6	[2]
			Pentachlorobenzene	608-93-5	0.055	[2]	37	[2]

			Pentachlorodibenzo-furans	---	0.000063	[3]	0 001	[3]
			Pentachlorodibenzo-p-dioxins	---	0 000063	[3]	0 001	[3]
			Pentachloronitrobenzene	82-68-8	0 055	[2]	4 8	[2]
			Pentachlorophenol	87-86-5	0 089	[2]	7 4	[2]
			Phenacetin	62-44-2	0 081	[2]	16	[2]
			Phenanthrene	85-01-8	0.059	[2]	3 1	[2]
			Phenol	108-95-2	0 039	[2]	6 2	[2]
			Phorate	298-02-2	0 021	[2]	4 6	[2]
			Phthalic anhydride	85-44-9	0 069	[2]	NA	
			Pronamide	23950-58-5	0 093	[2]	1.5	[2]
			Pyrene	129-00-0	0 067	[2]	8.2	[2]
			Pyridine	110-86-1	0 014	[2]	16	[2]
			Safrole	94-59-7	0 081	[2]	22	[2]
			Silvex (2,4,5-TP)	93-72-1	0 72	[2]	7 9	[2]
			2,4,5-T	93-76-5	0 72	[2]	7.9	[2]
			1,2,4,5,-Tetrachlorobenzene	95-94-3	0 055	[2]	19	[2]
			Tetrachlorodibenzo-furans	---	0.000063	[2]	0 001	[2]
			Tetrachlorodibenzo-p-dioxins	---	0 000063	[2]	0 001	[2]
			1,1,1,2-Tetrachloroethane	630-20-6	0 057	[2]	42	[2]
			1,1,2,2-Tetrachloroethane	79-34-6	0 057	[2]	42	[2]
			Tetrachloroethylene	127-18-4	0 056	[2]	5.6	[2]
			2,3,4,6-Tetrachlorophenol	58-90-2	0 030	[3]	37	[3]
			Toluene	108-88-3	0 080	[2]	28	[2]
			Toxaphene	8001-35-1	0 0095	[3]	1.3	[3]
			1,2,4-Trichlorobenzene	120-82-1	0 055	[3]	19	[3]
			1,1,1-Trichloroethane	71-55-6	0 054	[3]	5 6	[3]
			1,1,2-Trichloroethane	79-00-5	0 054	[3]	5 6	[3]
			Trichloroethylene	79-01-6	0 054	[3]	5 6	[3]
			2,4,5-Trichlorophenol	95-95-4	0 18	[3]	37	[3]
			2,4,6-Trichlorophenol	88-06-2	0 035	[3]	37	[3]
			1,2,3-Trichloropropane	96-18-4	0 85	[2]	28	[2]
			1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	0 057	[2]	28	[2]
			Tris(2,3-dibromopropyl) phosphate	126-72-7	0 11	[2]	NA	
			Vinyl chloride	75-01-4	0.27	[2]	33	[2]
			Xylene(s)	---	0.32	[2]	28	[2]
			Cyanides (Total)	57-12-5	1.2	[3]	1 8	[3]
			Fluoride	16984-48-8	35	[3]	NA	
			Sulfide	8496-25-8	14	[3]	NA	
			Antimony	7440-36-0	1 9	[3]	NA	
			Arsenic	7440-38-2	1 4	[3]	NA	
			Barium	7440-39-3	1.2	[3]	NA	
			Beryllium	7440-41-7	0.82	[3]	NA	
			Cadmium	7440-43-9	0.20	[3]	NA	
			Chromium (Total)	7440-47-32	0.37	[3]	NA	
			Copper	7440-50-8	1.3	[3]	NA	
			Lead	7439-92-1	0.28	[3]	NA	
			Mercury	7439-97-6	0.15	[3]	NA	
			Nickel	7440-02-0	0.55	[3]	NA	

268.43 TABLE CCW.—CONSTITUENT CONCENTRATIONS IN WASTES—Continued

Waste code	Commercial chemical name	See also	Regulated hazardous constituent	CAS number for regulated hazardous constituent	Wastewaters		Nonwastewaters	
					Concentration (mg/l)	Notes	Concentration (mg/kg)	Notes
			Selenium	7782-49-2	0.82	(3)	NA	
			Silver	7440-22-4	0.29	(3)	NA	
			Thallium	7440-28-0	1.4	(3)	NA	
			Vanadium	7440-62-2	0.042	(3)	NA	
			Zinc	7440-66-6	1.0	(3)	NA	
K001	NA	Table CCWE in 268.41	Naphthalene	91-20-3	0.031	(3)	1.5	(3)
			Pentachlorophenol	87-86-5	0.18	(3)	7.4	(3)
			Phenanthrene					
			Pyrene	85-01-8	0.031	(3)	1.5	(3)
			Toluene	129-00-0	0.028	(3)	1.5	(3)
			Xylenes (Total)	108-88-3	0.028	(3)	28	(3)
			Lead		0.032	(3)	33	(3)
				7439-92-1	0.037		NA	
K002	NA	Table CCWE in 268.41	Chromium (Total)	7440-47-32	0.9	(3)	NA	
			Lead	7439-92-1	3.4	(3)	NA	
K003	NA	Table CCWE in 268.41	Chromium (Total)	7440-47-32	0.9	(3)	NA	
			Lead	7439-92-1	3.4	(3)	NA	
K004	NA	Table CCWE in 268.41	Chromium (Total)	7440-47-32	0.9	(3)	NA	
			Lead	7439-92-1	3.4	(3)	NA	⑥
K005	NA	Table CCWE in 268.41	Chromium (Total)	7440-47-32	0.9	(3)	NA	
			Lead	7439-92-1	3.4	(3)	NA	
			Cyanides (Total)	57-12-5	0.74	(3)	(4)	
K006	NA	Table CCWE in 268.41	Chromium (Total)	7440-47-32	0.9	3.4	(5)	NA
			Lead	7439-92-1			(5)	NA
K007	NA	Table CCWE in 268.41	Chromium (Total)	7440-47-32	0.9	(3)	NA	
			Lead	7439-92-1	3.4	(3)	NA	
			Cyanides (Total)	57-12-5	0.74	(3)	(4)	
K008	NA	Table CCWE in 268.41	Chromium (Total)	7440-47-32	0.9	(3)	NA	
			Lead	7439-92-1	3.4	(3)	NA	
K009	NA	NA	Chloroform	67-66-3	0.1		6.0	
K010	NA	NA	Chloroform	67-66-3	0.1		6.0	
K011	NA	NA	Acetonitrile	75-05-8	38		1.8	
			Acrylonitrile	107-13-1	0.06		1.4	
			Acrylamide	79-06-1	19		23	
			Benzene	71-43-2	0.02		0.03	
			Cyanide (Total)	57-12-5	21		57	
K013	NA	NA	Acetonitrile	75-05-8	38		1.8	
			Acrylonitrile	107-13-1	0.06		1.4	
			Acrylamide	79-06-1	19		23	
			Benzene	71-43-2	0.02		0.03	
			Cyanide (Total)	57-12-5	21		57	
K014	NA	NA	Acetonitrile	75-05-8	38		1.8	
			Acrylonitrile	107-13-1	0.06		1.4	
			Acrylamide	79-06-1	19		23	
			Benzene	71-43-2	0.02		0.03	
			Cyanide (Total)	57-12-5	21		57	
K015	NA	Table CCWE in 268.41.	Anthracene	120-12-7	0.059		6.2	
			Benzal Chloride	98-87-3	0.28		3.4	
			Sum of Benzo(b) fluoranthene and Benzo(k) fluoranthene	207-08-9	0.055			
			Phenanthrene	85-01-8	0.059		3.4	
			Toluene	108-88-3	0.08		6.0	
			Chromium (Total)	7440-47-32	0.32		NA	
			Nickel	7440-02-0	0.44		NA	
K016	NA		Hexachlorobenzene	118-74-1	0.055		28	
			Hexachlorobutadiene	87-68-3	0.055		5.6	
			Hexachlorocyclopentadiene	77-47-4	0.057		5.6	
			Hexachloroethane	67-72-1	0.055		28	
			Tetrachloroethane	127-18-4	0.056		6.0	
K017	NA	NA	1,2-Dichloropropane	78-87-5	0.85	(12)	18	
			1,2,3-Trichloropropane	96-18-4	0.85	(12)	28	
			Bis(2-chloroethyl)ether	111-44-4	0.033	(12)	7.2	
K018	NA		Chloroethane	76-00-3	0.27		6.0	
			Chloromethane	74-87-3	0.19		NA	
			1,1-Dichloroethane	75-34-3	0.059		6.0	
			1,2-Dichloroethane	107-06-2	0.21		6.0	
			Hexachlorobenzene	118-74-1	0.055		28	
			Hexachlorobutadiene	87-68-3	0.055		5.6	
			Pentachloroethane	76-01-7	NA		5.6	
			1,1,1-Trichloroethane	71-55-6	0.054		6.0	
			Hexachloroethane	67-72-1	0.055		28	
K019	NA		Bis(2-chloroethyl) ether	111-44-4	0.033		5.6	
			Chlorobenzene	108-90-7	0.057		6.0	
			Chloroform	67-66-3	0.046		6.0	
			p-Dichlorobenzene	106-46-7	0.09		NA	
			1,2-Dichloroethane	107-06-2	0.21		6.0	
			Fluorene	86-73-7	0.059		NA	
			Hexachloroethane	67-72-1	0.055		28	
			Naphthalene	91-20-3	0.059		5.6	
			Phenanthrene	85-01-8	0.059		5.6	
			1,2,4,5-Tetrachlorobenzene	95-94-3	0.055		NA	
			Tetrachloroethane	127-18-4	0.056		6.0	
			1,2,4-Trichlorobenzene	120-82-1	0.055		19	
			1,1,1-Trichloroethane	71-55-6	0.054		6.0	
K020	NA		1,2-Dichloroethane	106-93-4	0.21		6.0	
			1,1,2,2-Tetrachloroethane	79-34-6	0.057		5.6	
			Tetrachloroethene	127-18-4	0.056		6.0	
K021	NA	Table CCWE in 268.41.	Chloroform	67-66-3	0.046	(3)(3)	6.2	
			Carbon tetrachloride	56-23-5	0.057		6.2	
			Antimony	7440-36-0	0.60		NA	

268.43 TABLE CCW.—CONSTITUENT CONCENTRATIONS IN WASTES—Continued

Waste code	Commercial chemical name	See also	Regulated hazardous constituent	CAS number for regulated hazardous constituent	Wastewaters		Nonwastewaters	
					Concentration (mg/l)	Notes	Concentration (mg/kg)	Notes
K022	NA	Table CCWE in 268.41.	Toluene	108-88-3	0.080	[2]	0.034	[2]
			Acetophenone	96-86-2	0.010		19	[2]
			Diphenylamine	22-39-4	0.52		NA	
			Diphenylnitrosamine	86-30-6	0.40	[2]	NA	
			Sum of Diphenylamine and Diphenylnitrosamine		NA		13	[3]
			Phenol	108-95-2				
			Chromium (Total)	7440-47-32	0.039		12	[2]
			Nickel	7440-02-0	0.35		NA	
K023	NA		Phthalic anhydride (measured as Phthalic acid)	85-44-9	0.47		NA	
K024	NA		Phthalic anhydride (measured as Phthalic acid)	85-44-9	0.069		28	[3]
K028	NA	Table CCWE in 268.41.	1,1-Dichloroethane trans-1,2-Dichloroethane	75-34-3	0.059		6.0	[3]
			Hexachlorobutadiene	87-63-3	0.054		5.6	[3]
			Hexachloroethane	67-72-1	0.055		28	[3]
			Pentachloroethane	76-01-7	0.055		5.6	[3]
			1,1,1,2-Tetrachloroethane	630-20-6	NA		5.6	[3]
			1,1,2,2-Tetrachloroethane	79-34-5	0.057		5.6	[3]
			1,1,1,-Trichloroethane	71-55-6	0.057		5.6	[3]
			1,1,2-Trichloroethane	79-00-5	0.054		6.0	[3]
			Tetrachloroethylene	127-18-4	0.054		6.0	[3]
			Cadmium	7440-43-8	0.056		6.0	[3]
			Chromium (Total)	7440-47-32	6.4		NA	
			Lead	7439-92-1	0.35		NA	
			Nickel	7440-02-0	0.037		NA	
K029	NA	NA	Chloroform	67-66-3	0.47		NA	
			1,2-Dichloroethane	107-06-2	0.046		6.0	[3]
			1,1-Dichloroethylene	75-35-4	0.21		6.0	[3]
			1,1,1-Trichloroethane	71-55-6	0.025		6.0	[3]
			Vinyl chloride	75-01-4	0.054		6.0	[3]
K030	NA		o-Dichlorobenzene	95-50-1	0.27		6.0	[3]
			p-Dichlorobenzene	106-46-7	0.088		NA	
			Hexachlorobutadiene	87-68-3	0.09		NA	
			Hexachloroethane	67-72-1	0.055		5.6	[3]
			Hexachloropropene	1888-71-7	0.055		28	[3]
			Pentachlorobenzene	608-93-5	NA		19	[3]
			Pentachloroethane	76-01-7	NA		28	[3]
			1,2,4,5-Tetrachlorobenzene	95-94-3	NA		5.6	[3]
			Tetrachloroethene	127-18-4	0.055		14	[3]
			1,2,4-Trichlorobenzene	120-82-1	0.056		6.0	[3]
					0.055		19	[3]

K031	NA	Table CCWE in 268.41	Arsenic	7440-38-2	0.79		NA	[3]
K032	NA	NA	Hexachlorocyclopentadiene	77-47-4	0.057	[3]	2.4	[3]
			Chlordane	57-74-9	0.0033	[3]	0.26	[3]
			Heptachlor	76-44-8	0.0012	[3]	0.066	[3]
			Heptachlor epoxide	1024-57-3	0.016	[3]	0.066	[3]
K033	NA	NA	Hexachlorocyclopentadiene	77-47-4	0.057	[3]	2.4	[3]
K034	NA	NA	Hexachlorocyclopentadiene	77-47-4	0.057	[3]	2.4	[3]
K035	NA	NA	Acenaphthene	83-32-9	NA		3.4	[3]
			Anthracene	120-12-7	NA		3.4	[3]
			Benz(a)anthracene	56-55-3	0.059	[3]	3.4	[3]
			Benzo(a)pyrene	50-32-8	NA		3.4	[3]
			Chrysene	218-01-9	0.059	[3]	3.4	[3]
			Dibenz(a,h)anthracene	53-70-3	NA		3.4	[3]
			Fluoranthene	206-44-0	0.068	[3]	3.4	[3]
			Fluorene	86-73-7	NA		3.4	[3]
			Indeno(1,2,3-cd)pyrene	193-39-5	NA		3.4	[3]
			Cresols (m- and p-isomers)		0.77	[3]	NA	
			Naphthalene	91-20-3	0.059	[3]	3.4	[3]
			o-cresol	95-48-7	0.11	[3]	NA	
			Phenanthrene	85-01-8	0.059	[3]	3.4	[3]
			Phenol	108-95-2	0.039		NA	
			Pyrene	129-00-0	0.067	[3]	8.2	[3]
K036	NA	NA	Disulfoton	298-04-4	0.025	[3]	0.1	[3]
K037	NA	NA	Disulfoton	298-04-4	0.025	[3]	0.1	[3]
			Toluene	108-88-3	0.080	[3]	28	[3]
K038	NA	NA	Phorate	298-02-2	0.025	[3]	0.1	[3]
K040	NA	NA	Phorate	298-02-2	0.025	[3]	0.1	[3]
K041	NA	NA	Toxaphene	8001-35-1	0.0095	[3]	2.6	[3]
K042	NA	NA	1,2,4,5-Tetrachlorobenzene	95-94-3	0.055	[3]	4.4	[3]
			o-Dichlorobenzene	95-50-1	0.088	[3]	4.4	[3]
			p-Dichlorobenzene	106-46-7	0.090	[3]	4.4	[3]
			Pentachlorobenzene	608-93-5	0.055	[3]	4.4	[3]
			1,2,4-Trichlorobenzene	120-82-1	0.055	[3]	4.4	[3]
K043	NA		2,4-Dichlorophenol	120-83-2	0.044		0.38	[3]
			2,6-Dichlorophenol	187-65-0	0.044		0.34	[3]
			2,4,5-Trichlorophenol	95-95-4	0.18		8.2	[3]
			2,4,6-Trichlorophenol	88-06-2	0.035		7.6	[3]
			Tetrachlorophenols (Total)		NA		0.68	[3]
			Pentachlorophenol	87-86-5	0.089		1.9	[3]
			Tetrachloroethene	79-01-6	0.056		1.7	[3]
			Hexachlorodibenzo-p-dioxins		0.000063		0.001	[3]
			Hexachlorodibenzofurans		0.000063		0.001	[3]
			Pentachlorodibenzo-p-dioxins		0.000063		0.001	[3]
			Pentachlorodibenzo furans		0.000063		0.001	[3]
			Tetrachlorodibenzo-p-dioxins		0.000063		0.001	[3]
			Tetrachlorodibenzofurans		0.000063		0.001	[3]
K045	NA	Table CCWE in 268.41.	Lead	7439-92-1	0.037		NA	
K048	NA	Table CCWE in 268.41.	Benzene	71-43-2	0.14	[3]	14	[3]

268.43 TABLE CCW.—CONSTITUENT CONCENTRATIONS IN WASTES—Continued

Waste code	Commercial chemical name	See also	Regulated hazardous constituent	CAS number for regulated hazardous constituent	Wastewaters		Nonwastewaters	
					Concentration (mg/l)	Notes	Concentration (mg/kg)	Notes
K049	NA	Table CCWE in 268.41	Benzo(a)pyrene	50-32-8	0.061	(2)	12	(2)
			Bis(2-ethylhexyl) phthalate	117-81-7	0.28	(2)	7.3	(2)
			Chrysene	218-01-9	0.059	(2)	15	(2)
			Di-n-butyl phthalate	84-74-2	0.057	(2)	3.6	(2)
			Ethylbenzene	100-41-4	0.057	(2)	14	(2)
			Fluorene	86-73-7	0.059	(2)	NA	
			Naphthalene	91-20-3	0.059	(2)	42	(2)
			Phenanthrene	85-01-8	0.059	(2)	34	(2)
			Phenol	108-95-2	0.039	(2)	3.6	(2)
			Pyrene	129-00-0	0.067	(2)	36	(2)
			Toluene	108-88-3	0.080	(2)	14	(2)
			Xylene(s)		0.32	(2)	22	(2)
			Cyanides (Total)	57-12-5	0.028	(2)	1.8	(2)
			Chromium (Total)	7440-47-32	0.2		NA	
			Lead	7439-92-1	0.037		NA	
			Anthracene	120-12-7	0.059	(2)	28	(2)
K050	NA	Table CCWE in 268.41.	Benzene	71-43-2	0.14	(2)	14	(2)
			Benzo(a)pyrene	117-81-7	0.061	(2)	12	(2)
			Bis(2-ethylhexyl) phthalate	75-150-0	0.28	(2)	7.3	(2)
			Carbon disulfide	75-15-0	0.014	(2)	NA	
			Chrysene	2218-01-9	0.059	(2)	15	(2)
			2,4-Dimethyl phenol	105-67-9	0.036	(2)	NA	
			Ethylbenzene	100-41-4	0.057	(2)	14	(2)
			Naphthalene	91-20-3	0.059	(2)	42	(2)
			Phenanthrene	85-01-8	0.059	(2)	34	(2)
			Phenol	108-95-2	0.039	(2)	3.6	(2)
			Pyrene	129-00-0	0.067	(2)	36	(2)
			Toluene	108-88-3	0.08	(2)	14	(2)
			Xylene(s)		0.32	(2)	22	(2)
			Cyanides (Total)	56-12-5	0.028	(2)	1.8	(2)
			Chromium (Total)	7440-47-32	0.2		NA	
			Lead	7439-92-1	0.037		NA	
K051	NA	Table CCWE in 268.41	Benzo(a)pyrene	50-32-8	0.061	(2)	12	(2)
			Phenol	108-95-2	0.039	(2)	3.6	(2)
			Cyanides (Total)	57-12-5	0.028	(2)	1.8	(2)
			Chromium (Total)	7440-47-32	0.2		NA	
			Lead	7439-29-1	0.037		NA	
			Acenaphthene	83-32-9	0.059	(2)	NA	
			Anthracene	120-12-7	0.059	(2)	28	(2)
			Benzene	71-43-2	0.14	(2)	14	(2)
			Benzo(a) anthracene	50-32-8	0.059	(2)	20	(2)
K052	NA	Table CCWE in 268.41	Benzo(a)pyrene	117-81-7	0.061	(2)	12	(2)
			Bis(2-ethylhexyl) phthalate	75-15-0	0.28	(2)	7.3	(2)
			Chrysene	2218-01-9	0.059	(2)	15	(2)
			Di-n-butyl phthalate	105-67-9	0.057	(2)	3.6	(2)
			Ethylbenzene	100-41-4	0.057	(2)	14	(2)
			Fluorene	86-73-7	0.059	(2)	NA	
			Naphthalene	91-20-3	0.059	(2)	42	(2)
			Phenanthrene	85-01-8	0.059	(2)	34	(2)
			Phenol	108-95-2	0.039	(2)	3.6	(2)
			Pyrene	129-00-0	0.067	(2)	36	(2)
			Toluene	108-88-3	0.08	(2)	14	(2)
			Xylene(s)		0.32	(2)	22	(2)
			Cyanides (Total)	57-12-5	0.028	(2)	1.8	(2)
			Chromium (Total)	7440-47-32	0.2		NA	
			Lead	7439-92-1	0.037		NA	
			Benzene	71-43-2	0.14	(2)	14	(2)
			Benzo(a)pyrene	50-32-8	0.061	(2)	12	(2)
			o-Cresol	95-48-7	0.11	(2)	6.2	(2)
			p-Cresol	106-44-5	0.77	(2)	6.2	(2)
			2,4-Dimenthylphenol	105-67-9	0.036	(2)	NA	
			Ethylbenzene	100-41-4	0.057	(2)	14	(2)
			Naphthalene	91-20-3	0.059	(2)	42	(2)
			Phenanthrene	85-01-8	0.059	(2)	34	(2)
			Phenol	108-95-2	0.039	(2)	3.6	(2)
			Toluene	108-88-3	0.08	(2)	14	(2)
			Xylenes		0.32	(2)	22	(2)
			Cyanides (Total)	56-12-5	0.028	(2)	1.8	(2)
			Chromium (Total)	7440-47-32	0.2		NA	
			Lead	7439-92-1	0.037		NA	
K060	NA	NA	Benzene	71-43-2	0.17	(12)	0.071	(2)
			Benzo(a)pyrene	50-32-8	0.035	(12)	3.6	(2)
			Naphthalene	91-20-3	0.028	(12)	3.4	(2)
			Phenol	108-95-2	0.042	(12)	3.4	(2)
			Cyanides (Total)	57-12-5	1.9		1.2	
K061	NA	Table CCWE in 268.41.	Cadmium	7440-43-9	1.61		NA	
			Chromium (Total)	7440-47-32	0.32		NA	
			Lead	7439-92-1	0.51		NA	
			Nickel	7440-02-0	0.44		NA	
K062	NA	Table CCWE in 268.41	Chromium (Total)	7440-47-32	0.32		NA	
			Lead	7439-92-1	0.04		NA	
			Nickel	7440-02-0	0.44		NA	
K069	NA	Table CCWE in 268.41 and Table 2 in 268.42.	Cadmium	7440-43-9	1.6		NA	
			Lead	7439-92-1	0.51		NA	
K071	NA	Table CCWE in 268.41.	Mercury	7439-97-6	0.030		NA	
K073	NA	NA	Carbon tetrachloride	56-23-5	0.057	(2)	6.2	(2)
			Chloroform	67-66-3	0.046	(2)	6.2	(2)
			Hexachloroethane	67-72-1	0.055	(2)	30	(2)

268.43 TABLE CCW.—CONSTITUENT CONCENTRATIONS IN WASTES—Continued

Waste code	Commercial chemical name	See also	Regulated hazardous constituent	CAS number for regulated hazardous constituent	Wastewaters		Nonwastewaters	
					Concentration (mg/l)	Notes	Concentration (mg/kg)	Notes
			Tetrachloroethane	127-18-4	0.056	[2]	6.2	[3]
K083	NA	Table CCWE in 268.41	1,1,1-Trichloroethane	71-55-6	0.054	[3]	6.2	[3]
			Benzene	71-43-2	0.14	[4]	6.6	[3]
			Aniline	62-53-3	0.81		14	
			Diphenylamine	22-39-4	0.52	[2]	NA	
			Diphenylnitrosamine	86-30-6	0.40	[2]	NA	
			Sum of Diphenylamine and Diphenylnitrosamine		NA		14	[1]
			Nitrobenzene					
			Phenol					
			Cyclohexanone					
			Nickel					
				98-95-3	0.068	[2]	14	[3]
				108-95-2	0.039		5.6	[3]
				108-94-1	0.36		NA	
K084	NA	NA		7440-02-0	0.47		NA	
K085	NA	NA	Arsenic	7440-38-2	0.79		NA	
			Benzene	71-43-2	0.14	[2]	4.4	[3]
			Chlorobenzene	108-90-7	0.057	[2]	4.4	[3]
			o-Dichlorobenzene	95-50-1	0.088	[3]	4.4	[3]
			m-Dichlorobenzene	541-73-1	0.036	[2]	4.4	[3]
			p-Dichlorobenzene	106-46-7	0.090	[3]	4.4	[3]
			1,2,4-Trichlorobenzene	120-82-1	0.055	[2]	4.4	[3]
			1,2,4,5-Tetrachlorobenzene	95-94-3	0.055	[3]	4.4	[3]
			Pentachlorobenzene	608-93-5	0.055	[3]	4.4	[3]
			Hexachlorobenzene	118-74-1	0.055	[3]	4.4	[3]
			Aroclor 1016	12674-11-2	0.013	[3]	0.92	[3]
			Aroclor 1221	11104-28-2	0.014	[3]	0.92	[3]
			Aroclor 1232	11141-16-5	0.013	[3]	0.92	[3]
			Aroclor 1242	53469-21-9	0.017	[3]	0.92	[3]
			Aroclor 1248	12672-29-6	0.013	[3]	0.92	[3]
			Aroclor 1254	11097-69-1	0.014	[3]	1.8	[3]
			Aroclor 1260	11096-82-5	0.014	[3]	1.8	[3]
K086	NA	Table CCWE in 268.41.	Acetone	67-64-1	0.28		160	
			Acetophenone	96-86-2	0.010		9.7	[3]
	Bis(2-ethylhexyl) phthalate			117-81-7	0.28	[2]	[1]	
	n-Butyl alcohol			71-36-3	5.6		[1]	
	Butylbenzylphthalate			85-68-7	0.017	[2]	7.9	
	Cyclohexanone			108-94-1	0.36		NA	
	1,2 Dichlorobenzene			95-50-1	0.088		[3]	
	Diethyl phthalate			84-66-2	0.20	[2]	28	[3]

	Dimethyl phthalate			131-11-3	0.047	[2]	28	[3]
	Di-n-butyl phthalate			84-74-2	0.057	[2]	28	[3]
	Di-n-octyl phthalate			117-84-0	0.017	[2]	28	[3]
	Ethyl acetate			141-78-6	0.34	[2]	33	[3]
	Ethylbenzene			100-41-4	0.057	[2]	6.0	
	Methanol			67-56-1	5.6	[2]	NA	[3]
	Methyl isobutyl ketone			108-10-1	0.14		33	[3]
	Methyl ethyl ketone			78-93-3	0.28		36	[3]
	Methylene chloride			75-09-2	0.089	[2]	33	[3]
	Naphthalene			91-20-3	0.059	[2]	3.1	[3]
	Nitrobenzene			98-95-3	0.068	[2]	14	[3]
	Toluene			108-88-3	0.080	[2]	28	[3]
	1,1,1-Trichloroethane			71-55-6	0.054	[2]	5.6	[3]
	Trichloroethylene			79-01-6	0.054	[2]	5.6	[3]
	Xylenes (Total)				0.32	[2]	28	[3]
	Cyanides (Total)			57-12-5	1.9		1.5	
	Chromium (Total)			7440-47-32	0.32		NA	
	Lead			7439-92-1	0.037		NA	
K087	NA	Table CCWE in 268.41	Acenaphthalene	208-96-8	0.059	[2]	3.4	
			Lead	7439-92-1	0.037		NA	
			Chrysene	218-01-9	0.059	[2]	3.4	[3]
			Fluoranthene	206-44-0	0.068	[2]	3.4	[3]
			Indeno (1,2,3-cd) pyrene	193-39-5	0.0055	[2]	3.4	[3]
			Naphthalene	91-20-3	0.059	[2]	3.4	[3]
			Phenanthrene	85-01-8	0.059	[2]	3.4	[3]
			Toluene	108-88-3	0.08	[2]	0.65	[3]
			Xylenes		0.32	[2]	0.07	[3]
			Benzene	71-43-2	0.14	[2]	0.071	[3]
			Lead	7439-92-1	0.037		NA	
K093	NA		Phthalic anhydride (measured as Phthalic acid)	85-44-9	0.069		28	[3]
K094	NA		Phthalic anhydride (measured as Phthalic acid)	85-44-9	0.069		28	[3]
K095	NA	NA	1,1,1,2-Tetrachloroethane	630-20-6	0.057		5.6	[3]
			1,1,2,2-Tetrachloroethane	79-34-6	0.057		5.6	[3]
			Tetrachloroethene	127-18-4	0.056		6.0	[3]
			1,1,2-Trichloroethane	79-00-5	0.054		6.0	[3]
			Trichloroethylene	79-01-6	0.054		5.6	[3]
			Hexachloroethane	67-72-1	0.055		28	[3]
			Pentachloroethane	76-01-7	0.055		5.6	[3]
K096	NA	NA	1,1,1,2-Tetrachloroethane	630-20-6	0.057		5.6	[3]
			1,1,2,2-Tetrachloroethane	79-34-6	0.057		5.6	[3]
			Tetrachloroethene	127-18-4	0.056		6.0	[3]
			1,1,2-Trichloroethane	79-00-5	0.054		6.0	[3]
			Trichloroethene	79-01-6	0.054		5.6	[3]
			Trichloroethylene	79-01-6	0.054		5.6	[3]
			1,3-Dichlorobenzene	541-73-1	0.036		5.6	[3]

268 43 TABLE CCW.—CONSTITUENT CONCENTRATIONS IN WASTES—Continued

Waste code	Commercial chemical name	See also	Regulated hazardous constituent	CAS number for regulated hazardous constituent	Wastewaters		Nonwastewaters	
					Concentration (mg/l)	Notes	Concentration (mg/kg)	Notes
K097	NA	NA	Pentachloroethane	76-01-7	0 055		5 6	(1)
			1,2 4-Trichlorobenzene	120-82-1	0 055		19	(1)
			Hexachlorocyclopentadiene	77-47-4	0 057	(2)	2 4	(1)
			Chlordane	57-74-9	0 0033	(2)	0.26	(1)
			Heptachlor	76-44-8	0 0012	(2)	0 066	(1)
K098	NA	NA	Heptachlor epoxide	1024-57-3	0 016	(2)	0 066	(1)
K099	NA	NA	Toxaphene	8001-35-1	0 0095	(2)	2 6	(1)
			2 4 Dichlorophenoxyacetic acid	94-75-7	1 0	(1)	1 0	(1)
			Hexachlorodibenzo-p-dioxins		0 001	(1)	0 001	(1)
			Hexachlorodibenzofurans		0 001	(1)	0 001	(1)
			Pentachlorodibenzo-p-dioxins		0 001	(1)	0 001	(1)
			Pentachlorodibenzofurans		0 001	(1)	0 001	(1)
			Tetrachlorodibenzo-p-dioxins		0 001	(1)	0 001	(1)
			Tetrachlorodibenzofurans		0 001	(1)	0 001	(1)
K100	NA	Table CCWE in 268 41	Cadmium	7440-43-9	1 6		NA	
			Chromium (Total)	7440-47-32	0.32		NA	
			Lead	7439-92-1	0 51		NA	
K101	NA	NA	o-Nitroaniline		0 27	(1)	14	(1)
			Arsenic	7440-38-2	0 79		NA	
			Cadmium	7440-43-9	0 24		NA	
			Lead	7439-92-1	0 17		NA	
			Mercury	7439-97-6	0 082		NA	
K102	NA	Table CCWE in 268 41	o-Nitrophenol		0 028	(1)	13	(1)
			Arsenic	7440-38-2	0 79		NA	
			Cadmium	7440-43-9	0.24		NA	
			Lead	7439-92-1	0 17		NA	
			Mercury	7439-97-6	0 082		NA	
K103	NA	NA	Aniline	62-53-3	4 5		5 6	(1)
			Benzene	71-43-2	0 15		6 0	(1)
			2 4-Dinitrophenol	51-28-5	0 61		5 6	(1)
			Nitrobenzene	98-95-3	0 073		5 6	(1)
			Phenol	108-95-2	1 4		5 6	(1)
K104	NA	NA	Aniline	62-53-3	4 5		5 6	(1)
			Benzene	71-43-2	0 15		6.0	(1)
			2 4-Dinitrophenol	51-28-5	0 61		5 6	(1)
			Nitrobenzene	98-95-3	0 073		5 6	(1)
			Phenol	108-95-2	1 4		5 6	(1)
			Cyanides (Total)	57-12-5	2 7		1 8	(1)
K105	NA	NA	Benzene	71-43-2	0 14		4 4	(1)
			Chlorobenzene	108-90-7	0 057		4 4	(1)
			o-Dichlorobenzene	95-50-1	0 088		4 4	(1)
			p-Dichlorobenzene	106-46-7	0 090		4 4	(1)
			2,4,5-Trichlorophenol	95-95-4	0 18		4.4	(1)
			2 4 6-Trichlorophenol	88-06-2	0 035		4 4	(1)
			2-Chlorophenol	95-57-8	0 044		4 4	(1)
			Phenol	108-95-2	0 039		4 4	(1)
K106	NA	Table CCWE in 268 41 and Table 2 in 268 42	Mercury	7439-97-6	0 030		NA	
K111	NA	Table CCWE in 268 41.	2 4-Dinitrotoluene	121-14-2	0 32		140	(1)
K115	NA		Nickel	7440-02-0	0 47		NA	
K117	NA		Ethylene dibromide	106-93-4	0 028		15	(1)
			Methyl bromide	74-83-9	0 11		15	(1)
			Chloroform	67-66-3	0 046		5 6	(1)
K118	NA		Ethylene dibromide	106-93-4	0 028		15	(1)
			Methyl bromide	74-83-9	0 11		15	(1)
			Chloroform	67-66-3	0 046		5 6	(1)
K131	NA		Methyl bromide	74-83-9	0 11		15	(1)
K132	NA		Methyl bromide	74-83-9	0 11		15	(1)
K136	NA		Ethylene dibromide	106-93-4	0 028		15	(1)
			Methyl bromide	74-83-9	0 11		15	(1)
			Chloroform	67-66-3	0 046		5 6	(1)
P004	Aldrin	NA	Aldrin	309-00-2	0 021	(2)	0 066	(1)
P010	Arsenic acid	Table CCWE in 268.41.	Arsenic	7440-38-2	0 79		NA	
P011	Arsenic pentoxide	Table CCWE in 268 41	Arsenic	7440-38-2	0 79		NA	
P012	Arsenic trioxide	Table CCWE in 268 41.	Arsenic	7440-38-2	0 79		NA	
P013	Barium cyanide	Table CCWE in 268 41	Cyanides (Total)	57-12-5	1.9		110	
			Cyanides (Amenable)	57-12-5	0.1		9 1	
P020	2-sec-Butyl-4 6-dinitrophenol (Dinoseb)	NA	2-sec-Butyl-4,6-dinitrophenol (Dinoseb).	88-85-7	0.066		2.5	(1)
P021	Calcium cyanide	NA	Cyanides (Total)	57-12-5	1 9		110	
			Cyanides (Amenable)	57-12-5	0 1		9 1	
P022	Carbon disulfide	Table 2 in 268 42	Carbon disulfide	75-15-0	0.014		NA	
P024	p-Chloroaniline	NA	p-Chloroaniline	106-47-8	0 46		16	(1)
P029	Copper cyanide	NA	Cyanides (Total)	57-12-5	1 9		110	
			Cyanides (Amenable)	57-12-5	0 1		9 1	
P030	Cyanides (soluble salts and complexes.	NA	Cyanides (Total)	57-12-5	1 9		110	
			Cyanides (Amenable)	57-12-5	0 1		9 1	
P036	Dichlorophenylarsine	Table CCWE in 268.41	Arsenic	7440-38-2	0.79		NA	
P037	Dieldrin	NA	Dieldrin	60-67-1	0.017	(2)	0 13	(1)
P038	Diethylarsine	Table CCWE in 268.41.	Arsenic	7440-38-2	0.79		NA	
P039	Disulfoton	NA	Disulfoton	298-04-4	0.017		0 1	(1)
P047	4,6-Dinitro-o-cresol	NA	4,6-Dinitro-o-cresol	534-52-1	0.28	(2)	160	(1)
P048	2,4-Dinitrophenol	NA	2,4-Dinitrophenol	51-28-5	0 12	(2)	160	(1)
P050	Endosulfan	NA	Endosulfan I	939-98-8	0 023	(2)	0 066	(1)

268.43 TABLE CCW.—CONSTITUENT CONCENTRATIONS IN WASTES—Continued

Waste code	Commercial chemical name	See also	Regulated hazardous constituent	CAS number for regulated hazardous constituent	Wastewaters		Nonwastewaters	
					Concentration (mg/l)	Notes	Concentration (mg/kg)	Notes
			Endosulfan II	33213-6-5	0.029	[2]	0.13	[1]
			Endosulfan sulfate	1031-07-8	0.029	[2]	0.13	[1]
P051	Endrin	NA	Endrin	72-20-8	0.0028	[4]	0.13	[1]
			Endrin aldehyde	7421-93-4	0.025	[2]	0.13	[1]
P056	Fluoride	Table 2 in 268.42	Fluoride	16984-48-8	35		NA	
P059	Heptachlor	NA	Heptachlor	76-44-8	0.0012	[2]	0.066	[1]
			Heptachlor epoxide	1024-57-3	0.016	[2]	0.066	[1]
P060	Isodrin	NA	Isodrin	465-73-6	0.021	[2]	0.066	[1]
P063	Hydrogen cyanide	NA	Cyanides (Total)	57-12-5	1.9		110	
			Cyanides (Amenable)	57-12-5	0.10		9.1	
P065	Mercury fulminate	Table CCWE in 268.41 and Table 2 in 268.42.	Mercury	7439-97-6	0.030		NA	
P071	Methyl parathion	NA	Methyl parathion	298-00-0	0.025		0.1	[1]
P073	Nickel carbonyl	Table CCWE in 268.41	Nickel	7440-02-0	0.32		NA	
P074	Nickel cyanide	Table CCWE in 268.41.	Cyanides (Total)	57-12-5	1.9		110	
			Cyanides (Amenable)	57-12-5	0.10		9.1	
			Nickel	7440-02-0	0.44		NA	
P077	p-Nitroaniline	NA	p-Nitroaniline	100-01-6	0.028	[2]	28	[1]
P082	N-Nitrosodimethylamine	Table 2 in 268.42	N-Nitrosodimethylamine	62-75-9	0.40	[2]	NA	
P089	Parathion	NA	Parathion	56-38-2	0.025		0.1	[1]
P092	Phenylmercury acetate.	Table CCWE in 268.41 and Table 2 in 268.42	Mercury	7439-97-6	0.030		NA	
P094	Phorate	NA	Phorate	298-02-2	0.025		0.1	[1]
P097	Famphur	NA	Famphur	52-85-7	0.025		0.1	[1]
P098	Potassium cyanide	NA	Cyanides (Total)	57-12-5	1.9		110	
			Cyanides (Amenable)	57-12-5	0.10		9.1	
P099	Potassium silver cyanide	Table CCWE in 268.41	Cyanides (Total)	57-12-5	1.9		110	
			Cyanides (Amenable)	57-12-5	0.1		9.1	
			Silver	7440-22-4	0.29		NA	
P101	Ethyl cyanide (Propanenitrile)	NA	Ethyl cyanide (Propanenitrile)	107-12-0	0.24	[2]	360	[1]
P103	Selenourea	Table CCWE in 268.41.	Selenium	7782-49-2	1.0	[2]	NA	
P104	Silver cyanide	Table CCWE in 268.41	Cyanides (Total)	57-12-5	1.9		110	
			Cyanides (Amenable)	57-12-5	0.10		9.1	
			Silver	7440-22-4	0.29		NA	
P106	Sodium cyanide	NA	Cyanides (Total)	57-12-5	1.9		110	
			Cyanides (Amenable)	57-12-5	0.10		9.1	
P110	Tetraethyl lead	Table CCWE in 268.41 and Table 2 in 268.42	Lead	7439-92-1	0.040		NA	
P113	Thallic oxide	Table 2 in 268.42	Thallium	7440-28-0	0.14	[2]	NA	
P114	Thallium selenite	Table CCWE in 268.41	Selenium	7782-49-2	1.0		NA	
P115	Thallium(I)sulfate	Table 2 in 268.42	Thallium	7440-28-0	0.14	[2]	NA	
P119	Ammonia vanadate	Table 2 in 268.42	Vanadium	7440-62-2	28	[2]	NA	
P120	Vanadium pentoxide	Table 2 in 268.42	Vanadium	7440-62-2	28	[2]	NA	
P121	Zinc cyanide	NA	Cyanides Total)	57-12-5	1.9		110	
			Cyanides (Amenable)	57-12-5	0.10		9.1	
P123	Toxaphene	NA	Toxaphene	8001-35-1	0.0095	[2]	1.3	[1]
U002	Acetone	NA	Acetone	67-64-1	0.28		160	[1]
U003	Acetonitrile	Table 2 in 268.42	Acetonitrile	75-05-8	0.17		0.17	
U004	Acetophenone	NA	Acetophenone	98-86-2	0.010	[1]	9.7	[1]
U005	2-Acetylaminofluorene.	NA	2-Acetylaminofluorene	53-96-3	0.059	[2]	140	[1]
U009	Acrylonitrile	NA	Acrylonitrile	107-13-1	0.24	[2]	84	[1]
U012	Aniline	NA	Aniline	62-53-3	0.81		14	[1]
U018	Benz(a)anthracene	NA	Benz(a)anthracene	56-55-3	0.059	[2]	8.2	[1]
U019	Benzene	NA	Benzene	71-43-2	0.14	[2]	35	[1]
U022	Benzo(a)pyrene	NA	Benzo(a)pyrene	50-32-8	0.061	[2]	8.2	[1]
U024	Bis(2-chloroethoxy) methane	NA	Bis(2-chloroethoxy) methane	111-91-1	0.036		7.2	[1]
U025	Bis(2-chloroethyl) ether	NA	Bis(2-chloroethyl) ether	111-44-4	0.033		7.2	[1]
U027	Bis(2-chloroisopropyl) ether	NA	Bis(2-chloroisopropyl) ether	39638-32-9	0.055	[2]	7.2	[1]
U028	Bis(2-ethylhexyl) phthalate		Bis(2-ethylhexyl) phthalate	117-81-7	0.28		28	[1]
U029	Bromomethane (Methyl bromide).	NA	Bromomethane (Methyl bromide).	74-83-9	0.11	[1]	15	[1]
U030	4-Bromophenyl phenyl ether	NA	4-Bromophenyl phenyl ether	101-55-3	0.055	[1]	15	[1]
U031	n-Butyl alcohol	NA	n-Butyl alcohol	71-36-3	5.6		2.6	[1]
U032	Calcium chromate	Table CCWE in 268.41	Chromium (Total)	7440-47-32	0.32		NA	
U036	Chlordane (alpha and gamma)	NA	Chlordane (alpha and gamma).	57-74-9	0.0033	[2]	0.13	[1]
U037	Chlorobenzene	NA	Chlorobenzene	108-90-7	0.057	[2]	5.7	[1]
U038	Chlorobenzilate	Table 2 in 268.42	Chlorobenzilate	510-15-6	0.10	[2]	NA	
U039	p-Chloro-m-cresol	NA	p-Chloro-m-cresol	59-50-7	0.018	[2]	14	[1]
U043	Vinyl chloride	NA	Vinyl chloride	75-01-4	0.27	[2]	33	[1]

268.43 TABLE CCW.—CONSTITUENT CONCENTRATIONS IN WASTES—Continued

Waste code	Commercial chemical name	See also	Regulated hazardous constituent	CAS number for regulated hazardous constituent	Wastewaters		Nonwastewaters	
					Concentration (mg/l)	Notes	Concentration (mg/kg)	Notes
U044	Chloroform	NA	Chloroform	67-66-3	0.046	[2]	5.6	[1]
U045	Chloromethane (Methyl chloride)	NA	Chloromethane (Methyl chloride)	74-87-3	0.19	[2]	33	[1]
U047	2-Chloro- naphthalene	NA	2-Chloro-naphthalene	91-58-7	0.055	[2]	5.6	[1]
U048	2-Chlorophenol	NA	2-Chlorophenol	95-57-8	0.044	[2]	5.7	[1]
U050	Chrysene	NA	Chrysene	218-01-9	0.059	[2]	8.2	[1]
U051	Creosote	Table CCWE in 268.41.	Naphthalene	91-20-3	0.031		1.5	[1]
			Pentachlorophenol	87-86-5	0.18		7.4	[1]
			Phenanthrene					
			Pyrene	85-01-8	0.031		1.5	[1]
			Toluene	129-00-0	0.028		1.5	[1]
			Xylenes (Total)	108-88-3	0.028		28	[1]
			Lead	7439-92-1	0.032		33	[1]
U052	Cresols (Cresylic acid)	NA	o-Cresol	95-48-7	0.037		NA	
			Cresols (m- and p- isomers)		0.11	[2]	5.6	[1]
U057	Cyclohexanone	Table 2 in 268.42	Cyclohexanone	108-94-1	0.77	[2]	3.2	[1]
U060	DDD	NA	o,p'-DDD	53-19-0	0.36		NA	
			p,p'-DDD	72-54-8	0.023		0.087	[1]
U061	DDT	NA	o,p'-DDT	789-02-6	0.023		0.087	[1]
			p,p'-DDT	50-29-3	0.0039	[3]	0.087	[1]
			o,p'-DDD	53-19-0	0.0039	[3]	0.087	[1]
			p,p'-DDD	72-54-8	0.023	[3]	0.087	[1]
			o,p'-DDE	3424-82-6	0.023	[3]	0.087	[1]
			p,p'-DDE	72-55-9	0.031	[3]	0.087	[1]
U063	Dibenzo-(a,h)-anthracene	NA	Dibenzo(a,h) anthracene	53-70-3	0.031	[3]	0.087	[1]
U066	1,2-Dibromo-3-chloropropane	NA	1,2-Dibromo-3-chloropropane	96-12-8	0.055	[2]	8.2	[1]
U067	1,2-Dibromo ethane (Ethylene dibromide).	NA	1,2-Dibromo-ethane (Ethylene dibromide)	106-93-4	0.11	[2]	15	[1]
U068	Dibromomethane	NA	Dibromomethane	74-95-3	0.028	[2]	15	[1]
U069	Di-n-butyl phthalate		Di-n-butyl phthalate	84-74-2	0.11	[2]	15	[1]
U070	o-Dichlorobenzene	NA	o-Dichlorobenzene	95-50-1	0.057		28	[1]
U071	m-Dichlorobenzene	NA	m-Dichlorobenzene	541-73-1	0.088	[2]	6.2	[1]
U072	p-Dichlorobenzene	NA	p-Dichlorobenzene	106-46-7	0.036		6.2	[1]
U075	Dichlorodifluoromethane.	NA	Dichlorodifluoromethane	75-71-8	0.090	[2]	6.2	[1]
					0.23	[2]	7.2	[1]
U076	1,1-Dichloroethane	NA	1,1-Dichloroethane	75-34-3	0.059	[2]	7.2	[1]
U077	1,2-Dichloroethane	NA	1,2-Dichloroethane	107-06-2	0.21	[2]	7.2	[1]
U078	1,1-Dichloroethylene	NA	1,1-Dichloroethylene	75-35-4	0.025	[2]	33	[2]

U079	1,2-Dichloroethylene	NA	trans-1,2-Dichloroethylene	156-60-5	0.054	[2]	33	[1]
U080	Methylene chloride	NA	Methylene chloride	75-09-2	0.089	[2]	33	[1]
U081	2,4-Dichlorophenol	NA	2,4-Dichlorophenol	120-83-2	0.044	[2]	14	[1]
U082	2,6-Dichlorophenol	NA	2,6-Dichlorophenol	87-65-0	0.044	[2]	14	[1]
U083	1,2-Dichloropropane	NA	1,2-Dichloropropane	78-87-5	0.85	[2]	18	[1]
U084	1,3-Dichloropropene	NA	cis-1,3-Dichloropropylene	10061-01-5	0.036	[2]	18	[1]
			trans-1,3-Dichloropropylene	10061-02-6	0.036	[2]	18	[1]
U088	Diethyl phthalate		Diethyl phthalate	84-66-2	0.2		28	[1]
U093	p-Dimethylaminoazo-benzene	Table 2 in 268.42	p-Dimethylaminoazobenzene	60-11-7	0.13	[2]	NA	
U101	2,4-Dimethylphenol	NA	2,4-Dimethylphenol	105-67-9	0.036	[2]	14	[1]
U102	Dimethyl phthalate		Dimethyl phthalate	131-11-3	0.047		28	[1]
U105	2,4-Dinitrotoluene	NA	2,4-Dinitrotoluene	121-14-2	0.32	[2]	140	[1]
U106	2,6-Dinitrotoluene	NA	2,6-Dinitrotoluene	606-20-2	0.55	[2]	28	[1]
U107	Di-n-octyl phthalate		Di-n-octyl phthalate	117-84-0	0.017		28	[1]
U108	1,4-Dioxane	NA	1,4-Dioxane	123-91-1	0.12	[2]	170	[1]
U111	Di-n-propylnitrosoamine.	NA	Di-n-propylnitrosoamine	621-64-7	0.40	[2]	14	[1]
U112	Ethyl acetate	NA	Ethyl acetate	141-78-6	0.34	[2]	33	[1]
U117	Ethyl ether	NA	Ethyl ether	60-29-7	0.12	[2]	160	[1]
U118	Ethyl methacrylate	NA	Ethyl methacrylate	97-63-2	0.14	[2]	160	[1]
U120	Fluoranthene	NA	Fluoranthene	206-44-0	0.068	[2]	8.2	[1]
U121	Trichloromonofluoro-methane	NA	Trichloromonofluoromethane	75-69-4	0.020	[2]	33	[1]
U127	Hexachlorobenzene	NA	Hexachlorobenzene	118-74-1	0.055	[2]	37	[1]
U128	Hexachlorobutadiene	NA	Hexachlorobutadiene	87-68-3	0.055	[2]	28	[1]
U129	Lindane	NA	alpha-BHC	319-84-6	0.00014	[2]	0.66	[1]
			beta-BHC	319-85-7	0.00014	[2]	0.66	[1]
			Delta-BHC	319-86-8	0.023	[2]	0.66	[1]
			gamma-BHC (Lindane)	58-89-9	0.0017	[2]	0.66	[1]
U130	Hexachlorocyclo-pentadiene.	NA	Hexachlorocyclopentadiene	77-47-7	0.057	[2]	3.6	[1]
U131	Hexachloroethane	NA	Hexachloroethane	67-72-1	0.055	[2]	28	[1]
U134	Hydrogen fluoride	Table 2 in 268.42	Fluoride	16964-48-8	35		NA	
U136	Cacodylic acid	Table CCWE in 268.41.	Arsenic	7440-38-2	0.79		NA	
U137	Indeno(1,2,3-c,d)pyrene.	NA	Indeno(1,2,3-c,d)pyrene	193-39-5	0.0055	[2]	8.2	[1]
U138	Iodomethane	NA	Iodomethane	74-88-4	0.19	[2]	65	[1]
U140	Isobutyl alcohol	NA	Isobutyl alcohol	78-83-1	5.6		170	[1]
U141	Isosafrole	NA	Isosafrole	120-58-1	0.081		2.6	[1]
U142	Kepone	NA	Kepone	143-50-8	0.0011		0.13	[1]
U144	Lead acetate	Table CCWE in 268.41.	Lead	7439-92-1	0.040		NA	
U145	Lead phosphate	Table CCWE in 268.41.	Lead	7439-92-1	0.040		NA	
U146	Lead subacetate	Table CCWE in 268.41.	Lead	7439-92-1	0.040		NA	

268.43 TABLE CCW.—CONSTITUENT CONCENTRATIONS IN WASTES—Continued

Waste code	Commercial chemical name	See also	Regulated hazardous constituent	CAS number for regulated hazardous constituent	Wastewaters		Nonwastewaters	
					Concentration (mg/l)	Notes	Concentration (mg/kg)	Notes
U151	Mercury	Table CCWE in 268.41 and Table 2 in 268.42.	Mercury	7439-97-6	0.030		NA	
U152	Methacrylonitrile	NA	Methacrylonitrile	126-98-7	0.24	[1]	84	[1]
U154	Methanol	NA	Methanol	67-56-1	5.6		NA	
U155	Methapyrilene	NA	Methapyrilene	91-80-5	0.081		1.5	[1]
U157	3-Methylcholanthrene.	NA	3-Methylcholanthrene	56-49-5	0.0055	[2]	15	[1]
U158	4,4'-Methylenebis(2-chloroaniline)	NA	4,4'-Methylenebis(2-chloroaniline)	101-14-4	0.50	[4]	35	[1]
U159	Methyl ethyl ketone	NA	Methyl ethyl ketone	78-93-3	0.28		36	[1]
U161	Methyl isobutyl ketone	NA	Methyl isobutyl ketone	108-10-1	0.14		33	[1]
U162	Methyl methacrylate	NA	Methyl methacrylate	80-62-6	0.14		160	[1]
U165	Naphthalene	NA	Naphthalene	91-20-3	0.059	[2]	3.1	[1]
U168	2-Naphthylamine	Table 2 in 268.42	2-Naphthylamine	91-59-8	0.52	[2]	NA	
U169	Nitrobenzene	NA	Nitrobenzene	98-95-3	0.068	[2]	14	[1]
U170	4-Nitrophenol	NA	4-Nitrophenol	100-02-7	0.12	[2]	28	[1]
U172	n-Nitrosodi-n-butylamine	NA	n-Nitrosodi-n-butylamine	924-16-3	0.40	[4]	17	[1]
U174	N-Nitrosodiethylamine.	NA	n-Nitrosodiethylamine	55-18-5	0.40	[2]	28	[1]
U179	N-Nitrosopiperidine	NA	n-Nitrosopiperidine	100-75-4	0.013	[2]	35	[1]
U180	N-Nitrosopyrrolidine	NA	n-Nitrosopyrrolidine	930-55-2	0.013	[2]	35	[1]
U181	5-Nitro-o-toluidine	NA	5-Nitro-o-toluidine	99-55-8	0.32	[2]	28	[1]
U183	Pentachlorobenzene	NA	Pentachlorobenzene	608-93-5	0.055	[2]	37	[1]
U185	Pentachloronitrobenzene	NA	Pentachloronitrobenzene	82-68-8	0.055	[2]	4.8	[1]
U187	Phenacetin	NA	Phenacetin	62-44-2	0.081		16	[1]
U188	Phenol	NA	Phenol	108-95-2	0.039		6.2	[1]
U190	Phthalic anhydride (measured as Phthalic acid).		Phthalic anhydride (measured as Phthalic acid)	85-44-9	0.069		28	[1]
U192	Pronamide	NA	Pronamide	23950-58-5	0.093		1.5	[1]
U196	Pyridine	NA	Pyridine	110-86-1	0.014	[2]	16	[1]
U203	Safrole	NA	Safrole	94-59-7	0.081		22	[1]
U204	Selenium dioxide	Table CCWE in 268.41	Selenium	7782-49-2	1.0		NA	
U205	Selenium sulfide	Table CCWE in 268.41.	Selenium	7782-49-2	1.0		NA	
U207	1,2,4,5-Tetrachlorobenzene	NA	1,2,4,5-Tetrachlorobenzene	95-94-3	0.055	[2]	19	[1]
U208	1,1,1,2-Tetrachloroethane	NA	1,1,1,2-Tetrachloroethane	630-20-6	0.057		42	[1]
U209	1,1,2,2-Tetrachloroethane	NA	1,1,2,2-Tetrachloroethane	79-34-5	0.057	[2]	42	[1]
U210	Tetrachloroethylene	NA	Tetrachloroethylene	127-18-4	0.056	[2]	5.6	[1]
U211	Carbon tetrachloride	NA	Carbon tetrachloride	56-23-5	0.057	[2]	5.6	[1]
U214	Thallium(I)acetate	Table 2 in 268.42	Thallium	7440-28-0	0.14	[2]	NA	
U215	Thallium(I) carbonate	Table 2 in 268.42	Thallium	7440-28-0	0.14	[2]	NA	
U216	Thallium(I)chloride	Table 2 in 268.42	Thallium	7440-28-0	0.14	[2]	NA	
U217	Thallium(I)nitrate	Table 2 in 268.42	Thallium	7440-28-0	0.14	[2]	NA	
U220	Toluene	NA	Toluene	108-88-3	0.080	[2]	28	[1]
U225	Tribromomethane (Bromoform)	NA	Tribromomethane (Bromoform)	75-25-2	0.63	[2]	15	[1]
U226	1,1,1-Trichloroethane	NA	1,1,1-Trichloroethane	71-55-6	0.054	[2]	5.6	[1]
U227	1,1,2-Trichloroethane	NA	1,1,2-Trichloroethane	79-00-5	0.054	[2]	5.6	[1]
U228	Trichloroethylene	NA	Trichloroethylene	79-01-6	0.054	[2]	5.6	[1]
U235	tris-(2,3-Dibromopropyl) phosphate	NA	tris-(2,3-Dibromopropyl)-phosphate	126-72-7	0.025		0.10	[1]
U239	Xylenes	NA	Xylenes		0.32	[2]	28	[1]
U240	2,4-Dichlorophenoxyacetic acid.	NA	2,4-Dichlorophenoxyacetic acid	94-75-7	0.72		10	[1]
U243	Hexachloropropene	NA	Hexachloropropene	1888-71-7	0.035	[2]	28	
U247	Methoxychlor	NA	Methoxychlor	72-43-5	0.25	[2]	0.18	[1]

[1] Treatment standards for this organic constituent were established based upon incineration in units operated in accordance with the technical requirements of 40 CFR part 264 subpart O or part 265 subpart O, or based upon combustion in fuel substitution units operating in accordance with applicable technical requirements. A facility may certify compliance with these treatment standards according to provisions in 40 CFR Section 268.7.

[2] Based on analysis of composite samples.

[3] As analyzed using SW-846 Method 9010 or 9012, sample size 10 gram; distillation time one hour and fifteen minutes.

[4] Reserved

Note NA means Not Applicable.

No Land Disposal for

K005 Nonwastewaters generated by the process described in the waste listing description, and disposed after June 8, 1989, and not generated in the course of treating wastewater forms of these wastes (Based on No Generation)

K007 Nonwastewaters generated by the process described in the waste listing description, and disposed after June 8, 1989, and not generated in the course of treating wastewater forms of these wastes. (Based on No Generation)

K021 Nonwastewater forms of these wastes generated by the process described in the waste listing description and disposed after August 17, 1988, and not generated in the course of treating wastewater forms of these wastes (Based on No Generation)

K025 Nonwastewater forms of these wastes generated by the process described in the waste listing description and disposed after August 17, 1988, and not generated in the course of treating wastewater forms of these wastes (Based on No Generation)

K036 Nonwastewater forms of these wastes generated by the process described in the waste listing description and disposed after August 17, 1988, and not generated in the course of treating wastewater forms of these wastes (Based on No Generation)

K044 (Based on Reactivity)

K045 (Based on Reactivity)

K047 (Based on Reactivity)

K060 Nonwastewater forms of these wastes generated by the process described in the waste listing description and disposed after August 17, 1988, and not generated in the course of treating wastewater forms of these wastes (Based on No Generation)

K061 Nonwastewaters—High Zinc Subcategory (greater than or equal to 15% total zinc) (Based on Recycling) effective 8/8/90

K069 Non-Calcium Sulfate Subcategory—Nonwastewater forms of these wastes generated by the process described in the waste listing description and disposed after August 17, 1988, and not generated in the course of treating wastewater forms of these wastes (Based on Recycling)

K100 Nonwastewater forms of these wastes generated by the process described in the waste listing description and disposed after August 17, 1988, and not generated in the course of treating wastewater forms of these wastes (Based on No Generation)

(b) When wastes with differing treatment standards for a constituent of concern are combined for purposes of treatment, the treatment residue must meet the lowest treatment standard for the constituent of concern.

(c) Notwithstanding the prohibitions specified in paragraph (a) of this section, treatment and disposal facilities may demonstrate (and certify pursuant to §268.7(b)(5)) compliance with the treatment standards for organic constituents specified by a footnote in Table CCW in this section, provided the following conditions are satisfied:

(1) The treatment standards for the organic constituents were established based on incineration in units operated in accordance with the technical requirements of 40 CFR part 264, subpart O, or part 265, subpart O, or based on combustion in fuel substitution units operating in accordance with applicable technical requirements;

(2) The treatment or disposal facility has used the methods referenced in paragraph (c)(1) of this section to treat the organic constituents, and

(3) The treatment or disposal facility has been unable to detect the organic constituents despite using its best good-faith efforts as defined by applicable Agency guidance or standards. Until such guidance or standards are developed, the treatment or disposal facility may demonstrate such good-faith efforts by achieving detection limits for the regulated organic constituents that do not exceed an order of magnitude of the treatment standards specified in this section.

[53 FR 31218, Aug. 17, 1988, as amended at 54 FR 26649, June 23, 1989, 55 FR 22701, June 1, 1990; 56 FR 3892, Jan. 31, 1991; 57 FR 37274, Aug. 18, 1992; 58 FR 29886, May 24, 1993]

§268.44 Variance from a treatment standard.

(a) Where the treatment standard is expressed as a concentration in a waste or waste extract and a waste cannot be treated to the specified level, or where the treatment technology is not appropriate to the waste, the generator or treatment facility may petition the Administrator for a variance from the treatment standard. The petitioner must demonstrate that because the physical or chemical properties of the waste differs significantly from wastes analyzed in developing the treatment standard, the waste cannot be treated to specified levels or by the specified methods.

(b) Each petition must be submitted in accordance with the procedures in §260.20.

(c) Each petition must include the following statement signed by the petitioner or an authorized representative:

I certify under penalty of law that I have personally examined and am familiar with the information submitted in this petition and all attached documents, and that, based on my inquiry of those individuals immediately responsible for obtaining the information, I believe that the submitted information is true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment.

(d) After receiving a petition for variance from a treatment standard, the Administrator may request any additional information or samples which he may require to evaluate the petition. Additional copies of the complete petition may be requested as needed to send to affected states and Regional Offices.

(e) The Administrator will give public notice in the FEDERAL REGISTER of the intent to approve or deny a petition and provide an opportunity for public comment. The final decision on a variance from a treatment standard will be published in the FEDERAL REGISTER.

(f) A generator, treatment facility, or disposal facility that is managing a waste covered by a variance from the treatment standards must comply with the waste analysis requirements for restricted wastes found under §268.7.

(g) During the petition review process, the applicant is required to comply with all restrictions on land disposal under this part once the effective date for the waste has been reached.

(h) Where the treatment standard is expressed as a concentration in a waste or waste extract and a waste generated under conditions specific to only one site cannot be treated to the specified level, or where the treatment technology is not appropriate to the waste, the generator or treatment facility may apply to the Administrator, or his delegated representative, for a site-specific variance from a treatment standard. The applicant for a site-specific variance must demonstrate that because the physical or chemical properties of the waste differs significantly from the waste analyzed in developing the treatment standard, the waste cannot be treated to specified levels or by the specified methods.

(i) Each application for a site-specific variance from a treatment standard must include the information in §260.20(b)(1)–(4);

(j) After receiving an application for a site-specific variance from a treatment standard, the Assistant Administrator, or his delegated representative, may request any additional information or samples which may be required to evaluate the application.

(k) A generator, treatment facility, or disposal facility that is managing a waste covered by a site-specific variance from a treatment standard must comply with the waste analysis requirements for restricted wastes found under §268.7.

(l) During the application review process, the applicant for a site-specific variance must comply with all restrictions on land disposal under this part once the effective date for the waste has been reached.

(m)—(n) [Reserved]

(o) The following facilities are excluded from the treatment standard under §268.43(a), Table CCW, and are subject to the following constituent concentrations:

TABLE—WASTES EXCLUDED FROM THE TREATMENT STANDARDS UNDER §268.43(a)

Facility name[1] and address	Waste code	See also	Regulated hazardous constituent	Wastewaters		Nonwastewaters	
				Concentration (mg/l)	Notes	Concentration (mg/kg)	Notes
Craftsman Plating and Tinning, Corp., Chicago IL	F006	Table CCWE in 268.41	Cyanides (Total)	1.2	(2)	1800	(4)
			Cyanides (Amenable)	86	(2 and 3)	30	(4)
			Cadmium	1.6		NA	
			Chromium	32		NA	
			Lead	.040		NA	
			Nickel	44		NA	
Northwestern Plating Works Inc Chicago IL	F006	Table CCWE in 268.41	Cyanides (Total)	1.2	(2 and 3)	970	(4)
			Cyanides (Amenable)	86	(2)	30	(4)
			Cadmium	1.6		NA	
			Chromium	32		NA	
			Lead	.040		NA	
			Nickel	44		NA	

(1)—A facility may certify compliance with these treatment standards according to provisions in 40 CFR 268.7.
(2)—Cyanide Wastewater Standards for F006 are based on analysis of composite samples.
(3)—These facilities must comply with 0.86 mg/l for amenable cyanides in the wastewater using the alkaline chlorination system. These facilities must also comply with 40 CFR §268.7 a 4 for appropriate monitoring frequency consistent with the facilities' waste analysis plan.
(4)—Cyanide nonwastewaters are analyzed using SW–846 Method 9010 or 9012, sample size 10 grams distillation time, 1 hour and 15 minutes.
Note: NA means Not Applicable

[51 FR 40642, Nov. 7, 1986; 52 FR 21017, June 4, 1987, as amended at 53 FR 31221, Aug. 17, 1988; 54 FR 36972, Sept. 6, 1989; 56 FR 12355, Mar. 25, 1991]

§ 268.45 Treatment standards for hazardous debris.

(a) *Treatment standards.* Hazardous debris must be treated prior to land disposal as follows unless EPA determines under §261.3(e)(2) of this chapter that the debris is no longer contaminated with hazardous waste or the debris is treated to the waste-specific treatment standard provided in this subpart for the waste contaminating the debris:

(1) *General.* Hazardous debris must be treated for each "contaminant subject to treatment" defined by paragraph (b) of this section using the technology or technologies identified in Table 1 of this section.

(2) *Characteristic debris.* Hazardous debris that exhibits the characteristic of ignitability, corrosivity, or reactivity identified under §§261.21, 261.22, and 261.23 of this chapter, respectively, must be deactivated by treatment using one of the technologies identified in Table 1 of this section.

(3) *Mixtures of debris types.* The treatment standards of Table 1 in this section must be achieved for each type of debris contained in a mixture of debris types. If an immobilization technology is used in a treatment train, it must be the last treatment technology used.

(4) *Mixtures of contaminant types.* Debris that is contaminated with two or more contaminants subject to treatment identified under paragraph (b) of this section must be treated for each contaminant using one or more treatment technologies identified in Table 1 of this section. If an immobilization technology is used in a treatment train, it must be the last treatment technology used.

(5) *Waste PCBs* Hazardous debris that is also a waste PCB under 40 CFR part 761 is subject to the requirements of either 40 CFR part 761 or the requirements of this section, whichever are more stringent.

(b) *Contaminants subject to treatment.* Hazardous debris must be treated for each "contaminant subject to treatment." The contaminants subject to treatment must be determined as follows:

(1) *Toxicity characteristic debris.* The contaminants subject to treatment for debris that exhibits the Toxicity Characteristic (TC) by §261.24 of this chapter are those EP constituents for which the debris exhibits the TC toxicity characteristic.

(2) *Debris contaminated with listed waste.* The contaminants subject to treatment for debris that is contaminated with a prohibited listed hazardous waste are those constituents for which BDAT standards are established for the waste under §§268.41 and 268.43.

(3) *Cyanide reactive debris.* Hazardous debris that is reactive because of cyanide must be treated for cyanide.

(c) *Conditioned exclusion of treated debris.* Hazardous debris that has been treated using one of the specified extraction or destruction technologies in Table 1 of this section and that does not exhibit a characteristic of hazardous waste identified under subpart C, part 261, of this chapter after treatment is not a hazardous waste and need not be managed in a subtitle C facility. Hazardous debris contaminated with a listed waste that is treated by an immobilization technology specified in Table 1 is a hazardous waste and must be managed in a subtitle C facility.

(d) *Treatment residuals*—(1) *General requirements.* Except as provided by paragraphs (d)(2) and (d)(4) of this section:

(i) Residue from the treatment of hazardous debris must be separated from the treated debris using simple physical or mechanical means; and

(ii) Residue from the treatment of hazardous debris is subject to the waste-specific treatment standards provided by subpart D of this part for the waste contaminating the debris.

(2) *Nontoxic debris.* Residue from the deactivation of ignitable, corrosive, or reactive characteristic hazardous debris (other than cyanide-reactive) that is not contaminated with a contaminant subject to treatment defined by paragraph (b) of this section, must be deactivated prior to land disposal and is not subject to the waste-specific treatment standards of subpart D of this part.

(3) *Cyanide-reactive debris.* Residue from the treatment of debris that is re-

active because of cyanide must meet the standards for D003 under § 268 43.

(4) *Ignitable nonwastewater residue.* Ignitable nonwastewater residue containing equal to or greater than 10% total organic carbon is subject to the technology-based standards for D001: "Ignitable Liquids based on § 261.21(a)(1)" under § 268.42.

(5) *Residue from spalling* Layers of debris removed by spalling are hazardous debris that remain subject to the treatment standards of this section.

TABLE 1 —ALTERNATIVE TREATMENT STANDARDS FOR HAZARDOUS DEBRIS [1]

Technology description	Performance and/or design and operating standard	Contaminant restrictions [2]
A Extraction Technologies		
1 Physical Extraction		
a. *Abrasive Blasting* Removal of contaminated debris surface layers using water and/or air pressure to propel a solid media (e g , steel shot, aluminum oxide grit, plastic beads)	*Glass, Metal Plastic, Rubber* Treatment to a clean debris surface [3] *Brick, Cloth, Concrete, Paper, Pavement Rock, Wood* Removal of at least 0 6 cm of the surface layer; treatment to a clean debris surface [3]	*All Debris* None
b *Scarification, Grinding, and Planing* Process utilizing striking piston heads saws, or rotating grinding wheels such that contaminated debris surface layers are removed.	Same as above	Same as above
c *Spalling* Drilling or chipping holes at appropriate locations and depth in the contaminated debris surface and applying a tool which exerts a force on the sides of those holes such that the surface layer is removed The surface layer removed remains hazardous debris subject to the debris treatment standards.	Same as above	Same as above
d *Vibratory Finishing.* Process utilizing scrubbing media, flushing fluid, and oscillating energy such that hazardous contaminants or contaminated debris surface layers are removed.[4]	Same as above	Same as above
e *High Pressure Steam and Water Sprays* Application of water or steam sprays of sufficient temperature, pressure, residence time, agitation, surfactants, and detergents to remove hazardous contaminants from debris surfaces or to remove contaminated debris surface layers.	Same as above	Same as above.
2 Chemical Extraction		
a. *Water Washing and Spraying* Application of water sprays or water baths of sufficient temperature, pressure, residence time, agitation, surfactants, acids, bases, and detergents to remove hazardous contaminants from debris surfaces and surface pores or to remove contaminated debris surface layers	*All Debris.* Treatment to a clean debris surface [3], *Brick, Cloth, Concrete, Paper, Pavement, Rock, Wood* Debris must be no more than 1.2 cm (½ inch) in one dimension (i e., thickness limit,[5] except that this thickness limit may be waived under an "Equivalent Technology" approval under § 268 42(b);[6] debris surfaces must be in contact with water solution for at least 15 minutes	*Brick, Cloth, Concrete Paper, Pavement, Rock Wood.* Contaminant must be soluble to at least 5% by weight in water solution or 5% by weight in emulsion; if debris is contaminated with a dioxin-listed waste,[6] an "Equivalent Technology" approval under § 268 42(b) must be obtained [6]
b. *Liquid Phase Solvent Extraction* Removal of hazardous contaminants from debris surfaces and surface pores by applying a nonaqueous liquid or liquid solution which causes the hazardous contaminants to enter the liquid phase and be flushed away from the debris along with the liquid or liquid solution while using appropriate agitation, temperature, and residence time [4]	Same as above	*Brick, Cloth, Concrete Paper, Pavement, Rock, Wood* Same as above, except that contaminant must be soluble to at least 5% by weight in the solvent

TABLE 1 —ALTERNATIVE TREATMENT STANDARDS FOR HAZARDOUS DEBRIS [1]—Continued

Technology description	Performance and/or design and operating standard	Contaminant restrictions [2]
c. *Vapor Phase Solvent Extraction* Application of an organic vapor using sufficient agitation, residence time, and temperature to cause hazardous contaminants on contaminated debris surfaces and surface pores to enter the vapor phase and be flushed away with the organic vapor [4]	Same as above, except that brick, cloth, concrete, paper, pavement, rock and wood surfaces must be in contact with the organic vapor for at least 60 minutes	Same as above
3 Thermal Extraction		
a. *High Temperature Metals Recovery* Application of sufficient heat, residence time, mixing, fluxing agents, and/or carbon in a smelting, melting, or refining furnace to separate metals from debris	For refining furnaces treated debris must be separated from treatment residuals using simple physical or mechanical means,[9] and, prior to further treatment, such residuals must meet the waste-specific treatment standards for organic compounds in the waste contaminating the debris	*Debris contaminated with a dioxin-listed waste*[6] Obtain an "Equivalent Technology" approval under § 268 42(b) [6]
b *Thermal Desorption* Heating in an enclosed chamber under either oxidizing or nonoxidizing atmospheres at sufficient temperature and residence time to vaporize hazardous contaminants from contaminated surfaces and surface pores and to remove the contaminants from the heating chamber in a gaseous exhaust gas [7]	*All Debris* Obtain an "Equivalent Technology" approval under § 268 42(b),[6] treated debris must be separated from treatment residuals using simple physical or mechanical means,[9] and, prior to further treatment, such residue must meet the waste-specific treatment standards for organic compounds in the waste contaminating the debris *Brick, Cloth, Concrete, Paper, Pavement, Rock, Wood* Debris must be no more than 10 cm (4 inches) in one dimension (i e , thickness limit),[5] except that this thickness limit may be waived under the "Equivalent Technology" approval	*All Debris* Metals other than mercury
B Destruction Technologies		
1 *Biological Destruction (Biodegradation)* Removal of hazardous contaminants from debris surfaces and surface pores in an aqueous solution and biodegration of organic or nonmetallic inorganic compounds (i e., inorganics that contain phosphorus, nitrogen, or sulfur) in units operated under either aerobic or anaerobic conditions	*All Debris* Obtain an "Equivalent Technology" approval under § 268 42(b),[6] treated debris must be separated from treatment residuals using simple physical or mechanical means,[9] and, prior to further treatment, such residue must meet the waste-specific treatment standards for organic compounds in the waste contaminating the debris. *Brick, Cloth, Concrete, Paper, Pavement, Rock, Wood* Debris must be no more than 1.2 cm (½ inch) in one dimension (i e., thickness limit),[5] except that this thickness limit may be waived under the "Equivalent Technology" approval	*All Debris* Metal contaminants.
2 Chemical Destruction		
a *Chemical Oxidation* Chemical or electolytic oxidation utilizing the following oxidation reagents (or waste reagents) or combination of reagents—(1) hypochlorite (e g , bleach), (2) chlorine; (3) chlorine dioxide, (4) ozone or UV (ultraviolet light) assisted ozone; (5) peroxides, (6) persulfates, (7) perchlorates, (8) permanganates; and/or (9) other oxidizing reagents of equivalent destruction efficiency [4] Chemical oxidation specifically includes what is referred to as alkaline chlorination	*All Debris* Obtain an "Equivalent Technology" approval under § 268 42(b),[6] treated debris must be separated from treatment residuals using simple physical or mechanical means,[9] and, prior to further treatment, such residue must meet the waste-specific treatment standards for organic compounds in the waste contaminating the debris. *Brick, Cloth, Concrete, Paper, Pavement Rock, Wood* Debris must be no more than 1.2 cm (½ inch) in one dimension (i e , thickness limit),[5] except that this thickness limit may be waived under the "Equivalent Technology" approval	*All Debris* Metal contaminants

TABLE 1.—ALTERNATIVE TREATMENT STANDARDS FOR HAZARDOUS DEBRIS [1]—Continued

Technology description	Performance and/or design and operating standard	Contaminant restrictions [2]
b *Chemical Reduction* Chemical reaction utilizing the following reducing reagents (or waste reagents) or combination of reagents. (1) sulfur dioxide; (2) sodium, potassium, or alkali salts of sulfites, bisulfites, and metabisulfites, and polyethylene glycols (e.g., NaPEG and KPEG), (3) sodium hydrosulfide, (4) ferrous salts; and/or (5) other reducing reagents of equivalent efficiency [4]	Same as above	Same as above
3 *Thermal Destruction* Treatment in an incinerator operating in accordance with Subpart O of Parts 264 or 265 of this chapter; a boiler or industrial furnace operating in accordance with Subpart H of Part 266 of this chapter, or other thermal treatment unit operated in accordance with Subpart X, Part 264 of this chapter, or Subpart P, Part 265 of this chapter, but excluding for purposes of these debris treatment standards Thermal Desorption units	Treated debris must be separated from treatment residuals using simple physical or mechanical means,[6] and, prior to further treatment, such residue must meet the waste-specific treatment standards for organic compounds in the waste contaminating the debris	*Brick, Concrete, Glass, Metal, Pavement, Rock, Metal·* Metals other than mercury, except that there are no metal restrictions for vitrification *Debris contaminated with a dioxin-listed waste* [8] Obtain an "Equivalent Technology" approval under §268.42(b),[9] except that this requirement does not apply to vitrification
C Immobilization Technologies		
1 *Macroencapsulation·* Application of surface coating materials such as polymeric organics (e.g., resins and plastics) or use of a jacket of inert inorganic materials to substantially reduce surface exposure to potential leaching media.	Encapsulating material must completely encapsulate debris and be resistant to degradation by the debris and its contaminants and materials into which it may come into contact after placement (leachate, other waste, microbes).	None
2. *Microencapsulation·* Stabilization of the debris with the following reagents (or waste reagents) such that the leachability of the hazardous contaminants is reduced: (1) Portland cement; or (2) lime/pozzolans (e.g., fly ash and cement kiln dust). Reagents (e.g., iron salts, silicates, and clays) may be added to enhance the set/cure time and/or compressive strength, or to reduce the leachability of the hazardous constituents.[8]	Leachability of the hazardous contaminants must be reduced.	None.
3. *Sealing.* Application of an appropriate material which adheres tightly to the debris surface to avoid exposure of the surface to potential leaching media. When necessary to effectively seal the surface, sealing entails pretreatment of the debris surface to remove foreign matter and to clean and roughen the surface. Sealing materials include epoxy, silicone, and urethane compounds, but paint may not be used as a sealant.	Sealing must avoid exposure of the debris surface to potential leaching media and sealant must be resistant to degradation by the debris and its contaminants and materials into which it may come into contact after placement (leachate, other waste, microbes).	None.

[1] Hazardous debris must be treated by either these standards or the waste-specific treatment standards for the waste contaminating the debris. The treatment standards must be met for each type of debris contained in a mixture of debris types, unless the debris is converted into treatment residue as a result of the treatment process. Debris treatment residuals are subject to the waste-specific treatment standards for the waste contaminating the debris.

[2] Contaminant restriction means that the technology is not BDAT for that contaminant. If debris containing a restricted contaminant is treated by the technology, the contaminant must be subsequently treated by a technology for which it is not restricted in order to be land disposed (and excluded from Subtitle C regulation).

[3] "Clean debris surface" means the surface, when viewed without magnification, shall be free of all visible contaminated soil and hazardous waste except that residual staining from soil and waste consisting of light shadows, slight streaks, or minor discolorations, and soil and waste in cracks, crevices, and pits may be present provided that such staining and waste and soil in cracks, crevices, and pits shall be limited to no more than 5% of each square inch of surface area.

[4] Acids, solvents, and chemical reagents may react with some debris and contaminants to form hazardous compounds. For example, acid washing of cyanide-contaminated debris could result in the formation of hydrogen cyanide. Some acids may also react violently with some debris and contaminants, depending on the concentration of the acid and the type of debris and contaminants. Debris treaters should refer to the safety precautions specified in Material Safety Data Sheets for various acids to avoid applying an incompatible acid to a particular debris/contaminant combination. For example, concentrated sulfuric acid may react violently with certain organic compounds, such as acrylonitrile.

[5] If reducing the particle size of debris to meet the treatment standards results in material that no longer meets the 60 mm minimum particle size limit for debris, such material is subject to the waste-specific treatment standards for the waste contaminating the material, unless the debris has been cleaned and separated from contaminated soil and waste prior to size reduction. At a minimum, simple physical or mechanical means must be used to provide such cleaning and separation of nondebris materials to ensure that the debris surface is free of caked soil, waste, or other nondebris material.

[6] Dioxin-listed wastes are EPA Hazardous Waste numbers FO20, FO21, FO22, FO23, FO26, and FO27.

[7] Thermal desorption is distinguished from Thermal Destruction in that the primary purpose of Thermal Desorption is to volatilize contaminants and to remove them from the treatment chamber for subsequent destruction or other treatment.

[8] The demonstration "Equivalent Technology" under §268.42(b) must document that the technology treats contaminants subject to treatment to a level equivalent to that required by the performance and design and operating standards for other technologies in this table such that residual levels of hazardous contaminants will not pose a hazard to human health and the environment absent management controls.

[9] Any soil, waste, and other nondebris material that remains on the debris surface (or remains mixed with the debris) after treatment is considered a treatment residual that must be separated from the debris using, at a minimum, simple physical or mechanical means. Examples of simple physical or mechanical means are vibratory or trommel screening or water washing. The debris surface need not be cleaned to a "clean debris surface" as defined in note 3 when separating treated debris from residue, rather, the surface must be free of caked soil, waste, or other nondebris material. Treatment residuals are subject to the waste-specific treatment standards for the waste contaminating the debris.

[57 FR 37277, Aug. 18, 1992]

§ 268.46 Alternative treatment standards based on HTMR.

Table 1 identifies alternative treatment standards for F006 and K062 nonwastewaters.

TABLE 1.—ALTERNATIVE TREATMENT STANDARDS

Waste code	See also	Regulated hazardous constituent	CAS No. for regulated hazardous constituent	Nonwastewaters concentration (mg/1) TCLP
F006	Table CCWE in 268.41 and Table CCW in 268.43	Antimony	7440-36-0	21
		Arsenic	7440-38-2	0.055
		Barium	7440-39-3	7.6
		Beryllium	7440-41-7	0.014
		Cadmium	7440-43-9	0.19
		Chromium (total)	7440-47-32	0.33
		Cyanide (mg/kg) (total)	57-12-5	1.8
		Lead	7439-92-1	0.37
		Mercury	7439-97-6	0.009
		Nickel	7440-02-0	5.0
		Selenium	7782-49-2	0.16
		Silver	7440-22-4	0.30
		Thallium		0.078
		Zinc	7440-66-6	5.3
K062	Table CCWE in 268.41 and Table CCW in 268.43	Antimony	7440-36-0	21
		Arsenic	7440-38-2	0.055
		Barium	7440-39-3	7.6
		Beryllium	7440-41-7	0.014
		Cadmium	7440-43-9	0.19
		Chromium (total)	7440-47-32	0.33
		Lead	7439-92-1	0.37
		Mercury	7439-97-6	0.009
		Nickel	7440-02-0	5.0
		Selenium	7782-49-2	0.16
		Silver	7440-22-4	0.30
		Thallium		0.078
		Zinc	7440-66-6	5.3

[57 FR 37280, Aug. 18, 1992]

Subpart E—Prohibitions on Storage

§ 268.50 Prohibitions on storage of restricted wastes.

(a) Except as provided in this section, the storage of hazardous wastes restricted from land disposal under subpart C of this part of RCRA section 3004 is prohibited, unless the following conditions are met:

(1) A generator stores such wastes in tanks, containers, or containment buildings on-site solely for the purpose of the accumulation of such quantities of hazardous waste as necessary to facilitate proper recovery, treatment, or disposal and the generator complies with the requirements in § 262.34 and parts 264 and 265 of this chapter.

(2) An owner/operator of a hazardous waste treatment, storage, or disposal facility stores such wastes in tanks, containers, or containment buildings solely for the purpose of the accumulation of such quantities of hazardous waste as necessary to facilitate proper recovery, treatment, or disposal and:

(i) Each container is clearly marked to identify its contents and the date each period of accumulation begins;

(ii) Each tank is clearly marked with a description of its contents, the quantity of each hazardous waste received, and the date each period of accumulation begins, or such information for each tank is recorded and maintained in the operating record at that facility. Regardless of whether the tank itself is marked, an owner/operator must comply with the operating record requirements specified in § 264.73 or § 265.73.

(3) A transporter stores manifested shipments of such wastes at a transfer facility for 10 days or less.

(b) An owner/operator of a treatment, storage or disposal facility may store such wastes for up to one year unless the Agency can demonstrate that such storage was not solely for the purpose of accumulation of such quantities of hazardous waste as are necessary to facilitate proper recovery, treatment, or disposal.

(c) A owner/operator of a treatment, storage or disposal facility may store such wastes beyond one year; however, the owner/operator bears the burden of proving that such storage was solely for the purpose of accumulation of such quantities of hazardous waste as are necessary to facilitate proper recovery, treatment, or disposal

(d) If a generator's waste is exempt from a prohibition on the type of land disposal utilized for the waste (for example, because of an approved case-by-case extension under § 268.5, an approved § 268.6 petition, or a national capacity variance under subpart C), the prohibition in paragraph (a) of this section does not apply during the period of such exemption.

(e) The prohibition in paragraph (a) of this section does not apply to hazardous wastes that meet the treatment standards specified under §§ 268.41, 268.42, and 268.43 or the treatment standards specified under the variance in § 268.44, or, where treatment standards have not been specified, is in compliance with the applicable prohibitions specified in § 268.32 or RCRA section 3004.

(f) Liquid hazardous wastes containing polychlorinated biphenyls (PCBs) at concentrations greater than or equal to 50 ppm must be stored at a facility that meets the requirements of 40 CFR 761.65(b) and must be removed from storage and treated or disposed as required by this part within one year of the date when such wastes are first placed into storage. The provisions of paragraph (c) of this section do not apply to such PCB wastes prohibited under § 268.32 of this part.

[51 FR 40642, Nov. 7, 1986; 52 FR 21017, June 4, 1987, as amended at 52 FR 25791, July 8, 1987; 54 FR 36972, Sept. 6, 1989; 57 FR 37281, Aug. 18, 1992]

APPENDIX I TO PART 268—TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)

NOTE. The TCLP is published in appendix II of part 261.

[55 FR 11876, Mar. 29, 1990]

APPENDIX II—TREATMENT STANDARDS (AS CONCENTRATIONS IN THE TREATMENT RESIDUAL EXTRACT)

NOTE The treatment standards for F001-F005 Spent Solvent Wastes appear in §§ 268.41, 268.42, 268.43

[57 FR 37281, Aug 18, 1992]

Appendix III

Appendix III
Associated Design and Larry Jackson's TCLP Bench Sheets and Calculations

Guidelines for the Conduct of the

Toxicity Characteristic Leaching Procedure

These guidelines have been prepared by Associated Design and Manufacturing Company for the informational use of environmental professionals engaged in the conduct of the Toxicity Characteristic Leaching Procedure (TCLP). They are intended to focus attention on important data collection activities associated with the TCLP. They are for guidance only and are not intended to replace sound professional judgment or regulatory requirements.

The guidelines are presented in the form of laboratory worksheets that can be used to document some of the most important points of the procedure. Each worksheet is supported by a discussion and recommendations of the data that should be recorded to document of the TCLP. The discussion is keyed to the worksheet for easy reference.

The paragraph references (¶ x.x.x.x) given in this document refer to the version of the TCLP which appeared in the July 29, 1990, Federal Register, p. 26986. These references have been updated to reflect the SW 846 Method 1311, July 1992 which has been corrected for typographical errors.

April 8, 1994
Revision: 3

TCLP Worksheet No. 1

Sample Description

Laboratory Sample No.					
Field Sample No.					
A. Sample Description					
Number of phases					
1. solid					
2. liquid					
a. lighter than water					
b. water					
c. heavier than water					
B. Percent Solid Phase					
1. weight of filter					
2. weight of subsample					
3. weight of filtrate					
4. weight percent solids (wet)[1]					
5. weight percent solids (dry)[2]					
6 volume of initial aqueous filtrate					
7. volume of initial organic filtrate					

1. The weight percent wet solids is given by the equation:

$$\frac{\textit{weight of subsample} - \textit{weight of filtrate}}{\textit{weight of subsample}} \times 100$$

2. The weight percent dry solids is given by the equation:

$$\frac{(\textit{weight of dry waste} + \textit{filter}) - \textit{weight of filter}}{\textit{weight of subsample}} \times 100$$

Discussion and Recommendations

TCLP Worksheet No. 1

Sample Description

This worksheet documents important information regarding the general description of the sample and the number of phases observed in the sample as received from the field. This information is used to determine the amount of leaching fluid used to leach solid materials and the weighting factors used when calculating final analyte concentrations from multi-phasic samples.

A. Sample Description

Number of phases -- The number of phases present in the sample determine how the TCLP is conducted. Solid materials having no visible liquid phase are extracted as received from the field and the analyte concentration found in the leachate is the reported value. Liquid materials having no measurable solids content (< 0.5 wt. % dry solids) are defined as the TCLP extract (¶ 2.1) and are filtered and analyzed directly.

Multi-phase samples must be separated (¶ 7.1.1.2) and each phase treated individually. Aqueous phases may be combined with the leachate from solid phase materials before analysis if the two aqueous materials are compatible (¶ 7.2.13.2). If the two aqueous materials are not compatible, than each liquid must be analyzed by the appropriate methods and the results combined numerically to determine the final reported value (¶7.2.14).

A.1. Solid -- record the visible presence of a solid material heavier than water. If the sample contains more than one solid phase (example, wood chips and sediment mixed with water) record the information in the laboratory notebook.

A.2. Liquid -- record the number of liquid phases observed in the sample according to their apparent density. It may be impossible to distinguish apparent density if only one liquid phase is observed and there is no indication on the accompanying chain-of-custody form (COC). If this is the case, record it as aqueous material and let the subsequent analytical record show if the liquid is organic after the container is opened at the appropriate time.

B. Percent of Solid / Liquid Phase(s) -- paragraphs 7.1.1 through 7.1.2.3 of the method describe the procedure to follow for the determination of the percent solids of the samples. It is also convenient to measure the percent of any non-miscible liquid phases at this point because the information is required in ¶ 7.2.14.

Laboratory subsampling of the material delivered to the laboratory must be thoroughly documented. The total contents of the sample container should be considered as "the sample" and care must be taken to ensure the representativeness of any subsample.

Heterogeneous and multi-phasic materials can be difficult to subsample properly and frequently require significant judgment on the part of the analyst.

Discussion – At this point , it is important to review the COC and confirm the number of containers of each sample provided to the laboratory and the types of analyses requested. If the analysis of volatile components is requested, the determination of percent solids in multi-phasic samples must be completed before proceeding to the leaching of the solid material in the zero headspace extractor (ZHE) to prevent overfilling the ZHE. It is best if a separate sample has been provided for this purpose (¶ 6.2). The laboratory should establish an SOP to address how to proceed if only one container is available.

It is common that when more than one container of multi-phasic materials is received from the field, each container will show different amounts of each phase. This provides a challenge to the laboratory which must report the data based on percent phase composition of the sample. A practical solution is to record the depth (measured from outside the container) of the layers in the each container after the contents have been allowed to settle and determine the combined volume of each phase in all the containers. Then measure the phase composition on a single container (after thorough mixing to obtain a representative subsample). Combine these two sets of values to determine the correct volume/mass adjustments on the TCLP results.

The laboratory should also establish an SOP on how to proceed when only a limited amount of sample is available and the analyses requested exceed the amount of sample provided.

B.1. Weight of filter -- This value must be measured before loading the filter into the filter holder because the mass of the filter is used in performing the calculation for percent dry solids.

B.2 Weight of sample aliquot -- a representative 100 gram sample (¶ 7.1.1.5) is withdrawn from the sample container for filtration. If liquid material is decanted from the sample before subsampling, its volume/weight must be recorded and factored into the calculations of percent solids.

Discussion -- Many multi-phasic samples are difficult to filter. This is especially true of oily wastes and sludges. The method directs that any material retained by the filter after following the instructions is defined as solid waste (¶ 7.1.18). Experience has shown that the reproducibility of the percent solids determination with these types of samples is highly variable. Subsequent steps in the extraction procedure (¶ 7.2.5 and 7.3.4.2) use the % solids value to estimate the mass of the original waste used to obtain an appropriate sized subsample of the solid for extraction.

The method directs that the material retained by the filter be dried at 100 ± 20 °C (¶ 7.1.2.2) to determine the percent dry solids. This may not be achievable for organic multi-phasic materials because of safety considerations and the fact that many organic liquids boil considerably higher than water and it may be impossible to achieve a constant weight for successive weighings (± 1%).

The laboratory should establish a standard operating procedure (SOP) addressing these types of samples. Basically, the laboratory has three choices of how to proceed. It may

- *attempt to dry all samples as directed by the method;*
- *dry samples containing only water as the liquid phase; and/or*
- *define the retained material as a dry solid for the purpose of further testing.*

This decision may have significant impact on the amount of material selected for leach testing and on the reported analyte values. The laboratory should consider discussing this issue with their clients and any regulatory groups to whom the data will be submitted.

B. 4 Weight percent solids(wet) equals:

$$\frac{\textit{weight of subsample} - \textit{weight of filtrate}}{\textit{weight of subsample}} \times 100$$

The procedure defines the material retained by the filter as the solid phase of the waste (¶ 7.1.1.8). This value is used to calculate the volume of the original multi-phasic material which must be filtered to yield the proper amount of solid waste for the extraction procedure.

B.5 Weight percent solids (dry) -- the total mass of the filtered solids and the filter are removed from the filtration apparatus and dried at 100 $\pm$ 20 °C until a constant weight is achieved (¶ 7.1.2.2). This value is used to calculate the dry solids content of the waste. *Use caution when drying samples that may contain flammable material.* It is important to factor in the tare weight of the filter for samples that have low solids values.

The weight percent solids (dry) is calculated by the equation:

$$\frac{(\textit{weight of dry waste} + \textit{filter}) - \textit{weight of filter}}{\textit{weight of subsample}} \times 100$$

If the weight percent dry solids is > 0.5%, the total waste is defined as a solid waste and steps must be taken to collect the appropriate weight of solid material for extraction (¶ 7.1.2.4).

B.6 Volume of initial aqueous filtrate -- this value is used in ¶ 7.2.14 and 7.3.14 in the final calculation of analyte concentration.

B.7 Volume of initial organic filtrate -- this value is used in ¶ 7.2.14 and 7.3.14 in the final calculation of analyte concentration.

TCLP Worksheet No. 2

Selection of Extraction Fluid

Laboratory Sample No.					
Field Sample No.					
C. ~~Extraction Fluid Determination~~ -- ~~does not apply to determination of volatile organic components~~					
1. particle size reduction? yes/no					
2. sample weight, ✓ if 5.0 ± 0.1 grams					
3. volume of water, ✓ if 96.5 ± 1.0 mL added					
4. initial pH (after 5 min. mixing time)					
5. if pH > 5.0, ✓ if 3.5 mL 1N HCl added					
6. ✓ if heated and held at 50 °C for ten minutes					
7. secondary pH (at room temp.)					
D. ~~Selection of Extraction Fluid~~					
1. ✓ if pH from C.4 or C.7 is < 5.0, use extraction fluid No. 1.					
2. ✓ if pH from C.7 is > 5.0, use extraction fluid No. 2					

Discussion and Recommendations

TCLP Worksheet No. 2

Selection of Extraction Fluid for Metals, Semi-volatile Organic Components, and Pesticides/Herbicides

This worksheet documents the important steps which should be followed to correctly determine the appropriate extraction fluid for leaching solid wastes for the analysis of metals, semi-volatile organic components, and pesticides/herbicides. This procedure does not apply to the determination of volatiles using the zero headspace extractor (ZHE).

Discussion -- the Environmental Protection Agency's "worst case" waste disposal model assumes mismanaged wastes will be co-disposed with municipal solid waste in a 5:95 ratio. These wastes will be exposed to leaching by the acidic fluids formed in municipal landfills. The EPA's model further assumes the acid/base characteristics of the waste will be dominated by the landfill fluids. The TCLP laboratory procedure directs that alkaline wastes be extracted with a stronger acidic leach fluid than acid or neutral wastes so that the alkaline nature of the waste will not control the leaching chemistry of the TCLP test. This is in keeping with the waste disposal model's assumption that the acid fluids in the landfill will dominate leaching chemistry over time.

The procedure described in ¶ 7.1.4 of the method addresses the determination of the appropriate extraction fluid. It is a short term test whose results can have a significant impact of the final analytical results if the wrong extraction fluid is selected. This is especially true for metals determinations because of their sensitivity to the pH of the leach medium. The following discussion examines each step of the procedure and points out some sensitive technical points and how they can affect the results.

¶ 7.1.4.1 Particle size of test material -- The requirement to use 1mm particle size material in the test recognizes the fact that in a short term reaction between a liquid and a solid, high surface area is the most important characteristic of the solid. The rate of the reaction is controlled by the rate of diffusion of the liquid into the pores of the solid so a high surface area is necessary if the results of a short term test are to be reliable. Therefore, failure to take a representative subsample of the solid material and perform the necessary particle size reduction can result in significant bias. This is especially true if the waste contains a wide range of particle sizes and only the fines are selected for testing.

¶ 7.1.4.3 Heating of the reaction mixture -- The method specifies that the waste/acid slurry is to be held at 50°C for ten (10) minutes. Care should be taken to heat the sample to 50 °C as rapidly as possible without overheating. When the sample has completed the ten minute period at temperature, it should be allowed to cool and the pH determined as soon as possible. The longer the reaction between the acid solution and the solid waste is allowed to continue, the more likely that a falsely high pH reading will result. This will result in improper selection of the more acidic extraction fluid. Failure to reach and hold the required temperature can result in an artificially low pH reading for the test solution, leading the incorrect selection of the less acidic extraction fluid.

C. Extraction Fluid Determination (¶ 7.1.4)

C.1. Indicate if particle size reduction is required for the sample.

Discussion -- the laboratory should consider establishing an SOP to address the particle size reduction requirements for the TCLP procedure. Most solid samples will not be received from the field with a particle size of 1mm as required for this step of the procedure (¶ 7.1.4.1). Many multi-phasic samples will not be amenable to size reduction because of the nature of the sample. Samples containing pebbles, rocks, or debris may be difficult to size reduce if the larger particles are hard. Proper subsampling of the waste may be difficult if the waste is heterogeneous.

C.2. Sample weight -- check the box if 5.0g of sample is used in the test. Record the actual weight if a different sized sample is used.

C.3. Volume of water -- the volume of water used in the test is dependant on the weight of sample being tested. If the sample weight (above) is 5g and 96.5 mL of water is added, check the box. If the weight is not 5g, record the volume of water added. (# of grams X 19.3mL).

C.4. Initial pH -- record the pH of the slurry after a five minute mixing period. Use narrow range pH indicator paper if organic material is observed floating on the top of the slurry to avoid damage to pH electrodes.

C.5. Procedure for alkaline wastes -- if the initial pH of the slurry is > 5.0, add 3.5 mL of 1N HCl to determine if the alkalinity of the waste is sufficient to require the use of the stronger acid extraction fluid.

C.6. Neutralization reaction conditions -- the slurry should be heated to 50 °C and held for ten minutes.The laboratory should consider validating their procedure to confirm these conditions are met. A bench procedure specifying the hot plate setting (or other source of heat), the time required to reach the desired temperature, the ten minute time at temperature, and the time required to return to room temperature should be established. This will assure the maximum degree of reproducibility in the determination of the alkaline potential of the wastes tested.

C.7. Secondary pH -- record the pH of the slurry after it has completed the cooling cycle.

D. Selection of Extraction Fluid

D.1. If either the initial pH or the secondary pH is < 5.0, select Extraction Fluid #1 as the leaching medium.

D.2. If the secondary pH is >5.0, select Extraction Fluid #2 as the leaching medium.

TCLP Worksheet No. 3

Determination of Extraction Fluid Volume for Metals, Semi-Volatile Organic Components and Pesticides/Herbicides

Laboratory Sample No.					
Field Sample No.					
E. Determination of Sample Size for Leach Testing -- the method requires a minimum 100 gram sample size for extraction (¶ 7.2.5).					
1. particle size reduction? yes/no					
2. amount of dry solids (100g min.)					
3. amount of multi-phasic sample[1]					
a. weight of material					
b. weight of filtrate					
c. weight of solid material					
F. Determination of Amount of Extraction Fluid -- the selection of the correct extraction fluid is found in Section D, Worksheet No. 2:					
1. for dry solids (20X sample wt.)					
2. for multi-phasic samples[2]					
G. Record of Extraction Test -- the extraction period is specified as 18 ± 2 hours.					
1. extraction start time					
2. extraction stop time					
3. filtration complete time					
4. pH of filtrate					
5. volume of filtrate					

1. The theoretical amount of multi-phasic waste necessary to yield a 100g sample is given by:

   $$\textit{Amount of multi-phasic material} = (10^4)/(\textit{wt. percent wet solids})$$

2. The amount of extraction fluid needed to extract the solid material from a filtered multi-phasic waste is given by:

   $$\textit{Amount of extraction fluid} = 20\ (\textit{weight of material filtered} - \textit{weight of filtrate})$$

Discussion and Recommendations

TCLP Worksheet No. 3

TCLP Extraction Procedure for Metals, Semi-volatile Organic Components, and Pesticides/Herbicides

This worksheet documents the performance of the TCLP extraction procedure for metals, semi-volatile organic compounds and pesticides/herbicides.

E. Determination of Sample Size for Leaching -- the specified size of sample for the leaching test is a minimum of 100g (¶ 7.2.5). The regulatory control limit for defining if the waste is hazardous is based on the levels of analytes reported in the leachate based on this size sample and a twenty to one (20:1) liquid to solid ratio. If the amount of waste subjected to extraction is not 100g, than the volume of extraction fluid must be adjusted to preserve the liquid to solid ratio.

E.2. Amount of dry solids -- record the weight of dry solids.

E.3. Amount of multi-phasic sample -- the amount of multi-phasic waste material necessary to produce a 100g sample after filtration can be estimated by the equation:

$$\textit{Amount of multi-phasic material} = (10^4)/(\textit{wt. percent wet solids})$$

F. Determination of the Amount of Leaching Fluid

F.1. Dry solids -- for dry solids containing no filtrable fluids, the calculation of the correct volume of leaching fluid is straightforward. The amount is equal to twenty (20) times the mass of solid being leached. Note that the method specifies a 20:1 ratio based on the weight of extraction fluid required (¶ 7.2.1.1). If the laboratory elects to use extraction fluid volume, rigorous adherence to the method requires a one time specific gravity correction to convert the required weight into the appropriate volume.

F.2. Multi-phasic samples -- the method says (¶ 7.2.11) the percent wet solids can be used to calculate the weight of extraction fluid used to extract the solid waste resulting from the filtration of a known weight of multi-phasic waste. The equation for this calculation is:

$$\textit{Amount of extraction fluid} = 0.2\ (\textit{percent wet solids})\ (\textit{weight of waste filtered})$$

This assumes there is no subsampling error between the original determination of the weight percent solid phase (wet) and the subsequent selection of a weight of the multi-phasic waste for filtration and extraction. This is frequently not so. The nature of many multi-phasic wastes and/or the necessity to use more than one sample container for the two determinations means that subsampling error can be

significant. This error can be eliminated if the actual weight of filtered solids is determined at the time the material is separated for extraction. The equation for this calculation is:

$$\textit{Amount of extraction fluid} = 20\ (\textit{weight of material filtered} - \textit{weight of filtrate})$$

The actual filtration procedure is detailed in ¶'s 7.2.2 though 7.2.8. Requirements for sample particle size reduction are given in ¶ 7.1.3 and 7.2.10. These should be followed as closely as the nature of the samples will allow and all departures from the instructions should be described in the laboratory notebook.

G. Record of the TCLP Extraction Test -- the period of the extraction test is given as 18 ± 2 hours (¶ 7.2.12). Extraction should be started so the resulting slurry can be filtered as soon as possible after the 18 hours has past. The filtration effectively stops the extraction process. If the extraction fluid is left in contact with the waste for longer than the specified period (overnight or over the weekend), the extraction process continues and may lead to elevated levels of contaminants.

G.1. Extraction start time -- record the time and date the extraction begins.

G.2. Extraction stop time -- record the time and date the extraction is completed.

G.3. Filtration completion time -- record the time and date the filtration is complete.

G.4. pH of filtrate -- while not required by the method, this is a good indicator of test performance when performing duplicate laboratory analysis or analyzing field replicates. It can be a reliable measure of sample heterogeneity.

G.5. Volume of filtrate -- record the total volume of filtrate collected from the sample. This value is required to make the appropriate volume corrections when reporting the results from multi-phasic wastes.

TCLP Worksheet No. 4

Zero Headspace Extraction (ZHE)

Laboratory Sample No.					
Field Sample No.					
H. Determination of Sample Size for Leach Testing – maximum 25 grams					
1. amount of dry solids					
2. amount of multi-phasic sample[1]					
I. Determination of Amount of Extraction Fluid No. 1					
1. for dry solids (20X sample wt.)					
2. for multi-phasic samples[2]					
a. weight of material					
b. weight of filtrate					
c. weight of solid material					
J. Record of ZHE Extraction Test – the extraction period is as 18 ± 2 hours (¶ 7.3.12.3).					
1. extraction start time					
2. starting pressure					
3. extraction stop time					
4. ✓ if positive pressure					
5. filtration completion time					
6. pH of filtrate					
7. volume of filtrate					

1. Determination of amount of multi-phasic sample for extraction:
   a. if weight percent dry solids is < 5% (from Worksheet No. 1, B. 5), the waste is filtered and the filtrate is defined as the TCLP leachate (¶ 7.3.4).
   b. if weight percent dry solids is > 5% (from Worksheet No. 1, B. 5), the amount of multi-phasic material which should be filtered to yield a 25 gram sample is given by:

$$\textit{Amount of multi-phasic material} = (2.5 \times 10^3)(\textit{wt. percent dry solids})$$

2. The amount of extraction fluid #1 needed to extract the solid material from the filtered multi-phasic waste (H.2) is given by:

$$\textit{Amount of extraction fluid} = 20\ (\textit{weight of material filtered} - \textit{weight of filtrate})$$

Discussion and Recommendations

TCLP Worksheet No. 4

Zero Headspace Extraction for Determination of Volatile Organic Compounds

This worksheet describes the important information regarding the conduct of the zero headspace extraction (ZHE) of solid waste materials for volatile organic compounds.

Samples containing < 5.0 % dry solids are NOT subjected to ZHE leaching procedure. They are filtered in the ZHE device and the resulting filtrate is defined as the TCLP leachate and analyzed directly (¶ 7.3.4).

H. Determination of Sample Size for Leach Testing -- the maximum sample size for this test is limited by the volume of the ZHE to approximately 25g (¶ 7.3).

H.1. Amount of dry solids -- record the weight of dry solids charged to the ZHE but do not exceed 25g.

H.2. Amount of multi-phasic sample -- the amount of multi-phasic waste material necessary to produce a 25g sample after filtration can be estimated by the equation:

$$\textit{Amount of multi-phasic material} = (2.5 \times 10^3)(\textit{wt. percent wet solids})$$

I. Determination of the Amount of Leaching Fluid #1

I.1. Dry solids -- for dry solids containing no filterable fluids, the calculation of the correct volume of leaching fluid is straightforward. The amount is equal to twenty (20) times the mass of solid being leached. Note that the method specifies a 20:1 ratio based on the weight of extraction fluid required (¶ 7.3.11). If the laboratory elects to use extraction fluid volume, rigorous adherence to the method requires a one time specific gravity correction to convert the required weight into the appropriate volume.

I.2. Multi-phasic samples -- the method indicates (¶ 7.3.11) that the percent wet solids can be used to calculate the weight of extraction fluid used to extract the solid waste resulting from the filtration of a known weight of multi-phasic waste. The equation for this calculation is:

$$\textit{Amount of extraction fluid} = \frac{20\ (\textit{percent wet solids})\ (\textit{weight of waste filtered})}{100}$$

This assumes there is no subsampling error between the original determination of the weight percent solid phase (wet) and the subsequent selection of a weight of the multi-phasic waste for filtration and extraction. This is frequently not the case. The nature of many multi-phasic wastes and/or the necessity to use more than one sample container for the two determinations means that subsampling error can be significant. This error can be eliminated if the actual weight of filtered solids is determined at the time the material is separated for extraction. The equation for this calculation is:

$$\textit{Amount of extraction fluid} = 20\ (\textit{weight of material filtered} - \textit{weight of filtrate})$$

The actual filtration procedure is detailed in ¶'s 7.3.7 though 7.3.9. Requirements for sample particle size reduction are given in ¶ 7.3.5 and 7.3.6. These should be followed as closely as the nature of the samples will allow and all departures from the instructions should be described in the laboratory notebook.

The addition of extraction fluid #1 to the ZHE is described in detail in ¶ 7.3.12.

J. Record of the ZHE Extraction Test -- the period of the extraction test is given as 18 ± 2 hours (¶ 7.3.12.3). Extraction should be started so the resulting slurry can be filtered as soon as possible after the 18 hours has past. The filtration effectively stops the extraction process. If the extraction fluid is left in contact with the waste for longer than the specified extraction period (overnight or over the weekend), the extraction process continues and may lead to elevated levels of contaminants.

J.1. Extraction start time -- record the time and date the extraction begins.

J.2. Starting pressure -- the method requires the ZHE be pressurized to approximately 10 psi at the beginning of the test.

J.3. Extraction stop time -- record the time and date the extraction is completed.

J.4. Positive final pressure – the method requires that the ZHE retain positive pressure at the conclusion of the extraction period or the test must be repeated (¶ 7.3.13). Loss of pressure is an indication the ZHE leaked during the test resulting in a loss of volatile components.

J.5. Filtration completion time – record the time and date the filtration is complete.

J.6. pH of filtrate – while not required by the method, this is a good indicator of test performance when performing duplicate laboratory analysis or analyzing field replicates. It can be a reliable measure of sample heterogeneity.

J.7. Volume of filtrate – record the total volume of filtrate collected from the sample. This value is required to make the appropriate volume corrections when reporting the results from multi-phasic wastes. The filtration of oily wastes may be especially difficult.

Appendix IV
USEPA Region II
Organic, Inorganic and TCLP Data Validation Methods

Evaluation of Metals Data for the Contract Laboratory Program (CLP)

based on

SOW. 3/90

(SOP Revision XI)

PREPARED BY: _____ DATE: 1-30-92
Hanif Sheikh, Quality Assurance Chemist
Toxic and Hazardous Waste Section

APPROVED BY: _____ DATE: 1-30-92
Kevin Kubik, Chief
Toxic and Hazardous Waste Section

APPROVED BY: _____ DATE: 1/31/92
Robert Runyon, Chief
Monitoring Management Branch

Title: Evaluation of Metals Data for the Contract Laboratory Program

Date: Sept. 1991
Number: HW-2
Revision: 11

1.0 **Scope**

1.1 This procedure is applicable to inorganic data obtained from contractor laboratories working for Hazardous Waste Site Contract Laboratory Program (CLP).

1.2 The data validation is based upon analytical and quality assurance requirements specified in Statement of Work (SOW) 3/90.

2.0 **Responsibilities** - Data reviewers will complete the following tasks as assigned by the Data Review Coordinator:

2.1. For a **total review**:

2.1.1 **Data Assessment** - **"Total Review-Inorganics" Checklist Appendix (A.1).**
The reviewer must answer every question on the checklist.

2.1.2 **Data Assessment** - **Data Assessment Narrative (Appendix A.2)**
The answer on the checklist must match the action in the narrative (appendix A.2) and on Form I's. Do not use pencil to write the narrative.

2.1.3 **Contract Non-Compliance** - **SMO Report (Appendix A.3)**
This report is to be completed only when a serious contract violation is encountered, or upon the request of the Data Review Manager or Deputy Project Officer (DPO). Forward 5 copies: one each for internal files, appropriate Regional DPO, Sample Management Office (SMO) and last two addresses of Mailing List for Data Reviewers (Appendix A.4). In other cases, all contract violations should be appended to end of Data Assessment Narrative (Sec. A.2.2).

2.1.4 **Data Summary Sheet** - **Summary of Inorganic Quality Control Data (Appendix A.5).**
Enter in ink on Data Summary Sheet required QC values from Forms I through IX. Circle all values that require data qualification "Action".

2.1.5 **CLP Data Assessment Summary Forms**

2.1.5.1 Appendix A.6
Fill in the total number of analytes analyzed by different analyses and the number of analytes rejected or flagged as estimated due to corresponding quality control criteria. Place an "X" in boxes where analyses were not performed, or criteria do not apply.

1.5.2 Appendix A.7
Data reviewer is also required to fill out Inorganic Regional Data Assessment

form (Appendix A.7) provided by EPA Headquarters. Codes listed on the form will be used to describe the Data Assessment Summary.

2.1.6 **Data Review Log:** It is recommended that each data reviewer should maintain a log of the reviews completed to include:

a. date of start of case review
b. date of completion of case review
c. site
d. case number
e. contract laboratory
f. number of samples
g. matrix
h. hours worked
i. reviewer's initials

2.1.7 **Telephone Record Log** - the data reviewer should enter the bare facts of inquiry, before initiating any phone conversation with CLP laboratory. After the case review has been completed, mail white copy of Telephone Record Log to the laboratory and pink copy to SMO. File yellow copy in the Telephone Record Log folder, and attach a xerox copy of the Telephone Record Log to the completed Data Assessment Narrative (Appendix A.2).

2.1.8 **Forwarded Paperwork**

2.1.8.1 Upon completion of review, the following are to be forwarded to the Regional Sample Control Center (RSCC) located in the Surveillance and Monitoring Branch:

a. data package
b. completed data assessment checklist (Appendix A.1,original)
c. SMO Contract Compliance Screening (CCS)
d. Data Summary Sheet (Appendix A.5) along with completed Data Assessment Narrative (Appendix A.2)
e. Record of Communication (copy)
f. CLP Reanalysis Request/Approval Record (original + 3 copies)
g. Appendix A.7 (original).

2.1.8.2 Forward 2 copies of completed Data Assessment Narrative (Appendix A.2) along with 2 copies of the Inorganic Data Assessment Form (Appendix A.7) and Telephone Record Log , if any,: one each for appropriate Regional TPO, and the other one to EPA EMSL office in Las Vegas. The addresses of TPOs and EPA office in Las Vegas are given in Appendix A-4.

2.1.9 **Filed Paperwork** - Upon completion of review, the following are to be filed within MMB files:
a. Two copies of completed Data Assessment Narrative (Appendix A.2) each carrying Appendix A.7.
b. Telephone Record Log (copy)
c. SMO Report (copy Appendix A-3)
d. CLP Reanalysis Request/Approval Record (copy)

3.0 **Data Completeness**
Each data package is checked by a Regional Sample Control Coordinator (RSSC) for completeness. A data package is assumed to be complete when all the deliverables required under the contract are present. If a data package is incomplete,the RSSC would call the laboratory for missing document(s). If the laboratory does not respond within a week, SMO and MMB coordinator of Region II will be notified.

4. **Rejection of Data** - All values determined to be unacceptable on the Inorganic Analysis Data Sheet (Form I) must be lined over with a red pencil. As soon as any review criteria causes data to be rejected, that data can be eliminated from any further review or consideration.

5.0 **Acceptance Criteria** - In order that reviews be consistent among reviewers, acceptance criteria as stated in Appendix A.1 (pages 4-25) should be used. Additional guidance can be found in the National Inorganic Functional Guidelines of October 1, 1989.

6.0 **SMO Contract Compliance Screening (CCS)** - This is intended to aid reviewer in locating any problems, both corrected and uncorrected. However, the validation should be carried out even if CCS is not present. Resubmittals received from laboratory in response to CCS must be used by the reviewer.

7.0 **Request for Reanalysis** - Data reviewers must note all items of contract non-compliance within Data Assessment Narrative.If holding times and sample storage times have not been exceeded, TPO may request reanalysis if items of non-compliance are critical to data assessment. Requests are to be made on "CLP Re-Analysis Request/Approval Record".

8.0 **Record of Communication** - Provided by the Regional Sample Control Center (RSCC) to indicate which data packages have been received and are ready to be reviewed.

9. **Rounding off numbers** - The data reviewer will follow the standard practice.

	YES	NO	N/A
A.1.1 **Contract Compliance Screening Report** (CCS) - Present?	[___]	___	___
ACTION: If no, contact RSCC.			
A.1.2 **Record of Communication (from RSCC)** - Present?	[___]	___	___
ACTION: If no, request from RSCC.			
A.1.3 **Trip Report** - Present and complete?	[___]	___	___
ACTION: If no, contact RSCC for trip report.			
A.1.4 **Sample Traffic Report** - Present?	[___]	___	___
Legible?	[___]	___	___
ACTION: If no, request from Regional Sample Control Center (RSCC).			
A.1.5 **Cover Page** - Present?	[___]	___	___
Is cover page properly filled in and signed by the lab manager or the manager's designee?	[___]	___	___
ACTION: If no, prepare Telephone Record Log, and contact laboratory.			
Do numbers of samples correspond to numbers on Record of Communication?	[___]	___	___
Do sample numbers on cover page agree with sample numbers on:			
(a) Traffic Report Sheet?	[___]	___	___
(b) Form I's?	[___]	___	___
ACTION: If no for any of the above, contact RSCC for clarification.			

A.1.6	**Form I to IX**	Yes	No	N/A
A.1.6.1	Are all the Form I through Form IX labeled with:			
	Laboratory name?	[___]	___	___
	Case/SAS number?	[___]	___	___
	EPA sample No.?	[___]	___	___
	SDG No.?	[___]	___	___
	Contract No.?	[___]	___	___
	Correct units?	[___]	___	___
	Matrix?	[___]	___	___

ACTION: If no for any of the above, note under Contract Problem/Non-Compliance section of the "Data Assessment Narrative".

A.1.6.2	Do any computation/transcription errors exceed 10% of reported values on Forms I-IX for:			
	(**NOTE:** Check all forms against raw data.)			
	(a) all analytes analyzed by ICP?	[___]	___	___
	(b) all analytes analyzed by GFAA?	[___]	___	___
	(c) all analytes analyzed by AA Flame?	[___]	___	___
	(d) Mercury?	[___]	___	___
	(e) Cyanide?	[___]	___	___

ACTION: If yes, prepare Telephone Log, contact laboratory for corrected data and correct errors with red pencil and initial.

Title: Evaluation of Metals Data for the Contract Laboratory Program
Appendix A.1: Data Assessment - Contract Compliance (Total Review)

Date: Sept. 1991
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A.1.7 **Raw Data**

A.1.7.1	Digestion Log* for flame AA/ICP (Form XIII) present?	[___]	___	___
	Digestion Log for furnace AA Form XIII present?	[___]	___	___
	Distillation Log for mercury Form XIII present?	[___]	___	___
	Distillation Log for cyanides Form XIII present?	[___]	___	___
	Are pH values (pH<2 for all metals, pH>12 for cyanide) present?	[___]	___	___
	*Weights, dilutions and volumes used to obtain values.			
	Percent solids calculation present for soils/sediments?	[___]	___	___
	Are preparation dates present on sample preparation logs/bench sheets?	[___]	___	___
A.1.7.2	Measurement read out record present? ICP	[___]	___	___
	Flame AA	[___]	___	___
	Furnace AA	[___]	___	___
	Mercury	[___]	___	___
	Cyanides	[___]	___	___
A.1.7.3	Are all raw data to support all sample analyses and QC operations present?	[___]	___	___
	Legible?	[___]	___	___
	Properly Labeled?	[___]	___	___

ACTION: If no for any of the above questions in sections A.1.7.1 through A.1.7.3, write Telephone Record Log and contact laboratory for resubmittals. data as estimated if pH

A.1.8 **Holding Times** - (aqueous and soil samples)

(Examine sample traffic reports and digestion/distillation logs.)

Mercury analysis (28 days). . . . . exceeded? ___ [___] ___

Cyanide distillation (14 days). . . . . exceeded? ___ [___] ___

Other Metals analysis (6 months). . . . exceeded? ___ [___] ___

NOTE: Prepare a list of all samples and analytes for which holding times have been exceeded. Specify the number of days from date of collection to the date of preparation (from raw data). Attach to checklist.

ACTION: If yes, reject (red-line) values less than Instrument Detection Limit (IDL) and flag as estimated (J) the values above IDL even though sample(s) was preserved properly.

A.1.8.2 Is pH of aqueous samples for:

Metals Analysis >2
Cyanides Analysis <12

Action: If yes, flag the associated metals and cyanides as estimated.

A.1.9 **Form I (Final Data)**

Are all Form I's present and complete? [___] ___ ___

ACTION: If no, prepare telephone record log and contact laboratory for submittal.

Are correct units (ug/l for waters and mg/kg for soils) indicated on Form I's? [___] ___ ___

Are soil sample results for each parameter corrected for percent solids? [___] ___ ___

Are EPA sample # s and corresponding laboratory sample ID # s the same as on the Cover Page, Form I's and in the raw data? [___] ___ ___

Are all "less than IDL" values properly coded with "U"? [___] ___ ___

Was a brief physical description of samples given on Form I's? [___] ___ ___

		YES	NO	N/A
	Were the correct concentration qualifiers used with final data?	[___]	___	___
	ACTION: If no for any of the above, prepare Telephone Record Log, and contact laboratory for corrected data.			
	Were any samples diluted beyond the requirements of contract?	___	[___]	___
	If yes, were dilutions noted on Form I's?	[___]	___	___
	ACTION: If no, note under Contract-Problem/Non-Compliance of the"Data Assessment Narrative".			
A.1.10	**Calibration**			
A.1.10.1	Is record of at least 2 point calibration present for ICP analysis?	[___]	___	___
	Is record of 5 point calibration present for Hg analysis?	[___]	___	___
	ACTION: If no for any of the above, write in the Contract Problem/Non-Compliance section of the "Data Assessment Narrative".			
A.1.10.2	Is record of 4 point calibration present for:			
	Flame AA?	[___]	___	___
	Furnace AA?	[___]	___	___
	Cyanides?	[___]	___	___

NOTE: 1. If less than 4 standards are measured in absorbance mode, then the remaining standards in concentration mode must be run immediately after calibration and be within ±10% of true value.
2. For all AA (except Hg) and Cyanide analyses, one calibration standard is at CRDL level. If not, write in the Contract-Problem/Non-Compliance section

of the "Data Assessment Narrative".

		YES	NO	N/A
	ACTION: Flag associated data as estimated if standards are not within ±10% of true values. Do not flag the data as estimated in linear range indicated by good recovery of standard(s).			
A.1.10.3	Is correlation coefficient* less than 0.995 for:			
	Mercury Analysis?	___	[___]	___
	Cyanide Analysis?	___	[___]	___
	Atomic Absorption Analysis?	___	[___]	___
	ACTION: If yes, flag the associated data as estimated.			
A.1.11	**Form II A (Initial and Continuing Calibration Verification)-**			
A.1.11.1	Present and complete for every metal and cyanide?	[___]	___	___
	Present and complete for AA and ICP when both are used for the same analyte?	[___]	___	___
	ACTION: If no for any of the above, prepare Telephone Record Log and contact laboratory.			
A.1.11.2	Circle on each Form II A all percent recoveries that are outside the contract windows. Are all calibration standards (initial and continuing) within control limits:			
	Metals- 90-110R%?	[___]	___	___
	Hg - 80-120R%?	[___]	___	___
	Cyanides- 85-115R%?	[___]	___	___

* The reviewer will calculate correlation coefficient.

	YES	NO	N/A
ACTION: Flag as estimated (J) all positive data (not flagged with a "U") analyzed between a calibration standard with %R between 75-89% (65-79% for Hg; 70-84% for CN) or 111-125% (121-135% for Hg; 116-130% for CN) recovery and nearest good calibration standard. Qualify results <IDL as estimated (UJ) if the ICV or CCV %R is 75-89% (CN, 70-84% ; HG, 65-79%). Reject (red-line) as unacceptable data if recovery of the ICV or CCV is outside the range 75-125% (CN, 70-130%; Hg, 65-135%). Qualify five samples on either side of verification standard out of control limits.			
Was continuing calibration performed every 10 samples or every 2 hours?	[___]	___	___
Was ICV for cyanides distilled?	[___]	___	___

ACTION: If no, for any of the above, write in the Contract-Problem/Non-Compliance section of the "Data Assessment Narrative".

A.1.12 **Form II B (CRDL Standards for AA and ICP)** -

A.1.12.1 Was a CRDL standard (CRA) analyzed after initial calibration for all AA metals (except Hg)?	[___]	___	___
Was a mid-range calib. verification standard distilled and analyzed for cyanide analysis?	[___]	___	___
Was a 2xCRDL (or 2xIDL when IDL>CRDL) analyzed (CRI) for each ICP run? (Note: CRI for AL,Ba,Ca,Fe,Mg,Na,or K is not required.)	[___]	___	___

ACTION: If no for any of the above, flag as estimated all data falling within the affected ranges. The affected ranges are:
AA Analysis - **True Value ± CRDL
ICP Analysis - **True Value ± 2CRDL
CN Analysis - **True Value ± 0.5 x True Value.

**True value of CRA, CRI or mid-range standard. Substitute IDL for CRDL when IDL > CRDL. Note the concentration of the missing mid-range standard from the calibration range.

		YES	NO	N/A
A.1.12.2	Was CRI analyzed after ICV/ICB and before the final CCV/CCB, and twice every eight hours of ICP run?	[___]	___	___
	ACTION: If no, write in Contract Problem/Non-Compliance Section of the "Data Assessment Narrative".			
A.1.12.3	Circle on each Form IIB all the percent recoveries that are outside the acceptance windows.			
	Are CRA and CRI standards within control limits:			
	Metals 80 - 120%R?	[___]	___	___
	Is mid-range standard within control limits:			
	Cyanide 80 - 120%R?	[___]	___	___
	ACTION: Flag as estimated all sample results within the affected ranges if the recovery of the standard is between 50-79%; flag only positive data if the recovery is between 121-150%; reject (red line) all data if the recovery is less than 50%; reject only positive data if the recovery is greater than 150%. Qualify 50% of the samples on either side of CRI standard outside the control limits.			
	Note: Flag or reject the final results only when sample raw data are within the affected ranges and the CRDL standards are outside the acceptance windows.			
A.1.13	**Form III (Initial and Continuing Calibration Blanks)**			
A.1.13.1	Present and complete?	[___]	___	___
	For both AA and ICP when both are used for the same analyte?	[___]	___	___
	Was an initial calibration blank analyzed?	[___]	___	___
	Was a continuing calibration blank analyzed after every 10 samples or every 2 hours (whichever is more frequent)?	[___]	___	___
	ACTION: If no, prepare Telephone Record Log, contact laboratory and write in the Contract-Problems/ Non-Compliance section of the "Data Assessment Narrative".			

		YES	NO	N/A
A.1.13.2	Circle on each Form III all calibration blank values that are above CRDL (or 2 x IDL when IDL > CRDL).			
	Are all calibration blanks (when IDL<CRDL) less than or equal to the Contract Required Detection Limits (CRDLs)?	[___]	___	___
	Are all calibration blanks less than two times Instrument Detection Limit (when IDL>CRDL)?	[___]	___	___
	ACTION: If no for any of the above, flag as estimated (J) positive sample results when raw sample value is less than or equal to calibration blank value analyzed between calibration blank with value over CRDL (or 2xIDL) and nearest good calibration blank. Flag five samples on either side of the calibration blank outside the control limits.			
.. .4	**FORM III (Preparation Blank) -** (**Note:** The preparation blank for mercury is the same as the calibration blank.)			
A.1.14.1	Was one prep. blank analyzed for: each 20 samples?	[___]	___	___
	each batch?	[___]	___	___
	each matrix type?	[___]	___	___
	both AA and ICP when both are used for the same analyte?	[___]	___	___
	ACTION: If no for any of the above, flag as estimated (J) all the associated positive data <10 x IDLs for which prep. blank was not analyzed. **NOTE:** If only one blank was analyzed for more than 20 samples, then first 20 samples analyzed do not have to be flagged as estimated (J).			
A.1.14.2	Is concentration of prep. blank value greater than the CRDL when IDL is less than or equal to CRDL?	___	[___]	___
	If yes, is the concentration of the sample with the least concentrated analyte less than 10 times the prep.blank?	___	[___]	___

		YES	NO	N/A
	ACTION: If yes, reject (red-line) all associated data greater than CRDL concentration but less than ten times the prep. blank value.			
A.1.14.3	Is concentration of prep. blank value (Form III) less than two times IDL, when IDL is greater than CRDL?	[___]	___	___
	ACTION: If no, reject (red-line) all positive sample results when sample raw data are less than 10 times the prep. blank value.			
A.1.14.4	Is concentration of prep. blank below the negative CRDL?	___	[___]	___
	ACTION: If yes, reject (red-line) all associated sample results less than 10xCRDL.			
A.1.15	**Form IV (ICP Interference Check Sample)**			
A.1.15.1	Present and complete?	[___]	___	___
	(**NOTE:** Not required for furnace AA, flame AA, mercury, cyanide and Ca, Mg, K and Na.)			
	Was ICS analyzed at beginning and end of run (or at least twice every 8 hours)?	[___]	___	___
	ACTION: If no, flag as estimated (J) all the samples for which AL, Ca, Fe, or Mg is higher than in ICS.			
A.1.15.2	Circle all values on each Form IV that are more than ± 20% of true or established mean value. Are all Interference Check Sample results inside the control limits (± 20%)?	[___]	___	___
	If no, is concentration of Al, Ca, Fe, or Mg lower than the respective concentration in ICS?	[___]	___	___
	ACTION: If no, flag as estimated (J) those positive results for which ICS recovery is between 121-150%; flag all sample results as estimated if ICS recovery falls within 50-79%; reject (red-line) those sample results for which ICS recovery is less than 50%; if ICS recovery is above 150%, reject positive results only (not flagged with a "U").			

Title: Evaluation of Metals Data for the Contract Laboratory Program
Appendix A.1: Data Assessment - Contract Compliance (Total Review)

Date: Sept. 1991
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		YES	NO	N/A
A.1.16	**Form V A (Spiked Sample Recovery - Pre-Digestion/Pre-Distillation)-** (**Note:** Not required for Ca, Mg, K, and Na (both matrices), Al, and Fe (soil only.)			
A.1.16.1	Present and complete for: each 20 samples?	[___]	___	___
	each matrix type?	[___]	___	___
	each conc. range (i.e. low, med., high)?	[___]	___	___
	For both AA and ICP when both are used for the same analyte?	[___]	___	___
	ACTION: If no for any of the above, flag as estimated (J) all the positive data less than four times the spiking levels specified in SOW for which spiked sample was not analyzed.			
	NOTE: If one spiked sample was analyzed for more than 20 samples, then first 20 samples analyzed do not have to be flagged as estimated (J).			
A.1.16.2	Was field blank used for spiked sample?	___	[___]	___
	ACTION: If yes, flag all positive data less than 4 x spike added as estimated (J) for which field blank was used as spiked sample.			
A.1.16.3	Circle on each Form VA all spike recoveries that are outside control limits (75% to 125%). Are all recoveries within control limits?	[___]	___	___
	If no, is sample concentration greater than or equal to four times spike concentration?	[___]	___	___
	ACTION: If yes, disregard spike recoveries for analytes whose concentrations are greater than or equal to four times spike added. If no, circle those analytes on Form V for which sample concentration is less than four times the spike concentration.			

	YES	NO	N/A
Are results outside the control limits (75-125%) flagged with "N" on Form I's and Form VA?	[___]	___	___

ACTION: If no, write in the Contract - Problem/Non - Compliance section of "Data Assessment Narrative".

A.1.16.4 **Aqueous**

Are any spike recoveries:			
(a) less than 30%?	___	[___]	___
(b) between 30-74%?	___	[___]	___
(c) between 126-150%?	___	[___]	___
(d) greater than 150%?	___	[___]	___

ACTION: If less than 30%, reject all associated aqueous data; if between 30-74%, flag all associated aqueous data as estimated (J); if between 126-150%, flag as estimated (J) all associated aqueous data not flagged with a "U"; if greater than 150%, reject (red-line) all associated aqueous data not flagged with a "U".

A.1.16.5 **Soil/Sediment**

Are any spike recoveries:			
(a) less than 10%?	___	[___]	___
(b) between 10-74%?	___	[___]	___
(c) between 126-200%?	___	[___]	___
(d) greater than 200%?	___	[___]	___

ACTION: If less than 10%, reject all associated data; if between 10-74%, flag all associated data as estimated; if between 126-200%, flag as estimated all associated data was not flagged with a "U"; if greater than 200%, reject all associated data not flagged with a "U".

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		YES	NO	N/A
A.1.17	**Form VI (Lab Duplicates)**			
A.1.17.1	Present and complete for: each 20 samples?	[___]	___	___
	each matrix type?	[___]	___	___
	each concentration range (i.e. low, med., high)?	[___]	___	___
	both AA and ICP when both are used for the same analyte?	[___]	___	___

ACTION: If no for any the above, flag as estimated (J) all the data ≥CRDL* for which duplicate sample was not analyzed.

Note: 1. If one duplicate sample was analyzed for more than 20 samples, then first 20 samples do not have to be flagged as estimated.
2. If percent solids for soil sample and its duplicate differ by more than 1%, prepare a Form VI for each duplicate pair, report concentrations in ug/L on wet weight basis and calculate RPD or Difference for each analyte.

A.1.17.2	Was field blank used for duplicate analysis?	___	[___]	___

ACTION: If yes, flag all data ≥CRDL* as estimated (J) for which field blank was used as duplicate.

A.1.17.3	Are all values within control limits (RPD 20% or difference ≤ ±CRDL)?	[___]	___	___
	If no, are all results outside the control limits flagged with an * on Form I's and VI?	[___]	___	___

ACTION: If no, write in the Contract - Problems/Non-Compliance section of "Data Assessment Narrative".

NOTE: 1. RPD is not calculable for an analyte of the sample - duplicate pair when both values are less than IDL.

Substitute IDL for CRDL when IDL > CRDL.

	YES	NO	N/A
2. If lab duplicate result is rejectable due to coefficient of correlation of MSA, analytical spike recovery, or duplicate injections criteria, do not apply precision criteria.			

A.1.17.4 **Aqueous**

Circle on each Form VI all values that are:

RPD > 50%, or

Difference > CRDL*

Is any RPD greater than 50% where sample and duplicate are both greater than or equal to 5 times *CRDL?	___	[___]	___
Is any difference** between sample and duplicate greater than *CRDL where sample and/or duplicate is less than 5 times *CRDL?	___	[___]	___

ACTION: If yes, flag the associated data as estimated.

A.1.17.5 **Soil/Sediment**

Circle on each Form VI all values that are:

RPD > 100%, or

Difference > 2 x CRDL*

Is any RPD (where sample and duplicate are both greater than or equal to 5 times *CRDL) : > 100%?	___	[___]	___
Is any **difference between sample and duplicate (where sample and/or duplicate is less than 5x*CRDL) : > 2x*CRDL?	___	[___]	___

* Substitute IDL for CRDL when IDL > CRDL.
** Use absolute values of sample and duplicate to calculate the difference.

		YES	NO	N/A
	ACTION: If yes, flag the associated data as estimated.			
A.1.18	**Field Duplicates**			
A.1.18.1	Were field duplicates analyzed?	[___]	___	___
	ACTION: If yes, prepare a Form VI for each aqueous field duplicate pair. Prepare a Form VI for each soil duplicate pair, if percent solids for sample and its duplicate differ by more than 1%; report concentrations of soils in ug/l on wet weight basis and calculate RPDs or Difference for each analyte.			
	NOTE: 1. Do not calculate RPD when both values are less than IDL. 2. Flag all associated data only for field duplicate pair.			
A.1.18.2	**Aqueous**			
	Circle all values on self prepared Form VI for field duplicates that are: RPD > 50%, or Difference > CRDL*			
	Is any RPD greater than 50% where sample and duplicate are both greater than or equal to 5 times *CRDL?	___	[___]	___
	Is any **difference between sample and duplicate greater than *CRDL where sample and/or duplicate is less than 5 times *CRDL?	___	[___]	___
	ACTION: If yes, flag the associated data as estimated.			

* Substitute IDL for CRDL when IDL > CRDL.
** Use absolute values of sample and duplicate to calculate the difference.

		YES	NO	N/A
A.1.18.3	**Soil/Sediment** Circle all values on self prepared Form VI for field duplicates that are: RPD >100%, or Difference > 2 x CRDL* Is any RPD (where sample and duplicate are both greater than 5 times *CRDL) : >100%?	___	[___]	___
	Is any **difference between sample and duplicate (where sample and/or duplicate is less than 5x *CRDL): >2x *CRDL?	___	[___]	___
	ACTION: If yes, flag the associated data as estimated.			
A.1.19	**Form VII (Laboratory Control Sample)** (Note: LCS - not required for aqueous Hg and cyanide analyses.)			
A.1.19.1	Was one LCS prepared and analyzed for: every 20 water samples?	[___]	___	___
	every 20 solid samples?	[___]	___	___
	both AA and ICP when both are used for the same analyte?	[___]	___	___
	ACTION: If no for any of the above, prepare Telephone Record Log and contact laboratory for submittal of results of LCS. Flag as estimated (J) all the data for which LCS was not analyzed.			
	NOTE: If only one LCS was analyzed for more than 20 samples, then first 20 samples close to LCS do not have to be flagged as estimated.			

* Substitute IDL for CRDL when IDL > CRDL.

** Use absolute values of sample and duplicate to calculate the difference.

		YES	NO	N/A
A.1.19.2	**Aqueous LCS**			
	Circle on each Form VII the LCS percent recoveries outside control limits (80 - 120%) except for aqueous Ag and Sb.			
	Is any LCS recovery: less than 50%?	___	[___]	___
	between 50% and 79%?	___	[___]	___
	between 121% and 150%?	___	[___]	___
	greater than 150%?	___	[___]	___
	ACTION: Less than 50%, reject (red-line) all data; between 50% and 79%, flag all associated data as estimated (J); between 121% and 150%, flag all positive (not flagged with a "U") results as estimated; greater than 150%, reject all positive results.			
A.1.19.3	**Solid LCS**			
	NOTE: 1. If "Found" value of LCS is rejectable due to duplicate injections or analytical spike recovery criteria, regardless of LCS recovery, flag the associated data as estimated (J).			
	2. If IDL of an analyte is equal to or greater than true value of LCS, disregard the "Action" below even though LCS is out of control limits.			
	Is LCS "Found" value higher than the control limits on Form VII?	___	[___]	___
	ACTION: If yes, qualify all associated positive data as estimated.			

		YES	NO	N/A
	Is LCS "Found" value lower than the Control limits on Form VII?	___	[___]	___
	ACTION: If yes, qualify all associated data as estimated.			
A.1.20	**Form IX (ICP Serial Dilution)** -			
	NOTE: Serial dilution analysis is required only for initial concentrations equal to or greater than 10 x IDL.			
A.1.20.1	Was Serial Dilution analysis performed for: each 20 samples?	[___]	___	___
	each matrix type?	[___]	___	___
	each concentration range (i.e. low, med.)?	[___]	___	___
	ACTION: If no for any of the above, flag as estimated all the positive data ≥ 10xIDLs or ≥ CRDL when 10xIDL ≤ CRDL for which Serial Dilution Analysis was not performed.			
A.1.20.2	Was field blank(s) used for Serial Dilution Analysis?	___	[___]	___
	ACTION: If yes, flag all associated data ≥ 10 x IDL as estimated (J). If 10xIDL ≤ CRDL, flag all data ≥ CRDL.			
A.1.20.3	Are results outside control limit flagged with an "E" on Form I's and Form IX when initial concentration on Form IX is equal to 50 times IDL or greater.	[___]	___	___

		YES	NO	N/A
	ACTION: If no, write in the Contract-Problem/Non-Compliance section of the "Data Assessment Narrative".			
A.1.20.4	Circle on each Form IX all percent difference that are outside control limits for initial concentrations equal to or greater than 10 x IDLs only. Are any % difference values:			
	> 10%?	___	[___]	___
	≥ 100%?	___	[___]	___
	ACTION: Flag as estimated (J) all the associated sample data ≥ 10xIDLs (or ≥ CRDL when 10xIDL ≥ CRDL) for which percent difference is greater than 10% but less than 100%. Reject (red-line) all the associated sample results equal to or greater than 10xIDLs (or ≥ CRDL when 10xIDL ≤ CRDL) for which PD is greater than or equal to 100%.			
	Note: Flag or reject on Form I's only the sample results whose associated raw data are ≥ 10xIDL (or ≥ CRDL when 10xIDL≤ CRDL)			
A.1.21	**Furnace Atomic Absorbtion (AA) QC Analysis**			
A.1.21.1	Are duplicate injections present in furnace raw data (except during full Method of Standard Addition) for each sample analyzed by GFAA?	[___]	___	___
	ACTION: If no, reject the data on Form I's for which duplicate injections were not performed.			
A.1.21.2	Do the duplicate injection readings agree within 20% Relative Standard Deviation (RSD) or Coefficient of Variation (CV) for concentration greater than CRDL?	[___]	___	___
	Was a dilution analyzed for sample with post digestion spike recovery less than 40%?	[___]	___	___
	ACTION: If no for any of the above, flag all the associated data as estimated (J).			

		YES	NO	N/A
A.1.21.3	Is *analytical spike recovery less than 10% or greater than 150% for any result?	___	[___]	___

ACTION: If yes, reject (red-line) the affected data if recovery is <10%; reject data not flagged with "U" if spike recovery is >150%.

NOTE: Reject or flag the data only when the affected sample(s) was not subsequently analyzed by Method of Standard Addition.

* Post digestion spike is not required on the pre-digestion spiked sample.

A.1.22 **Form VIII (Method of Standard Addition Results)**

A.1.22.1	Present?	[___]	___	___
	If no, is any Form I result coded with "S" or a "+"?	___	[___]	___

ACTION: If yes, write request on Telephone Record Log and contact laboratory for submittal of Form VIII.

A.1.22.2	Is coefficient of correlation for MSA less than 0.990 for any sample?	___	[___]	___

ACTION: If yes, reject (red-line) affected data.

A.1.22.3	Was *MSA required for any sample but not performed?	___	[___]	___
	Is coefficient of correlation for MSA less than 0.995?	___	[___]	___
	Are MSA calculations outside the linear range of the calibration curve generated at the beginning of the analytical run?	___	[___]	___

ACTION: If yes for any of the above, flag all the associated data as estimated (J).

A.1.22.4	Was proper quantitation procedure followed correctly as outlined in the SOW on page E-23?	[___]	___	___

ACTION: If no, note exception under Contract Problem/ Non-Compliance section of the "Data Assessment Narrative", and prepare a separate list.

		YES	NO	N/A
A.1.23	**Dissolved/Total or Inorganic/Total Analytes** -			
A.1.23.1	Were any analyses performed for dissolved as well as total analytes on the same sample(s).	___	[___]	___
	Were any analyses performed for inorganic as well as total (organic + inorganic) analytes on the same sample(s)?	___	[___]	___

* MSA is not required on LCS and prep. blank.

NOTE:
1. If yes, prepare a list comparing differences between all dissolved (or inorganic) and total analytes. Compute the differences as a percent of the total analyte only when dissolved concentration is greater than CRDL as well as total concentration.
2. Apply the following questions only if inorganic (or dissolved) results are (i) above CRDL, and (ii) greater than total constituents.
3. At least one preparation blank, ICS, and LCS should be analyzed in each analytical run.

A.1.23.2	Is the concentration of any dissolved (or inorganic) analyte greater than its total concentration by more than 10%?	___	[___]	___
A.1.23.3	Is the concentration of any dissolved (or inorganic) analyte greater than its total concentration by more than 50%?	___	[___]	___

ACTION: If more than 10%, flag both dissolved (or inorganic) and total values as estimated (J); if more than 50%, reject (red-line) the data for both values.

A.1.24	**Form I (Field Blank)** -			
A.1.24.1	Circle all field blank values on Data Summary Sheet that are greater than CRDL, (or 2 x IDL when IDL > CRDL).			
	Is field blank concentration less than CRDL (or 2 x IDL when IDL > CRDL) for all parameters of associated aqueous and soil samples?	[___]	___	___

Title: Evaluation of Metals Data for the Contract Laboratory Program
Appendix A.1: Data Assessment - Contract Compliance (Total Review)

Date: Sept. 1991
Number: HW-2
Revision: 11

	YES	NO	N/A
If no, was field blank value already rejected due to other QC criteria?	[___]	___	___

ACTION: If no, reject (except field blank results) all associated positive sample data less than or equal to five times the field blank value. Reject on Form I's the soil sample results that when converted to ug/L on wet basis are less than or equal to five times the field blank value.

A.1.25 **Form X, XI, XII (Verification of Instrumental Parameters).**

A 25.1 Is verification report present for:

Instrument Detection Limits (quarterly)?	[___]	___	___
ICP Interelement Correction Factors (annually)?	[___]	___	___
ICP Linear Ranges (quarterly)?	[___]	___	___

ACTION: If no, contact TPO of the lab.

A.1.25.2 **Form X (Instrument Detection Limits)** - (Note: IDL is not required for Cyanide.)

A.1.25.2.1 Are IDLs present for: all the analytes?	[___]	___	___
all the instruments used?	[___]	___	___
For both AA and ICP when both are used for the same analyte?	[___]	___	___

ACTION: If no for any of the above, prepare Telephone Record Log and contact laboratory.

		YES	NO	N/A
A.1.25.2.2	Is IDL greater than CRDL for any analyte?	___	[___]	___
	If yes, is the concentration on Form I of the sample analyzes on the instrument whose IDL exceeds CRDL, greater than 5 x CRDL.	[___]	___	___
	Action : If no, flag as estimated all values less than five times IDL of the instrument whose IDL exceeds CRDL.			
A.1.25.3	**Form XI (Linear Ranges)**			
A.1.25.3.1	Was any sample result higher than high linear range of ICP.	___	[___]	___
	Was any sample result higher than the highest calibration standard for non-ICP parameters?	___	[___]	___
	If yes for any of the above, was the sample diluted to obtain the result on Form I?	[___]	___	___
	ACTION: If no, flag the result reported on Form I as estimated(J).			
A.1.26	**Percent Solids of Sediments**			
A.1.26.1	Is soil content in sediment(s) less than 50%?	___	[___]	___
	ACTION: If yes, qualify as estimated all data not previously rejected or flagged due to other QC criteria.			

Title: Evaluation of Metals Data for the Contract Laboratory Program
Appendix A.2: Data Assessment Narrative

Date: Sept. 1991
Number: HW-2
Revision: 11

Case# _____ **Site** _____ **Matrix:** **Soil** _____

SDG# _____ **Lab** _____ **Water**_____

Contractor _____ **Reviewer** _____ **Other**_____

A.2.1 The case description and exceptions, if any, are noted below with reason(s) for rejection or qualification as estimated value(s) J.

_____

_____

_____

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_____

_____

Title: Evaluation of Metals Data for the Contract Laboratory Program
Appendix A.2: Data Assessment Narrative

Date: Sept. 1991
Number: HW-2
Revision: 11

A.2.1 (continuation)

A.2.1 (continuation)

A.2.2 Contract-Problem/Non-Compliance

MMB Reviewer: _____ Date: _____
Signature

Contractor Reviewer: _____ Date: _____
Signature

Verified by: _____ Date: _____

CONTRACT NON-COMPLIANCE
(SMO REPORT)

Regional Review of Uncontrolled Hazardous Waste
Site Contract Laboratory Data Package

CASE NO._____

The hardcopied (laboratory name)_____
Inorganic data package received at Region II has been reviewed and the quality assurance and performance data summarized. The data reviewed included:
SMO Sample No.:_____

Conc. & Matrix:_____

Contract No. WA87-K025,K026,K027(SOW787) requires that specific analytical work be done and that associated reports be provided by the contractor to the Regions, EMSL-LV, and SMO. The general criteria used to determine the performance were based on an examination of:

- Data Completeness
- Matrix Spike Results
- Calibration Standards Results
- Duplicate Analysis Results
- Blank Analysis Results
- MSA Results

[illegible] of non-compliance with the above contract are described below.

Comments:_____

_____

_____

_____

_____

_____

_____

_____

_____

_____ _____
Reviewer's Initial Date

Title: Evaluation of Metals Data for the Contract Laboratory Program
Appendix A.4: Mailing List for Data Reviewers

Date: Sept. 1991
Number: HW-2
Revision: 11

Title: Evaluation of Metals Data for the Contract Laboratory Program
Appendix A.5: Summary of Inorganics Quality Control Data

Date: Sept. 1991
Number: HW-2
Revision: 11

Title: Evaluation of Metals Data for the Contract Laboratory Program
Appendix A.6: CLP Data Assessment Summary Form (Inorganics)

Date: Sept. 1991
Number: HW-2
Revision: 11

Title: Evaluation of Metals Data for the Contract Laboratory Program
Appendix A.7: CLP Data Assessment Checklist
Inorganic Analysis

Date: Sept. 1991
Number: HW-2
Revision: 11

INORGANIC REGIONAL DATA ASSESSMENT Region_____

CASE NO._____ SITE_____

LABORATORY_____ NO. OF SAMPLES/ MATRIX_____

SDG#_____ REVIEWER (IF NOT ESD)_____

SOW#_____ REVIEWER'S NAME_____

DPO: ACTION_____ FYI_____ COMPLETION DATE_____

DATA ASSESSMENT SUMMARY

		ICP	AA	Hg	CYANIDE
1.	HOLDING TIMES				
2.	CALIBRATIONS				
3.	BLANKS				
4.	ICS				
5.	LCS				
6.	DUPLICATE ANALYSIS				
7.	MATRIX SPIKE				
8.	MSA				
9.	SERIAL DILUTION				
10.	SAMPLE VERIFICATION				
11.	OTHER QC				
12.	OVERALL ASSESSMENT				

O = Data has no problems/or qualified due to minor problems.
M = Data qualified due to major problems.
Z = Data unacceptable.
X = Problems, but do not affect data.

ACTION ITEMS:_____

AREAS OF CONCERN:_____

NOTABLE PERFORMANCE:_____

SOP NO. HW-6
Revision #8

CLP ORGANICS DATA REVIEW AND PRELIMINARY REVIEW

BY: _____ Date: January 2 1992
Leon Lazarus, Environmental Scientist
Toxic and Hazardous Waste Section

BY: _____ Date: January 3, 1992
George Karras, Chemist
Toxic and Hazardous Waste Section

BY: _____ Date: 1/3/1992
Stelios Gerazounis, Chemist
Toxic and Hazardous Waste Section

CONCURRED BY: _____ Date: 1/3/92
Kevin Kubik, Chief
Toxic and Hazardous Waste Section

APPROVED BY: _____ Date: 1/7/92
Robert Runyon, Chief
Monitoring Management Branch

YES NO N/A

PACKAGE COMPLETENESS AND DELIVERABLES
CASE NUMBER:_____ LAB:_____

SITE:_____

1.0 Data Completeness and Deliverables

1.1 Have any missing deliverables been received and added to the data package? [ ] ___ ___

ACTION: Call lab for explanation/resubmittal of any missing deliverables. If lab cannot provide them, note the effect on review of the package under the "Contract Problems/Non-Compliance" section of reviewer narrative.

1.2 Was SMO CCS checklist included with package? [ ] ___ ___

2.0 Cover Letter SDG Narrative

2.1 Is the Narrative or Cover Letter Present? [ ] ___ ___

2.2 Are Case Number and/or SAS number contained in the Narrative or Cover letter? [ ] ___ ___

3.0 Data Validation Checklist

The following checklist is divided into three parts. Part A is filled out if the data package contains any VOA analyses, Part B for any BNA analyses and Part C for Pesticide/PCBs.

Does this package contain:

VOA Data? ___ ___

BNA Data? ___ ___

Pesticide/PCB data? ___ ___

Action: Complete corresponding parts of checklist.

YES NO N/A

PART A: VOA ANALYSES

1.0 Traffic Reports and Laboratory Narrative

1.1 Are the Traffic Report Forms present for all samples? [ ] ___ ___

ACTION: If no, contact lab for replacement of missing or illegible copies.

1.2 Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data? ___ [ ] ___

ACTION: If any sample analyzed as a soil, other than TCLP, contains 50%-90% water, all data should be flagged as estimated (J). If a soil sample other than TCLP contains more than 90% water, all data should be qualified as unusable (R).

ACTION: If samples were not iced upon receipt at the laboratory, flag all positive results "J" and all Non-Detects "UJ".

ACTION: If both VOA vials for a sample have air bubbles or the VOA vial analyzed had air bubbles, flag all positive results "J" and all non-detects "R".

YES NO N/A

2.0 Holding Times

2.1 Have any VOA technical holding times, determined from date of collection to date of analysis, been exceeded? ___ [ ] ___

If unpreserved, aqueous samples maintained at 4°C which are to be analyzed for aromatic hydrocarbons must be analyzed within 7 days of collection. If preserved with HCl (pH<2) and stored at 4°C, then aqueous samples must be analyzed within 14 days of collection. If uncertain about preservation, contact sampler to determine whether or not samples were preserved.

The holding time for soils is 10 days.

Table of Holding Time Violations

			(See Traffic Report)		
Sample ID	Sample Matrix	Preserved?	Date Sampled	Date Lab Received	Date Analyzed

ACTION: If technical holding times are exceeded, flag all positive results as estimated ("J") and sample quantitation limits as estimated ("UJ"), and document in the narrative that holding times were exceeded. If analyses were done more than 14 days beyond holding time, either on the first analysis or upon re-analysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. At a minimum, all results must be qualified "J", but the reviewer may determine that non-detect data are unusable (R). If holding times are exceeded by more than 28 days, all non detect data are unusable (R).

		YES	NO	N/A
3.0	System Monitoring Compound (SMC) Recovery (Form II)			
3.1	Are the VOA SMC Recovery Summaries (Form II) present for each of the following matrices:			
	a. Low Water	[ ]	___	___
	b. Low Soil	[ ]	___	___
	c. Med Soil	[ ]	___	___
3.2	Are all the VOA samples listed on the appropriate System Monitoring Compound Recovery Summary for each of the following matrices:			
	a. Low Water	[ ]	___	___
	b. Low Soil	[ ]	___	___
	c. Med Soil	[ ]	___	___
	ACTION: Call lab for explanation/ resubmittals. If missing deliverables are unavailable, document effect in data assessments.			
3.3	Were outliers marked correctly with an asterisk?	[ ]	___	___
	ACTION: Circle all outliers in red.			
3.4	Was one or more VOA system monitoring compound recovery outside of contract specifications for any sample or method blank?	___	[ ]	___
	If yes, were samples re-analyzed?	[ ]	___	___
	Were method blanks re-analyzed?	[ ]	___	___

YES NO N/A

ACTION: If recoveries are > 10% but 1 or more compounds fail to meet SOW specifications:

1. All positive results are qualified as estimated (J).
2. Flag all non-detects as estimated detection limits ("UJ") where recovery is less than the lower acceptance limit.
3. If SMC recoveries are above allowable levels, do not qualify non-detects.

If any system monitoring compound recovery is <10% :

1. Flag all positive results as estimated ("J").
2. Flag all non-detects as unusable ("R").

Professional judgement should be used to qualify data that only have method blank SMC recoveries out of specification in both original and re-analyses. Check the internal standard areas.

3.5 Are there any transcription/calculation errors between raw data and Form II? ___ [ ] ___

ACTION: If large errors exist, call lab for explanation/resubmittal, make any necessary corrections and note errors in the data assessment.

4.0 Matrix Spikes (Form III)

4.1 Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III) present? [ ] ___ ___

YES NO N/A

4.2 Were matrix spikes analyzed at the required frequency for each of the following matrices:

a. Low Water [ ] ___ ___

b. Low Soil [ ] ___ ___

c. Med Soil [ ] ___ ___

ACTION: If any matrix spike data are missing, take the action specified in 3.2 above.

4.3 How many VOA spike recoveries are outside QC limits?

Water	Soils
_____ out of 10	_____ out of 10

4.4 How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?

Water	Soils
_____ out of 5	_____ out of 5

ACTION: No action is taken based on MS/MSD data alone. However, using informed professional judgement, the MS/MSD results may be used in conjunction with other QC criteria to determine the need for qualification of the data.

5.0 Blanks (Form IV)

5.1 Is the Method Blank Summary (Form IV) present? [ ] ___ ___

5.2 Frequency of Analysis: for the analysis of VOA TCL compounds, has a reagent/method blank been analyzed for each SDG or every 20 samples of similar matrix (low water, low soil, medium soil), whichever is more frequent? [ ] ___ ___

	YES	NO	N/A
5.3 Has a VOA method/instrument blank been analyzed at least once every twelve hours for each concentration level and GC/MS system used?	[ ]	___	___

ACTION: If any method blank data are missing, call lab for explanation/ resubmittal. If method blank data are not available, reject (R) all associated positive data. However, using professional judgement, the data reviewer may substitute field blank or trip blank data for missing method blank data.

5.4 Chromatography: review the blank raw data - chromatograms (RICs), quant reports or data system printouts and spectra. Is the chromatographic performance (baseline stability) for each instrument acceptable for VOAs?	[ ]	___	___

ACTION: Use professional judgement to determine the effect on the data.

6.0 Contamination

NOTE: "Water blanks", "drill blanks", and distilled water blanks" are validated like any other sample, and are not used to qualify data. Do not confuse them with the other QC blanks discussed below.

6.1 Do any method/instrument/reagent blanks have positive results (TCL and/or TIC) for VOAs? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample dilution factor and corrected for % moisture when necessary.	___	[ ]	___
6.2 Do any field/trip/rinse blanks have positive VOA results (TCL and/or TIC)?	___	[ ]	___

ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)

YES NO N/A

NOTE: All field blank results associated to a particular group of samples (may exceed one per case) must be used to qualify data. Trip blanks are used to qualify only those samples with which they were shipped and are not required for non-aqueous matrices. Blanks may not be qualified because of contamination in another blank. Field Blanks & Trip Blanks must be qualified for system monitoring compound, instrument performance criteria, spectral or calibration QC problems.

ACTION: Follow the directions in the table below to qualify TCL results due to contamination. Use the largest value from all the associated blanks. If any blanks are grossly contaminated, all associated data should be qualified as unusable (R).

	Sample conc > CRQL but < 10x blank value	Sample conc < CRQL & <10x blank value	Sample conc > CRQL & >10x blank value
Methylene Chloride Acetone Toluene 2-Butanone	Flag sample result with a "U;	Report CRQL & qualify "U"	No qualification is needed

	Sample conc > CRQL but < 5x blank	Sample conc < CRQL & is < 5x blank value	Sample conc > CRQL value & > 5x blank value
Other Contam-inants	Flag sample result with a "U"	Report CRQL & qualify "U"	No qualification is needed

NOTE: Analytes qualified "U" for blank contamination are still considered as "hits" when qualifying for calibration criteria.

YES NO N/A

ACTION: For TIC compounds, if the concentration in the sample is less than five times the concentration in the most contaminated associated blank, flag the sample data "R" (unusable).

6.3 Are there field/rinse/equipment blanks associated with every sample? [ ] ___ ___

ACTION: For low level samples, note in data assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

7.0 GC/MS Instrument Performance Check (Form V)

7.1 Are the GC/MS Instrument Performance Check Forms (Form V) present for Bromofluorobenzene (BFB)? [ ] ___ ___

7.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the BFB provided for each twelve hour shift? [ ] ___ ___

7.3 Has an instrument performance compound been analyzed for every twelve hours of sample analysis per instrument? [ ] ___ ___

YES NO N/A

ACTION: List date, time, instrument ID, and sample analysis for which no associated GC/MS tuning data are available.

DATE	TIME	INSTRUMENT	SAMPLE NUMBERS

ACTION: If lab cannot provide missing data, reject ("R") all data generated outside an acceptable twelve hour calibration interval.

7.4 Have the ion abundances been normalized to m/z 95? [ ] ___ ___

ACTION: If mass assignment is in error, qualify all associated data as unusable (R).

7.5 Have the ion abundance criteria been met for each instrument used? [ ] ___ ___

ACTION: List all data which do not meet ion abundance criteria (attach a separate sheet).

ACTION: If ion abundance criteria are not met, the Region II TPO must be notified.

7.6 Are there any transcription/calculation errors between mass lists and Form Vs? (Check at least two values but if errors are found, check more.) ___ [ ] ___

	YES	NO	N/A
7.7 Have the appropriate number of significant figures (two) been reported?	[ ]	___	___
ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and document effect in data assessments.			
7.8 Are the spectra of the mass calibration compound acceptable?	[ ]	___	___
ACTION: Use professional judgement to determine whether associated data should be accepted, qualified, or rejected.			

8.0 Target Compound List (TCL) Analytes

8.1 Are the Organic Analysis Data Sheets (Form I VOA) present with required header information on each page, for each of the following:			
a. Samples and/or fractions as appropriate	[ ]	___	___
b. Matrix spikes and matrix spike duplicates	[ ]	___	___
c. Blanks	[ ]	___	___
8.2 Are the VOA Reconstructed Ion Chromatograms, the mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?			
a. Samples and/or fractions as appropriate	[ ]	___	___
b. Matrix spikes and matrix spike duplicates (Mass spectra not required)	[ ]	___	___
c. Blanks	[ ]	___	___
ACTION: If any data are missing, take action specified in 3.2 above.			

		YES	NO	N/A
8.3	Are the response factors shown in the Quant Report?	[ ]	___	___
8.4	Is chromatographic performance acceptable with respect to:			
	Baseline stability?	[ ]	___	___
	Resolution?	[ ]	___	___
	Peak shape?	[ ]	___	___
	Full-scale graph (attenuation)?	[ ]	___	___
	Other: _____	[ ]	___	___

ACTION: Use professional judgement to determine the acceptability of the data.

8.5	Are the lab-generated standard mass spectra of the identified VOA compounds present for each sample?	[ ]	___	___

ACTION: If any mass spectra are missing, take action specified in 3.2 above. If lab does not generate their own standard spectra, make note in "Contract Problems/Non-compliance".

8.6	Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration?	[ ]	___	___
8.7	Are all ions present in the standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum?	[ ]	___	___

	YES	NO	N/A
8.8 Do sample and standard relative ion intensities agree within 20%?	[ ]	___	___

ACTION: Use professional judgement to determine acceptability of data. If it is determined that incorrect identifications were made, all such data should be rejected (R), flagged "N" (presumptive evidence of the presence of the compound) or changed to not detected (U) at the calculated detection limit. In order to be positively identified, the data must comply with the criteria listed in 8.6, 8.7, and 8.8.

ACTION: When sample carry-over is a possibility, professional judgement should be used to determine if instrument cross-contamination has affected any positive compound identification.

9.0 Tentatively Identified Compounds (TIC)

9.1 Are all Tentatively Identified Compound Forms (Form I Part B) present; and do listed TICs include scan number or retention time, estimated concentration and "JN" qualifier?	[ ]	___	___
9.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:			
a. Samples and/or fractions as appropriate	[ ]	___	___
b. Blanks	[ ]	___	___

ACTION: If any TIC data are missing, take action specified in 3.2 above.

ACTION: Add "JN" qualifier if missing.

	YES	NO	N/A
9.3 Are any TCL compounds (from any fraction) listed as TIC compounds (example: 1,2-dimethylbenzene is xylene- a VOA TCL analyte - and should not be reported as a TIC)?	___	[ ]	___
ACTION: Flag with "R" any TCL compound listed as a TIC.			
9.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% also present in the sample mass spectrum?	[ ]	___	___
9.5 Do TIC and "best match" standard relative ion intensities agree within 20%?	[ ]	___	___

ACTION: Use professional judgement to determine acceptability of TIC identifications. If it is determined that an incorrect identification was made, change identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate.

Also, when a compound is not found in any blank, but is detected in a sample and is a suspected artifact of a common laboratory contaminant, the result should be qualified as unusable (R). (i.e. Common Lab Contaminants: CO_2 (M/E 44), Siloxanes (M/E 73) Hexane, Aldol Condensation Products, Solvent Preservatives, and related by products - see Functional Guidelines for more guidance).

	YES	NO	N/A

10.0 Compound Quantitation and Reported Detection Limits

10.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Verify that the correct internal standard, quantitation ion, and RRF were used to calculate Form I result. Were any errors found? ___ [ ] ___

10.2 Are the CRQLs adjusted to reflect sample dilutions and, for soils, sample moisture? [ ] ___ ___

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and note errors under "Conclusions".

ACTION: When a sample is analyzed at more than one dilution, the lowest CRQLs are used (unless a QC exceedance dictates the use of the higher CRQL data from the diluted sample analysis). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and its associated value on the original Form I and substituting the data from the analysis of the diluted sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form I's that should not be used, including any in the summary package.

11.0 Standards Data (GC/MS)

11.1 Are the Reconstructed Ion Chromatograms, and data system printouts (Quant. Reports) present for initial and continuing calibration? [ ] ___ ___

ACTION: If any calibration standard data are missing, take action specified in 3.2 above.

	YES	NO	N/A

12.0 GC/MS Initial Calibration (Form VI)

12.1 Are the Initial Calibration Forms (Form VI) present and complete for the volatile fraction at concentrations of 10, 20, 50, 100, 200 ug/l? Are there separate calibrations for low water/med soils and low soil samples? [ ] ___ ___

ACTION: If any calibration standard forms are missing, take action specified in 3.2 above.

12.2 Were all low level soil standards, blanks and samples analyzed by heated purge? [ ] ___ ___

ACTION: If low level soil samples were not heated during purge, qualify positive hits "J" and non-detects "R".

12.3 Are response factors stable for VOA's over the concentration range of the calibration (%Relative Standard Deviation (%RSD) <30.0%)? [ ] ___ ___

ACTION: Circle all outliers in red.

NOTE: Although 11 VOA compounds have a minimum RRF and no maximum %RSD, the technical criteria are the same for all analytes.

ACTION: If %RSD > 30.0%, qualify associated positive results for that analyte "J" and non-detects using professional judgement. When RSD > 90%, flag all non-detects for that analyte R (unusable).

NOTE: Analytes previously qualified "U" for blank contamination are still considered as "hits" when qualifying for initial calibration criteria.

12.4 Are the RRFs above 0.05? [ ] ___ ___

Action: Circle all outliers in red.
Action: If any RRF are < 0.05, qualify associated non-detects (R) and flag associated positive data as estimated (J).

YES NO N/A

12.5 Are there any transcription/calculation errors in the reporting of average response factors (RRF) or %RSD? (Check at least 2 values, but if errors are found, check more.) ___ [ ] ___

13.0 GC/MS Continuing Calibration (Form VII)

13.1 Are the Continuing Calibration Forms (Form VII) present and complete for the volatile fraction? [ ] ___ ___

13.2 Has a continuing calibration standard been analyzed for every twelve hours of sample analysis per instrument? [ ] ___ ___

ACTION: List below all sample analyses that were not within twelve hours of the previous continuing calibration analysis.

_____

_____

_____

ACTION: If any forms are missing or no continuing calibration standard has been analyzed within twelve hours of every sample analysis, call lab for explanation/resubmittal. If continuing calibration data are not available, flag all associated sample data as unusable ("R").

13.3 Do any volatile compounds have a % Difference (% D) between the initial and continuing RRF which exceeds the ± 25% criteria? ___ [ ] ___

ACTION: Circle all outliers in red.

ACTION: Qualify both positive results and non-detects for the outlier compound(s) as estimated. When % D is above 90%, reject all non-detects for that analyte (R) unusable.

	YES	NO	N/A
13.4 Do any volatile compounds have a RRF <0.05?	[ ]	___	___

ACTION: Circle all outliers in red.

ACTION: If the RRF <0.05, qualify associated non-detects as unusable (R) and "J" associated positive values.

13.5 Are there any transcription/calculation errors in the reporting of average response factors (RRF) or %difference (%D) between initial and continuing RRFs? (Check at least two values but if errors are found, check more.)	___	[ ]	___

ACTION: Circle errors in red.

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and note errors under "Conclusions".

14.0 Internal Standard (Form VIII)

14.1 Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits (-50% to + 100%) for each continuing calibration?	[ ]	___	___

ACTION: List all the outliers below.

Sample #	Internal Std	Area	Lower Limit	Upper Limit
_____	_____	____	_____	_____
_____	_____	____	_____	_____
_____	_____	____	_____	_____
_____	_____	____	_____	_____
_____	_____	____	_____	_____

(Attach additional sheets if necessary.)

YES NO N/A

ACTION: 1. If the internal standard area count is outside the upper or lower limit, flag with "J" all positive results quantitated with this internal standard.

2. Non-detects associated with IS area counts > 100% should not be qualified.

3. If IS area is below the lower limit (< 50%), qualify all associated non-detects (U values) "J". If extremely low area counts are reported, (< 25%) or if performance exhibits a major abrupt drop off, flag all associated non-detects as unusable ("R").

14.2 Are the retention times of the internal standards within 30 seconds of the associated calibration standard? [ ] ___ ___

ACTION: Professional judgement should be used to qualify data if the retention times differ by more than 30 seconds.

15.0 Field Duplicates

15.1 Were any field duplicates submitted for VOA analysis? [ ] ___ ___

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between duplicate results must be addressed in the reviewer narrative. However, if large differences exist, identification of field duplicates should be confirmed by contacting the sampler.

YES NO N/A

PART B: BNA ANALYSES

1.0 Traffic Reports and Laboratory Narrative

1.1 Are the Traffic Report Forms present for all samples? [ ] ___ ___

ACTION: If no, contact lab for replacement of missing or illegible copies.

1.2 Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data? ___ [ ] ___

ACTION: If any sample analyzed as a soil, other than TCLP, contains 50%-90% water, all data should be flagged as estimated ("J"). If a soil sample, other than TCLP, contains more than 90% water, all data should be qualified as unusable (R).

ACTION: If samples were not iced upon receipt at the laboratory, flag all positive results "J" and all non-detects "UJ".

2.0 Holding Times

2.1 Have any BNA technical holding times, determined from date of collection to date of extraction, been exceeded? ___ [ ] ___

Continuous extraction of water samples for BNA analysis must be started within seven days of the date of collection. Soil/ sediment samples must be extracted within 7 days of collection. Extracts must be analyzed within 40 days of the date of extraction.

YES NO N/A

Table of Holding Time Violations

Sample	Sample Matrix	Date Sampled	(See Traffic Report) Date Lab Received	Date Extracted	Date Analyzed

ACTION: If technical holding times are exceeded, flag all positive results as estimated ("J") and sample quantitation limits as estimated ("UJ"), and document in the narrative that holding times were exceeded.

If analyses were done more than 14 days beyond holding time, either on the first analysis or upon reanalysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. At a minimum, all results should be qualified "J", but the reviewer may determine that non-detect data are unusable ("R"). If holding times are exceeded by more than 28 days, all non detect data are unusable (R).

3.0 Surrogate Recovery (Form II)

3.1 Are the BNA Surrogate Recovery Summaries (Form II) present for each of the following matrices:

a. Low Water [ ] ___ ___

b. Low Soil [ ] ___ ___

c. Med Soil [ ] ___ ___

	YES	NO	N/A
3.2 Are all the BNA samples listed on the appropriate Surrogate Recovery Summaries for each of the following matrices:			
a. Low Water	[ ]	___	___
b. Low Soil	[ ]	___	___
c. Low Soil	[ ]	___	___

ACTION: Call lab for explanation/resubmittals. If missing deliverables are unavailable, document effect in data assessments.

3.3 Were outliers marked correctly with an asterisk?	[ ]	___	___

ACTION: Circle all outliers in red.

3.4 Were two or more base-neutral <u>OR</u> acid surrogate recoveries out of specification for any sample or method blank?	[ ]	___	___
If yes, were samples reanalyzed?	[ ]	___	___
Were method blanks reanalyzed?	[ ]	___	___

ACTION: If all BNA surrogate recoveries are > 10% but two within the base-neutral or acid fraction do not meet SOW specifications, <u>for the affected fraction only (i.e. base-neutral or acid compounds)</u>:

1. Flag all positive results as estimated ("J").
2. Flag all non-detects as estimated detection limits ("UJ") when recoveries are less than the lower acceptance limit.
3. If recoveries are greater than the upper acceptance limit, do not qualify non-detects.

	YES	NO	N/A

If any base-neutral or acid surrogate has a recovery of <10%:

1. Positive results for the fraction with <10% surrogate recovery are qualified with "J".

2. Non-detects for that fraction should be qualified as unusable (R) .

Professional judgement should be used to qualify data that have method blank surrogate recoveries out of specification in both original and reanalyses. Check the internal standard areas.

3.5 Are there any transcription/calculation errors between raw data and Form II? ___ [ ] ___

ACTION: If large errors exist, call lab for explanation/resubmittal, make any necessary corrections and document effect in data assessments.

4.0 Matrix Spikes (Form III)

4.1 Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III) present? [ ] ___ ___

4.2 Were matrix spikes analyzed at the required frequency for each of the following matrices:

a. Low Water [ ] ___ ___

b. Low Soil [ ] ___ ___

c. Med Soil [ ] ___ ___

ACTION: If any matrix spike data are missing, take the action specified in 3.2 above.

YES NO N/A

4.3 How many BNA spike recoveries are outside QC limits?

Water	Soils
____ out of 22	____ out of 22

4.4 How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?

Water	Soils
____ out of 11	____ out of 11

ACTION: No action is taken on MS/MSD data alone. However, using informed professional judgement, the data reviewer may use the matrix spike and matrix spike duplicate results in conjunction with other QC criteria and determine the need for some qualification of the data.

5.0 Blanks (Form IV)

5.1 Is the Method Blank Summary (Form IV) present? [ ] ___ ___

5.2 Frequency of Analysis:

Has a reagent/method blank analysis been reported per 20 samples of similar matrix, or concentration level, and for each extraction batch? [ ] ___ ___

5.3 Has a BNA method blank been analyzed for each GC/MS system used? [ ] ___ ___
(See SOW p. D - 59/SV, Section 8.7)

ACTION: If any method blank data are missing, call lab for explanation/resubmittal. If not available, use professional judgement to determine if the associated sample data should be qualified.

YES NO N/A

5.4 Chromatography: review the blank raw data - chromatograms (RICs), quant reports or data system printouts and spectra.

Is the chromatographic performance (baseline stability) for each instrument acceptable for BNAs? [ ] ___ ___

ACTION: Use professional judgement to determine the effect on the data.

6.0 Contamination

Note: "Water blanks", "drill blanks" and "distilled water blanks" are validated like any other sample and are not used to qualify the data. Do not confuse them with the other QC blanks discussed below.

6.1 Do any method/instrument/reagent blanks have positive results (TCL and/or TIC) for BNAs? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample dilution factor and corrected for % moisture where necessary. ___ [ ] ___

6.2 Do any field/rinse/ blanks have positive BNA results (TCL and/or TIC)? ___ [ ] ___

ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)

Note: All field blank results associated to a particular group of samples (may exceed one per case) must be used to qualify data. Blanks may not be qualified because of contamination in another blank . Field Blanks must be qualified for surrogate, spectral, instrument performance or calibration QC problems.

YES NO N/A

ACTION: Follow the directions in the table below to qualify TCL results due to contamination. Use the largest value from all the associated blanks. If gross contamination exists, all data in the associated samples should be qualified as unusable (R).

Sample conc > CRQL but < 10x blank	Sample conc <CRQL & is< 10x blank value	Sample conc > CRQL value & >10x blank
Common Phthalate Esters		
Flag sample result with a "U";	Report CRQL & qualify "U"	No qualification is needed

Sample conc > CRQL but < 5x blank	Sample conc < CRQL & is < 5x blank value	Sample conc > CRQL value & >5 blank value
Other Contaminants		
Flag sample result with a "U";	Report CRQL & qualify "U"	No qualification is needed

NOTE: Analytes qualified "U" for blank contamination are still considered as "hits" when qualifying for calibration criteria.

ACTION: For TIC compounds, if the concentration in the sample is less than five times the concentration in the most contaminated associated blank, flag the sample data "R" (unusable).

6.3 Are there field/rinse/equipment blanks associated with every sample? [ ] ___ ___

ACTION: For low level samples, note in data assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

YES NO N/A

7.0 GC/MS Instrument Performance Check

7.1 Are the GC/MS Instrument Performance Check Forms (Form V) present for Decafluorotriphenylphosphine (DFTPP)? [ ] ___ ___

7.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the DFTPP provided for each twelve hour shift? [ ] ___ ___

7.3 Has an instrument performance check solution been analyzed for every twelve hours of sample analysis per instrument? [ ] ___ ___

ACTION: List date, time, instrument ID, and sample analyses for which no associated GC/MS tuning data are available.

DATE	TIME	INSTRUMENT	SAMPLE NUMBERS
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

ACTION: If lab cannot provide missing data, reject ("R") all data generated outside an acceptable twelve hour calibration interval.

ACTION: If mass assignment is in error, flag all associated sample data as unusable (R).

7.4 Have the ion abundances been normalized to m/z 198? [ ] ___ ___

	YES	NO	N/A
7.5 Have the ion abundance criteria been met for each instrument used?	[ ]	___	___
ACTION: List all data which do not meet ion abundance criteria (attach a separate sheet).			
ACTION: If ion abundance criteria are not met, the Region II TPO must be notified.			
7.6 Are there any transcription/calculation errors between mass lists and Form Vs? (Check at least two values but if errors are found, check more.)	___	[ ]	___
7.7 Have the appropriate number of significant figures (two) been reported?	[ ]	___	___
ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and document effect in data assessments.			
7.8 Are the spectra of the mass calibration compound acceptable?	[ ]	___	___
ACTION: Use professional judgement to determine whether associated data should be accepted, qualified, or rejected.			
8.0 Target Compound List (TCL) Analytes			
8.1 Are the Organic Analysis Data Sheets (Form I BNA) present with required header information on each page, for each of the following:			
a. Samples and/or fractions as appropriate	[ ]	___	___
b. Matrix spikes and matrix spike duplicates	[ ]	___	___
c. Blanks	[ ]	___	___

	YES	NO	N/A
8.2 Has GPC cleanup been performed on all soil/ sediment sample extracts?	[ ]	___	___
ACTION: If data suggests that GPC was not performed, use professional judgement. Make note in "Contract Problems/Non-Compliance".			
8.3 Are the BNA Reconstructed Ion Chromatograms, the mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?			
a. Samples and/or fractions as appropriate	[ ]	___	___
b. Matrix spikes and matrix spike duplicates (Mass spectra not required)	[ ]	___	___
c. Blanks	[ ]	___	___
ACTION: If any data are missing, take action specified in 3.2 above.			
8.4 Are the response factors shown in the Quant Report?	[ ]	___	___
8.5 Is chromatographic performance acceptable with respect to:			
Baseline stability?	[ ]	___	___
Resolution?	[ ]	___	___
Peak shape?	[ ]	___	___
Full-scale graph (attenuation)?	[ ]	___	___
Other:_____	[ ]	___	___
ACTION: Use professional judgement to determine the acceptability of the data.			

YES NO N/A

8.6 Are the lab-generated standard mass spectra of identified BNA compounds present for each sample? [ ] ___ ___

ACTION: If any mass spectra are missing, take action specified in 3.2 above. If lab does not generate their own standard spectra, make note in "Contract Problems/ Non-compliance". If spectra are missing, reject all positive data.

8.7 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration? [ ] ___ ___

8.8 Are all ions present in the standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum? [ ] ___ ___

8.9 Do sample and standard relative ion intensities agree within 20%? [ ] ___ ___

ACTION: Use professional judgement to determine acceptability of data. If it is determined that incorrect identifications were made, all such data should be rejected (R), flagged "N" (Presumptive evidence of the presence of the compound) or changed to not detected (U) at the calculated detection limit. In order to be positively identified, the data must comply with the criteria listed in 8.7, 8.8, and 8.9.

ACTION: When sample carry-over is a possibility, professional judgement should be used to determine if instrument cross-contamination has affected any positive compound identification.

9.0 Tentatively Identified Compounds (TIC)

9.1 Are all Tentatively Identified Compound Forms (Form I, Part B) present; and do listed TICs include scan number or retention time, estimated concentration and "JN" qualifier? [ ] ___ ___

	YES	NO	N/A
9.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:			
a. Samples and/or fractions as appropriate	[ ]	___	___
b. Blanks	[ ]	___	___

ACTION: If any TIC data are missing, take action specified in 3.2 above.

ACTION: Add "JN" qualifier if missing.

9.3 Are any TCL compounds (from any fraction) listed as TIC compounds (example: 1,2-dimethylbenzene is xylene a VOA TCL - and should not be reported as a TIC)?	___	[ ]	___

ACTION: Flag with "R" any TCL compound listed as a TIC.

9.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% also present in the sample mass spectrum?	[ ]	___	___
9.5 Do TIC and "best match" standard relative ion intensities agree within 20%?	[ ]	___	___

ACTION: Use professional judgement to determine acceptability of TIC identifications. If it is determined that an incorrect identification was made, change identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate. Also, when a compound is not found in any blank, but is a suspected artifact of a common laboratory contaminant, the result should be qualified as unusable (R).

YES NO N/A

10.0 Compound Quantitation and Reported Detection Limits

10.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Verify that the correct internal standard, quantitation ion, and RRF were used to calculate Form I result. Were any errors found? ___ [ ] ___

10.2 Are the CRQLs adjusted to reflect sample dilutions and, for soils, sample moisture? [ ] ___ ___

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and document effect in data assessments.

ACTION: When a sample is analyzed at more than one dilution, the lowest CRQLs are used (unless a QC exceedance dictates the use of the higher CRQL data from the diluted sample analysis). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and it's associated value on the original Form I and substituting the data from the analysis of the diluted sample. Specify which Form I is to be used, then draw a red " X" across the entire page of all Form I's that should not be used, including any in the summary package.

11.0 Standards Data (GC/MS)

11.1 Are the Reconstructed Ion Chromatograms, and data system printouts (Quant, Reports) present for initial and continuing calibration? [ ] ___ ___

ACTION: If any calibration standard data are missing, take action specified in 3.2 above.

YES NO N/A

12.0 GC/MS Initial Calibration (Form VI)

12.1 Are the Initial Calibration Forms (Form VI) present and complete for the BNA fraction? [ ] ___ ___

ACTION: If any calibration standard forms are missing, take action specified in 3.2 above.

12.2 Are response factors stable for BNAs over the concentration range of the calibration? (% Relative standard deviation (%RSD) < 30.0%) [ ] ___ ___

ACTION: Circle all outliers in red.

NOTE: Although 20 BNA compounds have a minimum RRF and no maximum %RSD, the technical criteria are the same for all analytes.

ACTION: If the % RSD is > 30.0%, qualify positive results for that analyte "J" and non-detects using professional judgement. When RSD > 90%, flag all non-detect results for that analyte R (unusable).

NOTE: Analytes previously qualified "U" due to blank contamination are still considered as "hits" when qualifying for calibration criteria.

12.3 Are all BNA compound RRFs > 0.05? [ ] ___ ___

ACTION: Circle all outliers in red.

ACTION: If any RRF < 0.05
1. "R" all non-detects.
2. "J" all positive results.

12.4 Are there any transcription/calculation errors in the reporting of average response factors (RRF) or % RSD? (Check at least two values but if errors are found, check more.) ___ [ ] ___

ACTION: Circle Errors in red.

YES NO N/A

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and note errors in data assessments.

13.0 GC/MS Continuing Calibration (Form VII)

13.1 Are the Continuing Calibration Forms (Form VII) present and complete for the BNA fraction? [ ] ___ ___

13.2 Has a continuing calibration standard been analyzed for every twelve hours of sample analysis per instrument? [ ] ___ ___

ACTION: List below all sample analyses that were not within twelve hours of a continuing calibration analysis for each instrument used.

_____

_____

_____

ACTION: If any forms are missing or no continuing calibration standard has been analyzed within twelve hours of every sample analysis, call lab for explanation/ resubmittal. If continuing calibration data are not available, flag all associated sample data as unusable ("R").

13.3 Do any semivolatile compounds have a % Difference (% D) between the initial and continuing RRF which exceeds the + 25.0% criteria? ___ [ ] ___

ACTION: Circle all outliers in red.

ACTION: Qualify both positive results and non-detects for the outlier compound(s) as estimated (J). When %D is above 90%, reject all non-detects for that analyte (R) unusable.

YES NO N/A

13.4 Do any semivolatile compounds have a RRF <0.05? ___ [ ] ___

ACTION: Circle all outliers in red.

ACTION: If RRF <0.05, qualify as unusable (R) associated non-detects and "J" associated positive values.

13.5 Are there any transcription/calculation errors in the reporting of average response factors (RRF) or % difference (%D) between initial and continuing RRFs? (Check at least two values but if errors are found, check more). ___ [ ] ___

ACTION: Circle errors in red.

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and document effect in data assessments.

14.0 Internal Standards (Form VIII)

14.1 Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits (-50% to + 100%) for each continuing calibration? [ ] ___ ___

ACTION: List all the outliers below.

Sample #	Internal Std	Area	Lower Limit	Upper Limit

(Attach additional sheets if necessary.)

ACTION: 1. If the internal standard area count is outside the upper or lower limit, flag with "J" all positive results and non-detects (U values) quantitated with this internal standard.

YES NO N/A

2. Non-detects associated with IS areas > 100% should not be qualified.

3. If the IS area is below the lower limit (<50%), qualify all associated non-detects (U-values) "J". If extremely low area counts are reported (<25%) or if performance exhibits a major abrupt drop off, flag all associated non-detects as unusable (R).

14.2 Are the retention times of the internal standards within 30 seconds of the associated calibration standard? [ ] ___ ___

ACTION: Professional judgement should be used to qualify data if the retention times differ by more than 30 seconds.

15.0 Field Duplicates

15.1 Were any field duplicates submitted for BNA analysis? [ ] ___ ___

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. However, if large differences exist, identification of field duplicates should be confirmed by contacting the sampler.

YES NO N/A

PART C: PESTICIDE/PCB ANALYSIS

1.0 Traffic Reports and Laboratory Narrative

1.1 Are Traffic Report Forms present for all samples? [ ] ___ ___

ACTION: If no, contact lab for replacement of missing or illegible copies.

1.2 Do the Traffic Reports or SDG Narrative indicate any problems with sample receipt, condition of the samples, analytical problems or special circumstances affecting the quality of the data? ___ [ ] ___

ACTION: If any sample analyzed as a soil, other than TCLP, contains 50%-90% water, all data should be qualified as estimated (J). If a soil sample, other than TCLP, contains more than 90% water, all data should be qualified as unusable (R).

ACTION: If samples were not iced upon receipt at the laboratory, flag all positive results "J" and all non-detects "UJ".

2.0 Holding Times

2.1 Have any PEST/PCB technical holding times, determined from date of collection to date of extraction, been exceeded? ___ [ ] ___

Water and soil samples for PEST/PCB analysis must be extracted within 7 days of the date of collection. Extracts must be analyzed within 40 days of the date extraction.

	YES	NO	N/A

ACTION: If technical holding times are exceeded, flag all positive results as estimated (J) and sample quantitation limits (UJ) and document in the narrative that holding times were exceeded. If analyses were done more than 14 days beyond holding time, either on the first analysis or upon re-analysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. At a minimum, all the data should at least be qualified "J", but the reviewer may determine that non-detects are unusable (R).

3.0 Surrogate Recovery (Form II)

3.1 Are the PEST/PCB Surrogate Recovery Summaries (Form II) present for each of the following matrices?

a. Low Water	[ ]	___	___
b. Soil	[ ]	___	___

3.2 Are all the PEST/PCB samples listed on the appropriate Surrogate Recovery Summary for each of the following matrices?

a. Low Water	[ ]	___	___
b. Soil	[ ]	___	___

ACTION: Call lab for explanation/resubmittals. If missing deliverables are unavailable, document effect in data assessments.

3.3 Were outliers marked correctly with an asterisk? [ ] ___ ___

ACTION: Circle all outliers in red.

3.4 Were surrogate recoveries of TCX or DCB outside of the contract specification for any sample or blank? (60-150%) ___ [ ] ___

YES NO N/A

ACTION: No qualification is done if surrogates are diluted out. If recovery for <u>both</u> surrogates is below the contract limit, but above 10%, flag all results for that sample 'J". If recovery is < 10% for either surrogate, qualify positive results 'J" and flag non-detects "R". If recovery is above the contract advisory limits for <u>both</u> surrogates qualify positive values "J".

3.5 Were surrogate retention times (RT) within the windows established during the initial 3-point analysis of Individual Standard Mixture A? [ ] ___ ___

ACTION: If the RT limits are not met, the analysis may be qualified unusable (R) for that sample on the basis of professional judgement.

3.6 Are there any transcription/calculation errors between raw data and Form II? ___ [ ] ___

ACTION: If large errors exist, call lab for explanation/resubmittal. Make any necessary corrections and document effect in data assessments.

4.0 <u>Matrix Spikes (Form III)</u>

4.1 Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III) present? [ ] ___ ___

4.2 Were matrix spikes analyzed at the required frequency for each of the following matrices? (1 MS/MSD must be performed for every 20 samples of similar matrix or concentration level)

a. Low Water [ ] ___ ___

b. Soil [ ] ___ ___

ACTION: If any matrix spike data are missing, take the action specified in 3.2 above.

YES NO N/A

4.3 How many PEST/PCB spike recoveries are outside QC limits?

Water	Soil
_____ out of 12	_____ out of 12

4.4 How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?

Water	Soil
_____ out of 6	_____ out of 6

ACTION: No action is taken on MS/MSD data alone. However, using informed professional judgement, the data reviewer may use the matrix spike and matrix spike duplicate results in conjunction with other QC criteria and determine the need for some qualification of the data.

5.0 Blanks (Form IV)

5.1 Is the Method Blank Summary (Form IV) present? [ ] ___ ___

5.2 Frequency of Analysis: For the analysis of Pesticide/PCB TCL compounds, has a reagent/ method blank been analyzed for each SDG or every 20 samples of similar matrix or concentration or each extraction batch, whichever is more frequent? [ ] ___ ___

ACTION: If any blank data are missing, take the action specified above in 3.2. If blank data is not available, reject (R) all associated positive data. However, using professional judgement, the data reviewer may substitute field blank data for missing method blank data.

5.3 Has a PEST/PCB instrument blank been analyzed at the beginning of every 12 hr. period following the initial calibration sequence? (minimum contract requirement)

YES NO N/A

ACTION: If any blank data are missing, call lab for explanation/resubmittals. If missing deliverables are unavailable, document the effect in data assessments.

5.4 Chromatography: review the blank raw data - chromatograms, quant reports or data system printouts.

Is the chromatographic performance (baseline stability) for each instrument acceptable for PEST/PCBs? [ ] ___ ___

ACTION: Use professional judgement to determine the effect on the data.

6.0 Contamination

NOTE: "Water blanks", "distilled water blanks" and "drilling water blanks" are validated like any other sample and are not used to qualify the data. Do not confuse them with the other QC blanks discussed below.

6.1 Do any method/instrument/reagent/cleanup blanks have positive results for PEST/PCBs? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample Dilution Factor and corrected for % moisture when necessary. ___ [ ] ___

6.2 Do any field/rinse blanks have positive PEST/PCB results? ___ [ ] ___

ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet)

NOTE: All field blank results associated to a particular group of samples (may exceed one per case or one per day) may be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field blanks must be qualified for surrogate, or calibration QC problems.

YES NO N/A

ACTION: Follow the directions in the table below to qualify TCL results due to contamination. Use the largest value from all the associated blanks.

Sample conc > CRQL but < 5x blank	Sample conc < CRQL & is < 5x blank value	Sample conc > CRQL & > 5x blank value
Flag sample result with a "U";	Report CRQL & qualify "U"	No qualification is needed

NOTE: If gross blank contamination exists, all data in the associated samples should be qualified as unusable (R).

6.3 Are there field/rinse/equipment blanks associated with every sample? [ ] ___ ___

ACTION: For low level samples, note in data assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

7.0 Calibration and GC Performance

7.1 Are the following Gas Chromatograms and Data Systems Printouts for both columns present for all samples, blanks, MS/MSD?

a. peak resolution check [ ] ___ ___

b. performance evaluation mixtures [ ] ___ ___

c. aroclor 1016/1260 [ ] ___ ___

d. aroclors 1221, 1232, 1242, 1248, 1254 [ ] ___ ___

e. toxaphene [ ] ___ ___

f. low points individual mixtures A & B [ ] ___ ___

g. med points individual mixtures A & B [ ] ___ ___

h. high points individual mixtures A & B [ ] ___ ___

	YES	NO	N/A
i. instrument blanks	[ ]	___	___

ACTION: If no, take action specified in 3.2 above.

7.2 Are Forms VI - PEST 1-4 present and complete for each column and each analytical sequence? [ ] ___ ___

ACTION: If no, take action specified in 3.2 above.

7.3 Are there any transcription/calculation errors between raw data and Forms VI? ___ [ ] ___

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and document effect in data assessments.

7.4 Do all standard retention times, including each pesticide in each level of Individual Mixtures A & B, fall within the windows established during the initial calibration analytical sequence? (For Initial Calibration Standards, Form VI - PEST - 1). [ ] ___ ___

ACTION: If no, all samples in the entire analytical sequence are potentially affected. Check to see if the chromatograms contain peaks within an expanded window surrounding the expected retention times. If no peaks are found and the surrogates are visible, non-detects are valid. If peaks are present and cannot be identified through pattern recognition or using a revised RT window, qualify all positive results and non-detects as unusable (R).
For aroclors, RT may be outside the RT window, but the aroclor may still be identified from the individual pattern.

7.5 Are the linearity criteria for the initial analyses of Individual Standards A & B within limits for both columns? (% RSD must be < 20.0% for all analytes except for the 2 surrogates, which must not exceed 30.0 % RSD). See Form VI PEST - 2. [ ] ___ ___

	YES	NO	N/A

ACTION: If no, qualify all associated positive results generated during the entire analytical sequence "J" and all non-detects "UJ". When RSD >90%, flag all non-detect results for that analyte R (unusable).

7.6 Is the resolution between any two adjacent peaks in the Resolution Check Mixture > 60.0% for both columns? (Form VI-PEST - 4) [ ] ___ ___

ACTION: If no, positive results for compounds that were not adequately resolved should be qualified "J". Use professional judgement to determine if non-detects which elute in areas affected by co-eluting peaks should be qualified "N" as presumptive evidence of presence or unusable (R).

7.7 Is Form VII - Pest-1 present and complete for each Performance Evaluation Mixture analyzed during the analytical sequence for both columns? [ ] ___ ___

ACTION: If no, take action as specified in 3.2 above.

7.8 Has the individual % breakdown exceeded 20.0% on either column. ___ [ ] ___

- for 4,4' - DDT? ___ [ ] ___

- for endrin? ___ [ ] ___

Has the combined % breakdown for 4,4'- DDT/ Endrin exceeded 30.0% on either column? (required in all instances) ___ [ ] ___

ACTION: 1. If any % breakdown has failed the QC criteria in either PEM in steps 2 and 17 in the initial calibration sequence (p. D-38/Pest SOW 3/90), qualify all sample analyses in the entire analytical sequence as described below.

YES NO N/A

2. If any % breakdown has failed the QC criteria in a PEM Verification calibration, review data beginning with the samples which followed the last in-control standard until the next acceptable PEM & qualify the data as described below.

a. 4,4'-DDT Breakdown: If 4,4'-DDT breakdown is greater than 20.%:

   i. Qualify all positive results for DDT with 'J". If DDT was not detected, but DDD and DDE are positive, then qualify the quantitation limit for DDT as unusable (R).

   ii. Qualify positive results for DDD and/or DDE as presumptively present at an approximated quantity (NJ).

b. Endrin Breakdown: If endrin breakdown is greater than 20.0%:

   i. Qualify all positive results for endrin with "J". If endrin was not detected, but endrin aldehyde and endrin ketone are positive, then qualify the quantitation limit for endrin as unusable (R).

   ii. Qualify positive results for endrin ketone and endrin aldehyde as presumptively present at an approximated quantity (NJ).

c. Combined Breakdown: If the combined 4,4'-DDT and endrin breakdown is greater than 30.0%:

   i. Qualify all positive results for DDT and endrin with "J". If endrin was not detected, but endrin aldehyde and endrin ketone are positive, then qualify the quantitation limit for endrin as unusable (R). If DDT was not detected, but DDD and DDE are positive, then qualify the quantitation limit for DDT as unusable (R).

YES NO N/A

ii. Qualify positive results for endrin ketone and endrin aldehyde as presumptively present at an approximated quantity (NJ). Qualify positive results for DDD and/or DDE as presumptively present at an approximated quantity (NJ).

7.9 Are the relative percent difference (RPD) values for all PEM analytes <25.0%? (Form VII-PEST-1) [ ] ___ ___

ACTION: If no, qualify all associated positive results generated during the analytical sequence "J" and sample quantitation limits "UJ".

NOTE: If the failing PEM is part of the initial calibration. all samples are potentially affected. If the offending standard is a verification calibration, the associated samples are those which followed the last in-control standard until the next passing standard.

7.10 Have all samples been injected within a 12 hr. period beginning with the injection of an Instrument Blank? [ ] ___ ___

ACTION: If no, use professional judgement to determine the severity of the effect on the data and qualify accordingly.

7.11 Is Form VII - Pest-2 present and complete for each INDA and INDB Verification Calibration analyzed? [ ] ___ ___

ACTION: If no, take action specified in 3.2 above.

7.12 Are there any transcription/calculation errors between raw data and Form VII - Pest-2? ___ [ ] ___

ACTION: If large errors exists, call lab for explanation/resubmittal, make any necessary corrections and document effect in data assessments. under "Conclusions".

YES NO N/A

7.13 Do all standard retention times for each INDA and INDB Verification Calibration fall within the windows established by the initial calibration sequence? [ ] ___ ___

ACTION: If no, beginning with the samples which followed the last in-control standard, check to see if the chromatograms contain peaks within an expanded window surrounding the expected retention times. If no peaks are found and the surrogates are visible, non-detects are valid. If peaks are present and cannot be identified through pattern recognition or using a revised RT window, qualify all positive results and non-detects as unusable (R).

7.14 Are RPD values for all verification calibration standard compounds < 25.0%? [ ] ___ ___

ACTION: If the RPD is >25.0% for the compound being quantitated, qualify all associated positive results "J" and non-detects "UJ". The "associated samples" are those which followed the last in-control standard up to the next passing standard containing the analyte which failed the criteria. If the RPD is >90%, flag all non-detects for that analyte R (unusable).

8.0 Analytical Sequence Check (Form VIII-PEST)

8.1 Is Form VIII present and complete for each column and each period of analyses? [ ] ___ ___

ACTION: If no, take action specified in 3.2 above.

8.2 Was the proper analytical sequence followed for each initial calibration and subsequent analyses? (see CLP SOW p. D-39 & D-41/PEST) [ ] ___ ___

ACTION: If no, use professional judgement to determine the severity of the effect on the data and qualify it accordingly. Generally, the effect is negligible unless the sequence was grossly altered or the calibration was also out of limits.

	YES	NO	N/A

9.0 Cleanup Efficiency Verification (Form IX)

9.1 Is Form IX - Pest-1 present and complete for each lot of Florisil Cartridges used? (Florisil Cleanup is required for all Pest/PCB extracts.) [ ] ___ ___

ACTION: If no, take action specified in 3.2 above. If data suggests that florisil cleanup was not performed, make note in "Contract Problems/Non-Compliance".

9.2 Are all samples listed on the Pesticide Florisil Cartridge Check Form? [ ] ___ ___

ACTION: If no, take action specified in 3.2 above.

9.3 If GPC Cleanup was performed, (mandatory for all soil sample extracts) is Form IX - Pest-2 present? [ ] ___ ___

ACTION: If no, take action specified in 3.2 above.

ACTION: If GPC was not performed when required, make note in" Contract Problems/Non-Compliance" section of data assessment.

9.4 Are percent recoveries (% R) of the pesticide and surrogate compounds used to check the efficiency of the cleanup procedures within QC limits:

80-120% for florisil cartridge check? [ ] ___ ___

80-110% for GPC calibration? [ ] ___ ___

Qualify only the analyte(s) which fail the recovery criteria as follows:

ACTION: If % R are < 80%, qualify positive results "J" and quantitation limits "UJ". Non-detects should be qualified "R" if zero %R was obtained for pesticide compounds. Use professional judgement to qualify positive results if recoveries are greater than the upper limit.

	YES	NO	N/A

NOTE: Sample data should be evaluated for potential interferences if recovery of 2,4,5-trichlorophenol was > 5% in the Florisil Cartridge Performance Check analysis. Make note in Contract Problems/ Non-Compliance section of reviewer narrative.

NOTE: The raw data of the GPC Calibration Check analysis is evaluated for pattern similarity with previously run Aroclor standards.

10.0 Pesticide/PCB Identification

10.1 Is Form X complete for every sample in which a pesticide or PCB was detected? [ ] ___ ___

ACTION: If no, take action specified in 3.2 above.

10.2 Are there any transcription/calculation errors between raw data and Forms 6E, 6G, 7E, 7D, 8D, 9A, B, 10A. ___ [ ] ___

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and note error under "Conclusions".

10.3 Are retention times (RT) of sample compounds within the established RT windows for both analyses? [ ] ___ ___

Was GC/MS confirmation provided when required (when compound concentration is > 10 ug/ml in final extract)? [ ] ___ ___

Action: Use professional judgement to qualify positive results which were not confirmed by GC/MS. Qualify as unusable (R) all positive results which were not confirmed by second GC column analysis. Also qualify as unusable (R) all positive results not meeting RT window unless associated standard compounds are similarly biased. (see Functional Guidelines) The reviewer should use professional judgement to assign an appropriate quantitation limit.

YES NO N/A

10.4 Is the percent difference (% D) calculated for the positive sample results on the two GC columns < 25.0%? [ ] ___ ___

ACTION: If the reviewer finds neither column shows interference for the positive hits, the data should be flagged as follows:

% Difference	Qualifier
25-50 %	J
50-90 %	JN
> 90 %	R

NOTE: The lower of the two values is reported on Form I. If using professional judgement, the reviewer determines that the higher result was more acceptable, the reviewer should replace the value and indicate the reason for the change in the data assessment.

10.5 Check chromatograms for false negatives, especially the multiple peak compounds toxaphene and PCBs. Were there any false negatives? ___ [ ] ___

ACTION: Use professional judgement to decide if the compound should be reported. If the appropriate PCB standards were not analyzed, qualify the data unusable (R).

11.0 Compound Quantitation and Reported Detection Limits

11.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Were any errors found? ___ [ ] ___

NOTE: Single-peak pesticide results can be checked for rough agreement between quantitative results obtained on the two GC columns. The reviewer should use professional judgement to decide whethera much larger concentration obtained on one column versus the other indicates the presence of an interfering compound. If an interfering compound is indicated, the lower of the two values should be reported and qualified as presumptively present at an approximated quantity (NJ). This necessitates a determination of an estimated concentration on the confirmation column. The narrative should indicate that the presence of interferences has interfered with the evaluation of the second column confirmation.

	YES	NO	N/A
11.2 Are the CRQLs adjusted to reflect sample dilutions and, for soils, % moisture?	[ ]	___	___

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and document effect in data assessments.

ACTION: When a sample is analyzed at more than one dilution, the lowest CRQLs are used (unless a QC exceedance dictates the use of the higher CRQL data from the diluted sample analysis). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" value on the original Form I and substituting it with data from the analysis of diluted sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form I's that should not be used, including any in the summary package.

ACTION: Quantitation limits affected by large, off-scale peaks should be qualified as unusable (R). If the interference is on-scale, the reviewer can provide an approximated quantitation limit (UJ) for each affected compound.

12.0 Chromatogram Quality

12.1 Were baselines stable?	[ ]	___	___
12.2 Were any electropositive displacement (negative peaks) or unusual peaks seen?	___	[ ]	___

ACTION: Address comments under System Performance of data assessment.

YES NO N/A

13.0 Field Duplicates

13.1 Were any field duplicates submitted for PEST/PCB analysis? [ ] ___ ___

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. However, if large differences exist, identification of field duplicates should be confirmed by contacting the sampler.

CLP DATA ASSESSMENT

Functional Guidelines for Evaluating Organic Analysis

Case No._____SDG No._____LABORATORY_____SITE_____

DATA ASSESSMENT:

The current Functional Guidelines for evaluating organic data have been applied.

All data are valid and acceptable except those analytes which have been qualified with a "J" (estimated), "N" (presumptive evidence for the presence of the material), "U" (non-detects), "R" (unusable),or "JN" (presumptive evidence for the presence of the material at an estimated value). All action is detailed on the attached sheets.

Two facts should be noted by all data users. First, the "R" flag means that the associated value is unusable. In other words, due to significant QC problems, the analysis is invalid and provides no information as to whether the compound is present or not. "R" values should not appear on data tables because they cannot be relied upon, even as a last resort. The second fact to keep in mind is that no compound concentration, even if it has passed all QC tests, is guaranteed to be accurate. Strict QC serves to increase confidence in data but any value potentially contains error.

Reviewer's
Signature:_____Date:___/____/199__

Verified By:_____Date:___/_____/199__

DATA ASSESSMENT

1. HOLDING TIME:

The amount of an analyte in a sample can change with time due to chemical instability, degradation, volatilization, etc. If the specified holding time is exceeded, the data may not be valid. Those analytes detected in the samples whose holding time has been exceeded will be qualified as estimated, "J". The non-detects (sample quantitation limits) will be flagged as estimated, "J", or unusable, "R", if the holding times are grossly exceeded.

The following analytes in the samples shown were qualified because of holding time:

DATA ASSESSMENT

2. BLANK CONTAMINATION:

Quality assurance (QA) blanks, i.e., method, trip, field, or rinse blanks are prepared to identify any contamination which may have been introduced into the samples during sample preparation or field activity. Method blanks measure laboratory contamination. Trip blanks measure cross-contamination of samples during shipment. Field and rinse blanks measure cross- contamination of samples during field operations. If the concentration of the analyte is less than 5 times the blank contaminant level (10 times for the common contaminants), the analytes are qualified as non- detects, "U". The following analytes in the samples shown were qualified with "U" for these reasons:

A) Method blank contamination

B) Field or rinse blank contamination ("water blanks" or "distilled water blanks" are validated like any other sample)

C) Trip blank contamination

DATA ASSESSMENT

3. MASS SPECTROMETER TUNING:

Tuning and performance criteria are established to ensure adequate mass resolution, proper identification of compounds, and to some degree, sufficient instrument sensitivity. These criteria are not sample specific. Instrument performance is determined using standard materials. Therefore, these criteria should be met in all circumstances. The tuning standard for volatile organics is bromofluorobenzene (BFB) and for semi-volatiles is decafluorotriphenyl- phosphine (DFTPP).

If the mass calibration is in error, or missing, all associated data will be classified as unusable, "R". The following samples shown were qualified with "R" because of tuning:

DATA ASSESSMENT

4. CALIBRATION:

Satisfactory instrument calibration is established to ensure that the instrument is capable of producing acceptable quantitative data. An initial calibration demonstrates that the instrument is capable of giving acceptable performance at the beginning of an experimental sequence. The continuing calibration verifies that the instrument is giving satisfactory daily performance.

A) RESPONSE FACTOR

The response factor measures the instrument's response to specific chemical compounds. The response factor for the VOA/BNA Target Compound List (TCL) must be $\geq$ 0.05 in both the initial and continuing calibrations. A value < 0.05 indicates a serious detection and quantitation problem (poor sensitivity). If the mean RRF of the initial calibration or the continuing calibration has a response factor <0.05 for any analyte, those analytes detected in environmental samples will be qualified as estimated, "J". All non-detects for those compounds will be rejected ("R"). The following analytes in the samples shown were qualified because of response factor:

DATA ASSESSMENT

5. CALIBRATION:

A) PERCENT RELATIVE STANDARD DEVIATION (%RSD) AND PERCENT DIFFERENCE (%D):

Percent RSD is calculated from the initial calibration and is used to indicate the stability of the specific compound response factor over increasing concentration. Percent D compares the response factor of the continuing calibration check to the mean response factor (RRF) from the initial calibration. Percent D is a measure of the instrument's daily performance. Percent RSD must be <30% and %D must be <25%. A value outside of these limits indicates potential detection and quantitation errors. For these reasons, all positive results are flagged as estimated, "J"; and non-detects are flagged "UJ". If %RSD and %D grossly exceed QC criteria, non-detect data may be qualified "R".

For the PCB/PESTICIDE fraction, if %RSD exceeds 20% for all analytes except for the 2 surrogates (which must not exceed 30% RSD), qualify all associated positive results "J" and non-detects "UJ".

The following analytes in the samples shown were qualified for %RSD and %D:

DATA ASSESSMENT

6. SURROGATES/ SYSTEM MONITORING COMPOUNDS (SMC):

All samples are spiked with surrogate/ SMC compounds prior to sample preparation to evaluate overall laboratory performance and efficiency of the analytical technique. If the measured surrogate/ SMC concentrations were outside contract specifications, qualifications were applied to the samples and analytes as shown below. The following analytes for the samples shown were qualified because of surrogate/ SMC recovery:

DATA ASSESSMENT

7. INTERNAL STANDARDS PERFORMANCE:

Internal Standard (IS) performance criteria ensure that the GC/MS sensitivity and response are stable during every experimental run. The internal standard area count must not vary by more than a factor of 2 (-50% to +100%) from the associated continuing calibration standard. The retention time of the internal standard must not vary more than ±30 seconds from the associated continuing calibration standard. If the area count is outside the (-50% to +100%) range of the associated standard, all of the positive results for compounds quantitated using that IS are qualified as estimated, "J", and all non-detects as "UJ" only if IS area is < 50%. Non detects are qualified as "R" if there is a severe loss of sensitivity (< 25% of associated IS area counts).

If an internal standard retention time varies by more than 30 seconds, the reviewer will use professional judgment to determine either partial or total rejection of the data for that sample fraction. The following analytes in the samples shown were qualified because of internal standards performance:

DATA ASSESSMENT

8. COMPOUND IDENTIFICATION:

A) VOLATILE AND SEMI-VOLATILE FRACTIONS

TCL compounds are identified on the GC/MS by using the analyte's relative retention time (RRT) and ion spectra. For the results to be a positive hit, the sample peak must be within ± 0.06 RRT units of the standard compound, and have an ion spectra which has a ratio of the primary and secondary m/e intensities within 20% of that in the standard compound. For tentatively identified compounds (TIC), the ion spectra must match accurately. In the cases where there is not an adequate ion spectrum match, the laboratory may have provided false positive identifications. The following analytes in the samples shown were qualified for compound identification:

B) PESTICIDE FRACTION:

The retention times of reported compounds must fall within the calculated retention time windows for the two chromatographic columns. The percent difference (%D) of the positive results obtained on the two GC columns should be ≤25% The following analytes in the samples shown were qualified because of compound identification:

DATA ASSESSMENT

9. MATRIX SPIKE/SPIKE DUPLICATE, MS/MSD:

The MS/MSD data are generated to determine the long-term precision and accuracy of the analytical method in various matrices. The MS/MSD may be used in conjunction with other QC criteria for some additional qualification of data. The following analytes, for the samples shown, were qualified because of MS/MSD:

DATA ASSESSMENT

10. OTHER QC DATA OUT OF SPECIFICATION:

11. SYSTEM PERFORMANCE AND OVERALL ASSESSMENT (continued on next page if necessary):

12. CONTRACTUAL NON-COMPLIANCE:

13. This package contains re-extraction, re-analysis or dilution. Upon reviewing the QA results, the following form I(s) are identified to be used:

DATA ASSESSMENT

11. SYSTEM PERFORMANCE AND OVERALL ASSESSMENT (continued):

SOP NO. HW-7
Revision # 3

TCLP DATA VALIDATION

BY: Leon Lazarus Date: March 12 1993
Leon Lazarus, Environmental Scientist
Toxic and Hazardous Waste Section

BY: George Karras Date: March 16, 1993
George Karras, Chemist
Toxic and Hazardous Waste Section

CONCURRED BY: Kevin W. Kubik Date: March 22, 1993
Kevin Kubik, Chief
Toxic and Hazardous Waste Section

APPROVED BY: Robert Runyon Date: March 22, 1993
Robert Runyon, Chief
Monitoring Management Branch

ALL LAND BAN TCLP ANALYSIS MUST USE SW-846 METHODS.

THIS SOP ONLY APPRAISES THE TCLP EXTRACTION PROCEDURE. TO COMPLETELY VALIDATE A TCLP ANALYSIS, YOU MUST ALSO USE THE REGION II SOPS FOR ORGANIC AND INORGANIC DATA VALIDATION.

BEFORE VALIDATING TCLP DATA, THE DATA VALIDATOR MUST DETERMINE IF ANY TOXICITY CHARACTERISTIC OR LAND BAN REGULATORY ACTION LEVELS ARE APPLICABLE.

	YES	NO	N/A
Was a ZHE vessel used for VOAs?	[___]	___	___
Was there any evidence of leakage?	___	[___]	___

Action: If a ZHE vessel leaked, or was not used, reject (R) all VOA data, except data which exceeds the regulatory level for any analyte. See attached list for TC regulatory levels. If other analytes are being validated, the validator must determine which, if any, Land Ban regulatory levels are applicable. The Land Ban TCLP regulatory levels are listed in 40CFR268.

Did the lab use proper bottles?	[___]	___	___

Action: If a plastic bottle was used, except for PTFE, reject (R) all non detect organic data. All positive organic values should be flagged as presumptively present at an estimated quantity (JN).

Did the lab correctly compute % solids?	[___]	___	___

Action: If the lab made an error, request revised data.

If appropriate, did the lab reduce particle size?	[___]	___	___

Action: If the lab did not perform a required particle size reduction, reject (R) all non detects. All positive values should be flagged as presumptively present at an estimated quantity (JN).

Was the correct extraction fluid used?	[___]	___	___
Was the pH of the extraction fluid correct? (4.88-4.98 for fluid #1) (2.83 - 2.93 for extraction fluid #2)	[___]	___	___

Action: If the extraction fluid pH was wrong, or the wrong fluid was used, reject (R) all non detects. All positive values should be flagged as presumptively present at an estimated quantity (JN).

	YES	NO	N/A
Was the correct weight of extraction fluid used?	[___]	___	___

Action: If the extraction fluid weight is not ±15% of the correct value, flag all results as estimated (J). If the extraction fluid weight is more than 30% above the correct value, reject (R) all non detects, and flag all positive values as presumptively present at an estimated quantity (JN).

For volatile analytes, was the sample weight 25 grams or less?	[___]	___	___

Action: If the sample weight is more than 25 grams, flag all data as estimated (J).

Were the TCLP extracts properly preserved? (Metals must be preserved to a pH <2 with HNO_3).	[___]	___	___

Action: If the preservative causes precipitation, the sample should not be preserved, but the sample should be analyzed as soon as possible after extraction. The use of organic preservatives is optional. If proper inorganic preservation procedures were not followed, reject (R) all non detects, and flag all positive values below regulatory action levels as presumptively present at an estimated quantity (JN). Positive data at concentrations above regulatory action levels should not be qualified.

Is there a TCLP blank with the appropriate TCLP fluid for every 20 samples? (This is in addition to the method blanks, which are required for each analytical method).	[___]	___	___

Action: If there is no TCLP blank, call laboratory for explanation/resubmittal. If not available, reject (R) all associated positive data. Contaminants in TCLP blanks should be treated as method blank contaminants when validating data.

Have samples been analyzed within TCLP holding times from date of collection ?	[__]	__	__

NOTE: CLP holding times do not apply to TCLP analysis. The following table lists TCLP holding times:

TCLP Holding Times

TCLP HOLDING TIMES (DAYS)	FROM COLLECTION TO TCLP EXTRACTION	FROM TCLP EXTRACTION TO PREPARATIVE EXTRACTION	FROM PREPARATIVE EXTRACTION TO ANALYSIS
VOA	14	N/A	14
ORGANIC EXTRACTABLES	14	7	40
MERCURY	28	N/A	28
OTHER METALS	180	N/A	180

HOLDING TIME DECISION TABLE

Have samples been analyzed within TCLP holding time?

If Yes.
Action: Do not qualify data because of holding time.

If No.
Action: In the sample, does any analyte exceed the regulatory level?
Toxicity Characteristic regulatory action levels are listed on page 5 of this SOP.
The Land Ban regulatory action levels are listed in 40CFR268.

If No.
Action: Reject (R) all analytes.

If Yes.
Action: Do not qualify analytes which exceed regulatory levels. Mention in data assessment that reported value represents the minimum concentration present.

Assume that TCLP analysis of TC analytes is for the purpose of determining compliance with the TC regulatory levels (attached). If other analytes are being validated, the validator must determine which, if any, Land Ban regulatory levels are applicable. The Land Ban TCLP regulatory levels are listed in 40CFR268.

ANALYTICAL DATA MUST BE VALIDATED ACCORDING TO THE REGIONAL ORGANIC AND INORGANIC DATA VALIDATION SOPS BEFORE THE FOLLOWING QUESTIONS MAY BE ADDRESSED.

	YES	NO	N/A
Have any acetates, acetic acid, or acetic anhydride been reported as TICs?	___	[___]	___
Action : If yes, reject (R) TICs.			
Are all organic compounds analyzed by the TCLP method properly calibrated?	[___]	___	___
Analytes on Form I that have not been calibrated should be qualified as follows: non-detects should be rejected (R); positive values should be reported as TICs, and flagged "JN".			
Have multi-phasic samples been properly analyzed? (Check to see if aqueous samples have > .5% solids.) If not, reject (R) all data below regulatory action levels.	[___]	___	____
Have adequate raw data deliverables been submitted?	[___]	___	___
Action: If not, contact the laboratory. If the raw data is not available, use professional judgement to qualify analytical data, and mention in data assessment.			
Was the method of standard additions properly utilized for analysis of metals?	[___]	___	___
Action: If not, all metals data should be qualified as estimated "J".			

THE FOLLOWING STATEMENT MUST BE ADDED TO ALL TCLP DATA VALIDATION REPORTS:

Analytical data qualified as "JN" or "R" may not be used to demonstrate compliance with Toxicity Characteristic or Land Ban Regulations.

TC ANALYTES AND THEIR REGULATORY LEVELS

Constituent	Regulatory Level (mg/l)	Constituent	Regulatory Level (mg/l)
benzene	0.5	hexachlorobenzene	0.13
carbon tetrachloride	0.5	hexachloro-1,3-butadiene	0.5
chlordane	0.03	hexachloroethane	3.0
chlorobenzene	100.0	methyl ethyl ketone	200.0
chloroform	6.0	nitrobenzene	2.0
o-cresol	200.0	pentachlorophenol	100.0
m-cresol	200.0	pyridine	5.0
p-cresol	200.0	tetrachloroethylene	0.7
1,4-dichlorobenzene	7.5	trichloroethylene	0.5
1,2-dichloroethane	0.5	2,4,5-trichlorophenol	400.0
1,1-dichloroethylene	0.7	2,4,6-trichlorophenol	2.0
2,4-dinitrotoluene	0.13	vinyl chloride	0.2
heptachlor	0.008		
arsenic	5.0	silver	5.0
barium	100.0	endrin	0.02
cadmium	1.0	lindane	0.4
chromium	5.0	methoxychlor	10.0
lead	5.0	toxaphene	0.5
mercury	0.2	2,4-D	10.0
selenium	1.0	2,4,5-TP (silvex)	1.0

Appendix V

Appendix V
References for Multi-phasic and Oily Waste

United States
Environmental Protection Agency

Workshop on

Predicting the Environmental

Impact of Oily Materials

July 14, 1992

Eighth Annual Waste Testing
And
Quality Assurance
Symposium

Arlington, VA

PREDICTING THE ENVIRONMENTAL IMPACT OF OILY MATERIALS: INTRODUCTION AND REGULATORY PERSPECTIVE

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BACKGROUND

Prevention of groundwater contamination has historically been one of the EPA's highest priorities in implementing the RCRA program. To that end, the Agency has developed and promulgated test methods, fate and transport models, and regulatory standards to control the management of wastes whose properties might pose a hazard to groundwater. Scientists are concerned with oily waste due to its volume, toxicity, and potential for causing severe ecological damage. Such wastes take many forms including: liquids of widely varying viscosity, contaminated soils, sludges, and tarry "plastic" masses.

Oily wastes have some unique properties. They can migrate like a liquid but appear to be a solid. Because they result from many commercial processes and applications, they are broadly distributed, of very large volume, and of tremendous commercial importance.

In developing the hazardous waste identification characteristics, EPA highlighted its concerns with protecting ground water resources by developing the Extraction Procedure Toxicity Characteristic (40 CFR 261.24). The characteristic relies on laboratory procedures to predict toxicant mobility.

Over the years a number of laboratory extraction methods have been applied to the problem of predicting what might migrate from oily wastes managed under landfill conditions. Among the test methods that have been developed and employed to identify those wastes which might pose an unacceptable hazard are: EPA methods 1310, 1311 and 1330 (Extraction Procedure, Toxicity Characteristic Leaching Procedure and Oily Waste Extraction Procedure).

The current approaches all have deficiencies with respect to predicting the mobility of toxic chemicals from oily wastes. Methods 1310 (EP) and Method 1311 (the TCLP) underestimate the mobility of many oily wastes due to filter clogging problems, their precision is less than desirable, and they have certain operational problems. Conversely, Method 1330 (OWEP) probably overestimates mobility since it emulates a worst possible case scenario. None of the available laboratory mobility procedures is thus totally satisfactory.

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PROBLEM

Given the importance of this issue, it is imperative that accurate, precise, and usable approaches to characterizing the mobility of oily materials be developed. That is what we are here for today.

The problem of mobility estimation is too large and complicated for us to try and solve all its aspects in a half day. Therefore, we will focus on just one aspect of the problem - predicting the initial source term. To put it another way, we want to predict the highest concentration of material that might be released from the waste to the soil immediately below the point of disposal for some reasonable amount of time. This information would then feed into the fate and transport models used to predict the final toxicant concentration at some distance away from the disposal area.

DISPOSAL SCENARIO OF CONCERN

The priority waste management facility scenario that EPA has selected to be modeled in this workshop is placement of the waste into or on the ground (e.g., landfill or lagoon). Within this scenario a number of parameters need to be considered. These include:

- Temperature (assume temperate conditions),
- Rainfall regime,
- Biodegradation,
- Hydrolysis,
- Soil types (assume soil underlying the waste management unit has a porosity similar to that of sand), and
- Amount of waste (assume amount is large enough so that it can be considered to be infinite).

FATE AND TRANSPORT MODEL CONSIDERATIONS

To properly manage oily wastes to protect ground water sources from contamination by waste constituents, an adequate model to predict the fate and transport in the subsurface environment is needed. The Agency is developing a model to simulate the migration of aqueous and nonaqueous phase liquids and the transport of individual chemical constituents which may move by convection and dispersion in each phase.

As input parameters, the model needs information on the amount and composition of both the aqueous and nonaqueous phase liquid portions of the waste as well as the composition of the leachate that might be generated by action of surface waters on any

"solid" material that may have initially been present in the waste material. At this time, the Agency does not have a precise way of defining either an "aqueous phase liquid" or a "nonaqueous phase liquid".

REGULATORY PERSPECTIVE

In an ideal situation, an effective approach to evaluating oily wastes would:

- Be simple to use (not require sophisticated equipment, nor constant attention by a highly trained technician),
- Be inexpensive to run,
- Take as little time as possible to perform (ideally no more than 24 hours),
- Be accurate (relative to predicting behavior of waste in the environment),
- Be precise (i.e., be reproducible),
- Be rugged (capable of characterizing a broad range of waste types and constituents of concern), and
- Not generate wastes (e.g., generate little if any solvent waste and waste contaminated media).

The characteristics that the approach should have (maximum desirable values for each parameter) are:

- A high degree of freedom from false negatives (any errors tend toward overestimation of threat to environment),
- Precision (RSD <50%),
- Relatively low cost ,
- Taking as little time as possible to perform (<24 hours), and
- Ruggedness.

OPTIONS FOR CONSIDERATION

I. Develop a two-component mobility test that determines the fraction of the waste which is flowable (capable of physical movement under the influence of gravity and overburden pressure) under the conditions of the test. Suggested conditions: room temperature and 50 psi. Define as mobile all material that is flowable under terms of the test plus the aqueous extract of the non-flowable fraction. Under this option, the procedures used are independent of waste properties and disposal environment.

II. Develop a single generic laboratory procedure to estimate what disposal point concentration would result from aqueous leaching of the hazardous constituents from both the mobile and non-mobile fraction of the material. Under this option, the procedures used are independent of waste properties and disposal environment.

III. Employ a series of laboratory test procedures to evaluate the waste material. These procedures would be keyed to the fate and transport model to be employed to evaluate the data. The procedure also would be independent of the properties of the material under evaluation.

The questions we would like you to address are:

- What would be the "best" approach to use in order to predict the nature and concentration of the components that would leach from oily wastes if the waste were to be placed in an unlined landfill environment?

- If the necessary tools are not presently available, how should such a test method or model be developed and evaluated?

- What form should a cooperative development program take? How could it be organized? Who might the cooperators be? How long would you expect it to take to develop the necessary tools?

If you think of any ideas, information, or suggestions that you feel the Agency should consider when addressing this issue, please send them to us. Send your comments to:

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We will need to receive your comments by August 21, 1992 in order for them to be incorporated into the conference final report.

PREDICTING THE ENVIRONMENTAL IMPACT OF OILY WASTE: INDUSTRY PERSPECTIVE

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STATEMENT OF ISSUE

Managing solid waste in an environmentally sound manner is a subject of high concern to industry, EPA and the public. However, we must have regulatory tools which accurately reflect the environmental hazard, and analysis tools which accurately assess the potential impact of various management approaches. We need to bring the best science to bear on the evaluation of the potential environmental threat from oily waste disposal, considering the likely management scenarios. One way EPA has decided to regulate certain oily waste in the past is by listing the waste as hazardous under RCRA. This approach identifies a material as an environmental threat based on possible (but not necessarily realistic) mismanagement scenarios. A second way to regulate the waste is to determine if it is hazardous using the toxicity characteristic (TC) and the Toxicity Characteristics Leaching Procedure (TCLP) test. This test is used to determine whether a waste is hazardous or not based upon a specific leachability test and municipal landfill disposal scenario. The options explored in this paper serve to promote thinking about new approaches for identifying the environmental threats and thereby to better focus regulations dealing with the management of these materials. This will be done by introducing and critiquing current most common predictive methods and presenting potential avenues for more accurate methods.

Although it is nearly impossible to precisely define the term "oily waste", the following analysis can provide a basis for further discussion:

a) An oil is generally an immiscible or relatively insoluble liquid, varying in composition but consisting of organic constituents. Petroleum oils principally consist of hydrocarbons; vegetable and animal oils are glycerides, and fatty acids; and essential oils are terpenes, alkaloids, etc.

b) An oily waste is an industrial process waste or residual bearing oil in visual and/or measurable proportions.

c) Oil in oily wastes can occur in any matrix, including: sorbed to dry solids; in sludges or slurries; multi-phase liquids or sludges/slurries with multi-phase

liquids, if water is present. Proper treatment and disposal of all such matrices is a concern of the petroleum industry.

d) Analysis of oils in oily wastes can be accomplished by techniques such as Total Petroleum Hydrocarbons (TPH) (not constituent-specific) or TCLP (constituent-specific). In a number of contexts, the procedures of methods such as these serve to define what is meant by "oil" and "oily waste."

e) Oily wastes possess a wide variety of compositions and physical and toxicological properties.

Some examples of oily waste include petroleum refinery sludges, such as oil- water separator sludge and dissolved air floatation froth, storage tank bottom sludge, used oil and others. Expanded beyond the petroleum community there are many types of oily wastes (POTW sludges, polymer plants, timber processing, iron and steel, pulp and paper, meat packing, slaughterhouse, leather tanning, coil coating,restaurants, and miscellaneous foods including meat, dairy and vegetable based oils and fats, etc.).

Currently there are a variety of state and local programs designed to address potential environmental impacts of the management of various types of oily materials, such as E&P wastes, spill residues and UST wastes. A number of RCRA listed and toxicity characteristic wastes are also regulated under federal programs. EPA is currently evaluating the possible listing of additional petroleum refining wastes.

Unfortunately, the current analytical methods for determination of the environmental threat of petroleum constituents in wastes and oily materials via the TCLP test and model and RCRA listing system remain controversial. USEPA, academia, and the regulated community are continuing efforts to identify a sound, reproducible methodology to accurately assess these threats. In fact, EPA has recently proposed a rule to address the over-regulation of listed wastes created by EPA's "mixture" and "derived-from" rules. This initiative, called the Hazardous Waste Identification Rule, could have major impacts on the classifications and management of hazardous and nonhazardous industrial wastes, including oily waste.

An approach that the American Petroleum Institute (API) has suggested to the Agency is concentration-based exclusion criterion coupled with contingent management. It is a two-tiered process for determining whether wastes captured under the RCRA listing rule should or should not continue to be regulated as hazardous. One tier would allow wastes with constituents below health based levels (with an appropriate multiplier to account for dilution and attenuation) to be deemed nonhazardous provided the waste does not exhibit a RCRA characteristic. A second tier would allow wastes that contain constituents below somewhat higher health based levels to be deemed as nonhazardous, provided these wastes are managed in certain environmentally protective ways. This second approach would alter the current system by basing a waste's classification on how it is actually managed and not how it could be hypothetically mismanaged.

On the test method side, many, including Environment Canada, ASTM and the USEPA, have been involved with the development of improved leachability and contaminant fate/transport tests and models. Despite this work, predicting the potential environmental hazard associated with oily wastes remains problematic.

TCLP APPROACH

The toxicity characteristic leaching procedure (TCLP) was developed as a way to evaluate the threat of solid waste disposal under "...a mismanagement scenario for toxic wastes which constitutes a prevalent form of improper management--namely, the co-disposal of toxic wastes in an actively decomposing municipal landfill which overlies a groundwater aquifer..." (Fed. Reg., May 8, 1990). The TCLP is a leaching and acidic aqueous extraction test. The test was designed to model mismanagement of the disposal of process wastes. The Toxicity Characteristic (TC) rule itself, and constituent-specific limits associated with the TC, define wastes as hazardous on the basis of the concentrations of certain toxic constituents. The TC and its constituent-specific limits were developed in large part to protect human health from contamination of drinking water aquifers. The TCLP test, as currently interpreted, is applicable to those wastes which produce a separate non-aqueous phase as well as those which do not.

Specifically the model system that forms the basis for the regulatory limits imposed by the current toxicity characteristic is one which assumes that the waste is disposed of in a municipal hazardous waste landfill where it is leached by acidic landfill liquids, emerges from the landfill bottom into underlying groundwater whereupon it migrates to an hydraulically down-gradient drinking water well (see Figure 1).

The current Toxicity Characteristic defined-method for determining environmental risks uses a three-part system, consisting of a physical model (TCLP), coupled to a mathematical model (EPACML), coupled to a toxicological model. The TCLP simulates constituent leaching from a landfill, EPACML simulates constituent transport from a landfill to a drinking water well, and the toxicological model relates drinking water concentration to health-effects. The TCLP was not designed as, and fails as, a multi-phase model. This is because the oil phase is simply treated as water. Additionally any multi-phase capability within EPACML was ignored due to the TCLP output.

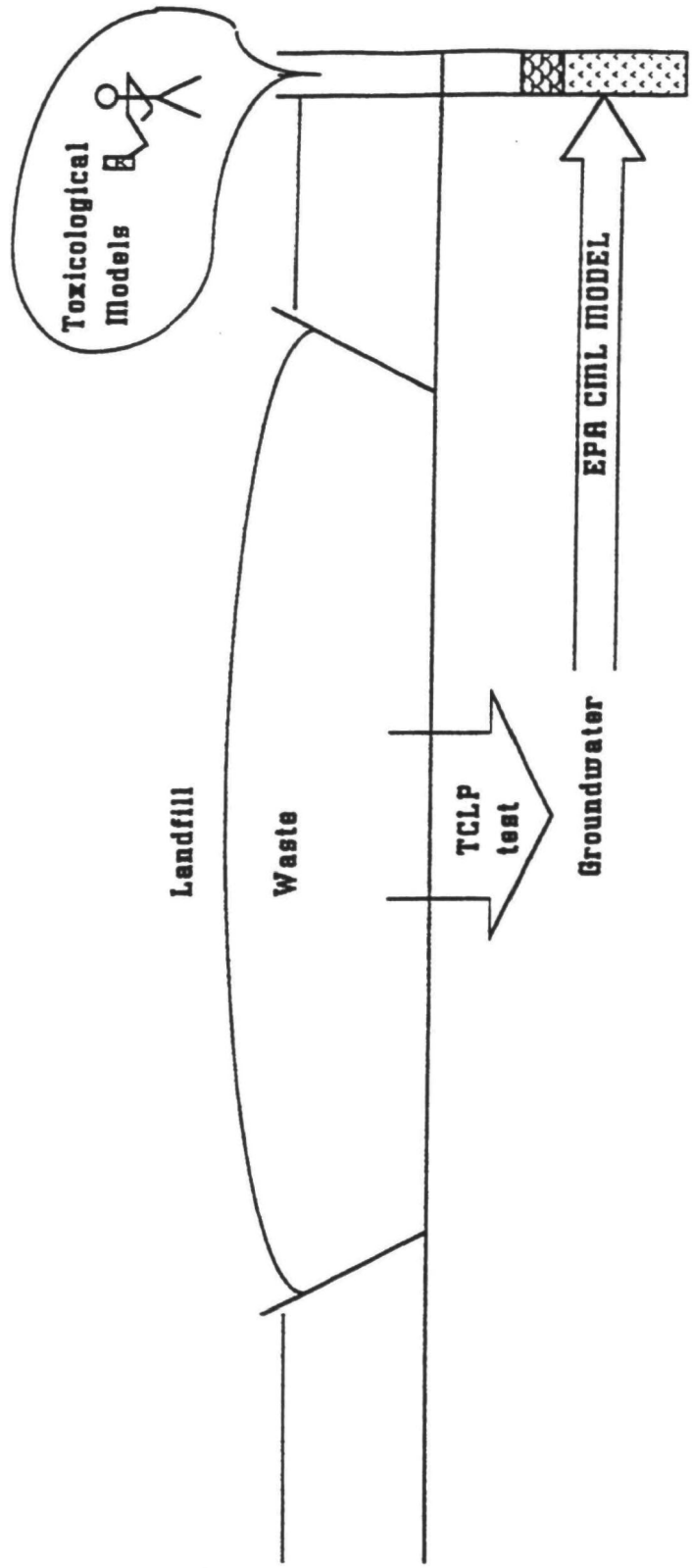

Figure 1: Components of TC "System"

Significant technical aspects of the TCLP simulation can be summarized as follows (see Figure 2).

- No vadose zone-bottom of the landfill is in direct contact with the ground water.
- The disposal of waste liquids (oil and water) are equally mobile.
- The liquids are not leached or diluted but elute directly from the landfill into the ground water.
- Infinite source-liquids continue to be released forever regardless of amount of liquids in the original waste.
- The solids are leached with a 20:1 volume of acidic "landfill leachate" which then enters the ground water.
- Infinite source - the hazardous constituent concentration in the initial 20:1 leachate volume continues to be leached from the material forever, regardless of mass of constituents in the original waste.
- The liquids and leachate travel through the ground water to a drinking water well. Attenuation and dilution reduce concentrations by a factor of 100.
- Oil moves as water.
- Hazardous constituent concentrations achieve steady-state in the well at which time the well-owner drinks two-liters/day for 70 years (oil and all).

VALIDITY OF THE TCLP APPROACH

Oily wastes provide a great challenge to those charged with evaluating their potential impact on the environment. Unfortunately the design of the TCLP test in concept, methodology and fate/transport modeling, inaccurately predicts the behavior of waste containing separate-phase oil and organic constituents. One shortcoming is that it forces a generic disposal scenario which may be reasonable for some cases but impossible for others. Specific problems include operational problems with the test procedure, the assumption that oil behaves identically to water in the environment, the validity of the disposal scenario, and invalid contaminant fate/transport assumptions.

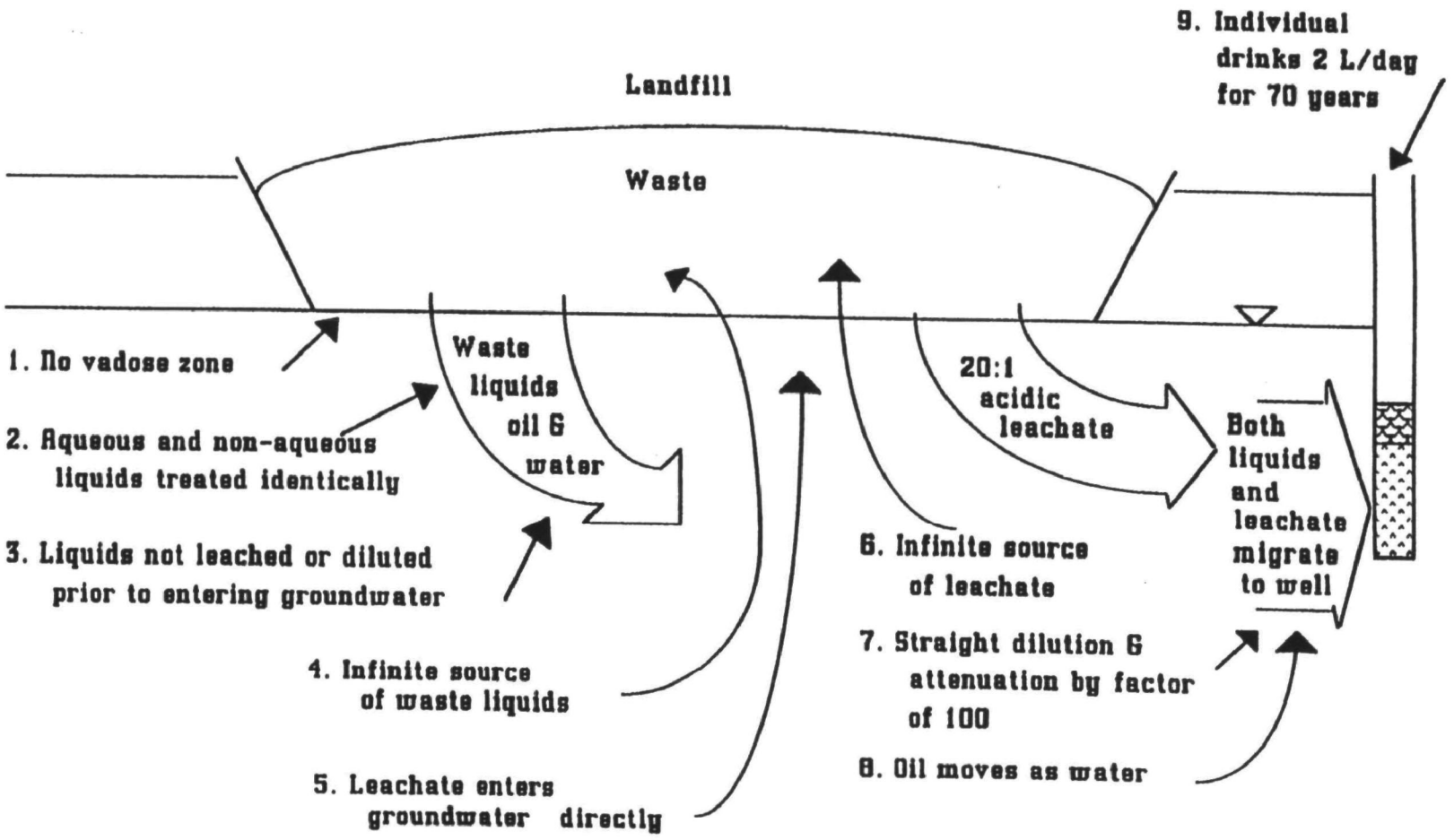

Figure 2: Issues inherent in TC scenario which work to introduce inaccuracy.

The test system was not designed for multi-liquid phase materials. This results in operational problems with the TCLP methodology including non-reproducible free oil breakthrough, filter clogging, and difficulties with volatiles equipment. The zero headspace extractor (ZHE) test equipment is difficult to clean. Some volatile chlorinated compounds are transformed within the TCLP extraction (Bricka, et al, 1991). EPA has, to date, not provided approved test methods which are validated for the analysis of metals in non-aqueous liquids (55 Fed.Reg. 4444).

One of the initial steps in the TCLP test is pressure filtration of the waste. For some oily wastes, non-aqueous liquid may be expressed. This liquid is segregated from the remaining solids which are then acid-leached. This acid leachate is combined with the non-aqueous liquid to produce the "TCLP leachate" which is compared to hazardous/nonhazardous criteria.

Implicit in this procedure is the assumption that both aqueous and non-aqueous liquids will behave identically, both within the landfill and upon their hypothetical release. EPA has been able to provide little, if any, support for this critical portion of the TCLP.

"The initial liquid/solid separation problems are due to the tendency for some material, such as certain types of oily wastes, to clog the 0.45um filter and prevent filtration..... This problem is serious, since materials which do not pass the 0.45 um filter are treated as solids even if they physically appear to be a liquid. These (liquid) wastes are then carried through EP extraction as a solid."

"This is particularly serious for oily wastes, since oils have been known to frequently migrate to ground waters. It is important for the liquid (sic)/solid separation to treat, as liquids, those materials which can behave as liquids in the environment."

"As indicated below, EPA believes that the liquid/solid separation technique.... reduces variability....and that it also provides a more adequate differentiation between those materials that behave as liquids in the environment, and those materials which behave as solids."(51 Fed. Reg. 21658)

As we gain experience with risk evaluations, we see that the risk posed by light, non-aqueous phase liquids (i.e. "oil") appears to be mostly due to dissolved contaminants in drinking water. The calculated risk due to free oil is not great due to the lack of exposure. As it moves through the soil, oil will be immobilized in the soil and from that point may partition into the water phase according to constituent solubilities. Any mobile oil migrating to a water well does not represent a 2L/day, 70 year hazard since it is not realistic to project that anyone will drink free-phase hydrocarbons daily for their entire lives. Therefore, it makes some technical sense to leach the oil fraction with the acidic medium along with the solids.

The disposal scenario as depicted by EPA is not an accurate description of current waste disposal practices. An EPA-OSW survey, several years old already, documents that liquid-type wastes are no longer being accepted by municipal landfills (51 Fed. Reg. 21655). New test methods based upon actual waste management situations would give more accurate results than those based on generic hypothetical scenarios.

Industry has commented upon the shortcomings of the current TCLP/CML model. For example, the infinite source assumptions require contaminant mass to continue to be available for introduction into ground water until steady state is achieved. This is unrealistic. One improvement would be to design transient, declining source terms into the model. Further, there is no consideration of a vadose zone although we know it exists and future landfill regulations will require the presence of a vadose zone. The TCLP is not designed to handle the separate organic phase flow. The current TCLP system does not take into account aerobic biodegradation, volatilization, or retardation. Hydrolysis is apparently being considered at this juncture, but is not currently part of this system.

The unilateral application of TCLP to multi-phase wastes, especially those containing oily materials, is unsupported and inappropriate. There is no evidence that non-aqueous liquids behave as aqueous liquids in a landfill. Indeed, such liquids have an affinity for the solid materials in the landfill which could cause contaminants to be less mobile than predicted by the TCLP.

Until work on the behavior of non-aqueous materials and the prediction of their movement is more mature, the non-aqueous liquids should be treated like the waste itself and be subjected to the same extraction with acidic fluid. To the extent that hazardous constituents are released into the extractant, they should be combined with the aqueous extract generated from the waste solids.

THE EFFECTS OF THE CURRENT TC SCENARIO MODEL - SOME IMPACTS

A number of wastes from the petroleum industry, such as waters from tank drawdowns, ground-water extraction, and hydrotesting, are or may be subject to the TC rule even though there is no conceivable way that these materials would ever find their way into a landfill.

The RCRA Corrective Action Program could potentially generate large quantities of petroleum-contaminated soils. On-site and in-situ management techniques are not accurately represented by the TCLP. In addition, a number of states currently have effective response programs for clean up of spills and other releases of petroleum into the environment. States are concerned that application of the TCLP (particularly if TCLP is a poor estimator of environmental threat) will seriously impact operation and effectiveness of these programs by adding unnecessary and unwarranted hazardous waste handling requirements to wastes which don't pose a threat. We can no longer afford to waste large sums of money handling solid waste in a manner which over estimates the actual environmental threat.

A WAY FORWARD - SAB LEACHABILITY SUBCOMMITTEE

Given the problems with the applicability of the TCLP to multi-phase waste, are there any alternatives? What are the potential ways forward?

Last year, a report was issued by the Science Advisory Board (Environmental Engineering Committee, Leachability Subcommittee), entitled "Recommendations and Rationale for Analysis for Contaminant Release." It contained nine recommendations:

- A variety of contaminant release tests and test conditions which in corporate adequate understanding of the important parameters that affect leaching should be developed and used to assess the potential lease of contaminants from sources of concern.

- Prior to developing or applying any leaching tests or models, the controlling mechanisms must be defined and understood.

- A consistent, repeatable and easily applied, physical, hydrologic and geochemical representation should be developed for the waste management scenario of concern.
- Leach tests and conditions (stresses) appropriate to the situations being evaluated should be used for assessing long-term contaminant release potential.

- Laboratory leach tests should be field-validated, and release test accuracy and precision established before tests are broadly applied.

- More and improved leaching models should be developed and used to complement laboratory tests.

- To facilitate the evaluation of risk implications of environmental releases, the Agency should coordinate the development of leach tests and the development of models in which release terms are used.

- The Agency should establish an inter-office, inter-disciplinary task group, including ORD to help implement these recommendations and devise an Agency-wide protocol for evaluating release scenarios, tests, procedures, and their applications.

- The task group should also be charged with recommending what the appropriate focal point(s), responsibilities, and organizational, budgetary and communication links should be within the Agency for the most effective, continued and ongoing support and pursuit of research, development, and utilization of methods and procedures.

To fully accomplish all of the recommendations will be costly and time- consuming. However there can be no alternate to core research on contaminant release and transport

methods. SAB identified approximately 30 leach tests which are used internationally to attempt to evaluate environmental threat of wastes. Unfortunately SAB concludes that each method suffers from shortcomings.

APPLICATION TO OILY WASTE

If we wish to improve upon the system, there are two basic options:

1. Stick with the physical/mathematical model basis of the TCLP and improve accuracy by modeling multi-phase transport and remove assumptions predicated on long-term human consumption of immiscible product.

2. Replace with a single alternative model, either physical or mathematical.

We understand that EPA is exploring various enhanced modeling systems for multi-phase disposal scenarios. These would include multi-phase flow within the unsaturated zone, and partitioning between aqueous, oil, and air phases within the soil. Also included would be saturated zone groundwater pollutant transport models which are more accurate. Industry favors these developments as tools to better understand oily waste disposal impact.

On the other hand, we must currently deal with an inappropriate TC rule. Currently, industry must comply with the TC rule, which means it must run TCLP tests on oily wastes. This has resulted in a disastrous situation. The TCLP was not designed to accurately assess the environmental threat of oily materials. Therefore it does not. However, decisions on the "proper" management of these wastes are being made on the basis of a flawed test.

Industry has had to deal with the TC for many years. We have modified our waste management approaches and strategies, we have complied with TC and land disposal requirements and we are preparing to fully comply with Corrective Action. Unfortunately, changes to the TC at this point may be just as disruptive and costly as compliance with the TC has been to date. Modifications must be done carefully and deliberately, always using the best possible science to ensure accuracy, not just consistency.

API supports, as mentioned in the Statement of Issue section, a concentration- based exclusion coupled with contingent management for exempting listed hazardous waste from subtitle C requirements. This would address the "inappropriate scenario" dilemma by incorporating elements of actual management approaches instead of one hypothetical approach.

The regulated community has volunteered to work with EPA both as individuals, individual companies, and through trade organizations. We will continue to offer such assistance. For myself, I see continued interaction between EPA- OSW and the American Petroleum Institute. Typical industrial support to EPA includes offering technical comment, procuring wastes, providing waste generation and characterization data, and participating in round-robin testing of new methods.

TABLE 1 - EXTRACTION TESTS

I. STATIC TESTS (LEACHING FLUID NOT RENEWED)

A AGITATED EXTRACTION TESTS

TEST METHOD	LEACHING FLUID	LIQUID-SOLID RATIO	MAXIMUM PARTICLE SIZE	NUMBER OF EXTRACTIONS	TIME OF EXTRACTIONS
TCLP (1311)	ACETIC ACID	20 1	9 5 mm	1	18 HOURS
	0 1 N ACETIC ACID SOLUTION, pH 2 9, FOR ALKALINE WASTES				
	0.1 M SODIUM ACETATE BUFFER SOLUTION, pH 5.0, FOR NON-ALKALINE WASTES				
EP TOX (1310)	0 5 N ACETIC ACID (pH=5.0)	16:1 DURING EXTRACTION 20.1 FINAL DILUTION	9 5 mm	1	24 HOURS
ASTM D3987-85	ASTM TYPE IV REAGENT WATER	20 1	AS IN ENVIRONMENT	1	18 HOURS
CALIFORNIA WET	0 2 M SODIUM CITRATE (pH=5.0)	10:1	2 0 mm	1	48 HOURS
LEACHATE EXTRACTION PROCEDURE (MOE, ONTARIO)	ACETIC ACID 2 MEQ/G	20·1	AS IN ENVIRONMENT	1	24 HOURS
QUEBEC R S Q (MOE, QUEBEC)	INORGANIC 0 02 MEQ/G ORGANIC DISTILLED WATER	10 1	GROUND	1	24 HOURS
FRENCH LEACH TEST (AFMOR, FRANCE)	DI WATER	10 1	9.5 mm	1	16 HOURS
EQUILIBRIUM EXTRACTION (ENVIRONMENT CANADA)	DISTILLED WATER	4 1	GROUND	1	7 DAYS
MULTIPLE BATCH LEACHING PROCEDURE (ENVIRONMENT CANADA)	ACETIC ACID BUFFER, pH 4 5	4.1 OR 2:1	9 5 mm	VARIABLE	24 HOURS

TABLE 1 - EXTRACTION TESTS (continued)

TEST METHOD	LEACHING FLUID	LIQUID:SOLID RATIO	MAXIMUM PARTICLE SIZE	NUMBER OF EXTRACTIONS	TIME OF EXTRACTIONS
MATERIAL CHARACTERIZATION CENTRE-4 (MATERIAL CHARACTERIZATION CENTRE)	CHOICE	10:1	2 FRACTIONS 74 - 149 mm	1 150 - 425 mm	20 DAYS TO 10 YEARS
OILY WASTE (1330)	SOXLET WITH THF AND TOLUENE EP ON REMAINING SOLIDS	100G.300 ML 20:1	9.5 mm	3	24 HOURS (EP)
SYNTHETIC PRECIPITATION LEACHING PROCEDURE (1312)	VARIABLE	20:1	9.5 mm	1	18 HOURS
EQUILIBRIUM LEACH TEST	DISTILLED WATER	4:1	150 um	1	7 DAYS

B. NON-AGITATED EXTRACTION TESTS

TEST METHOD	LEACHING FLUID	LIQUID:SOLID RATIO	MAXIMUM PARTICLE SIZE	NUMBER OF EXTRACTIONS	TIME OF EXTRACTIONS
STATIC LEACH TEST METHOD (MATERIAL CHACTERISTIC CENTRE-1)	CAN BE SITE SPECIFIC	VOL/SURFACE 10 um	40 mm^2 SURFACE AREA	1	>7 DAYS
HIGH TEMPERATURE STATIC LEACH TEST METHOD (MATERIAL CHACTERIZATION CENTRE-2)	SAME AS ABOVE BUT AT 100°C	VOL/SURFACE 10 um	40 mm^2 SURFACE AREA	1	>7 DAYS

C. SEQUENTIAL CHEMICAL EXTRACTION TESTS

TEST METHOD	LEACHING FLUID	LIQUID:SOLID RATIO	MAXIMUM PARTICLE SIZE	NUMBER OF EXTRACTIONS	TIME OF EXTRACTIONS
SEQUENTIAL EXTRACTION TESTS	0.04 M ACETIC ACID	50 1	9.5 mm	15	24 HOURS PER EXTRACTION

TABLE 1 - EXTRACTION TESTS (continued)

D. CONCENTRATION BUILD-UP TEST

TEST METHOD	LEACHING FLUID	LIQUID:SOLID RATIO	MAXIMUM PARTICLE SIZE	NUMBER OF EXTRACTIONS	TIME OF EXTRACTIONS
SEQUENTIAL CHEMICAL EXTRACTION	FIVE LEACHING SOLUTIONS INCREASING ACIDITY	VARIES FROM 16:1 TO 40:1	150 um	5	VARIES FROM 2 TO 24 HOURS
STANDARD LEACH TEST, PROCEDURE C (UNIVERSITY OF WISCONSIN)	DI WATER SYN LANDFILL LEACHATE	10:1, 5:1 7.5:1	AS IN ENVIRONMENT	3	3 OR 14 DAYS

II. DYNAMIC TESTS (LEACHING FLUID RENEWED)

A. SERIAL BATCH (PARTICLE)

TEST METHOD	LEACHING FLUID	LIQUID:SOLID RATIO	MAXIMUM PARTICLE SIZE	NUMBER OF EXTRACTIONS	TIME OF EXTRACTIONS
MULTIPLE EXTRACTION PROCEDURE (1320)	SAME AS EP TOX, THEN WITH SYNTHETIC ACID RAIN (SULFURIC ACID: NITRIC ACID IN 60 40% MIXTURE)	20:1	9.5 mm	9 (OR MORE)	24 HOURS PER EXTRACTION
MWEP (MONOFILL WASTE EXTRACTION PROCEDURE)	DISTILLED/DEIONIZED WATER OR OTHER FOR	10:1 PER EXTRACTION SPECIFIC SITE	9.6 mm OR MONOLITH	4	18 HOURS PER EXTRACTION
GRADED SERIAL BATCH (U.S. ARMY)	DISTILLED WATER	INCREASES FROM 2:1 TO 96:1	N/A	>7	UNTIL STEADY STATI
SEQUENTIAL BATCH ASTM D4793-88	TYPE IV REAGENT WATER	20:1	AS IN ENVIRONMENT	10	18 HOURS
WASTE RESEARCH UNIT LEACH TEST (HARWELL LABORATORY, UK)	ACETIC ACID BUFFERED pH=5	1 BED VOL 5 ELUTIONS 10 BED VOL >6 ELUTIONS	CRUSHING	>11	2 TO 80 HOURS
STANDARD LEACHING TEST: CASCADE TEST SOSUV, NETHERLANDS	DISTILLED WATER HNO_3 PH 4 0	20.1	CRUSHING	5	23 HOURS

TABLE 1 - EXTRACTION TESTS (continued)

B. FLOW AROUND TESTS

TEST METHOD	LEACHING FLUID	LIQUID:SOLID RATIO	MAXIMUM PARTICLE SIZE	NUMBER OF EXTRACTIONS	TIME OF EXTRACTIONS
IAEA DYNAMIC LEACH TEST (INTERNATIONAL ATOMIC ENERGY AGENCY)	DI WATER/SITE WATER	N/A	ONE FACE PREPARED	>19	>6 MONTHS
ISO LEACH TEST (INTERNATIONAL STANDARDS ORGANI-ZATION)	DI WATER/SITE WATER	N/A	SURFACE POLISHING	>10	>100 DAYS
ANSI/ANS 16.1 (AMERICAN NATIONAL STANDARD INSTITUTE/ AMERICAN NUCLEAR SOCIETY)	DI WATER	N/A	SURFACE WASHING	11	90 DAYS
DLT	DI WATER	N/A	SURFACE WASHING	18	196 DAYS

C. FLOW THROUGH TESTS

TEST METHOD	LEACHING FLUID	LIQUID:SOLID RATIO	MAXIMUM PARTICLE SIZE	NUMBER OF EXTRACTIONS	TIME OF EXTRACTIONS
STANDARD LEACHING TEST: COLUMN TEST (SOSUV, THE NETHERLANDS)	DI WATER HNO_3 pH=4	10:1	AS IN ENVIRONMENT	7	20 DAYS
COLUMN ASTM D4874-89	TYPE IV REAGENT WATER	ONE VOID VOLUME	AS IN ENVIRONMENT	1	24 HOURS

TABLE I EXTRACTION TESTS (continued)

III. OTHER TESTS

TEST METHOD	LEACHING FLUID	LIQUID:SOLID RATIO	MAXIMUM PARTICLE SIZE	NUMBER OF EXTRACTIONS	TIME OF EXTRACTIONS
MCC-5S SOXHLET TEST (MATERIAL CHARACTERISTIC CENTER)	DI/SITE WATER	100:1	CUT AND WASHED	1	0 2 ML/MIN
ACID NEUTRALIZATION CAPACITY	HNO, SOLUTIONS OF INCREASING STRENGTH	3:1	150 um	1	48 HOURS PER EXTRACTION

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2. Private discussions with Gail Hansen, Office of Solid Waste, U S EPA

PREDICTING THE ENVIRONMENTAL IMPACT OF OILY MATERIALS: SCIENTIFIC PERSPECTIVE

Larry P. Jackson

INTRODUCTION

This paper is intended to stimulate discussion into new or better ways to evaluate the potential release of regulated substances from oily wastes. The paper discusses some options to the currently approved procedures to determine the concentrations of regulated organic chemicals released into the groundwater regime from improperly managed oily waste. The paper also describes a proposed method to evaluate the fraction of an oily waste which is flowable under the influence of gravity or overburden pressure if the material is improperly disposed in a landfill. The options presented cover, in part, some of the major technical concerns of the Environmental Engineering Committee of the Environmental Protection Agency's (EPA) Science Advisory Board (SAB) in their October, 1991 recommendations to the EPA Administrator [1]

This paper is prepared from the perspective that accurate, reliable, and cost-effective analytical procedures can be developed to properly characterize and manage potentially hazardous oily wastes. The paper accepts the premise that the regulatory community must proceed carefully and the "worst case scenario" will be considered in any proposed solutions. The paper seeks to incorporate some of the suggestions of the EPA Science Advisory Board that methods should take into consideration real world factors such as:

- source matrix properties,
- contaminant properties,
- leachant properties,
- fluid dynamics,
- chemical and physical properties of the waste,
- temporal/spatial dependence,
- measurement methods, and
- physical models.

TECHNICAL ISSUES

Neither the regulatory nor the regulated community has successfully proposed methods to properly characterize the potential for environmental impact for oily wastes. Existing leaching tests are known to be technically and mechanically deficient, and no method exists to measure the amount of flowable, oily material which may be released from a waste. Solutions to these problems have not been discussed to any extent in the published literature nor in the proceedings of symposia and workshops. These issues are recognized as the major unaddressed problems in evaluating the pollution potential of oily wastes. Any scenario which proposes to assess the pollution potential of this class of wastes must address these issues. This section describes the current state of the technology in these areas.

The Oily Waste Extraction Procedure (OWEP, EPA Method 1330A)[2] is designed to evaluate the potential for an oily waste to release metals under aqueous leaching conditions. OWEP separates the solid material from the oil by solvent extraction. The solid phase is then leached by Method 1310A, Extraction Procedure Toxicity Test[2] and the extracted oil analyzed directly for the metals of interest. The results of the analyses of the two fractions are combined mathematically. It is generally conceded that this overestimates the leaching potential of the waste. If the method is applied to the analysis of regulated organic constituents, all of the analyte will be deemed leachable which is incorrect. It should be noted that the OWEP has never been suggested as appropriate for organic constituents.

The current approach for analyzing the leaching potential of solid waste, EPA Method 1311, Toxicity Characteristic Leaching Procedure (TCLP)[2] differs from the OWEP in that TCLP attempts to determine the aqueous leachability of the waste for both inorganic and organic constituents in a single leach test. It is very difficult to conduct in a reproducible manner. Mechanical problems with the test make it time consuming to perform and frequent reanalysis is required. Precision between replicate tests is very poor. Equipment cleanup is a major obstacle to laboratory productivity. Costs can run to several thousand dollars per sample for difficult-to-handle samples. The major problems found in conducting the TCLP are:

- Handling of the sample is messy, effecting weighing of proper amounts into the extraction vessels. Loss of volatiles occurs.
- Proper sub-sampling of multi-phasic materials is difficult. Samples frequently contain oil, water, and solids. Isolation of solids for extraction is arduous.
- The tumbling action of the two liter extraction vessels forms emulsions making isolation of the aqueous leachate difficult.
- Separation of the leachate from the solid residue after extraction is frequently impossible because the oily material clogs the filter. This is especially serious when using the zero headspace extractor (ZHE) since the test must be repeated if this happens.

- Oily wastes frequently yield aqueous leachates and free organic material which must be separated and analyzed separately, doubling or tripling the analytical costs.

- Equipment cleanup is very time consuming, minor amounts of residual organic material can carry over and contaminate succeeding samples.

These problems are sufficiently severe that both regulators and regulated community have lost confidence in the utility of the method to estimate the potential environmental hazard of oily wastes.

PROPOSED APPROACHES

This section discusses four proposed approaches to improving the technical and/or procedural methods for determining the potential environmental impact of oily materials. They are:

- Adopt a flowable materials test
- Modify the TCLP.
- Adopt a new method of contacting the leach medium with the waste.
- Devise a new model for determining the amount of a regulated substance released from an oily waste by aqueous leaching mechanisms.

Approach 1 - Flowable Materials Test

The EPA has laid the groundwork for a Flowable Materials Test (FMT) in the 1991 proposed rule making for the Liquid Release Test (LRT), EPA Method 9096[3]. The Agency has published two reports describing the test for its original application, namely to detect the release of any free liquid from material destined for land disposal[4,5]. The test places a 76mm diameter by 10mm high sample in a confined chamber under a 50 psi load for ten minutes to force the release of free liquids.

If the device is modified to provide an tight fitting piston/barrel arrangement (identical to the design of the zero headspace extractor, the ZHE) and the indicator paper holder is replaced with a reinforced screen and fluid collection vessel, it will be capable of applying the necessary degree of pressure to the sample necessary to simulate overburden pressure. The screen will allow for the effective escape and collection of the flowable material from the solid mass. Both the retained solids and the collected flowable material can be analyzed separately using one or more of the suggested experimental changes described in the following sections.

Approach 2 - Modify the TCLP

Modification 1 - Addition of Inert Substrate.

One basic problem with the ability to conduct TCLP is the physical nature of oily material and the impact that has on the conduct of the test as discussed above. The EPA has addressed this type of problem in Methods 3540 and 3550[2], Soxhlet Extraction and Sonication Extraction respectively, where inert adsorbents are added to the waste to provide a free flowing material with sufficient permeability to allow for efficient extraction. The same approach can be taken with the TCLP.

The addition of a high surface area, inert matrix like silica beads (or sand) will effectively immobilize the free phase organic material and provide a free flowing medium for sample preparation (sub-sampling) and extraction. The increased surface area will promote solubilization of the organic components into the extraction medium. This approach will be effective for both free liquids and oily solids. If an aqueous phase is also present, adsorption of the oily material should facilitate separation of the aqueous material prior to extraction. The presence of the substrate surface as a host site for oily material will minimize the formation of emulsions during tumbling of the waste/leachant mixture, provided the viscosity of the organic material is sufficiently high that the shear forces of the tumbling action do not separate the liquid material from the solid. After the tumbling sequence, the solid substrate and absorbed oily material will settle to the bottom of the leaching vessel and eliminate or minimize the amount of free organic liquid floating at the surface of the solution, making the filtration step much easier and clogging less likely.

This type of sorbent bed closely resembles the real world case of oily material spilled onto or migrating through soil columns until it no longer moves under the force of gravity. This model of oil coated soil represents the most common real world source of potential pollutant release from oily wastes.

Modification 2 - Use of Fritted Stainless Steel Filter.

Regardless of whether or not the method is modified by the addition of an inert substrate to immobilize the oily material, the filtration step of the method can be improved. Agency funded research of improved filtration media led to the development of a sintered stainless steel filter that overcame many of the clogging problems[6]. The modification has not been added to the method at this time, but it has been used without formal regulatory adoption, with some intractable wastes. Other approaches to improve filtering should be examined, such as the use of thick pads (several mms) of non-woven glass or plastic fibers and powdered filter aids as pre-filters. These are physical changes to the filter apparatus; they should be permitted as long as the modifications can be shown not to alter the composition of the filtrate by absorption of analytes or allow for the passage of particles with a nominal size greater than 0.7 micron.

Approach 3 - Adopt a New Leaching Technique

The mechanical forces that act on oily waste during the TCLP tend to separate the oil from the substrate that was part of the original waste material or the inert material added in Approach 2. This leads to the formation of emulsions and/or free liquid phases which coat and clog the filters during the filtration step. Column leaching configurations that are less physically aggressive than tumbling can be used as the leaching model for oily wastes. The permeability of the waste material is the key property of the waste which must be controlled if a column technique is to work (permeability also impacts the efficiency of any extraction process). Use of inert sorbents, as in Approach 2, can provide the necessary permeability to allow for uniform flow of the aqueous medium through the waste bed and promote effective leaching. Uniform flow would be provided by pumping the leachant through the system.

Flow rates can be adjusted to minimize the shear forces which might dislodge oily material from the waste. Flow direction can be changed based on the density of the organic fluids. Downward flow for materials lighter than water and upward flow for materials heavier than water will minimize the likelihood that oily material will separate from the substrate during testing. If the fluids do separate, they will not find their way into the leachate reservoir without passing through the substrate bed where they will re-deposit on the surface. Minimum flow volumes per unit mass of waste will become the operational control of the test rather than the tumbling time that is now used.

The dynamic flow conditions of the column test also allow for efficiencies in subsequent sample analysis. Modern solid phase sorbents for both organic and inorganic analytes can be placed between the pump outlet and the head of the column to collect and pre-concentrate the analytes for future analysis. The use of these sorbents also allows the introduction of "fresh leachant" to the top of the column of waste in a manner similar to the way fresh ground or surface water would contact the waste in the real world scenario. This would promote maximum release of the target analytes.

The column leach model proposed here resembles the real world case where ground or surface water percolates through oily material that adheres to the soil, more closely than does the tumbling action of the TCLP. The column leach approach can be extended to evaluate the attenuation of solubilized materials by a representative soil, by placing a soil layer in the same extraction column as the waste or by passing the leachate through a second column placed in series with the waste containing column. This allows for the development of a modular test sequence in which the same test used to characterize a waste leachate is used as the source term for attenuation studies, which may be conducted as part of a site-specific risk assessment. This strategy is in keeping with the SAB's recommendation to the EPA.

Approach 4 - Use a Totally Different Leaching Model

The current TCLP and the two options previously discussed are alternative physical models of the leaching process. Most problems resulting from these approaches center around sample handling, leaching, and filtering the leachate. To avoid some of these

problems, the Agency should consider using well-developed, existing theoretical and experimental models of the leaching of materials from oily matrices as well and the migration and interaction of the soluble components with soils. Both the EPA and the American Petroleum Institute (API) have published significant papers on the approach[7,8,9,10,11]. There is ample experimental evidence that these models are a good first order approximation of the amounts material actually found from aqueous leaching of oily materials. They apply to both oily solids and flowable oily materials. The models depend on the amount of the target analyte present in the waste, the analytes physical/chemical properties, and the chemical properties of the soil. Some of the more important features of these models are discussed below.

The American Petroleum Institute (API) published a review of historical data relating fuel composition to the aqueous solubility of its various components[7]. The review investigated the relationship between the solubility of the pure hydrocarbon components in water and the amount found in aqueous solutions that had been allowed to equilibrate with fuels (1:10 fuel/water ratio). The study defined the partition coefficient for this process K_{fw} by equation 1:

$$K_{fw} = \frac{C_f}{C_w} \qquad (1)$$

where: C_f = concentration of the component in the fuel, g/L
C_w = concentration of the component in the water, g/L

This property is related to the solubility of the pure component in water, S, for a group of six aromatic compounds by equation 2 which has a correlation coefficient of r^2 = 0.99:

$$Log\ K_{fw} = -0.884 \log S + 0.975 \qquad (2)$$

As should be expected, the relationship between S and K_{fw} is a function of the class of organic compounds being considered (aromatic, aliphatic, olefinic, etc.). When six additional compounds, one aromatic, two olefinic, and three aliphatic, were considered, the best fit equation describing the relationship between S and K_{fw} became

$$Log\ K_{fw} = -1.018 \log S + 0.706 \qquad (3)$$

and the correlation coefficient, r^2, dropped to 0.87. Table 1 compares the experimental data for eleven of the compounds for which data was experimentally determined with the data derived from equation 3. Most of the data compare favorably with the normal range of allowable differences between replicate analytical determinations.

TABLE 1
Comparison of Observed and Estimated Hydrocarbon Concentrations from a Standard Gasoline in Equilibrium with Water (1:10)

HYDROCARBON	CONCENTRATION	
	OBSERVED	ESTIMATED
Benzene	58.7	58.8
Toluene	33.4	37.8
2-Butene	2.4	3.2
2-Pentene	2.4	1.7
Ethylbenzene	4.3	3.2
o-Xylene	6.9	4.7
m-Xylene	11.0	9.2
Butane	2.7	5.2
1,2,4-Trimethylbenzene	1.1	1.8
2-Methylbutane	3.7	6.2
Pentane	1.0	2.2

This type of model works well for those cases where the oily waste matrix is the primary determinate in the partition coefficient in the matrix/water distribution. The 1984 API report discusses the situation where the amount of oil is very small compared to the total mass of organic carbon in the soil/sediment/waste matrix and in effect represents oil absorbed on soil[6]. Equation 4 applies to these situations.

$$K_p = (K_{oc})\,(f_{oc}) \qquad (4)$$

where: K_p = soil/water partition coefficient
K_{oc} = the organic carbon partition coefficient
f_{oc} = weight percent organic carbon in the substrate.

The organic carbon partition coefficient , K_{oc} , is related to the octanol water partition coefficient, K_{ow} , by equation 5.

$$LogK_{oc} = \log K_{ow} - 0.317 \qquad (5)$$

These models are based on the physical and chemical properties of the target analytes and the substrates with which they are associated, be it a flowable liquid, solid waste, or soil. Accurate and precise methods exist for experimentally determining the input variables to the models. These variables include but are not limited to:

- analyte concentration in total waste,
- percent organic carbon in soil or waste matrix,
- partition coefficients (fuel/water, soil/water, octanol/water), and
- water solubility of target analytes.

As the data base is expanded, relationships among classes of organic compounds, water, soils, and wastes will emerge. These relationships will lead to better empirical and theoretical understanding of the physical and chemical factors controlling the release of materials to the environment. New materials can be evaluated without detailed experimental studies by analogy with similar compounds, wastes, and soils.

Use of this type of approach helps fulfill the SAB's recommendation that more rigorous scientific procedures be used to determine the potential for release as well as environmental impact. This approach also meets the recommendation that rugged tests that are less susceptible to waste matrix effects be used. The approach also uses many of the same parameters used in determining fate and transport and important measures of environmental risk; therefore, a more unified model of environmental impact can be developed.

ROLE OF THE EPA AND PUBLIC SECTOR GROUPS

The EPA can serve as a catalyst for the necessary research studies for needed to improve and develop reliable analytical methods. EPA also can lead in measuring the important physical and chemical properties, of the analytes, wastes, and soils, that define analyte behavior in the environment. Members of the public sector can contribute laboratory support and technical expertise to developing the necessary methodology and demonstrating the applicability and ruggedness of the methods.

These workshops are an ideal expression of how this should work.

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Appendix VI

Appendix VI
Office of Solid Waste Methods Section
Memoranda #35, #36

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OFFICE OF
SOLID WASTE AND EMERGENCY RESPONSE

MEMORANDUM # 36

DATE: January 12, 1993

SUBJECT: Notes on RCRA Methods and QA Activities

From: Gail Hansen, Chief
Methods Section (OS-331)

This memo addresses the following topics:

- 1992 Symposium on Waste Testing and Quality Assurance
- Issue Discussion Groups
- Inorganic Methods Workgroup Meeting
- Organic Methods Workgroup Meeting
- QA Workgroup Meeting
- Miscellaneous Methods Workgroup Meeting
- ICP Discussion Group
- HPLC Methods Discussion Group
- SPA Methods Discussion Group
- SFE Methods Discussion Group
- SW-846 Update and TCLP Spike Recovery Correction Removal Notice Update
- Total Analysis Versus TCLP.

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The instrument manufacturers are working with the Agency to determine the optimum SFE conditions for the major classes of semivolatile analytes. This input will help expedite development of a broader scope for Method 3560.

For further information on SFE topics, please contact Barry Lesnik at (202) 260-7459.

SW-846 and TCLP Spike Recovery Correction Removal Notice

The final SW-846 Update I rule and the proposed Update II rule packages are both currently at the Office of Management and Budget (OMB) review step in the regulatory process. It is not known how long this review step will take. Once the review by OMB is complete, it is expected that the promulgation of Update I and the proposal of Update II will take at least 2 months.

The rule to delete the matrix spike correction requirement from the TCLP which was finalized on June 29, 1990, has been published (57 FR 55114-56117, November 24, 1992). This rule withdraws the spike recovery correction requirements from the TCLP and, except for a few technical and format changes made in the June 29, 1990 rule revising the TCLP, returns the QA provisions of the TCLP to those promulgated on March 29, 1990 (55 FR 11796). Specifically, this rule requires the method of standard additions as the quantitation method for metallic contaminants when appropriate as specified in the method.

For further information on SW-846 updates or the TCLP rule, please give Kim Kirkland a call at (202) 260-6722.

Totals Analysis Versus TCLP

Over the past year, the Agency has received a number of questions concerning the issue of total constituent analysis with respect to the TCLP. Section 1.2 of the TCLP allows for a compositional (total) analysis in lieu of the TCLP when the constituent of concern is absent from the waste, or if present, is at such a low concentration that the appropriate regulatory level could not be exceeded. A number of persons have contacted the MICE Service and have requested clarification on this issue with respect to a number of waste testing scenarios.

Wastes that contain less than 0.5% dry solids do not require extraction. The waste, after filtration, is defined as the TCLP extract. The filtered extract is then analyzed and the resulting concentrations are compared directly to the appropriate regulatory concentration.

For wastes that are 100% solid as defined by the TCLP, the maximum theoretical leachate concentration can be calculated by dividing the total concentration of the constituent by 20. The dilution factor of 20 reflects the liquid to solid ratio employed in the extraction procedure. This value then can be compared to the appropriate regulatory concentration. If this value is below the regulatory concentration, the TCLP need not be performed. If the value is above the regulatory concentration, the waste may then be subjected to the TCLP to determine its regulatory status.

The same principal applies to wastes that are less than 100% solid (i.e., wastes that have filterable liquid). In this case however, both the liquid and solid portion of the waste are analyzed for total constituency and the results are combined to determine the maximum leachable concentration of the waste. The following equation may be used to calculate this value.

$$\frac{[A \times B] + [C \times D]}{B + [20\frac{L}{kg} \times D]} = E$$

where: A = concentration of the analyte in liquid portion of the sample (mg/L)

B = Volume of the liquid portion of the sample (L)

C = Concentration of analyte in the solid portion of the sample (mg/kg)

D = Weight of the solid portion of the sample (kg)

E = Maximum theoretical concentration in leachate (mg/L)

To illustrate this point, the following example is provided:

An analyst wishes to determine if a lead processing sludge could fail the TC for lead. The sludge is reported to have a low concentration of lead, and the analyst decides to perform a compositional analysis of the waste instead of a full TCLP evaluation. A representative sample of waste is subjected to a preliminary percent solids determination as described in the TCLP. The percent solids is found to be 75%. Thus, for each 100 grams of this waste filtered, 25 grams of liquid and 75 grams of solid are obtained. It is assumed for the purpose of this calculation that the density of the filterable liquid is equal to one. The liquid and solid portion of the sample are then analyzed for total lead. The following data are generated:

Percent solids = 75%
Concentration of lead in the liquid phase = 0.023 mg/l
Volume of filtered liquid = 0.025 L
Concentration of lead in the solid phase = 85 mg/kg (wet weight)
Weight of the solid phase = 0.075 kg.

The calculated concentration is as follows:

$$\frac{[0.023\frac{mg}{L} \times 0.025L] + [85\frac{mg}{kg} \times 0.075kg]}{0.025\ L + [20\frac{L}{kg} \times 0.075kg]} = 4.18\ \frac{mg}{L}$$

In this case, the maximum leachable concentration is below the 5 mg/l regulatory concentration for lead, and the TCLP need not be performed.

Non-aqueous based wastes (i.e., oily wastes) may be calculated in the same manner as described above, except the concentration of constituents from the liquid portion of the waste (A in the above formula) are expressed in mg/kg units. Volumes also would be converted to weight units (kg). The final leachate concentration is expressed in mg/kg units.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OFFICE OF
SOLID WASTE AND EMERGENCY RESPONSE

MEMORANDUM # 35

DATE: June 12, 1992

SUBJECT: Notes on RCRA Methods and QA Activities

From: Gail Hansen, Chief
Methods Section (OS-331)

This memo addresses the following topics:

- o 1992 Symposium on Waste Testing and Quality Assurance
- o SW-846 Update
  - Final Rule for January 23, 1989 Proposed Rule
  - Notice, Proposed Rulemaking for the Second Update to the Third Edition
- o Chlorofluorocarbon 113 (CFC-113) Solvent Replacement Update
- o Environmental Monitoring Methods Index (EMMI)
- o Sampling Work Group Formation
- o MICE Update
- o Oily Waste Analysis
- o Electronic SW-846 Availability.

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Oily Waste Analysis

One of the most frequently asked questions on the MICE Service concerns the application of the TCLP, Method 1311, to oily wastes. Many callers request technical guidance on the extraction of oily wastes due to the difficulty in the filtration on these types of waste. In many cases, an oily waste does not filter completely due to premature clogging of the glass fiber filter. This can result in the retention of standing liquid on the glass fiber filter. Material that do not pass through the glass fiber filter at the conclusion of the filtration step is defined by the method as the solid phase of the waste. The solid phase is then subjected to the leaching procedure of the TCLP. For oily wastes, clogging of the glass fiber filter can result in an overestimation of the amount of solid material available for leaching.

To solve this problem, the Agency recommends a conservative approach, one that probably will overestimate the amount of leaching. Rather than performing the TCLP extraction on the unfiltered portion of the oily waste, assume the waste is 100% liquid (e.g., will pass through the glass fiber filter) and perform a totals analysis on the oily waste to determine if the oil exceeds the appropriate regulatory level.

Filterable waste oil generated during the TCLP must be analyzed for a variety of organic and inorganic analytes. The OSW recognizes the difficulty in achieving acceptable performance for the analysis of waste oil using methods currently provided in SW-846. As a result, the Agency will provide several new methods for the preparation and analysis of oil samples to the Organic Methods Workgroup in July. In addition, a microwave assisted digestion procedure should improve the analysis of metals and will be proposed as part of the Second Update of the Third Edition of SW-846. Brief descriptions of these techniques are provided below, for additional information on the organic procedures contact Barry Lesnik at (202) 260-7459. For additional information on microwave digestion contact Ollie Fordham (202) 260-4778.

The use of purge-and-trap (Method 5030) for volatiles in oil generally results in severe contamination of analytical instrumentation. Traps, transfer lines and chromatography columns may become contaminated with oil. This leads to elevated baselines, hydrocarbon background in subsequent analyses, and cross-contamination. Headspace (Method 3810) is currently allowed only as a screening procedure in SW-846. The Agency is evaluating the use of headspace in conjunction with isotope dilution mass spectrometry for the quantitative analysis of volatiles in oil. Headspace reduces interference problems encountered with purge-and-trap. However, headspace quantitation can be questionable because the distribution of analytes is not

the same in different types of samples. That difficulty appears to be minimized by the use of isotope dilution calculations. Headspace/isotope dilution analysis will require the promulgation of two new SW-846 methods: Method 5022, Volatiles by Automated Headspace, and Method 8266, Volatiles by Isotope Dilution GC/MS. Performance data for the analysis of motor oil will be presented to the Organics Workgroup and during a platform talk at the July Symposium. Draft methods should be available for limited distribution by September.

Headspace/isotope dilution will require that laboratories acquire hardware and provide additional analyst training. Therefore, an alternate Solvent Dilution Direct Injection (Method 3585) option for Method 8260 is also being evaluated. While use of the direct injection technique will result in more instrument contamination, it may be appropriate for laboratories that analyze only a limited number of oil samples. Method performance data will also be presented for direct injection during the symposium in July.

The analysis of semi-volatile target analytes is also difficult with present methods. While gel permeation cleanup (GPC) is effective, it can only be used for small oil samples (<0.5 g). Work is in progress to evaluate partition and extraction cleanup procedures for waste oil. Partitioning oil between dimethyl formamide (DMF) and hexane or extraction of oil with methanol/DMF followed by acid/base partitioning has proved successful prior to the analysis of chlorophenols in waste oil. A similar approach is being evaluated for the analysis of organochlorine pesticides. Work to date has demonstrated that steam distillation and vapor/vapor extraction procedures are not appropriate for petroleum products.

The Agency will propose a new digestion procedure (Method 3051) for inorganic samples in the Second Update of the Third Edition of SW-846. The procedure uses a microwave oven to heat the acid during digestion of sediment, sludge, soil and oil samples. The resulting digestate can be analyzed using atomic absorption (AA) or inductively coupled plasma (ICP) methods in SW-846. Microwave assisted digestion is suitable for all oils including oils that contain particulates. The only current inorganic preparation method suitable for oils is Method 3040, a dissolution procedure. In contrast to Method 3051, Method 3040 is suitable only for metals dissolved in oil. Method 3040 can be used to show that an oil is hazardous based on the concentrations of dissolved metals.

Electronic SW-846

SW-846 now can be purchased from private vendors in an electronic format. A brief description of each known package and information on how to obtain copies of each are given below.

The SW-846 Authority is published in electronic format by Virtual Media Corporation. The SW-846 Authority is an comprehensive electronic publication designed to track and manage regulatory issues. By using IQTP (Intelligent Query Text Processor), the SW-846 Authority utilizes a comprehensive index, allowing users full text and retrieval capabilities. Users have access to thousands of EPA generated regulatory documents and official notices, including the full text of Federal Register Preambles. Other features of the SW-846 Authority include:

- RCRA Act (SWDA)
- RCRA 40 CFR Parts 260-265, 270-272
- SW-846 Solid Waste Test Methods Manual
- RCRA Inspection Manual.

For information on the SW-846 Authority call (800) 645-4130 or write to:

Virtual Media Corporation
14455 North Handen Road, Suite 201
Scottsdale, AZ 85260

Electronic E.P.A. Methods™ 1.1 is offered by Chemsoft™ Corporation as an electronic database of all EPA methods. This program is designed for rapid search and retrieval of EPA methods by method number, analyte, title, type of instrumentation, or CAS number. Each program contains the full text of the methods as they appear in the appropriate EPA manual. Use of this software requires Windows 3.0. The following programs are available either separately or may be purchased as a single package:

- EPA SW-846 Series Methods
- EPA 500 Series Methods
- EPA 600 Series Methods
- EPA Water and Waste Methods.

For further information on Electronic E.P.A. Methods, call (800) 536-0404 or (707) 864-0845 or write to:

WindowChem Software, Inc.
1955 West Texas Street, Suite 7-288
Fairfield, CA 94533-4462

The Methods Section will provide additional information in future memoranda on other sources of electronic SW-846 media when we become aware of them.

Appendix VII

Appendix VII
Recommendations and Rationale for Analysis of Contaminant Release by the Environmental Engineering Committee
Science Advisory Board
October 1991

Miller

United States Environmental Protection Agency | Science Advisory Board (A-101F) | EPA-SAB-EEC-92-003 October 1991

EPA

Leachability Phenomena

Recommendations and Rationale for Analysis of Contaminant Release by the Environmental Engineering Committee

Printed on Recycled Paper

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

EPA-SAB-EEC-92-003

October 29, 1991

OFFICE OF
THE ADMINISTRATOR

Honorable William K. Reilly
Administrator
U.S. Environmental Protection Agency
401 M Street, S.W.
Washington, D.C. 20460

Subject: Leachability: Recommendations and Rationale for Analysis of Contaminant Release

Dear Mr. Reilly:

The Leachability Subcommittee (LS) of the Science Advisory Board's Environmental Engineering Committee (EEC) has prepared the attached recommendations and rationale on leachability, an important release term related to solid wastes and contaminated soils, for your consideration.

Over the past decade, the EEC has reviewed a number of EPA issues involving leachability phenomena and noted several problems relating to this release term that were common to a variety of EPA offices. The Committee believed that these common problems would be best called to the Agency's attention through a general review of leachability phenomena.

Drafts of this report on leachability have been reviewed at a series of Subcommittee, Committee, and Executive Committee meetings over the past 18 months. This included both a session on February 26, 1990, devoted to assessing the Agency's varied needs on leachability-related information, and a Technical Workshop on May 9, 1990. The workshop assisted in determining how leachability phenomena should be used to determine how a waste will leach when present under various scenarios in the environment.

The following recommendations have been developed. First, in regard to leachability test development we recommend:

a) incorporation of research on processes affecting leachability into EPA's core research program to better define and understand principal controlling mechanisms,

b) development of a variety of contaminant release tests, rather than focusing on mimicking a single scenario,

c) development of improved release and transport-transformation models of the waste matrix to complement the leaching tests, and

d) field validation of the tests and models, and establishment of release-test accuracy and precision before tests are broadly applied.

Next, in regard to the application of such tests and models, we recommend:

e) use of a variety of contaminant release tests and test conditions which incorporate adequate understanding of the important parameters that affect leaching in order to assess the potential release of contaminants from sources of concern. A medical analogy is that no physician would diagnose on the basis of one test showing only one aspect of the problem,

f) development of a consistent, easily applied, physical, hydrologic, and geochemical representation for the phenomenon or waste management scenario of concern,

g) identification and application of appropriate environmental conditions for tests in order to evaluate long-term contaminant release potential as required under varying statutes, and

h) coordination between the Agency's programs which develop leachability tests with those that develop the environmental models in which the release terms are used.

Finally, we recommend:

i) establishment by the Agency of an inter-office, inter-disciplinary task group, including ORD to help implement these recommendations, and

j) development of an Agency-wide protocol for evaluating release scenarios, tests, procedures, and their applications.

These recommendations are made with the anticipation that an improved understanding of the fundamental scientific principles that control contaminant release and transport within a waste matrix will allow better regulatory and technical decisions to be

made in cases where the potential exists for leaching of contaminants into the environment.

We are pleased to be of service to the Agency, and hope that you will find this effort useful. We look forward to your response to the recommendations cited above.

Raymond C. Loehr

Dr. Raymond C. Loehr, Chairman
Executive Committee

Richard A. Conway

Mr. Richard A. Conway, Chairman
Environ. Engineering Committee

Calvin H. Ward

Dr. C. H. Ward, Chairman
Leachability Subcommittee

ABSTRACT

The Leachability Subcommittee (LS) of the Environmental Engineering Committee (EEC) of the EPA Science Advisory Board (SAB) conducted a self-initiated study and prepared a report on the topic of leachability phenomena. The intent of this report is to provide recommendations and rationale for analysis of contaminant release to the staff in the various offices of the Environmental Protection Agency (EPA). The nine recommendations from the report are highlighted as follows:

1) A variety of contaminant release tests and test conditions which incorporate adequate understanding of the important parameters that affect leaching should be developed and used to assess the potential release of contaminants from sources of concern.

2) Prior to developing or applying any leaching tests or models, the controlling mechanisms must be defined and understood.

3) A consistent, replicable and easily applied, physical, hydrologic, and geochemical representation should be developed for the waste management scenario of concern.

4) Leach test conditions (stresses) appropriate to the situations being evaluated should be used for assessing long-term contaminant release potential.

5) Laboratory leach tests should be field-validated, and release test accuracy and precision established before tests are broadly applied.

6) More and improved leach models should be developed and used to complement laboratory tests.

7) To facilitate the evaluation of risk implications of environmental releases, the Agency should coordinate the development of leach tests and the development of models in which the release terms are used.

8) The Agency should establish an inter-office, interdisciplinary task group, including ORD to help implement these recommendations and devise an Agency-wide protocol for evaluating release scenarios, tests, procedures, and their applications.

9) Core research on contaminant release and transport within the waste matrix is needed.

Key Words: leachability, leachability phenomena, leach tests and methods, leaching chemistry, leaching models

I. EXECUTIVE SUMMARY

In waste management, including managing the effects of spills or other releases which are sources of underground contamination, a critical issue is the assessment of the potential for constituents to leach to the environment. The Environmental Engineering Committee (EEC) of the Science Advisory Board (SAB) undertook a study of this issue because it noted several common problems relating to this release term as it reviewed, over the past decade, various leaching tests and risk models for several EPA offices. Tests such as the Extraction Procedure (EP) and the Toxicity Characteristic Leaching Procedure (TCLP) had, and continue to have, scientific limitations, yet were being inappropriately and in some cases widely used. Often tests were developed without rigorous review. A self-initiated study seemed appropriate to define the leachability problem better and to offer advice on its resolution.

The EEC established a Leachability Subcommittee (LS) that addressed:

1) Needs of the Agency and regulated communities to quantify leachability (releases) of contaminants to the environment.

2) State-of-the-art and science related to fundamental principles and practice in predicting leaching of constituents from wastes, contaminated soils, and other sources.

3) Recommendations to improve the scientific understanding and application of leaching tests.

Workshops were held, literature was analyzed, and findings were discussed over an 18-month period leading to the preparation of this report.

The various needs for tests and models to predict leaching are defined. Tests developed and used in the U.S. and Canada are summarized. The scientific considerations important in design and interpretation of leachability tests are presented. This information, expert advice and analysis by workshop participants, and reviews by SAB members, resulted in guidance which should, if progressively implemented, significantly strengthen the Agency's ability to assess appropriately leaching of contaminants from hazardous wastes, contaminated soils and other sources.

This guidance, in the form of nine recommendations, is summarized as follows:

1) A variety of contaminant release tests and test conditions which incorporate adequate understanding of the important parameters that affect leaching should be developed

and used to assess the potential release of contaminants from sources of concern.

2) Prior to developing or applying any leaching tests or models, the controlling mechanisms must be defined and understood.

3) A consistent, replicable and easily applied, physical, hydrologic, and geochemical representation should be developed for the waste management scenario of concern.

4) Leach test conditions (stresses) appropriate to the situations being evaluated should be used for assessing long-term contaminant release potential.

5) Laboratory leach tests should be field-validated, and release test accuracy and precision established before tests are broadly applied.

6) More and improved leach models should be developed and used to complement laboratory tests.

7) To facilitate the evaluation of risk implications of environmental releases, the Agency should coordinate the development of leach tests and the development of models in which the release terms are used.

8) The Agency should establish an inter-office, inter-disciplinary task group, including ORD, to help implement these recommendations and devise an Agency-wide protocol for evaluating release scenarios, tests, procedures, and their applications. The task group should also be charged with recommending what the appropriate focal point(s), responsibilities, and organizational, budgetary and communication links should be within the Agency for the most effective, continued and ongoing support and pursuit of the research, development and utilization of methods and procedures.

9) Core research on contaminant release and transport within the waste matrix is needed.

II. INTRODUCTION

In both hazardous and non-hazardous waste management, one of the most critical issues is the assessment of the potential for constituents contained in the source material to leach or otherwise be released to the environment. Approaches to estimate potential release of organic and inorganic constituents and their subsequent environmental migration and associated health risks are important in many situations (e.g., pollution prevention, risk reduction, restoration-remediation and hazard identification).

Appendix VIII

Appendix VIII
USEPA Region II
Special Analytical Services Request

U.S. ENVIRONMENTAL PROTECTION AGENCY
CLP Sample Management Office
P.O. Box 818 - Alexandria, Virginia 22313
Phone: (703) 557-2490 - (FTS) 557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

☐ Regional Transmittal ☐ Telephone Request

A. EPA Region/Client:_____

B. RSCC Representatives:_____

C. Telephone Number:___(___)_____

D. Date of Request:_____

E. Site Name:_____

Please provide below description of your recent request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in a delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. **General Description of Analytical Service Requested:**

Analysis of soil samples by TCLP for TC analytes.
Analysis of aqueous blanks for TC analytes.

2. **Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):**

Number of Samples	Matrix	Concentration	Analysis
3	Soil	Low	TCL(plus pyridene and m-cresol), TAL, 2,4-D and 2,4,5-TP by TCLP
1	Water Field Blank	Low	TCL(plus pyridene and m-cresol), TAL, 2,4-D and 2,4,5-TP

3. **Purpose of analysis (specify whether Superfund (Enforcement or Remedial Action), RCRA, NPDES, etc.):**

4. **Estimated date(s) of collection:**

5. **Estimated date(s) and method of shipment:**

6. **Number of days analysis and data required after laboratory receipt of samples:**

Environmental samples must undergo TCLP extraction within the following time periods after sample receipt:

Mercury	26 days
Other Metals	178 days
Volatiles	12 days
Pest/Herb/BNA	12 days (7 additional days from TCLP extraction to preparative extraction)

Environmental TCLP sample extracts must be analyzed within the following time periods after extraction:

Mercury	28 days
Other Metals	180 days
Volatiles	14 days
Pest/Herb/BNA	40 days

Field and trip blanks must be analyzed within the following time periods after sample receipt:

Mercury	26 days
Other Metals	6 months
Volatiles	10 days
Pest/Herb/BNA	5 days to extraction, 40 days to analysis

The complete data package containing all the sample delivery groups (SDG) associated with this case must be submitted as one data package in its entirety within 35 days from the verified time of receipt of the last sample in this case.

7. **Analytical protocol required (attach copy if other than a protocol currently used in this program):**

Parameter	Matrix	Preparation	Analysis
TCLP Metals	soil	SW-846 1311	CLP ILM03.0
TCLP VOAs	soil	SW-846 1311	CLP OLM01.8
TCLP BNAs	soil	SW-846 1311	CLP OLM01.8
TCLP Pest	soil	SW-846 1311	CLP OLM01.8
TCLP Herb	soil	SW-846 1311	SW-846 8150A
Metals	water		CLP ILM03.0
VOAs	water		CLP OLM01.8
BNAs	water		CLP OLM01.8
Pest	water		CLP OLM01.8
Herb	water		SW-846 8150A

Only the 39 TC analytes shall be reported.

Revision 1 of Method 8150A, dated November 1990, shall be used to analyze herbicides.

The July 1992 version of Method 1311 shall be used.

8. **Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):**

All Fractions

If dilutions are necessary due to an analyte being out of calibration range, they must be done in increments of 10. The raw data of all the dilutions must be provided; the final result of the analyte shall be calculated from the least dilution that would bring the analyte concentration within the calibration range.

A TCLP blank must be carried through the extraction, digestion, and analytical procedures.

The maximum number of samples in a sample delivery group (SDG) is 20.

Field blanks and trip blanks do not require MS/MSD. The matrix spike shall be added to the TCLP extract, not the environmental sample.

Metals

The CRDLs for the TC metals shall be twenty times the CRDL in the current SOW, except for mercury, which will

be two hundred times higher. The matrix spike analytes will be spiked at five times the contract specified concentrations. The TCLP Section 8.4 criteria for method of standard additions shall be followed.

Organics

Volatile and semi volatile TCL fractions will be diluted five times before analysis, which will increase the CRQLs by a factor of five. Pesticides will be diluted ten times before analysis, which will increase the CRQLs by a factor of ten. The TCL surrogates will be spiked at five times the contract specified concentrations. The TCL matrix spike analytes shall consist of all the TC analytes except toxaphene, and shall be spiked at ten times the contract specified concentrations.

When analyzing BNA samples, the 2/88 CLP extraction procedure must be used. Initial and continuing calibrations are required for pyridine and m-cresol. There are no calibration acceptance criteria for pyridine or m-cresol.

Herbicides

Follow requirements in 8150A and 8000A.

Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain of Custody Documentation, etc). If not completed, format of results will be left to program discretion.

The following TCLP deliverables shall be supplied:

1. The TCLP and preparative extraction dates and analyses dates. Data to justify selection of TCLP extraction fluid.
2. A physical description of the samples.
3. The sample weights and the extraction fluids weights.
4. The final volume of TCLP extract and the volume of extract analyzed.
5. The calculations used to compute percent dry solids and the weight of the liquid phase (if applicable).
6. Extraction logs for each sample, indicating the volume and pH of acid added. Were inorganic sample extracts properly preserved?
7. A description of the materials of construction for extraction vessels, filtration devices, and ZHE extraction devices (i.e. glass, Teflon, PVC, stainless steel etc.).
8. The calculations used to compute TCLP extract concentrations for multiphasic samples.

9. When VOA samples consist of oily waste that cannot be filtered, describe how the TCLP extract is separated from the oily waste.
10. A copy of the sampling log.
11. Any evidence of leakage in the ZHE device.

TCLP Worksheets 1, 2, 3, and 4 (attached), must be submitted with the analytical data.

A TCLP bank must be analyzed in addition to method blanks.

The following analytical results shall be submitted for Method 8150 A analysis:

The laboratory must submit all documentation including: SAS packing lists, traffic reports, chain of custody forms, and sample preparation information. Analytical and QC results shall be submitted on the following modified CLP/SOW pesticide forms: Form I (Analytical Results), Form II (Surrogates), Form III (Matrix Spikes), Form IV (Method Blank), Form VI (Initial Calibration), Form VII (Calibration Verification), Form VIII (Analytical Sequence) and Form X (Identification Summary). All QA/QC information, including laboratory generated standards and sample chromatograms, must be submitted. A written narrative describing problems encountered in receipt or during analysis and corrective actions taken (including telephone logs, etc.) must be provided. All documents (modified CLP forms, raw data, etc.) related to re-extraction/re-analysis must also be submitted in its entirety.

10. **Other (Use additional sheets or attach supplementary information, as needed):**

The following requirements apply to method 8150A:

The laboratory must supply any information required to reproduce, during independent data review, all results reported by the laboratory. The laboratory must supply a detailed example calculation that clearly demonstrates the manner in which the initial and final results were derived. Where applicable, each component of the calculation must be explained (e.g., if the calculation include a dilution factor, it must be specified how each dilution occurred).

11. **Name of sampling/shipping contact:**

_____

Phone: ( )_____

12. **Data Requirements**

For VOAs, BNAs, pesticides and metals, follow CLP criteria.

The following requirements apply to 8150A herbicide analysis:

Parameter	Detection Limit	Precision Desired
2,4-D	As per method	As per method
2,4,5-TP	8150A	8150A

Estimated Quantitation Limits (EQL) can be computed from Table 1 & 2 of method 8150A for various parameters.

13. **QC Requirements**

For VOAs, BNAs, pesticides, and metals, follow CLP criteria.

The following requirements apply to 8150A herbicide analysis:

Audits Required	**Frequency of Audits**	**Limits (% or Concentration)**
Initial Calibration	See Method 8000A	
Continuing(mid-level std) Calibration	Every 10 samples	%D ≤25%
Surrogate	All samples, etc.	50-120% Recovery
Method Blank	1 per 20 samples	<MDL
Duplicate	1 per 20 samples	≤50% RPD
Matrix Spike	1 per 20 samples	60-140% Recovery

Limits

Audits Required **Frequency of Audits** **(% or Concentration)**

For VOAs, BNAs, pesticides, and metals, follow CLP criteria. For 8150A and TCLP, follow method criteria.

14. **Action Required if Limits are Exceeded**

For VOAs, BNAs, pesticides, and metals, follow CLP protocol.

For 8150A, reextract and reanalyze.

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions, or need any assistance, please contact your Regional representative at the Sample Management Office.

TCLP Worksheet No. 1

Sample Description

Laboratory Sample No.					
Field Sample No.					
A. Sample Description					
Number of phases					
1. solid					
2. liquid					
a. lighter than water					
b. water					
c. heavier than water					
B. Percent Solid Phase					
1. weight of filter					
2. weight of subsample					
3. weight of filtrate					
4. weight percent solids (wet)[1]					
5. weight percent solids (dry)[2]					
6 volume of initial aqueous filtrate					
7. volume of initial organic filtrate					

1. The weight percent wet solids is given by the equation:

$$\frac{\textit{weight of subsample - weight of filtrate}}{\textit{weight of subsample}} \times 100$$

2. The weight percent dry solids is given by the equation:

$$\frac{\textit{(weight of dry waste - filter) - weight of filter}}{\textit{weight of subsample}} \times 100$$

Discussion and Recommendations

TCLP Worksheet No. 1

Sample Description

This worksheet documents important information regarding the general description of the sample and the number of phases observed in the sample as received from the field. This information is used to determine the amount of leaching fluid used to leach solid materials and the weighting factors used when calculating final analyte concentrations from multi-phasic samples.

A. Sample Description

Number of phases -- The number of phases present in the sample determine how the TCLP is conducted. Solid materials having no visible liquid phase are extracted as received from the field and the analyte concentration found in the leachate is the reported value. Liquid materials having no measurable solids content (< 0.5 wt. % dry solids) are defined as the TCLP extract (¶ 2.1) and are filtered and analyzed directly.

Multi-phase samples must be separated (¶ 7.1.1.2) and each phase treated individually. Aqueous phases may be combined with the leachate from solid phase materials before analysis if the two aqueous materials are compatible (¶ 7.2.13.2). If the two aqueous materials are not compatible, than each liquid must be analyzed by the appropriate methods and the results combined numerically to determine the final reported value (¶7.2.14).

A.1. Solid -- record the visible presence of a solid material heavier than water. If the sample contains more than one solid phase (example, wood chips and sediment mixed with water) record the information in the laboratory notebook.

A.2. Liquid -- record the number of liquid phases observed in the sample according to their apparent density. It may be impossible to distinguish apparent density if only one liquid phase is observed and there is no indication on the accompanying chain-of-custody form (COC). If this is the case, record it as aqueous material and let the subsequent analytical record show if the liquid is organic after the container is opened at the appropriate time.

B. Percent of Solid / Liquid Phase(s) -- paragraphs 7.1.1 through 7.1.2.3 of the method describe the procedure to follow for the determination of the percent solids of the samples. It is also convenient to measure the percent of any non-miscible liquid phases at this point because the information is required in ¶ 7.2.14.

Laboratory subsampling of the material delivered to the laboratory must be thoroughly documented. The total contents of the sample container should be considered as "the sample" and care must be taken to ensure the representativeness of any subsample. Heterogeneous and multi-phasic materials can be difficult to subsample properly and frequently require significant judgment on the part of the analyst.

Discussion -- At this point , it is important to review the COC and confirm the number of containers of each sample provided to the laboratory and the types of analyses requested. If the analysis of volatile components is requested, the determination of percent solids in multi-phasic samples must be completed before proceeding to the

leaching of the solid material in the zero headspace extractor (ZHE) to prevent overfilling the ZHE. It is best if a separate sample has been provided for this purpose (¶ 6.2). The laboratory should establish an SOP to address how to proceed if only one container is available.

It is common that when more than one container of multi-phasic materials is received from the field, each container will show different amounts of each phase. This provides a challenge to the laboratory which must report the data based on percent phase composition of the sample. A practical solution is to record the depth (measured from outside the container) of the layers in the each container after the contents have been allowed to settle and determine the combined volume of each phase in all the containers. Then measure the phase composition on a single container (after thorough mixing to obtain a representative subsample). Combine these two sets of values to determine the correct volume/mass adjustments on the TCLP results.

The laboratory should also establish an SOP on how to proceed when only a limited amount of sample is available and the analyses requested exceed the amount of sample provided.

B.1. Weight of filter -- This value must be measured before loading the filter into the filter holder because the mass of the filter is used in performing the calculation for percent dry solids.

B.2 Weight of sample aliquote -- a representative 100 gram sample (¶ 7.1.1.5) is withdrawn from the sample container for filtration. If liquid material is decanted from the sample before subsampling, its volume/weight must be recorded and factored into the calculations of percent solids.

Discussion -- Many multi-phasic samples are difficult to filter. This is especially true of oily wastes and sludges. The method directs that any material retained by the filter after following the instructions is defined as solid waste (¶ 7.1.18). Experience has shown that the reproducibility of the percent solids determination with these types of samples is highly variable. Subsequent steps in the extraction procedure (¶ 7.2.5 and 7.3.4.2) use the % solids value to estimate the mass of the original waste used to obtain an appropriate sized subsample of the solid for extraction.

The method directs that the material retained by the filter be dried at 100 ± 20 °C (¶ 7.1.2.2) to determine the percent dry solids. This may not be achievable for organic multi-phasic materials because of safety considerations and the fact that many organic liquids boil considerably higher than water and it may be impossible to achieve a constant weight for successive weighings (± 1%).

The laboratory should establish a standard operating procedure (SOP) addressing these types of samples. Basically, the laboratory has three choices of how to proceed. It may

- *attempt to dry all samples as directed by the method;*
- *dry samples containing only water as the liquid phase; and/or*
- *define the retained material as a dry solid for the purpose of further testing.*

This decision may have significant impact on the amount of material selected for leach testing and on the reported analyte values. The laboratory should consider discussing this issue with their clients and any regulatory groups to whom the data will be submitted.

B. 3 Record weight of filtrate.

B. 4 Weight percent solids(wet) equals:

$$\frac{\textit{weight of subsample} - \textit{weight of filtrate}}{\textit{weight of subsample}} \times 100$$

The procedure defines the material retained by the filter as the solid phase of the waste (¶ 7.1.1.8). This value is used to calculate the volume of the original multi-phasic material which must be filtered to yield the proper amount of solid waste for the extraction procedure.

B.5 Weight percent solids (dry) -- the total mass of the filtered solids and the filter are removed from the filtration apparatus and dried at 100 $\pm$ 20 °C until a constant weight is achieved (¶ 7.1.2.2). This value is used to calculate the dry solids content of the waste. *Use caution when drying samples that may contain flammable material.* It is important to factor in the tare weight of the filter for samples that have low solids values.

The weight percent solids (dry) is calculated by the equation:

$$\frac{(\textit{weight of dry waste} + \textit{filter}) - \textit{weight of filter}}{\textit{weight of subsample}} \times 100$$

If the weight percent dry solids is $>$ 0.5%, the total waste is defined as a solid waste and steps must be taken to collect the appropriate weight of solid material for extraction (¶ 7.1.2.4).

B.6 Volume of initial aqueous filtrate -- this value is used in ¶ 7.2.14 and 7.3.14 in the final calculation of analyte concentration.

B.7 Volume of initial organic filtrate -- this value is used in ¶ 7.2.14 and 7.3.14 in the final calculation of analyte concentration.

TCLP Worksheet No. 2

Selection of Extraction Fluid

Laboratory Sample No.					
Field Sample No.					
C. Extraction Fluid Determination -- does not apply to determination of volatile organic components					
1. particle size reduction? yes/no					
2. sample weight, ✓ if 5.0 ± 0.1 grams					
3. volume of water, ✓ if 96.5 ± 1.0 mL added					
4. initial pH (after 5 min. mixing time)					
5. if pH > 5.0, ✓ if 3.5 mL 1N HCl added					
6. ✓ if heated and held at 50 °C for ten minutes					
7. secondary pH (at room temp.)					
D. Selection of Extraction Fluid					
1. ✓ if pH from C.4 or C.7 is < 5.0, use extraction fluid No. 1.					
2. ✓ if pH from C.7 is > 5.0, use extraction fluid No. 2					

Discussion and Recommendations

TCLP Worksheet No. 2

Selection of Extraction Fluid for Metals, Semi-volatile Organic Components, and Pesticides/Herbicides

This worksheet documents the important steps which should be followed to correctly determine the appropriate extraction fluid for leaching solid wastes for the analysis of metals, semi-volatile organic components, and pesticides/herbicides. This procedure does not apply to the determination of volatiles using the zero headspace extractor (ZHE).

Discussion -- the Environmental Protection Agency's "worst case" waste disposal model assumes mismanaged wastes will be co-disposed with municipal solid waste in a 5:95 ratio. These wastes will be exposed to leaching by the acidic fluids formed in municipal landfills. The EPA's model further assumes the acid/base characteristics of the waste will be dominated by the landfill fluids. The TCLP laboratory procedure directs that alkaline wastes be extracted with a stronger acidic leach fluid than acid or neutral wastes so that the alkaline nature of the waste will not control the leaching chemistry of the TCLP test. This is in keeping with the waste disposal model's assumption that the acid fluids in the landfill will dominate leaching chemistry over time.

The procedure described in ¶ 7.1.4 of the method addresses the determination of the appropriate extraction fluid. It is a short term test whose results can have a significant impact on the final analytical results if the wrong extraction fluid is selected. This is especially true for metals determinations because of their sensitivity to the pH of the leach medium. The following discussion examines each step of the procedure and points out some sensitive technical points and how they can affect the results.

¶ 7.1.4.1 Particle size of test material -- The requirement to use 1mm particle size material in the test recognizes the fact that in a short term reaction between a liquid and a solid, high surface area is the most important characteristic of the solid. The rate of the reaction is controlled by the rate of diffusion of the liquid into the pores of the solid so a high surface area is necessary if the results of a short term test are to be reliable. Therefore, failure to take a representative subsample of the solid material and perform the necessary particle size reduction can result in significant bias. This is especially true if the waste contains a wide range of particle sizes and only the fines are selected for testing.

¶ 7.1.4.3 Heating of the reaction mixture -- The method specifies that the waste/acid slurry is to be held at 50°C for ten (10) minutes. Care should be taken to heat the sample to 50 °C as rapidly as possible without overheating. When the sample has completed the ten minute period at temperature, it should be allowed to cool and the pH determined as soon as possible. The longer the reaction between the acid solution and the solid waste is allowed to continue, the more likely that a falsely high pH reading will result. This will result in improper selection of the more acidic extraction fluid. Failure to reach and hold the required temperature can result in an artificially low pH reading for the test solution, leading the incorrect selection of the less acidic extraction fluid.

C. Extraction Fluid Determination (¶ 7.1.4)

C.1. Indicate if particle size reduction is required for the sample.

Discussion -- the laboratory should consider establishing an SOP to address the particle size reduction requirements for the TCLP procedure. Most solid samples will not be received from the field with a particle size of 1mm as required for this step of the procedure (¶ 7.1.4.1). Many multi-phasic samples will not be amenable to size reduction because of the nature of the sample. Samples containing pebbles, rocks, or debris may be difficult to size reduce if the larger particles are hard. Proper subsampling of the waste may be difficult if the waste is heterogeneous.

C.2. Sample weight -- check the box if 5.0g of sample is used in the test. Record the actual weight if a different sized sample is used.

C.3. Volume of water -- the volume of water used in the test is dependant on the weight of sample being tested. If the sample weight (above) is 5g and 96.5 mL of water is added, check the box. If the weight is not 5g, record the volume of water added. (# of grams X 19.3mL).

C.4. Initial pH -- record the pH of the slurry after a five minute mixing period. Use narrow range pH indicator paper if organic material is observed floating on the top of the slurry to avoid damage to pH electrodes.

C.5. Procedure for alkaline wastes -- if the initial pH of the slurry is > 5.0, add 3.5 mL of 1N HCl to determine if the alkalinity of the waste is sufficient to require the use of the stronger acid extraction fluid.

C.6. Neutralization reaction conditions -- the slurry should be heated to 50 °C and held for ten minutes.The laboratory should consider validating their procedure to confirm these conditions are met. A bench procedure specifying the hot plate setting (or other source of heat), the time required to reach the desired temperature, the ten minute time at temperature, and the time required to return to room temperature should be established. This will assure the maximum degree of reproducibility in the determination of the alkaline potential of the wastes tested.

C.7. Secondary pH -- record the pH of the slurry after it has completed the cooling cycle.

D. Selection of Extraction Fluid

D.1. If either the initial pH or the secondary pH is < 5.0, select Extraction Fluid #1 as the leaching medium.

D.2. If the secondary pH is >5.0, select Extraction Fluid #2 as the leaching medium.

TCLP Worksheet No. 3
Determination of Extraction Fluid Volume
for
Metals, Semi-Volatile Organic Components and Pesticides/Herbicides

Laboratory Sample No.					
Field Sample No.					
E. Determination of Sample Size for Leach Testing -- the method requires a minimum 100 gram sample size for extraction (¶ 7.2.5).					
1. particle size reduction? yes/no					
2. amount of dry solids (100g min.)					
3. amount of multi-phasic sample[1]					
a. weight of material					
b. weight of filtrate					
c. weight of solid material					
F. Determination of Amount of Extraction Fluid -- the selection of the correct extraction fluid is found in Section D, Worksheet No. 2.					
1. for dry solids (20X sample wt.)					
2. for multi-phasic samples[2]					
G. Record of Extraction Test -- the extraction period is specified as 18 ± 2 hours.					
1. extraction start time					
2. extraction stop time					
3. filtration complete time					
4. pH of filtrate					
5. volume of filtrate					

1. The theoretical amount of multi-phasic waste necessary to yield a 100g sample is given by:

$$\textit{Amount of multi-phasic material} = \frac{(10^4)}{(\textit{wt. percent wet solids})}$$

2. The amount of extraction fluid needed to extract the solid material from a filtered multi-phasic waste is given by:

$$\textit{Amount of extraction fluid} = 20\ (\textit{weight of material filtered} - \textit{weight of filtrate})$$

Discussion and Recommendations

TCLP Worksheet No. 3

TCLP Extraction Procedure for Metals, Semi-volatile Organic Components, and Pesticides/Herbicides

This worksheet documents the performance of the TCLP extraction procedure for metals, semi-volatile organic compounds and pesticides/herbicides.

E. Determination of Sample Size for Leaching -- the specified size of sample for the leaching test is a minimum of 100g (¶ 7.2.5). The regulatory control limit for defining if the waste is hazardous is based on the levels of analytes reported in the leachate based on this size sample and a twenty to one (20:1) liquid to solid ratio. If the amount of waste subjected to extraction is not 100g, than the volume of extraction fluid must be adjusted to preserve the liquid to solid ratio.

E.1. Amount of dry solids -- record the weight of dry solids.

E.2. Amount of multi-phasic sample -- the amount of multi-phasic waste material necessary to produce a 100g sample after filtration can be estimated by the equation:

$$\textit{Amount of multi-phasic material} = \frac{(10^4)}{(\textit{wt. percent wet solids})}$$

F. Determination of the Amount of Leaching Fluid

F.1. Dry solids -- for dry solids containing no filtrable fluids, the calculation of the correct volume of leaching fluid is straightforward. The amount is equal to twenty (20) times the mass of solid being leached. Note that the method specifies a 20:1 ratio based on the weight of extraction fluid required (¶ 7.2.1.1). If the laboratory elects to use extraction fluid volume, rigorous adherence to the method requires a one time specific gravity correction to convert the required weight into the appropriate volume.

F.2. Multi-phasic samples -- the method says (¶ 7.2.11) the percent wet solids can be used to calculate the weight of extraction fluid used to extract the solid waste resulting from the filtration of a known weight of multi-phasic waste. The equation for this calculation is:

$$\textit{Amount of extraction fluid} = 0.2\ (\textit{percent wet solids})\ (\textit{weight of waste filtered})$$

This assumes there is no subsampling error between the original determination of the weight percent solid phase (wet) and the subsequent selection of a weight of

the multi-phasic waste for filtration and extraction. This is frequently not so. The nature of many multi-phasic wastes and/or the necessity to use more than one sample container for the two determinations means that subsampling error can be significant. This error can be eliminated if the actual weight of filtered solids is determined at the time the material is separated for extraction. The equation for this calculation is:

Amount of extraction fluid = 20 (*weight of material filtered - weight of filtrate*)

The actual filtration procedure is detailed in ¶'s 7.2.2 though 7.2.8. Requirements for sample particle size reduction are given in ¶ 7.1.3 and 7.2.10. These should be followed as closely as the nature of the samples will allow and all departures from the instructions should be described in the laboratory notebook.

G. Record of the TCLP Extraction Test -- the period of the extraction test is given as 18 ± 2 hours (¶ 7.2.12). Extraction should be started so the resulting slurry can be filtered as soon as possible after the 18 hours has past. The filtration effectively stops the extraction process. If the extraction fluid is left in contact with the waste for longer than the specified period (overnight or over the weekend), the extraction process continues and may lead to elevated levels of contaminants.

G.1. Extraction start time -- record the time and date the extraction begins.

G.2. Extraction stop time -- record the time and date the extraction is completed.

G.3. Filtration completion time -- record the time and date the filtration is complete.

G.4. pH of filtrate -- while not required by the method, this is a good indicator of test performance when performing duplicate laboratory analysis or analyzing field replicates. It can be a reliable measure of sample heterogeneity.

G.5. Volume of filtrate -- record the total volume of filtrate collected from the sample. This value is required to make the appropriate volume corrections when reporting the results from multi-phasic wastes.

TCLP Worksheet No. 4

Zero Headspace Extraction (ZHE)

Laboratory Sample No.					
Field Sample No.					
H. Determination of Sample Size for Leach Testing – maximum 25 grams					
1. amount of dry solids					
2. amount of multi-phasic sample[1]					
I. Determination of Amount of Extraction Fluid No. 1					
1. for dry solids (20X sample wt.)					
2. for multi-phasic samples[2]					
a. weight of material					
b. weight of filtrate					
c. weight of solid material					
J. Record of ZHE Extraction Test – the extraction period is as 18 ± 2 hours (¶ 7.3.12.3).					
1. extraction start time					
2. starting pressure					
3. extraction stop time					
4. ✓ if positive pressure					
5. filtration completion time					
6. pH of filtrate					
7. volume of filtrate					

1. Determination of amount of multi-phasic sample for extraction:
   a. if weight percent dry solids is < 0.5% (from Worksheet No. 1, B. 4), the waste is filtered and the filtrate is defined as the TCLP leachate (¶ 7.3.4).
   b. if weight percent wet solids is > 5% (from Worksheet No. 1, B. 4), the amount of multi-phasic material which should be filtered to yield a 25 gram sample is given by:

$$\textit{Amount of multi-phasic material} = \frac{(2.5 \times 10^3)}{(\textit{wt. percent wet solids})}$$

2. The amount of extraction fluid #1 needed to extract the solid material from the filtered multi-phasic waste (H.2) is given by:

$$\textit{Amount of extraction fluid} = 20\ (\textit{weight of material filtered} - \textit{weight of filtrate})$$

Discussion and Recommendations

TCLP Worksheet No. 4

Zero Headspace Extraction for Determination of Volatile Organic Compounds

This worksheet describes the important information regarding the conduct of the zero headspace extraction (ZHE) of solid waste materials for volatile organic compounds.

Samples containing < 0.5 % dry solids are NOT subjected to ZHE leaching procedure. They are filtered in the ZHE device and the resulting filtrate is defined as the TCLP leachate and analyzed directly (¶ 7.3.4).

H. Determination of Sample Size for Leach Testing -- the maximum sample size for this test is limited by the volume of the ZHE to approximately 25g (¶ 7.3).

H.1. Amount of dry solids -- record the weight of dry solids charged to the ZHE but do not exceed 25g.

H.2. Amount of multi-phasic sample -- the amount of multi-phasic waste material necessary to produce a 25g sample after filtration can be estimated by the equation:

$$\textit{Amount of multi-phasic material} = \frac{(2.5 \times 10^3)}{(\textit{wt. percent wet solids})}$$

I. Determination of the Amount of Leaching Fluid #1

I.1. Dry solids -- for dry solids containing no filterable fluids, the calculation of the correct volume of leaching fluid is straightforward. The amount is equal to twenty (20) times the mass of solid being leached. Note that the method specifies a 20:1 ratio based on the weight of extraction fluid required (¶ 7.3.11). If the laboratory elects to use extraction fluid volume, rigorous adherence to the method requires a one time specific gravity correction to convert the required weight into the appropriate volume.

I.2. Multi-phasic samples -- the method indicates (¶ 7.3.11) that the percent wet solids can be used to calculate the weight of extraction fluid used to extract the solid waste resulting from the filtration of a known weight of multi-phasic waste. The equation for this calculation is:

$$\textit{Amount of extraction fluid} = \frac{20\ (\textit{percent wet solids})\ (\textit{weight of waste filtered})}{100}$$

This assumes there is no subsampling error between the original determination of the weight percent solid phase (wet) and the subsequent selection of a weight of the multi-phasic waste for filtration and extraction. This is frequently not the case. The nature of many multi-phasic wastes and/or the necessity to use more than one sample container for the two determinations means that subsampling error can be significant. This error can be eliminated if the actual weight of filtered solids is determined at the time the material is separated for extraction. The equation for this calculation is:

$$\textit{Amount of extraction fluid} = 20\ (\textit{weight of material filtered} - \textit{weight of filtrate})$$

The actual filtration procedure is detailed in ¶'s 7.3.7 though 7.3.9. Requirements for sample particle size reduction are given in ¶ 7.3.5 and 7.3.6. These should be followed as closely as the nature of the samples will allow and all departures from the instructions should be described in the laboratory notebook.

The addition of extraction fluid #1 to the ZHE is described in detail in ¶ 7.3.12.

J. Record of the ZHE Extraction Test -- the period of the extraction test is given as 18 ± 2 hours (¶ 7.3.12.3). Extraction should be started so the resulting slurry can be filtered as soon as possible after the 18 hours has past. The filtration effectively stops the extraction process. If the extraction fluid is left in contact with the waste for longer than the specified extraction period (overnight or over the weekend), the extraction process continues and may lead to elevated levels of contaminants.

J.1. Extraction start time -- record the time and date the extraction begins.

J.2. Starting pressure -- the method requires the ZHE be pressurized to approximately 10 psi at the beginning of the test.

J.3. Extraction stop time -- record the time and date the extraction is completed.

J.4. Positive final pressure -- the method requires that the ZHE retain positive pressure at the conclusion of the extraction period or the test must be repeated (¶ 7.3.13). Loss of pressure is an indication the ZHE leaked during the test resulting in a loss of volatile components.

J.5. Filtration completion time -- record the time and date the filtration is complete.

J.6. pH of filtrate -- while not required by the method, this is a good indicator of test performance when performing duplicate laboratory analysis or analyzing field replicates. It can be a reliable measure of sample heterogeneity.

J.7. Volume of filtrate -- record the total volume of filtrate collected from the sample. This value is required to make the appropriate volume corrections when reporting the results from multi-phasic wastes. The filtration of oily wastes may be especially difficult.

Appendix IX
Office of Solid Waste Methods Section
Required Uses of SW 846

The following information regarding required uses of SW-846 was compiled by the Methods Section, OSW, U.S. EPA Headquarters.

Several of the hazardous waste regulations under Subtitle C of RCRA require that specific testing methods described in SW-846 be employed for certain applications. Any reliable analytical method may be used to meet other requirements in 40 **CFR** parts 260 through 270. For the convenience of the reader, the Agency lists below a number of the sections found in 40 **CFR** parts 260 through 270 that require the use of a specific method for a particular application, or the use of appropriate SW-846 methods in general:

(1) Section 260.22(d)(1)(i) - Submission of data in support of petitions to exclude a waste produced at a particular facility (i.e., delisting petitions);

(2) Sections 261.22(a)(1) and (2) - Evaluation of waste against the corrosivity characteristic;

(3) Sections 261.24(a) - Leaching procedure for evaluation of waste against the toxicity characteristic;

(4) Sections 264.190(a), 264.314(c), 265.190(a), and 265.314(d) - Evaluation of waste to determine if free liquid is a component of the waste;

(5) Section 266.112(b)(1) - Certain analyses in support of exclusion from the definition of a hazardous waste of a residue which was derived from burning hazardous waste in boilers and industrial furnaces;

(6) Section 268.32(i) - Evaluation of a waste to determine if it is a liquid for purposes of certain land disposal prohibitions;

(7) Sections 268.40(a), 268.41(a), and 268.43(a) - Leaching procedure for evaluation of waste to determine compliance with Land Disposal treatment standards;

(8) Sections 270.19(c)(1)(iii) and (iv), and 270.62(b)(2)(i)(C) and (D) - Analysis and approximate quantification of the hazardous constituents identified in the waste prior to conducting a trial burn in support of an application for a hazardous waste incineration permit; and

(9) Sections 270.22(a)(2)(ii)(B) and 270.66(c)(2)(i) and (ii) - Analysis conducted in support of a destruction and removal efficiency (DRE) trial burn waiver for boilers and industrial furnaces burning low risk wastes, and analysis and approximate quantitation conducted for a trial burn in support of an application for a permit to burn hazardous waste in a boiler and industrial furnace.

Appendix X

Appendix X
Stabilization/Solidification: Is It Always Appropriate?
and
Stabilization/Solidification of Wastes Containing Volatile Organic Compounds in Commercial Cementitious Waste Forms

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Solidification/Stabilization: Is It Always Appropriate?

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ABSTRACT: The findings of recent research and evaluation efforts are assessed to determine whether solidification/stabilization (S/S) has been properly and appropriately applied for different types of hazardous wastes. Results from these studies are mixed and, as a result, the need for proper test procedures and for treatability studies is emphasized. Technical guidance is given for assessing the appropriateness of using S/S for any specific wastes.

KEY WORDS: solidification, stabilization, immobilization, hazardous waste, toxic waste, waste treatment, remediation, treatability studies

The increasing use of solidification/stabilization (S/S) technologies in the United States, especially for remediation of sites under the Superfund program, has raised several questions about the overall appropriateness of S/S. For many types of hazardous waste, notably for heavy metals, S/S usually gives excellent results for long-term immobilization, as measured by existing physical and chemical protocols. However, results of several studies, as well as data from remediation of several Superfund sites, have raised concerns about whether S/S is a valid technology for treating organic-bearing wastes. Even when applied to heavy metals, S/S requires careful choice of proper binders (recognizing the amphoteric behavior of certain metals) and good quality control throughout the process. Lack of good investigative procedures has diminished the value of data for evaluating S/S for some metals. Furthermore, studies also provide evidence that tests other than the regulatory extraction tests [for example, toxicity characteristic leaching procedure (TCLP)] will be required to evaluate the effectiveness of S/S, especially when applied to organic wastes. Suggestions are offered for improving treatability studies used for evaluating S/S applied to selected metals. Approaches are also provided for determining the appropriateness of S/S applied to organic contaminants. This paper will focus on evaluating chemical leaching behavior as a measure of S/S effectiveness.

Mechanisms for Containing Contaminants by Solidification/Stabilization

At present, the most prevalently used S/S technologies are cement-based or pozzolan-based. Such processes result in several chemical and physical mechanisms that combine, capture, and/or immobilize the waste contaminant. The chemical mechanisms can include chemical form change (such as the chemical change of a soluble salt of a hazardous metal to a relatively insoluble silicate, hydroxide, or carbonate form), chemical incorporation within a crystal, simple adsorption mechanisms, and similar chemical processes. The physical mechanisms involve the capture (microencapsulation) of hazardous material within the resulting physical structure of the solidified waste form. Release of physically contained contaminants is impeded by effects such as a decrease in the permeability and porosity compared to that of the "native" soil, an increase in the tortuosity of escape paths, and decreased surface area. These physical characteristics aid in eliminating or controlling water transport through the solidified product and thus the transport of the contaminant to the environment. However, if the waste exists (or can exist) in a liquid or gaseous form at ambient temperatures and pressures, then the possibility of contaminant escape in a form other than in an aqueous solution must also be considered.

Solidification/stabilization has been repeatedly demonstrated as an acceptable method to immobilize heavy metal wastes, as well as soils and sludges contaminated with heavy metals. This treatment technique is not without potential pitfalls. Technologies such as pozzolanic solidification, in which the hazardous metal is incorporated into an insoluble crystalline structure of the solid product, are probably the most reliable for long-term immobilization. Other techniques, such as lime stabilization, may not be as reliable. Careful consideration of waste characteristics versus binder mechanism is absolutely necessary when considering any S/S process.

The opportunities to capture and contain organic materials in a cement-based solidification process are limited. Most organics do not form insoluble precipitates. Moreover, most organic molecules are too large and do not readily enter into the crystal structure of most crystalline solids. Therefore, sorption processes and various physical containment mechanisms are the major mechanisms that immobilize organics. It should be noted that at least one S/S vendor claims to have a proprietary chemical that incorporates large organic molecules into a clathrate, a structural capture analogous to capture within a crystal.

Evaluations of whether a contaminant has been successfully immobilized by a particular S/S process are usually based on how much of the contaminant is removed by an extraction test. For metals, such extraction tests have been based on aqueous or weak acid extractions. Test procedures for the evaluation of the immobilization of organic materials must take into consideration the manner in which organic materials can escape. Most hazardous organic materials are only slightly soluble in water or in the weak acids used in the common regulatory extraction test procedures. Thus, these tests may measure the ability of the material to escape in an aqueous medium but not its ability to escape in its liquid or gaseous form. Many organics are not readily mobile in an aqueous medium and yet can be quite mobile in liquid or gaseous form. The U.S. Environmental Protection Agency (EPA) is sponsoring studies of test and evaluation procedures that may lead to improved technical evaluations of S/S applied to a broad range of waste types, both organic and inorganic.

S/S Applied to Toxic Metals

The chemical mechanisms for binding and incorporating hazardous constituents into S/S products are exceedingly complex. Review of data from several S/S applications reveals a lack of consistency in reporting which makes it difficult to interpret and compare results. Furthermore, many S/S technologies on the market include proprietary additives whose function can vary from useless to absolutely necessary. The EPA is supporting research to further the understanding of S/S approaches. Even without a complete understanding of S/S chemistry, it is obvious that the chemical character of the waste constituents and possible chemical interactions between the waste, the binder materials, and any interfering chemicals are of paramount importance.

For example, consider three toxic metals of common concern: arsenic, chrome, and lead.

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Arsenic under oxidizing conditions will be in the pentavalent form and will be soluble over a broad range of pH values (pH from ≃ 2 to 14). Thus, in order to stabilize arsenic chemically, it may be necessary to reduce it to the trivalent or elemental form. In this case, the pH must be maintained below 11, as As(III) becomes soluble at a pH of > 11.

Chromium is similar to arsenic in its chemical behavior. The oxidized, soluble form of chrome is the hexavalent form Cr(VI). In order to apply many of the common S/S technologies, the operator first must chemically reduce Cr(VI) to Cr(III), which is relatively insoluble over a pH range of about 5 to 13. Again, controlling final pH in the process is necessary because Cr(III) is soluble outside this range.

Lead is amphoteric, that is, it is soluble under both basic and acidic conditions. Lead is fairly insoluble at pH values between 7 and 12 (Fig. 1). At a pH below about 7, lead is soluble as Pb^{++}, while at a pH above 12, it can be soluble as anionic plumbate ion. Chemical reduction of the lead is ineffective; proper control of pH is usually more important.

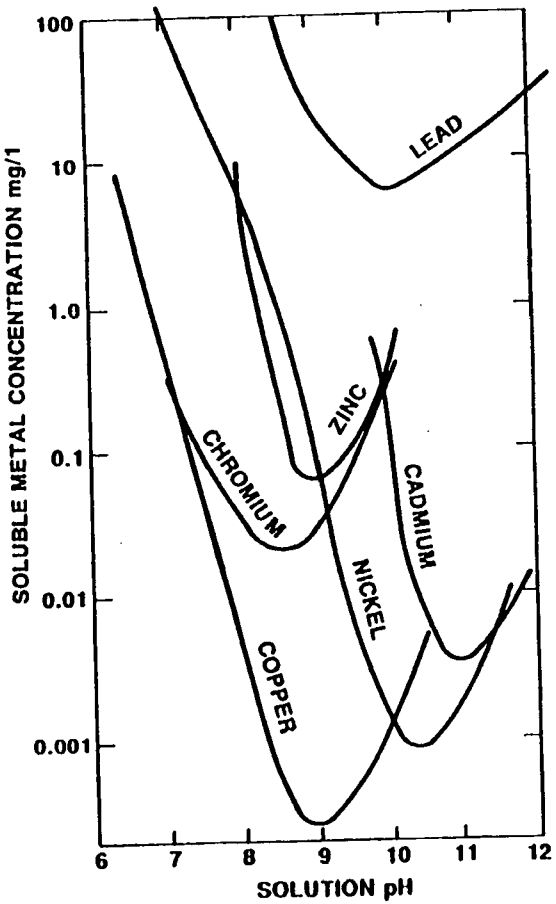

FIG. 1—*Solubilities of metal hydroxides as a function of pH.*

While these shortcomings do not preclude the use of S/S for these metals, they do emphasize the importance of performing carefully controlled treatability studies before applying specific S/S techniques to specific waste streams. Furthermore, to improve the interpretation of treatability study results, certain guidelines must be followed. Preliminary rules of thumb for assessing S/S technology applied to metals, at the bench-scale level, are as follows.

1. Characterize the original waste as well as possible. This characterization should include chemical characteristics, mineral composition, physical properties, and so on, as well as the normal analyses for pollutant levels.
2. Identify the target contaminants and tailor the binder system to low solubility regions of the metal of interest.
3. Perform appropriate extraction tests on the untreated waste to serve as a benchmark for subsequent data on the treated waste. Report extraction test data for both treated and untreated forms. Consider incorporating leaching data into a site-specific groundwater model.
4. Analyze and record pH on the extracts from the untreated waste and treated product.
5. Analyze additives for presence of hazardous constituents. Identify critical characteristics of all additives to establish a quality control program. Carefully control and record all additives during treatability tests. Small variations in additives can greatly affect leachability.
6. When reporting leachate data on treated samples, show the total percent reduction achieved and the extent to which simple dilution has contributed to such reduction.

These are not complete guidelines for performing treatability studies, merely some rules to follow that will make S/S data easier to interpret and transfer. The importance of conducting properly designed and controlled treatability studies for any waste cannot be overemphasized. The EPA is currently evaluating protocols for conducting such treatability studies.

S/S Applied to Wastes Containing Volatile Organics

It is common to distinguish organics as volatile, semivolatile, and nonvolatile. Typically, volatile organic compounds (VOC) are considered to be those having boiling points below 150°C (301°F). When one considers the operational steps in the S/S process, it is obvious that during the mixing of binder, water, and waste, as well as during the curing process, VOCs are lost because of the mixing and because of any rise in temperature. The EPA's Office of Air Quality Planning and Standards (OAQPS) is presently developing guidance for the Resource Conservation and Recovery Act (RCRA) Treatment, Storage and Disposal (TSD) facilities, indicating that solidification processes, even for very low levels of volatile constituents, must incorporate capture and control mechanisms to ensure that there is no release of volatile constituents to the atmosphere. Furthermore, since the capture mechanisms of cement-based processes depend so heavily on physical containment, it is almost a certainty that volatiles can escape from the solidified form, no matter how great the retardation introduced by reductions in pore space or increases in tortuosity. Thus, as a general rule, sites contaminated with only volatile constituents should not be considered as candidates for S/S. Sites contaminated with a mix of materials, including volatiles as well as other organics and inorganics, will require careful characterization before selecting S/S as the principal technology. Whether or not S/S should be used will depend upon the relative levels of volatiles and other constituents present. If the site contains metals or other constituents above specific levels of concern, then additional treatment by S/S may be required.

The quantities of such organics acceptable for S/S should be based on a risk assessment for the given site and/or on the result of a treatability study that includes a mass balance on the organics before, during, and after treatment. The treatability study should include a "harsh" extraction procedure (for example, total waste analysis) to evaluate the migration of organics in both treated and untreated material because many organics are not soluble in water or weak acid media. The risk assessment should assume that none of the highest risk compounds will be retained by the S/S process and/or that all such compounds will be lost via air emissions during the S/S processing, unless an air pollution control mechanism is in place. This scenario is very conservative as it does not assume any retardation of the compound that can occur by physical mechanisms in the solidified waste form. Conversely, the mass balance approach will give some credit for physical characteristics or other bonding mechanisms in the solidified waste form. Even so, this approach is also conservative in that a total waste analysis (TWA) or similar extraction procedure requires grinding of the sample. This grinding results in destroying or altering and reducing the effectiveness of some physical properties that can impede the release of the organics under actual field conditions. However, we believe these conservative approaches are necessary based upon available S/S data as applied to organics and the apparent inadequacies in the sampling, analysis, and extraction tests used to evaluate the treatment of organics at Superfund sites.

Therefore, except for the case when 100% hazardous volatile constituents are present, the amount of organics present may not be the most important factor in deciding whether or not to evaluate S/S as part of the total treatment process. The important factors are the concentration and characteristics of remaining constituents of concern that will require treatment, if all the volatiles were removed and/or destroyed. In other words, pretreatment, removal, and/or capture and treatment of volatile organic constituents will be required prior to or during S/S.

S/S Applied to Wastes Containing Semivolatile Organics

Evaluating the effectiveness of S/S processes to capture and immobilize semivolatile organics is more complicated. After mixing, the solidification process that takes place over a period of time is called "curing." During the curing process, the water present is used in hydration reactions. The final solidified waste form, especially for cement-based processes, is usually a monolithic structure incorporating hydrated silicates and carbonates in an agglomeration of crystalline structures that incorporate and/or microencapsulate the hazardous constituents, the original soils, and any aggregate that may have been added to enhance the product's strength. Solidification processes are normally exothermic because of the hydration reactions. Typically, the heat given off is sufficient to raise the temperature of the matrix by 30 to 40°C in the early stages of curing. In cases where kiln dust or other lime-based products (that is, those containing large amounts of quicklime (CaO)) are used, temperature rises can be significantly higher yet. Thus, it is possible that some of the low-boiling semivolatile compounds volatilize and escape during the curing process.

Once curing is complete, the effectiveness of cement-based solidification processes to contain semivolatile organic compounds is currently unclear. The application of the TCLP test to organically contaminated soils before and after treatment with cement-based solidification processes in the Superfund Innovative Technology Evaluation (SITE) program has yielded inconclusive results. This situation is due largely to the fact that the solubility of the organics in the extraction fluid is so low that even small quantities of these organic contaminants quickly saturate the TCLP liquid. Thus, no measurable difference between the leachability of organic contaminants from untreated soils and from the solidified soil product can be determined. [illegible] adequate measure of the probable mobility of these

contaminants in aqueous-phase leaching processes, it does not address the potential escape of organics in liquid or gaseous form. Since the liquid and gaseous forms of these organics can flow, it is not unreasonable to expect that escape will occur.

Thus, the same basic approach is probably necessary for semivolatile organics, as is suggested for volatile organics. If the level of nonvolatile and/or inorganic constituents is present above some agreed-upon acceptable threshold, then S/S processes will probably be required as part of a complete treatment train. Stabilization/solidification would follow some earlier stage of treatment for the removal and/or destruction of the volatile and semivolatile constituents. The levels of residual semivolatile compounds remaining in the waste prior to a solidification process must be determined based on maximum concentration limits allowable for each such constituent. Note that in this context, S/S technologies should be deemed inappropriate for sites contaminated only with volatile and/or semivolatile organic compounds, unless some specialized S/S process can be demonstrated to be effective. At present, such demonstration should be based on a TWA and should include a mass balance for the waste constituents. As stated earlier, protocols for evaluating S/S applied to organics are presently being evaluated by EPA.

Discussion of Extraction Tests

Extraction tests are used to determine or estimate the levels of targeted contaminants that can be extracted from waste under selected conditions. None of the presently existing leach tests were developed for, nor validated for use in, assessing the leachability of organic compounds. Table 1 lists several leach tests and some comments on their applicability for organic contaminants. Technically, the TCLP and similar extraction test methods are not adequate for identifying all classes of organics that might be available for escape to the environment. The harsh extractions, such as total waste analysis, do not represent realistic field conditions nor do they recognize immobilization due to the physical characteristics of the solidified waste form. What is needed is a leaching method sensitive to all phases of organics that are not adequately bound in the solidified matrix. This test also needs to represent realistic field conditions and be relatively easy to perform. Unfortunately, no such test currently exists. However, EPA is investigating potential test methods for this purpose.

Caldwell, Côté, and Chao [*1*] have developed leaching procedures based on sequential batch leaching, with the development of a partition coefficient between the solid and liquid phases. The technique appears to work well for low levels of organic contaminants (up to 1000 μg per gram of solidified waste), but does not fully address the concern about escape in a nonaqueous medium. Through EPA support, the U.S. Army Corps of Engineers, Waterways Experiment Station and others have performed several studies aimed at evaluating different leach procedures for use in solidification of organic contaminated soils and sediments [*2–4*]. In general, this work indicates that a sequential batch extraction process gives a better measure of the total leachable quantity of contaminants present than do the single-exposure tests.

Summary of Selected Results for Organic Wastes

Several studies have been performed that strongly indicate the inadvisability of using S/S as the principal remediation technology for organic wastes. Kolvites and Bishop [*5*] conducted column leaching tests on cement-based solidified samples containing known amounts of phenol and trichloroethylene (TCE). So much of the TCE was lost in preparation and mixing that the results were inconclusive. However, the results for phenol give a good measure of the amount of this semivolatile organic compound that is retained in the solidified product. Table 2 gives the leach test results on a sample that was allowed to cure for three days and Table 3

TABLE 1—*Some extraction tests used for evaluating stabilization/solidification of organic-contaminated waste*

Extraction Tests	Peer Reviewed for Organic Stabilization	Utilized for Evaluating Organic Stabilization	Comments
TCLP	No	Several Superfund Sites	Grinding of monolith, Codisposal scenario only may not show organics Most organics not soluble in leachate
TCLP Cage	No	ASTM Committee D-34	No grinding Erosion caused by cage does not represent "real world" Poor reproducibility noted by ASTM Most organics not soluble in leachate
TCLP Acid Rain	No	Limited number of Superfund sites	Used synthetic acid rain with TCLP apparatus
TCLP Acetone	No	Limited number of Superfund sites	Acetone to extract PCBs not realistic Conservative worst case does not represent "real world"
MEP	No	Delisting (metals)	Somewhat realistic Simulates acid rain
Total Waste Analysis (TWA)	No	R&D	Methylene chloride or hexane Conservative worst case, does not represent "real world"
ANSI 16.1	No	Several Superfund sites	May not show organics
ANSI 16.1 plus groundwater	No	Limited number of Superfund sites	May not show organics
Acid Rain (1312)	No	Unknown	May not be representative of real acid rain May not be applicable for organics

for a sample that was allowed to cure 28 days before leach testing. These tables show a complete mass balance indicating the amount of phenol retained in the solidified product. The enhanced retention in the 28-day cured product is probably due to a decrease in pore size in the product as curing proceeds. Twenty-eight days is a typical curing time for cement-based solidification processes. Even so, only about 40% of the phenol is retained in the solidified product.

Bricka, Holmes, and Cullinane [6] of the U.S. Army Corps of Engineers, Waterways Experiment Station, in a study performed for EPA, showed a comparison of the EP and TCLP leaching extracts for two solidified sludges and one nonsolidified, organic-based sludge (Table 4). A metal hydroxide sludge (identified as WES) was stabilized by addition of cement. A second material (identified as WTC) was a metal solution containing chromium chloride, cadmium nitrate, l[illegible]itrate, sodium arsenite, and phenol at a pH of 2.5. This synthetic metal solution was sol[illegible]y adding portland cement, Type F fly ash, and soil in equal quantities to the [illegible]. The third material, a PCE sludge, was a still-bottom waste resulting from the

TABLE 2—*Mass balance leach test No. 1* [5]

Leaching Period	Phenol Released, mg	Leachant Volume, mL
Days 1 and 2: leachate	27.42	245
vapor	0.00	
Days 3 and 4: leachate	6.13	339
vapor	0.00	
Days 5 and 6: leachate	2.54	385
vapor	0.00	
Days 7 and 8: leachate	1.04	377
vapor	0.00	
Total phenol leached from 30 g of cement product	37.13	
Phenol adsorbed to liner of cement mold	0.05	
Phenol mixed into 30 g of cement product	40.00	
Phenol retained in cement product (calculated by difference)	2.74	

distillation of "dirty" perchloroethylene for solvent recovery. Each of these wastes was spiked with 1000 to 10 000 mg/L of twelve organic compounds and then treated. The treated materials were then subjected to the two extraction procedures and their leachate analyzed. The data presented in Table 4 show that, except for the polar compounds butanone and 4-methylpentanone, less than 3% of the spiking contaminant was recovered in either extract, even from the nonsolidified PCE organic-based sludge. This result clearly indicates that aqueous-phase leach tests are not truly measuring the potential for escape of low-solubility organic compounds.

In an attempt to enhance the capture and retention of organic materials in cement-based solidified waste, Sheriff et al. [7] of the Imperial College of London added activated charcoal and various clays to specific organic waste streams prior to cement-based solidification. Figure 2 is an example of the results of mixing 40 g of cement with 1 g of charcoal and 40 mg of phenol and two chlorinated phenols. The controls indicated on the graph are the same concentration of organic contaminants in cement alone with no charcoal added.

In a study evaluating the loss of volatile organic material during solidification, Weitzman et al. [8] presented several graphs showing the loss of specific volatile organic compounds during mixing and curing. Figure 3 is typical of their results and shows the results for two solidi-

TABLE 3—*Mass balance leach test No. 2* [5]

Leaching Period	Phenol Released, mg	Leachant Volume, mL
Days 1 and 2: leachate	13.54	245
vapor	0.00	
Days 3 and 4: leachate	5.08	228
vapor	0.00	
Days 5 and 6: leachate	3.08	219
vapor	0.00	
Days 7 and 8: leachate	1.60	192
vapor	0.00	
Total phenol leached from 30 g of cement product	23.30	
Phenol adsorbed to liner of cement mold	0.02	
[illegible]enol mixed into 30 g of cement product	40.00	
[illegible]ol retained in cement product (calculated by difference)	16.68	

TABLE 4—*Study B average toxicity characteristic leaching procedure (TCLP) and extraction procedure (EP) extract concentration for the organic spike contaminants (averaged over the three replicate extract samples)* [6])

Sludge	Level of Spike Added, mg/L	Extract Concentration, mg/L		Organic Recovery, %	
		EP	TCLP	EP	TCLP
		Chloroform			
WES	1 000	0 88	1 40	0 088	0 140
	10 000	13 97	27 27	0.140	0 273
PCE	1 000	1 01	1 56	0 101	0 156
	10 000	23 77	32 70	0.238	0 327
WTC	1 000	0 22	0 20	0 022	0 020
	10 000	8 98	9 13	0 090	0 091
		1,2-Dichloroethane			
WES	1 000	1 57	1 27	0 157	0 127
	10 000	38 70	61 37	0 387	0 614
PCE	1 000	3 61	4 23	0 361	0 423
	10 000	57.30	71 40	0 573	0 714
WTC	1 000	0 76	0 49	0 076	0 049
	10 000	45 03	44 23	0 450	0 442
		1,1,1-Trichloroethanl			
WES	1 000	0 96	1 93	0 096	0 193
	10 000	18 33	46 80	0 183	0 468
PCE	1 000	0 55	4 80	0 055	0 480
	10 000	15 07	25 07	0 151	0 251
WTC	1 000	0 29	0 45	0 029	0 045
	10 000	15 07	24.83	0 151	0 248
		Carbon Tetrachloride			
WES	1 000	0 42	0 89	0 042	0 089
	10 000	3 93	7 60	0 039	0 076
PCE	1 000	0 23	0 50	0 023	0 050
	10 000	10 00	10 00	0 100	0 100
WTC	1 000	0 10	0 20	0 010	0 020
	10 000	5 00	5 00	0 050	0 050
		Trichloroethene			
WES	1 000	3 47	6 90	0 347	0 690
	10 000	64 63	134 33	0 646	1 343
PCE	1 000	1 48	3 54	0 148	0 354
	10 000	33 73	39 97	0 337	0 400
WTC	1 000	2.32	2 55	0 232	0 255
	10 000	98 07	135 67	0 981	1 357
		Benzene			
WES	1 000	1 60	2 30	0 160	0 230
	10 000	42 97	85 33	0 430	0 853
PCE	1 000	2 62	5 29	0 262	0 529
	10 000	54 17	76 57	0 542	0 766
WTC	1 000	0 91	0 79	0 091	0 079
	10 000	55 23	62 40	0 552	0 624
		1,1,2,2-Trichloroethanl			
WES	1 000	0 25	0 22	0 025	0 022
	10 000	1 00	5 00	0 010	0 050
	1 000	7 31	9 04	0 731	0 904

TABLE 4—*Continued*

Sludge	Level of Spike Added, mg/L	Extract Concentration, mg/L		Organic Recovery, %	
		EP	TCLP	EP	TCLP
WTC	1 000	0 10	0 20	0 010	0 020
	10 000	, 5 00	5 00	0 050	0 050
		Tetrachloroethene			
WES	1 000	3 10	7 00	0 310	0 700
	10 000	25 97	38 67	0 260	0 387
PCE	1 000	3 03	3 19	0 303	0 319
	10 000	28 30	13 37	0 283	0 134
WTC	1 000	1 00	1 60	0 100	0 160
		18 87	39 87	0 189	0 399
		Toluene			
WES	1 000	3.03	4 43	0 303	0 443
	10 000	55 43	93 67	0 554	0 937
PCE	1 000	1 37	2 50	0 137	0 250
		36 67	35 77	0 367	0 358
WTC	1 000	1.24	1 39	0 124	0 139
	10 000	65 67	89 57	0 657	0 896
		Ethylbenzene			
WES	1 000	5 27	17 33	0 527	1 733
	10 000	33 83	47 33	0 338	0 473
PCE	1 000	2 03	2 33	0 203	0 233
	10 000	34 53	20.93	0 345	0 209
WTC	1 000	2 93	3 94	0 293	0 394
	10 000	36 10	95 60	0 361	0 956
		Butanone			
WES	1 000	35 80	17 00	3 580	1 700
	10 000	188 00	256 67	1 880	2 567
PCE	1 000	5 19	5 39	0 519	0 539
	10 000	133 33	134 33	1 333	1 343
WTC	1 000	9 59	6 29	0 959	0 629
	10 000	163 00	165 67	1 630	1 657
		4-Methyl-2-Pentanone			
WES	1 000	41 33	13 33	4 133	1 333
	10 000	192 67	313 33	1 927	3 133
PCE	1 000	11 63	10 63	1 163	1 063
	10 000	233 00	247 00	2 330	2 470
WTC	1 000	7 67	4 88	0 767	0 488
		298 00	306 00	2 980	3 060

fication processes, one based on portland cement and fly ash, the other on lime kiln dust and fly ash. Both are compared with a blank wherein the organic materials were mixed with non-reactive solid material to simulate the solidification mixing process. This work and additional field tests are being used by EPA to develop regulations and guidance on the control of VOC emissions during S/S treatment processes used at RCRA TSD facilities.

Recently, research with modified clay has shown promise for a strong bonding between semivolatile compounds and organophilic clay material [9]. The extraction data presented in Table 5 and bonding evaluation techniques indicated strong bonding.

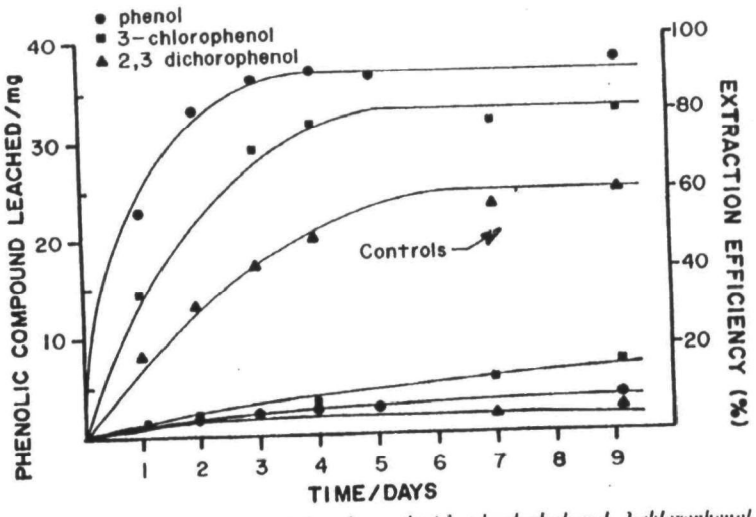

FIG. 2—*Leach tests on cement containing charcoal with adsorbed phenol, 3-chlorophenol, and 2,3-dichlorophenol.*

Recommendations for Determining Appropriateness of S/S for Organics

The following recommendations are based upon current knowledge about using S/S for treating organics. Generally, S/S should not be used to treat a site containing only organic waste. Alternative technologies (for example, incineration, steam stripping, vacuum extraction, and so on) should be used to remove and/or destroy the organics. If residues remain after this primary treatment that still require additional treatment, S/S can be effectively used. However, a well-designed and controlled treatability study should be conducted to assess S/S effectiveness and to select and design a proper S/S process.

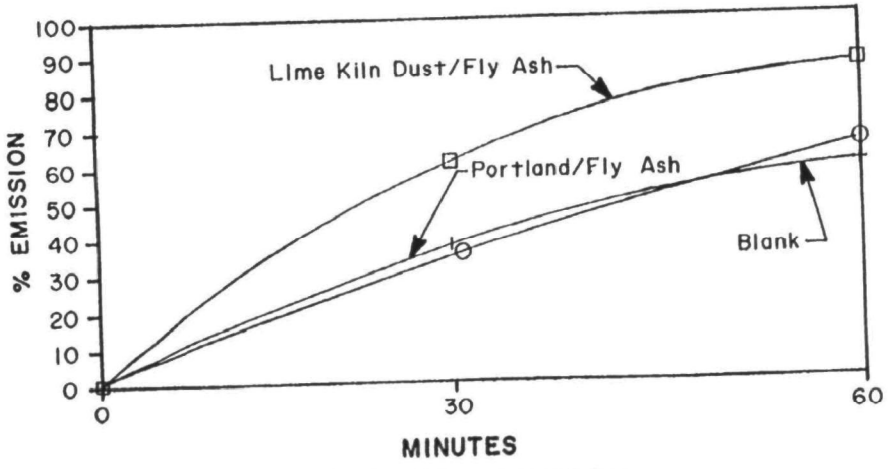

FIG. 3—*Cumulative acetone emissions.*

TABLE 5—*Extraction data upon waste stabilized with organophilic clays.*

Compound	Raw Waste Mean Concentration, μg/kg	Treated Waste Mean Concentration, μg/kg
Bis-(2-chloroisopropyl) ether	8 528	ND[a]
Naphthalene	18 060	1445[b]
Phenanthrene	20 184	ND
Benzo (α) anthracene	30 460	ND

[a] ND = not detected.
[b] Value corrected for dilution.

On strictly technical considerations, there may be exceptions to this general approach. For example, if the organic is generally not mobile through air, soil, and water [for example, polychlorinated biphenyls (PCBs)] then some approaches to S/S may be acceptable cost-effective treatment alternatives for a given site, depending on the remediation goals. Careful attention must be paid to any existing state and federal environmental regulations concerning the particular organic contaminant (for example, dioxins, and so on). Treatability studies must be performed that incorporate appropriate test methods to evaluate the organic waste's potential for escape.

Based on existing data, volatile organic compounds cannot be immobilized by current S/S technology. Whether or not a site containing VOCs can be treated by S/S will depend upon specific conditions existing at the site. Available data also indicate that semivolatile organic compounds cannot be effectively treated by current S/S techniques. Whether or not a site containing semivolatile organics, along with other wastes, should be treated using S/S also depends upon site-specific factors.

Figure 4 provides a general decision tree for determining if S/S should be considered for treating a site containing organics. The approach is as follows:

1. Determine the amount of organics (relative to inorganic contaminants and other materials) and their chemical and physical characteristics. Determine whether the organics of concern are above acceptable levels (for example, above regulatory limits or above levels causing unacceptable risks to exposed populations).
2. Based upon this information and other site characterization data, further determine what inorganic compounds, materials, and so on, would remain if all the organics were destroyed or removed.
3. Determine the concentrations and the chemical and physical characteristics of the residuals remaining if organics were removed/destroyed.
4. If the site contains only organics, do not consider S/S unless the organics are not mobile (in air, water, or soil) and do not require special handling/disposal/treatment because of special regulations (for example, TOSCA, and so on) or the organics are below some predetermined acceptable level based upon a risk assessment.

   a. If organics are not mobile and do not require special treatment and management because of environmental laws, unacceptable risks, or other special considerations, consider S/S.
   b. If organics are volatile or semivolatile, are hazardous and are above levels of concern, then they must be destroyed and/or removed prior to S/S. However, depending upon the amounts of volatile and/or semivolatile organics present, it may be feasible to collect and treat the organics as part of the S/S treatment train.

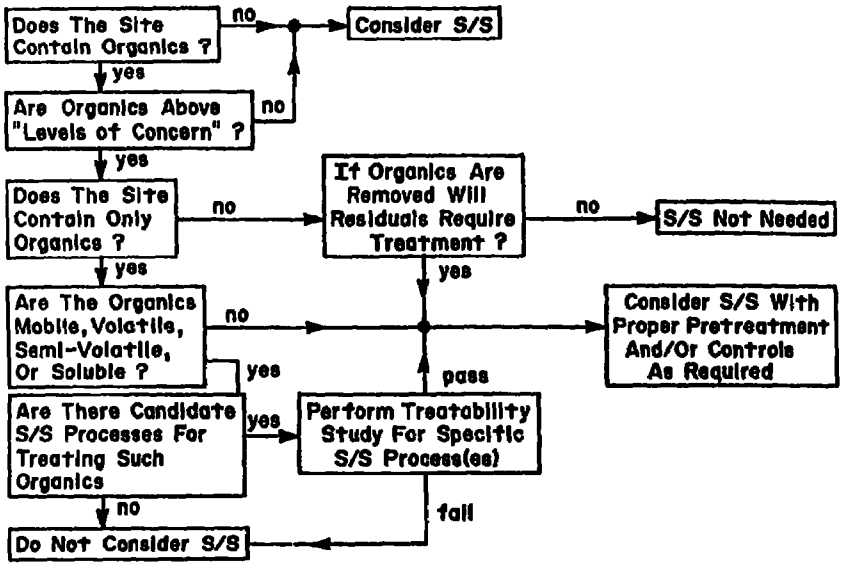

FIG 4—*General decision tree for S/S applied to organics*

c. If organics are mobile and/or volatile but not hazardous (which is unlikely at Superfund sites) then S/S is a potentially feasible treatment

Determining Levels of Volatile/Semivolatile Organics Acceptable to S/S

There are two approaches for determining the maximum amounts of volatile/semivolatile organics acceptable for S/S as the whole treatment or as part of a treatment train. One is based on results of risk assessment and an assumption that S/S will not provide treatment. The second is based on results of a treatability study and a comparison of organic levels in the untreated waste with those in the solidified waste using a total waste analysis or similar tests. The first approach, based on risk assessment, is as follows:

1. Determine the concentration of the organics present in the waste to be treated
2. Identify the compound that poses the highest health or environmental risk (quantity and toxicity)
3. Determine a threshold concentration of the highest risk compound below which it will not pose a health or environmental risk at the given site
4. Assume that
   a. The S/S process will not treat or contain the selected compound and therefore all of it will be released from the solidified waste, and/or
   b. That all of the compound will be released as air emissions during the S/S process
5. If these concentrations are above those levels determined to pose a health/environmental risk at the given site, then pretreatment to remove or destroy the organics will be required and/or air emission controls and treatment will be needed on the S/S treatment train

Regulatory limits have not yet been established but could be based on some multiples of the drinking water standards, on limits being established by EPA for air emission controls, or on some other standard.

This approach is very conservative and does not account for the retardation of the release of organics caused by the physical characteristics of the solidified waste form. A second approach, based on "harsh" extraction test concepts, can be envisioned as follows:

1. Use a total waste analysis, methylene chloride, or similar harsh extraction procedure to determine the organic content of the untreated waste
2. Perform the S/S treatability study, but ensure that proper controls are used to collect all organic air emissions. Continue collection of air emissions during the curing phase.
3. Perform extraction/leaching tests on solidified waste forms after curing. Collect organic air emissions
4. By comparing the organic levels in the untreated waste, the collected air emissions, the solidified waste form, and in the extract from the extractions test, one can determine if acceptable treatment has been achieved and also whether VOC emission controls will be necessary during full-scale application of the S/S process
5. Compare the levels of organics in the extract from the solidified waste form to some predetermined level (for example, 100 times drinking water standards) established as acceptable for the site. If the levels are acceptable, then S/S can be used. However, one must also determine if controls are necessary to collect and treat VOC emissions during the S/S process.

This second approach is also conservative, but will give some credit to any S/S processes which claim to be able to treat organics. Because of the grinding required for the harsh extraction tests, this approach provides little credit for physical retardation of releases of the organics that may result from the improved physical properties of the solidified waste form.

Summary

This paper discussed some approaches for determining whether or not organic contaminated soils should be treated by S/S technologies. The approaches are conservative and give little recognition to the physical characteristics of the solidified waste forms in the immobilization process. These approaches are also based upon technical rather than regulatory considerations after reviewing available information on the S/S of organic wastes. Several instances have been reported where processors have claimed treatment of organics by S/S. In most, if not all of these, the experimental approach was too limited. Measuring organic content before treatment and after treatment without controls to collect and analyze air emissions is not acceptable. Many, if not all, of the volatile and semivolatile organics will "disappear" during the process because they volatilize. Much more sound scientific evidence is required before S/S of organic contaminated waste can become routine practice. The EPA is addressing this issue as well as the development of reasonably quick and inexpensive protocols for conducting S/S treatability studies involving organics.

Also presented were some general rules for conducting treatability studies for metal-bearing wastes. The need for this has become evident in reviewing many studies, including data from the conduct of treatability studies at Superfund sites. The purpose of these rules is to improve the quality and applicability of the data.

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Guidelines for Evaluation of the Permanence of a Stabilization/Solidification Technology

REFERENCE: Soundararajan, R., "Guidelines for Evaluation of the Permanence of a Stabilization/Solidification Technology," *Stabilization and Solidification of Hazardous, Radioactive, and Mixed Wastes, 2nd Volume, ASTM STP 1123*, T. M. Gilliam and C. C. Wiles, Eds., American Society for Testing and Materials, Philadelphia, 1992, pp 33–39

ABSTRACT: Stabilization/solidification technologies are used widely for treating both inorganic and organic waste materials Unfortunately, some of these processes have turned out to be simple adsorption/dilution phenomena. For a true stabilization/solidification process, the binder and the waste must interact chemically to create chemical bonding Based on our past investigations in this area, some guidelines for evaluating the permanence of a stabilization/solidification process are presented in this paper

KEY WORDS: stabilization, solidification, adsorption, dilution, chemical bonding, Fourier transform infrared spectra (FTIR), thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), bond energy, heat of solution

Stabilization/solidification (S/S) technologies are emerging as viable alternatives for other waste management processes. However, there have been a number of questions raised about the technology's effectiveness and permanence by the Office of Technology Assessment. Several reports critical of the "science" involved in this process have been published in the recent past It becomes imperative to take a hard look at the technology from a purely scientific standpoint and to gather evidence about the permanence of this process in order to preserve the integrity of the technology In the past, we have conducted a series of investigations on various aspects of S/S technology. Based on our findings, we present a few protocols that would assess the permanence of an S/S process. Most of the research work presented in this paper was funded by the Office of Research and Development of the U.S. Environmental Protection Agency (EPA), Cincinnati, OH

Discussion

It is worth mentioning that all S/S technologies are governed by the fundamental laws of chemistry. A thorough understanding of the chemistry of the waste to be stabilized results in a better stabilization process In general, the S/S process of inorganics and organics may be explained as follows: In the inorganic S/S process, the metallic ion to be fixed is converted into its most insoluble form and then placed inside a cementitious matrix [*1*].

In the case of stabilization of lead, for example, the following facts were considered. The solubility product of lead hydroxide [$Pb(OH)_2$] is known to be 1.2×10^{-15}. We need this infor-

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Stabilization/Solidification of Wastes Containing Volatile Organic Compounds in Commercial Cementitious Waste Forms

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ABSTRACT: Stabilization/solidification (S/S) is one of the most widely used techniques for waste treatment and remedial actions, but does not have regulatory approval for treating organics. Application with volatile organic compounds (VOCs) is particularly controversial since it was believed that the necessary mechanical mixing and exothermic cementitious reactions would vaporize the VOCs. The objective of this study was to establish whether S/S is a viable alternative for a sludge heavily contaminated (about 1%) with relatively immobile metals, but lightly contaminated (<0.04%) with VOCs that were contaminating the groundwater. The mass balance indicated that >50% of the VOCs were retained in the laboratory preparation of cementitious samples cured for 28 days. The performance tests indicated the commercial products could attain leachability indices from 7 to >9 for the eight VOCs studied and distribution coefficients of >10 could be attained for all eight and >100 for some compounds.

KEY WORDS: stabilization, solidification, VOCs, organic, immobilization, cement, grout, waste form, trichloroethene, acetone, methyl ethyl ketone, 1,2-dichloroethene, chloroform, benzene, chlorobenzene, perchloroethene

Stabilization/solidification (S/S) is one of the most widely used techniques for the treatment and ultimate disposal of both radioactive and chemically hazardous wastes because of low processing costs, compatibility with a variety of disposal scenarios, and ability to meet stringent processing and performance requirements. S/S is accepted as the best demonstrated available technology (BDAT) for many applications involving metals contamination, but not involving organic contamination [*1,2*]. The sludge used in this study was contaminated (see Table 1) mainly with metals, to a lesser extent with semivolatile organics, and to an even lesser extent volatile organic compounds (VOCs). Only the VOCs were observed to be migrating in the groundwater at the site from which the sludge was excavated; hence, VOCs were the focal point of the study. In situ S/S is an attractive remedial action alternative in this case because incineration will result in little or no volume reduction for this sludge, incineration may result in more mobile metallic species, excavation may result in evaporation of most of the VOCs, and in situ S/S may achieve the environmental protection desired with the least disturbance to the site and environment and the maximum protection to operating personnel and residents. To pursue this alternative, the sponsor needed to demonstrate the immobilization potential of the

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TABLE 1—*Measured concentrations in the unspiked sludge (Note: Some volatiles were lost in the process of excavating and homogenizing the large sample. The table values reflect what was present in the laboratory prior to spiking.)*[a]

Compound	Concentration, mg/kg	Site Maximum
VOLATILE ORGANIC COMPOUNDS (VOC)		
Acetone	1 7	9
1,2-DCE	0 02	100
Chloroform	1 7	17
MEK	5 6	4
TCE	15	130
Benzene	0 55	3
PERC	7.5	59
Chlorobenzene	3 9	20
BASE/NEUTRAL/ACID ORGANIC COMPOUNDS (BNA)		
Phenol	3 7	
1,3-Dichlorobenzene	130	
1,4-Dichlorobenzene	120	
1,2-Dichlorobenzene	<6 1	
2,4-Dimethylphenol	110	
1,2,4-Trichlorobenzene	100	
2-Methylnaphthalene	190	
Di-n-butylphthalate	160	
Bis(2-ethylhexyl)phthalate	190	
METALS		
Barium	140	
Cadmium	560	
Chromium	6 200	
Lead	760	
Silver	4 7	
Nickel	210	
Aluminum	28 000	
Calcium	3 000	
Iron	82 000	
Phosphorus	5 400	
Silicon	1 500	

[a] A 46 4 wt% loss was observed upon drying the unspiked sludge

commercially available waste forms. Fortunately, recent investigations have indicated true bonding between organic wastes and some binders as required in the Environmental Protection Agency's (EPA) interpretation of the Hazardous and Solid Waste Amendments (HSWA) to the Resource Conservation and Recovery Act (RCRA) [*1,3,4*].

This paper presents the results of a study done at Oak Ridge National Laboratory (ORNL) on the VOC immobilization potential of commercial cementitious waste forms through the Hazardous Waste Remedial Actions Program (HAZWRAP) in support of the sponsor. Although the toxicity characteristic leaching procedure (TCLP) is the regulatory test for these organics, it was not considered a satisfactory test of the immobilization potential for these waste forms because this test was not designed for waste that had been stabilized/solidified, and, at the time of this study, regulatory guidance was not clear on sample handling and preparation when VOCs were involved (that is, VOC losses prior to the initiation of the extraction procedure may have given erroneously good results). A static leach test was utilized that enabled the calculation of both the mass transfer resistance of the waste forms and the affinity of the waste forms for the VOCs. Crucial to the accuracy of this test was the development of techniques to estimate the VOCs actually retained in the sample at the start of leaching. This was accomplished indirectly by measuring the VOCs that evaporated during sample preparation. Although the TCLP was done and is reported in this paper, the main measure of immobilization potential is considered to be the leachability index and distribution coefficient reported for each VOC and each of the following four vendors: Vendor A, RMC Environmental and Analytical Laboratories Co.; Vendor B, Wastech, Inc.; Vendor C, International Waste Technologies; and Vendor D, Silicate Technology Corp.

Procedures

It is standard practice in studies of this type to compare the TCLP performance of the waste before and after treatment. This approach was not used in this study because much of the VOCs were lost in the process of taking the large sample needed, and the objective of the study was measurement of the VOC immobilization potential. In the former approach, it is quite all right, desirable even, to have leachate, or extract, concentrations below the detection limits as a gross measure of effective treatment for a specific site; but one learns little about the true immobilization potential of a particular waste form that can be extrapolated to other conditions or sites. Since the VOC concentration of the waste sample in the laboratory was not representative of the site VOC concentration, spiking of the laboratory sample was required, and, to ensure obtaining measurable concentrations in the leachates, spiking was designed to give concentrations not only higher than the site average concentrations, but also higher than site maximum concentrations. Eight VOCs—acetone, 1,2-dichloroethene (1,2-DCE), chloroform, methyl ethyl ketone (MEK), benzene, trichloroethene (TCE), chlorobenzene, and perchloroethene (PERC)—were selected among the possible candidates to be spiked into the sludge and studied.

Sample Preparation

The procedure consisted of spiking the sludge using a Hobart mixer outside the glove box, taking three sludge samples for analysis by standard U.S. Environmental Protection Agency (EPA) Contract Laboratory Program (CLP) protocols (EPA CLP Statement of Work for Organics Analysis Multimedia, Multicomponent), immediately placing the spiked sludge inside the glove box, mixing the spiked sludge with the vendor materials according to the vendor instructions (grout), filling the stainless steel molds with the grout and covering with stainless steel endplates, removing the molds from the glove box and quickly sealing inside the stainless steel curing pipe, measuring the VOC concentration of the glove box air using a gas chromatograph with a flame ionization detector (GC-FID), curing the samples for 28 days, measuring the VOC concentration of the curing pipe air with the GC-FID, removing the cured samples from the pipe, and quickly removing the endplates, weighing, and sealing inside a zero-headspace extraction vessel (ZHEV). (Grout means the cementitious waste form in this document.) The amount of VOCs retained in the sample prior to leaching was estimated by subtracting the amount of VOCs that evaporated into the glove box and curing pipe from the amount measured in the sludge by the EPA protocol. (Rubbers and plastics, including Teflon®, should be avoided.)

Static Leaching

The static leaching procedure consisted of suspending each cured sample in 603 g of deionized water inside a ZHEV. Each sample consisted of a flat disk (6 2-cm diameter by 1.6-cm thick) inside a stainless steel ring with no covering on either flat face (the leaching surfaces). Two types of static leaching were done. First, three samples were leached separately for the entire time period with periodic in-house leachate analysis. Second, five separate samples were leached, each for a different time period. At the end of the time period, the leachate was removed and submitted to an EPA approved laboratory for analysis.

Leachate Analysis

The in-house analysis was done using a Tekmar liquid sample concentrator (LSC) in conjunction with a Perkin-Elmer GC ion trap detector (ITD) capable of measuring concentrations as low as 1 mg/Mg (PPB) within 15% of the true value. The submitted samples were measured using an LSC with GC mass spectrometer following EPA contract laboratory procedures (CLP), capable of measuring concentrations as low as 1 mg/Mg within 10% of the true value.

Results

Grout Composition and Properties

Only about 10 to 50% of the VOCs added during spiking was mixed into and retained in the sludge; presumably, the remainder evaporated during the process of mixing the spike into the sludge. This was expected based on the development tests and was the reason analysis of the sludge VOC concentration was required after spiking. The sludge analysis technique consisted of extraction followed by analysis of the extract. The extractant (typically hexane) did not always extract all of the VOCs in the sludge; thus, the VOC content of the spiked sludge tended to be underestimated for this study. Tables 2 and 3 list the average VOC concentrations, and their standard deviations, measured in the spiked sludge used to make the static leach samples and the TCLP samples, respectively. The acetone concentration was left as an unknown for the Vendor A static leach samples because its concentration was measured to be negligible, well below the amount that leached during the static leaching. The concentrations listed in Table 3 were more representative of maximum site concentrations, as intended. The concentrations listed in Table 2 were designed to give measurable leachate concentrations during static leaching.

TABLE 2—*The VOC sludge concentrations measured after spiking for the static leach samples, mg/kg (average of three with the standard deviation in parentheses)*

Compound	Vendor A	Vendor B	Vendor C	Vendor D
Acetone		123 (25)	223 (9)	210 (119)
1,2-DCE	927 (139)	170 (16)	485 (304)	953 (143)
Chloroform	1033 (125)	190 (16)	313 (180)	1150 (147)
MEK	253 (69)	137 (9)	430 (59)	257 (74)
TCE	623 (56)	193 (12)	184 (94)	573 (82)
Benzene	787 (98)	173 (17)	327 (188)	820 (91)
PERC	963 (119)	537 (115)	767 (201)	887 (246)
Chlorobenzene	550 (51)	547 (52)	220 (64)	477 (37)

[a] Left as an unknown.

TABLE 3—*The VOC sludge concentrations measured after spiking for the TCLP samples, mg/kg (average of three with the standard deviation in parentheses)*

Compound	Vendor A	Vendor B	Vendor C	Vendor D
Acetone	148 (74)	180 (28)	183 (24)	163 (26)
1,2-DCE	457 (127)	313 (19)	303 (47)	363 (9)
Chloroform	31 (1)	29 (2)	27 (1)	27 (3)
MEK	763 (29)	198 (128)	210 (262)	637 (202)
TCE	101 (63)	180 (24)	183 (21)	103 (12)
Benzene	6 (2)	6 (0 3)	4 (0 4)	8 (0 5)
PERC	233 (66)	197 (17)	106 (21)	170 (36)
Chlorobenzene	48 (4)	38 (4)	36 (4)	40 (3)

than while curing for 28 days inside the curing pipe. Although the vapor VOC concentration inside the curing pipe was significant, little mass was lost to the small headspace of these pipes. The concentration retained in the cured grout was estimated from the concentration measured in the spiked sludge correcting for the evaporation losses measured during mixing and curing and for the dilution of the spiked sludge in the grout product. Table 4 lists the grout composition for each vendor's product along with some other physical properties. The estimated VOC concentration retained in the samples after curing and percent retention is listed in Tables 5 and 6 for the static leach samples and the TCLP samples, respectively.

Static Leaching

Figure 1 illustrates the scatter in the leaching data. Despite the scatter, the general trend in Fig. 1 was obvious and was typical of this data. This trend was analyzed using the average leachate concentration for all the samples at a given time. The leachate for Vendor B's product foamed during analysis, resulting in invalidation of much of the early data for in-house analysis until this problem was solved. As a consequence, only the single set of data for the submitted samples exist for this early time period for Vendor B.

TABLE 4—*Cementitious waste form composition and physical properties*

	Vendor A	Vendor B	Vendor C	Vendor D
	COMPOSITION, WT%			
Sludge[a]	39 8	9 1	62 5	73 5
Water added	15 9	18 5	6 9	8 1
Solid additives	44 3	63 3	13 9	18 4
Liquid additives	0 0	9 1	16 7	0 0
	PROPERTIES			
Density, kg/L	1 61	1 62	1 22	1 39
Volume increase, %	121	784	93	40
28-day unconfined compressive strength, kPa	6447	12 797	83	414

[a] This reflects the wt% of the unspiked sludge. The spike was added as a water emulsion, but this extra mass of water is included under "water added."

TABLE 5—*Estimated VOC concentration (mg/kg) retained in the cured cementitious samples for the static leach test. (The value in parentheses is the wt% of the VOC that was estimated to be retained through mixing and curing.)*

Compound	Vendor A	Vendor B	Vendor C	Vendor D
Acetone	...[a]	9 (75%)	148 (98%)	108 (63%)
1,2-DCE	277 (67%)	8 (44%)	245 (75%)	482 (62%)
Chloroform	352 (76%)	13 (66%)	144 (68%)	710 (76%)
MEK	83 (73%)	11 (76%)	286 (98%)	146 (70%)
TCE	226 (81%)	13 (64%)	101 (81%)	392 (84%)
Benzene	270 (77%)	12 (67%)	174 (79%)	480 (72%)
PERC	412 (95%)	39 (72%)	510 (98%)	702 (97%)
Chlorobenzene	233 (95%)	50 (90%)	122 (82%)	370 (95%)

[a] Left as an unknown.

TCLP

Table 7 lists the results of the TCLP test. (Table 7 lists only the VOCs that were spiked, but the other compounds were below their TCLP limits.) The extract for Vendor A did not exceed any TCLP limits. The TCE limit was exceeded for Vendors B, C, and D, and the PERC limit was exceeded for Vendor C. Referring to Tables 1 and 3, the spiked concentration exceeded the site maximum concentration in all cases, except for TCE for Vendors A and D. The ratio of the site maximum to the spiked sludge concentration of TCE for Vendor A was 1.29. Multiplying the TCE extract concentration for Vendor A by this factor yields 0.032 mg/L, still below the limit of 0.07 mg/L. The equivalent factor for Vendors B and C was 0.72, giving a reduced TCE concentration of 0.051 mg/L (below the TCLP limit) for Vendor B and 0.22 mg/L (still above the TCLP limit) for Vendor C. A similar pro rata reduction for PERC and Vendor C gave 0.06 mg/L compared to the limit of 0.1 mg/L. Thus, correcting the TCLP extract concentrations by the ratio of the site maximum concentration to the measured sludge concentration resulted in concentrations below the TCLP limits for Vendors A and B, but TCE was still above the TCLP limit for Vendors C and D. The ratio of the TCE limit to the measured extract concentration was 0.23 and 0.37, respectively, for Vendors C and D. Assuming the extract concentration is proportional to the measured sludge concentration, the sludge concentration would have to be lower than 43 mg/kg (33% of the site maximum) for Vendor C and 38 mg/kg (29% of the site maximum) for Vendor D. The average of the TCE concentrations measured for the samples taken from the buried lagoon during site characterization

TABLE 6—*Estimated VOC concentration (mg/kg) retained in the cured cementitious samples for the TCLP test. (The value in parentheses is the wt% of the VOC that was estimated retained through mixing and curing.)*

Compound	Vendor A	Vendor B	Vendor C	Vendor D
Acetone	68 (98%)	18 (92%)	124 (99%)	131 (98%)
1,2-DCE	146 (68%)	25 (73%)	160 (78%)	212 (71%)
Chloroform	13 (85%)	2 (74%)	16 (89%)	19 (87%)
MEK	360 (100%)	22 (99%)	142 (100%)	520 (100%)
TCE	28 (58%)	10 (52%)	98 (79%)	56 (66%)
Benzene	2 (67%)	0.4 (36%)	3 (76%)	5 (83%)
PERC	107 (97%)	19 (88%)	67 (93%)	135 (97%)
Chlorobenzene	21 (94%)	4 (93%)	23 (9%)	31 (95%)

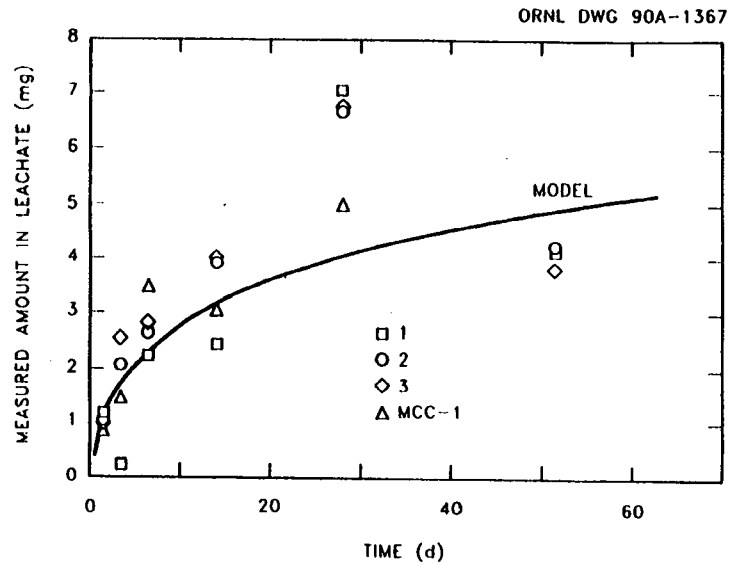

FIG. 1—*Static leaching of TCE from Vendor A's product.*

was 5.8 mg/kg (4% of the site maximum). Thus, all of the vendors may pass the TCLP test depending on what basis of comparison is allowed by the regulatory agencies.

Regardless of these number games about the TCLP limits for this particular site, it is important to note that this level of sludge concentration (10 to1000 mg/kg) did lead to measurable extract concentrations (0.004 to 0.3 mg/L), in some cases in the vicinity of the TCLP limits for these compounds. Undoubtedly, the performance of these products could be improved, for example, by targeting the additive for a specific compound or compounds, but it does seem to imply that application to some organic species present at as high as, or higher than, 0.1 wt% would be questionable, at least for this sludge. On the other hand, the performance was impressive for some compounds (for example, MEK was spiked in the sludge as high as 763 mg/kg, but never exceeded 0.5 mg/L in the extract, despite being a hydrophilic compound) compared to others (for example, the extract TCE concentration was uncomfortably close to or greater than the TCLP limit, despite being a hydrophobic compound). An additive such as organo-

TABLE 7—*Concentrations measured in the extracts for the TCLP test (mg/L).*

Component	Vendor A	Vendor B	Vendor C	Vendor D	TCLP Limit
Acetone	0.15	0.18	0.25	0.18	0.59
1,2-DCE	0.13	0.08	0.10	0.19	...[a]
Chloroform	0.005	0.004	0.009	0.004	0.07
MEK	0.014	0.008	0.048	0.032	7.2
TCE	0.025	0.071	0.30	0.19	0.07
Benzene	0.002	0.009	0.005	0.007	0.07
PERC	0.026	0.028	0.11	0.083	0.1
Chlorobenzene	0.074	0.027	0.11	0.14	1.4

[a] No limit has been set for this compound.

philic clay could be targeted for TCE to improve performance; in fact, the clay could be modified into two or more different forms to target different types of organic molecules. The performance of such an approach needs to be studied and the economics compared to other alternatives.

Analysis of Results

This section concerns estimation of the effective diffusion coefficients and distribution coefficient from the static leach results. The static leach test involved leaching from the surfaces of a slab. The initial concentration was assumed to be uniformly distributed in the slab and zero for the leachate. The leaching is assumed to be simple diffusion from the slab as defined by Fick's second law, with the diffusion coefficient for the slab much less than the leachate film diffusion coefficient. The leachates were not replaced (static leaching), and, hence, leaching is assumed to approach an equilibrium distribution of each specie between the slab and the leachate. Crank gives the following solution for this diffusion problem [5]

$$\frac{A}{(A_o - A_f)} = 1 - \sum_{n=1}^{\infty} \frac{2\alpha(1+\alpha)}{1+\alpha+\alpha^2 q_n^2} e^{-Dq_n^2 t/l^2} \qquad (1)$$

where the q_n's are the nonzero positive roots of

$$\tan q_n = -\alpha q_n$$

and

A = total mass leached in time t, mg,
A_o = initial mass in slab, mg,
A_f = final mass in slab in equilibrium with leachate, mg,
D = effective diffusion coefficient, cm²/s,
t = time, s,
l = half the thickness of the slab, cm,
$\alpha = V_1/KV_s = (A_o - A_f)/A_f$, and
K = equilibrium distribution coefficient, slab concentration/leachate concentration.

The distribution coefficient was defined as

$$K = (A_f V_1)/[V_s(A_o - A_f)] \qquad (2)$$

where

V_1 = geometric volume of leachate outside the slab, cm³, and
V_s = geometric volume of slab, cm³.

By basing the distribution coefficient on the geometric volume, the porosity, or void volume, of the porous solid body of the slab can remain unknown. Since most, if not all, of this void volume will be occupied by the leachate, A_f can approach the specie mass in the pore solution, but not zero. Mathematically, this means K can approach the ratio of the void volume (occupied by pore solution) to the leachate volume, but the variability of the data made such a low K statistically indistinguishable from zero. Thus, the least squares estimates of A_f and hence K, were allowed to go to zero. The reader may substitute a nonzero value representative of this ratio (say, 0.04) for the K values of zero in Table 8, if it is desired to keep K mathematically correct as defined for this paper. For the purposes of this paper and considering the

TABLE 8—*NEWBOX estimates of the mass transfer parameters (the time delay for Vendor B is given in parenthesis)*

Vendor	D, cm²/s	A_f, mg	A_o, mg	Leachability Index	K, mg/L grout per mg/L water
		Acetone			
A	3.0×10^{-7}	0.00[a]	5.0[a]	6.5	0.0[a]
B (17.6 days)	3.5×10^{-8}	0.06	0.8	7.5	1.0
C	[b]	7.00	7.2	[b]	439.7
D	3.0×10^{-7}	0.00[a]	16.0[a]	6.5	0.0[a]
		1,2-Dichloroethene			
A	7.0×10^{-8}	10.0	24.0	7.2	9.0
B (16.7 days)	6.0×10^{-8}	0.00	0.7	7.2	0.0
C	1.0×10^{-6}	4.00	12.0	6.0	6.3
D	1.0×10^{-7}	13.0	36.0	7.0	7.1
		Chloroform			
A		30.0[d]	30.6	[c]	628.1[d]
B (18.8 days)	4.0×10^{-8}	0.00	1.1	7.4	0.0
C	1.0×10^{-6}	0.70[e]	7.0	6.0	1.4[e]
D	2.0×10^{-8}	46.0	54.0	7.7	75.0
		Methyl ethyl ketone			
A	4.0×10^{-7}	1.30	7.2	6.4	2.8
B (12.2 days)	4.0×10^{-8}	0.55	0.9	7.4	19.7
C	1.0×10^{-8}	8.00	14.0	8.0	16.8
D	1.0×10^{-6}	1.25	11.0	6.0	1.6
		Trichloroethene			
A	2.0×10^{-8}	13.0	20.0	7.7	23.3
B (17.4 days)	1.3×10^{-8}	0.00	1.1	7.9	0.0
C	5.0×10^{-7}	1.70	4.9	6.3	6.7
D	1.5×10^{-8}	17.0	30.0	7.8	16.4
		Benzene			
A	7.0×10^{-8}	11.0	23.0	7.2	11.5
B (12.9 days)	3.5×10^{-8}	0.00	1.0	7.5	0.0
C	8.0×10^{-7}	1.40	8.5	6.1	2.5
D	8.0×10^{-8}	14.0	16.0	7.0	8.0
		Perchloroethene			
A	6.0×10^{-10}	33.50	36.0	9.2	168.3
B (12.8 days)	2.0×10^{-9}	0.00	3.4	8.7	0.0
C	1.0×10^{-9}	22.0	25.0	9.0	92.1
D	4.0×10^{-10}	51.50	53.0	9.4	431.3
		Chlorobenzene			
A	4.8×10^{-9}	16.50	20.0	8.3	59.2
B (0.5 days)	1.0×10^{-9}	0.90	4.3	9.0	3.3
C	6.0×10^{-8}	0.00	5.9	7.2	0.0
D	1.8×10^{-9}	21.00	28.0	8.7	37.7

[a] The amount leached exceeded the estimated A_o, thus, A_f was set as negligible and NLWBOX estimated t_o and D. The low acetone concentration measured in the sludge used to prepare Vendor A's samples was suspected of being erroneous.

[b] This data set was particularly poor for evaluating D. Using all the data, NLWBOX selected a D of 1×10^{-5}, which is not realistic, especially combined with the high K. This value is left unknown until a more thorough analysis will hopefully give a defensible value.

[c] NLWBOX estimated a suspiciously high D of 1×10^{-11} using the graphical estimate for A_f but NLWBOX's estimate for both (a D of 1×10^{-12} and a negligible A_f) was considered even worse.

data scatter, a value of zero to a few units for K means little or no significant interaction of the specie with the solid body. A value of about 10 probably means there was significant interaction with the solid body. The equivalent of the analytical solution is included in NEWBOX, a computer program that estimates the least squares fit for up to five parameters for leaching problems and that includes the equivalent for several analytical solutions to Fick's second law [6] In general, the average mass measured in the leachate for each vendor and specie at each time and the mass of each specie estimated retained in the slab (Table 5) were used as input to NEWBOX to estimate two parameters, D and A_f The distribution coefficient, K, was calculated from this A_f and the mass estimated retained in the sample after curing, A_o One exception to this approach was for the acetone in Vendor A's product In this case A_o was left unknown and estimated by NEWBOX, and A_f was assumed to be zero.

The trend for Vendor B's product was to leach little or no mass for a few days, followed by leach rates comparable to the other vendors This was not a simple diffusion process and could not be handled by NEWBOX. The NEWBOX model was modified for Vendor B by assuming no leaching for a set time, followed by a simple diffusion, that is, a time offset in Eq 1. The time delay was estimated as the time intercept of the least squares fit for the linearized form of the data. The linearized form of the data comes from the much simpler solution of the diffusion model for dynamic leaching of semi-infinite medium. From this solution, a plot of the total amount leached versus the square root of time is linear. Finite geometries and static leaching will deviate significantly from linearity, but the early leaching behavior will mimic this model Thus, the quantity leached was plotted against the square root of time, the linear portion subjectively selected, and least squares analysis used on this portion to find the time intercept for each specie. These time delays were subtracted from the times for Vendor B's data and these corrected times used as input to NEWBOX to get estimates for D and K. Table 8 lists NEWBOX's estimates for D and K, as well as the leachability index defined as

$$L = -\log(D) \tag{3}$$

where

L = leachability index with D in cm^2/s.

Discussion

The leachability index for these commercial products varied from 6 0 to 9 4, and the distribution coefficient varied from 0 to 628 From past experience with mobile species such as nitrate and some radionuclides, a leachability index of 6 to 7 is not very good for these waste forms and indicates that unless a strong interaction exists the specie will readily leach out of the porous solid body (usually there is a correlation between the leachability index and the distribution coefficient). Cementitious waste forms usually can achieve a leachability index of 7 to 8 and the better ones can get as high as 9 or better for the more difficult species. Some species are more readily immobilized in cementitious waste forms, or are just not very mobile, and have a leachability index of 10 or higher.

Taking each specie individually, one would expect acetone to be quite difficult to control because of its affinity for water, and this indeed proved to be the case. The leachability index and distribution coefficient were low for three (although Vendor B had a fair leachability index of 7 5) out of the four vendors, but Vendor C's product never leached much of the acetone estimated retained. The leachate concentration stabilized at a low level quickly, indicating both a high interaction and low leachability index. (In fact, it stabilized so quickly that NEWBOX estimated the leachability index as 5. This seemed unreasonably low because water has

a leachability index of about 5 and the distribution coefficient was estimated so high) Although NEWBOX estimates the two as independent parameters, D and K are expected to be related as follows

$$D = D_p/g(1 + K) \tag{4}$$

where

D_p = true diffusivity in the pore solution, cm^2/s, and
g = geometric factor, dimensionless.

This expectation raised questions about why the acetone concentration stabilized at this low value so quickly for Vendor C's product. Several explanations are possible, including the amount retained was in error and very little was actually in the sample, the original acetone was divided into two or more species, one of which was "unleachable" (strongly sorbed or disappeared by reaction); and the concentration was actually slowly increasing but the analytical technique was unable to detect this (the analytical variability has already been mentioned and acetone was one of the harder compounds to follow at these concentrations) There is not enough evidence at this time to rule out or verify any of these explanations or other possible explanations All that is known is that little of the acetone estimated retained in the Vendor C sample leached into the water according to analysis of the leachate.

For chloroform, although three of the leachability indices were still less than 8, Vendor D had a high K of 75 and Vendor A was even better at 628. NEWBOX estimated the leachability index at 11 for Vendor A, quite high for cementitious waste forms. This case was similar to the one for acetone with Vendor C in that not much leached Considering the variability and low concentrations, the index estimates for these two cases may be in considerable error.

The best performance for 1,2-dichloroethene was observed for Vendor A's product with a leachability index of 7.2 and a K of 9.0 Methyl ethyl ketone, like acetone, has an affinity for water, but had a better performance than 1,2-dichloroethene with an index of 8 and a K of 16 8 for Vendor C. The best performance for trichloroethene was for Vendor A with an index of 7.7 and K of 23.3. Vendor B had an index of 7.5 for benzene and a K of 0, but Vendor A had an index of 7 2 and a K of 11.5 The performance for perchloroethene and chlorobenzene was better than for the other compounds, up to 9.4 and 9.0, respectively, for the index and 431 and 59 for K

Conclusions

The TCLP was judged not satisfactory for testing the immobilization potential of cementitious waste forms Consequently, the immobilization potential was tested by static leaching and subsequent estimation of the leachability index and distribution coefficient.

Although open mechanical mixers were required to mix the sludge and treatment solids, the resulting reactions were exothermic, and the products were cured for 28 days; most of the VOCs were retained in the cured samples. The large uncertainty in VOC analysis means the error bars were large for the parameter estimation. The estimated error for leachate analysis was from 5 to 15%, depending on the compound, but the error in the parameter estimation was also affected by the errors in spiking and subsequent sludge analysis. Part of the problem was the inability of the standard extraction technique to extract some compounds completely from the sludge for analysis As a consequence, the VOCs tended to be underestimated in the sludge and the subsequent testing and parameter estimation tended to be conservative because more VOC's were actually present in the samples than estimated It is not unusual for the leachability index to vary by 0 2 without such large analytical errors The leachability index range

for this study may be 1.0 or more, with the reported estimates at the low end, and the estimates for K should probably be considered order of magnitude estimates.

Strong interactions were estimated for acetone, chloroform, and perchloroethene and significant interactions for 1,2-dichloroethene, methyl ethyl ketone, trichloroethene, benzene, and chlorobenzene, but no single product appears to interact significantly for all eight compounds. This implies that it is possible for the vendors to formulate for a combination of VOCs using a mixture of additives unless the different additives used by the vendors interfere with each other. Despite the evidence of interaction, the estimated leachability indices were disappointingly low except for perchloroethene and chlorobenzene. It is possible that some, but not all, of a given VOC was strongly sorbed and that the "free" VOCs quickly leached out, giving a relatively low index but a high or moderate K. Nevertheless, these low or moderate indices agree with the observation that the sludge concentration must be limited at about the spiked levels or less (<1000 mg/kg) to pass the TCLP test. The TCLP results proved that a commercially available cementitious waste form can pass the TCLP criteria for sludge concentrations indicative of this site. Based on these results, stabilization/solidification is a viable alternative for a sludge or soil that is heavily contaminated with metals and lightly contaminated with VOCs. In such a case, incineration is an expensive alternative that will likely convert the metals into a more mobile form requiring further treatment of the incinerated sludge/soil. Currently available additives proved capable of handling VOCs with limited success, albeit good enough to pass TCLP for this site. Perhaps better additives will be developed that will handle even higher organic concentrations if S/S is accepted for such applications.

Acknowledgment

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Solidification and Stabilization of Phenol and Chlorinated Phenol Contaminated Soils

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ABSTRACT: Phenol and chlorinated phenols are toxic compounds found in many treated wood products such as fence posts and railroad ties, hence soils near such treatment facilities or near old railroad yards can be quite contaminated. This study investigated the results of using sodium bentonite clay modified with dimethyl di(hydrogenated tallow) ammonium chloride mixed with Type I portland cement to immobilize a series of phenols (phenol, 2,4,6-trichlorophenol, and pentachlorophenol) from a sandy Shawano B horizon (typic Udipsamment) soil.

The test soil was mixed with the various phenols at a contaminant concentration of 1000 mg/kg. An admixture of contaminated soil and modified clay (0 to 10% w/w organoclay) was prepared. Final solidification was done by adding cement at concentrations of 16 to 39% w/w. Toxicity characteristic leaching procedure (TCLP) and unconfined compressive strength determinations were made. The results of these tests indicate potential for using these organoclay admixtures for treating soils contaminated with various chlorinated phenols.

KEY WORDS: organoclay, phenols, chlorinated phenols, stabilization, solidification, site remediation, toxic wastes

Unintentional contamination of soil with toxic chemicals is a continuing problem. The U.S. General Accounting Office estimates that as many as half of the 5000 hazardous waste sites regulated by the U.S. Environmental Protection Agency (EPA) may be leaking hazardous materials into local earth materials and then to groundwater [*1*].

Many of these toxic chemicals are organic in nature, some with potentially severe adverse effects. They can be carcinogenic or mutagenic or both in man and animals, toxic to aquatic life, and generally degrade the quality of water for human consumption. Hence, there is a need to develop new and alternate control technologies capable of preventing these toxic chemicals from dispersing through soil into aquifers.

A variety of different stabilization and solidification technologies have been developed in recent years [*2*]. Suggestion has been made that site remediation can be accomplished by using an organically modified clay (organoclay) [*3*]. This study focuses on the possibility of using an organoclay to stabilize phenol and chlorinated phenols in contaminated soils.

To be feasible for stabilizing and solidifying contaminants, a treatment procedure must produce a waste that (1) withstands a pressure of 0.344 MPa (50 psi) when applied in an uncon-

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Appendix XI

Appendix XI
Army Waste Classification Guidance for Building Demolition Debris Containing Lead Based Paint

WASTE CLASSIFICATION GUIDANCE FOR BUILDING DEMOLITION DEBRIS CONTAINING LEAD BASED PAINT

PURPOSE: This guidance provides two different acceptable methods for the characterization of the solid waste generated during demolition operations through sampling and Toxicity Characteristic Leaching Procedure (TCLP) analyses. Using either method, demolition debris can be characterized as hazardous or non-hazardous waste as defined by RCRA. These methods apply to demolition debris only and do not apply to heavy metal bearing wastestreams that are generated from other specific operations (e.g., paint scrapings, sandblast residues, etc.). Throughout this document, lead contamination, the most common TCLP debris concern, is used as an example, although these methods can be used to classify other TCLP wastes as well.

METHODS: Method I- The **Sampling/Statistical Analyses Method** requires sampling and TCLP analyses of both the paint and building material. The data is analyzed using conventional statistical methods to transform the results to a confidence interval (CI) of 80%. Method II- The **Mass Balance TCLP Method** requires only sampling and TCLP analyses of the paint and adjusts the TCLP data by a factor based the calculated uncontaminated mass in the total debris.

Method I. Sampling/Statistical Analyses:

SCOPE:

a. Before characterizing the waste, it is necessary to define the wastestream. The wastestream is the debris generated during a given demolition project at a given site/installation. While all buildings/structures generating demolition debris constitute the wastestream, only a percentage of these buildings need be sampled. Details on how to determine the appropriate number of buildings to sample are presented in the "PROCEDURE" section below.

PROCEDURE: During demolition debris waste characterization, several site-specific determinations need to be made. The following steps explain how:

a. Defining Individual Wastestreams/Populations: As defined above, the wastestream consists of all the debris generated during a specified demolition project. A list of the buildings should be recorded, including the building components undergoing demolition , and notations of buildings that will be generating the same type of debris because they are the same or similar design and construction and are undergoing the same type of demolition or rehabilitation. Information should also be gathered regarding the demolition and disposal procedures.

For instance, if the structures are set on cement foundations it would be necessary to determine whether the cement is to be demolished and disposed of with the rest of the debris. If such foundations were to be left in place they would not be considered as debris; otherwise, they would be included in the wastestream and would be sampled in accordance with the procedures discussed below.

b. Determining the number of Samples: Based on EPA guidance (EPA/600/8-89/046, March 1989, Soil Sampling Quality Assurance User's Guide, 2nd Edition), a statistical approach will be used to determine the number of buildings that need to be sampled. This approach is based on the assumption that the buildings are all relatively uniform and that the analytical results of the study will be normally distributed. The EPA manual SW-846--Test Methods for Evaluating Solid Wastes, Section 9.1.1.3, Basic Sampling Strategies (Attachment A), requires that the number of samples used to characterize a wastestream ensure an 80 percent confidence level in the resulting determination (in this case, hazardous or nonhazardous waste determination). The enclosed Table 1 is based on these guidelines and should be used to determine the number of buildings to be sampled at a given project site.

c. Sample Buildings Selection: Once the number of buildings to be sampled has been determined, the specific buildings to be sampled need to be identified. For buildings generating identical or similar debris (made of the same construction materials and painted with the same paint) a random approach should be used in the selection process. Buildings may be randomly selected using building numbers or placement on maps. However, when one or more groups of buildings generating identical or similar debris constitutes a separate and distinct segment within the total number of buildings, an appropriate percentage of buildings should be selected from the individual group(s).

d. Sampling Strategy: The objective is to obtain one representative composite sample from the debris wastestream from each building selected for sampling. The composite sample must include appropriate proportions of each debris that the wastestream is made of. Figure 1 depicts various areas of a building that may be constructed of different materials and must be sampled if they are part of the debris wastestream. Areas that will not be part of the debris wastestream do not have to be sampled.

(1) Debris components, such as glass, screen, or wiring, that are difficult to sample and comprise a very small (de minimis) percentage of the debris wastestream, do not have to be sampled. Also materials such as aluminum siding, large metal ductwork, light ballasts, utility equipment, and asbestos insulation need not be sampled if these materials are separated from the demolition debris and recycled/reused (e.g., scrap metal). In general, the most commonly sampled components will be wood, brick, cement and plaster/wallboard.

TABLE 1- STATISTICAL DETERMINATION OF THE NUMBER OF BUILDINGS TO BE SAMPLED

NO. OF TOTAL BUILDINGS AT ONE PROJECT SITE	NO. OF BUILDINGS TO SAMPLE*
1 - 9	ALL
11 - 15	10
16 - 20	13
21 - 30	16
31 - 40	21
41 - 100	26
> 100	32

* It should be noted that a sample is defined as a composite of subsamples from one building. 20-30 subsamples should be sufficient to make one sample.

2) The proportional size of the various building debris components based on (estimated) square footage must be determined. For instance, a building may be 70 feet long, 40 feet wide and 12 feet high; if all four of the exterior walls are to be demolished and are made of the same material, there is 2640 ft^2 of that debris component. Window and door space should be subtracted out from the exterior-interior walls and considered as separate debris components. The estimated area of each debris component (e.g., exterior wall, interior plaster board wall, interior plywood/panelling wall, floor, cinder block supports,

etc.) should be compared to one another in order to establish ratios. The ratios will determine the number of subsamples to obtain from each individual component. For instance, if 20% of the area is cinder block, than 20% of the subsamples should consist of cinder block samples.

Generally, 20 to 30 subsamples are necessary to make-up one 110-gram composite sample for each building. This number will vary based on the number of components that make up the debris. For instance, if building demolition debris consists of 60% walls, 30% floor and 10% doors (based on surface area), 18 subsamples (60/100 x 30) could appropriately be from the walls, 9 from the floor and 3 from the doors.

e. Sampling Methodology:

(1) Using a 1-inch bit drill or similar device, "core" subsamples should be obtained from each component of the building demolition debris. The number of subsamples taken for each debris component relative to the other debris components will be based on the ratios calculated above. The subsamples should be collected into a disposable container (such as large sheets of paper) as the drilling is done. The sampling crew should -- to the extent possible -- drill through the entire thickness of each component (e.g., doors, floor, etc). For building debris components such as cinder block or cement, a hammer drill should be used. The number of drill holes obtained from each component should be recorded. If the amount of material collected for the total composite sample for the building is not enough (i.e., less than 110 grams) for the TCLP, additional subsamples should be obtained from each of the specific components, with the number of additional subsamples based on the above calculated ratios. [NOTE: For at least 5 percent of the samples (and a minimum of 1 sample) taken for each demolition project approximately 300 grams should be obtained for adequate split laboratory analyses.]

(2) Field duplicates, equaling 5 percent of the number of actual composite samples (at a minimum of one), taken after each demolition project, should be obtained to check the sampling practice. The duplicate(s) should be obtained by simultaneously filling two sample containers during the sample process (i.e., for each subsample within a sample building, two adjacent cores should be obtained and placed into two separate containers).

f. Collection and Labelling: The sample material from each building debris should be collected onto a (disposable) container (such as sheets of unused paper, paper plates, etc.). From this collection container, the materials should be emptied into clean (new) plastic baggies and labelled with the project/installation name and or identification number, sample (building) number, sample date, and sampling personnel's name.

g. Decontamination: Nondedicated sampling equipment such as the drill bit should be decontaminated between sampling of individual buildings. The sampling crew should first brush excess material from the equipment and then wash using tap water and soap. This should be followed by a final rinse with distilled, deionized, filtered (DDIF) water. To ensure the equipment was properly decontaminated, a used rinse water sample should be taken and analyzed.

LABORATORY ANALYSES:

a. Packaging and Transportation: All samples should be properly packaged before transporting them to the certified analytical laboratory.

b. Laboratory Preparation: To ensure thorough mixing of the composite sample, the laboratory should be requested to thoroughly mix/homogenize the sample before preparing it for analyses. This will minimize the "settling" that may occur during transportation. This procedure is extremely important when excess sample has been obtained and the laboratory will only be using a portion of the overall sample.

c. Analytical Methodology: All solid material being analyzed (wood/plaster/paintchip, etc.) should be extracted using EPA Method 1311 (TCLP). The samples should be analyzed using either EPA Method 6010A [Inductively Coupled Plasma (ICP)-Atomic Emission Spectroscopy] or EPA Method 7421, the Atomic Absorption, Furnace Technique for lead. The ICP procedure is recommended due to lower cost, but either method will satisfy EPA requirements. The rinsate sample should also be analyzed using one of these methods.

DATA ANALYSES:

a. The TCLP laboratory results should be statistically analyzed to assess the variability among the buildings of the demolition or rehabilitation project and overall normality of the TCLP lead concentration distribution. If the analytical results do not indicate a normal distribution (i.e., the arithmetic mean is not greater than the variance), the raw data should be transformed. After normality has been achieved through an appropriate transformation, the 80 percent confidence interval (CI) should be calculated and compared to the (similarly transformed) regulatory threshold (RT) of 5.0 mg/L for lead. (See SW-846--Test Methods for Evaluating Solid Wastes, Section 9.1.1.3, Basic Sampling Strategies).

b. Additional procedures may be necessary to address potential "statistical outliers," or buildings that yield unusually high TCLP lead concentrations that dramatically skew the 80 percent CI. If necessary, such buildings may be addressed as a separate population.

An example of a sampling/data analyses program is attached below (Example 1.0). See Test Methods for Evaluating Solid Waste, EPA Manual SW-846, Vol. II, Chapter 9, November 1986 (Attachment A) for detailed calculations.

EXAMPLE 1.0
STATISTICAL ANALYSES
Building Debris Samples: Collected May-June 1992

Bldg #	Pb Values (mg/L)	square root of Pb values	
4711	1.08	1.039	
4900	1.11	1.054	
4901	14.7	3.834	
4905	10.0	3.162	
4713	1.35	1.162	
3644	3.11	1.764	
3626	1.40	1.183	
3635	1.63	1.277	
3641	1.53	1.237	
3628	1.76	1.327	
3639	1.38	1.175	
3640	0.50	0.707	
3629	0.51	0.716	
3632	1.06	1.030	
3637	0.91	0.952	
3627	0.82	0.907	
4904	2.56	1.600	
3634	0.50	0.707	
mean	2.55	1.38	
std deviation	3.61	0.80	
std err	0.85	0.19	
normal	No	Yes	
80% CI*	N/A	1.63	*80% Confidence Interval = mean + ($t_{.20}$*std err); where $t_{.20}$=1.333 for df=17
trsfd RT	N/A	2.24	
Hazardous waste	N/A	No	

By performing a square root transformation of the values, the data shows a NORMAL distribution (the mean > the STD squared).

Since the 80% CI is LESS than the square root of the regulatory level of lead (5 mg/l) the waste is not hazardous.

Reference: Test Methods for Evaluating Solid Waste, EPA Manual SW-846, Vol. II, Chapter 9, November 1986. (Attachment A)

Method II. MASS BALANCE TCLP CALCULATION METHOD:

SCOPE:

a. This method is based on the assumption that for building demolition debris, only the paint will contain heavy metals (e.g., lead) while the unpainted building construction materials will contain no heavy metals. In this instance, TCLP sampling and analyses of only the paint is required. If the above assumption is not valid, then the Sampling/Statistical Analyses Method (Method I) must be used.

b. Before characterizing the waste, it is necessary to define the wastestream. The wastestream is the debris generated during a given demolition project at a given site/installation. While debris from all buildings/structures being demolished or rehabilitated constitute the wastestream, only a percentage of these buildings need be sampled. Details on how to determine the appropriate number of buildings to sample are presented in the "PROCEDURE" section below.

PROCEDURE: During a demolition debris waste characterization study, several site-specific determinations will need to be made. The following steps are detailed to the extent possible.

a. Defining Individual Wastestreams/Populations: As defined above, the wastestream consists of all the debris generated during a specified demolition project. A list of the buildings should be recorded, including the building components undergoing demolition, and notations of buildings that will be generating the same type of debris because they are the same or similar design and construction and are undergoing the same type of demolition or rehabilitation. Information should also be gathered regarding the demolition and disposal procedures.

For instance, if the structures are set on cement foundations it would be necessary to determine whether the cement is to be demolished and disposed of with the rest of the debris. If such foundations were to be left in place they would not be considered as debris; otherwise, they would be included in the wastestream and would be sampled in accordance with the procedures discussed below.

b. Determining the number of Samples: Based on EPA guidance (EPA/600/8-89/046, March 1989, Soil Sampling Quality Assurance User's Guide, 2nd Edition), a statistical approach will be used to determine the number of buildings that need to be sampled. This approach is based on the assumption that the buildings are all of a relatively uniform construction and that the analytical results of the study will be normally distributed.

The EPA manual SW-846--Test Methods for Evaluating Solid Wastes, Section 9.1.1.3, Basic Sampling Strategies (Attachment A), requires that the number of samples used to characterize a wastestream ensure an 80 percent confidence level in the resulting determination (in this case, hazardous or nonhazardous waste determination). Table 1 (Method I) is based on these guidelines and should be used to determine the number of buildings to be sampled.

c. Sample Buildings Selection: Once the number of buildings to be sampled has been determined, the specific buildings to be sampled need to be identified. For buildings generating identical or similar debris (made of the same construction materials and painted with the same paint) a random approach should be used in the selection process. Buildings may be randomly selected using building numbers or placement on maps. However, when one or more groups of buildings generating identical or similar debris constitutes a separate and distinct segment within the total number of buildings, an appropriate percentage of buildings should be selected from the individual group(s).

d. Sampling Strategy: The objective is to obtain one composite sample of paint from each building selected for sampling. Only samples of paint from the building components that will be part of the debris wastestream should be sampled.

e. Sampling Methodology:

(1) Using a scraper or similar device, paint samples should be obtained from each component of the demolition debris. Typically, one should sample each component of the debris that contains lead paint and combine the samples into one composite sample (knowing that at least a 100-110g composite sample is needed for a TCLP analyses and that at least a 10-15g sample must be obtained from each debris component). The choice of and number of samples should be approximately in proportion to the areas of each building component that has been painted with the lead paint and that will make up the debris wastestream. Table 2 below provides an example of the percentage of total building debris area that each debris component comprises and the number of grab samples that should be taken of each component. Again, if any of these building components will not be part of the debris wastestream, they should not be sampled. [NOTE: For at least 5 percent of the samples (and a minimum of 1 sample) taken for each demolition project approximately 300 grams should be obtained for adequate split laboratory analyses.]

TABLE 2- PERCENTAGES OF AREAS COVERED BY LEAD PAINT

Category	Area	Percentage	# of Subsamples
Interior			
Ceiling	519.6	13%	3
Walls	1137	28.4	6
Doors	352.4	8.9	2
Windows	132	5.8	1
Cabinets	46		1
Shelves	55		1
Exterior			
Siding, Attic	201.6	5.0	1
Siding, Walls	1000	25.0	5
Roof, Underside	200	5.0	1
Porch	355.5	8.9	2
Totals	3999.1	100.0%	23

(2) Field duplicates, equaling 5 percent of the number of actual composite samples (at a minimum of one), taken during each demolition project, should be obtained to check the sampling practice. The duplicate(s) should be obtained by simultaneously filling two sample containers during the sample process (i.e., for each sample within a sample building, an adjacent grab sample should be obtained and placed into a separate container).

f. Collection and Labelling: The sample material from each building debris should be collected onto a (disposable) container (such as sheets of unused paper, paper plates, etc.). From this collection container, the materials should be emptied into clean (new) plastic baggies and labelled with the project/installation name and or identification number, sample (building) number, sample date, and sampling personnel's name.

g. Decontamination: Nondedicated sampling equipment such as the drill bit should be decontaminated between sampling of individual buildings. The sampling crew should first brush excess material from the equipment and then wash using tap water and soap. This should be followed by a final rinse with distilled, deionized, filtered (DDIF) water. To ensure the equipment was properly decontaminated, a used rinse water sample should be taken and analyzed.

LABORATORY ANALYSES:

a. Packaging and Transportation: All samples should be properly packaged before transporting them to the certified analytical laboratory.

b. Laboratory Preparation: To ensure thorough mixing of the composite sample, the laboratory should be requested to thoroughly mix/homogenize the sample before preparing it for analyses. This will minimize the "settling" that may occur during transportation. This procedure is extremely important when excess sample has been obtained and the laboratory will only be using a portion of the overall sample.

c. Analytical Methodology: All paint being analyzed should be extracted using EPA Method 1311 (TCLP). The samples should be analyzed using either EPA Method 6010A [Inductively Coupled Plasma (ICP)-Atomic Emission Spectroscopy] or EPA Method 7421, the Atomic Absorption, Furnace Technique for lead. The ICP procedure is recommended due to lower cost, but either method will satisfy EPA requirements. The rinsate sample should also be analyzed using one of these methods.

DATA ANALYSES:

I) TCLP DATA ANALYSES:

a. If only one building was sampled (e.g., the same paint was used in the other buildings) no statistical analyses need be performed on the TCLP data. If more than one building was sampled the TCLP laboratory results should be statistically analyzed to assess the variability among the buildings of the demolition project and overall normality of the TCLP lead concentration distribution. If the analytical results do not indicate a normal distribution (i.e., the arithmetic mean is not greater than the variance), the raw data should be transformed. After normality has been achieved through an appropriate transformation, the 80 percent confidence interval (CI) should be calculated.

b. Additional procedures may be necessary to address potential "statistical outliers," or buildings that yield unusually high TCLP lead concentrations that dramatically skew the 80 percent CI. If necessary, such buildings may be addressed as a separate wastestream.

II) TOTAL WASTESTREAM TCLP DETERMINATION:

After obtaining statistically acceptable TCLP of the paint from step I, above (where only one building had to be sampled, the TCLP value for the one composite sample is used), the TCLP level of the total debris wastestream can then be estimated from a TCLP sample. The key relationship can be derived as follows:

TCLPwaste = TCLPpaint x mp / mw,

Where mp is the mass of the paint on the building surfaces, and mw is the total mass of the debris wastestream generated. This simple proportion will provide a reasonable estimate of the TCLP for the debris wastestream which can be directly compared to the regulatory standard (e.g., 5.0 mg/l for lead).

a. Estimation of the Mass of Paint and Total Wastes:

Two basic formulas will be defined to estimate the parameters, mp and mw:

$mp(kg) = Ap(ft^2) \times dp(ft) \times rp(g/cc) \times 28.3\ (kg/ft^3)$

where Ap is the surface area of paint; dp is the depth of the paint surface; and rp is the paint density and 28.3 (kg/ft^3) is the density of water.

$mw(kg) = Vi(ft^3) \times ri(g/cc) \times 28.3\ (kg/ft^3)$

where i are the various debris components, such as wood, concrete, brick, etc, Vi is the volume and ri is the density of each debris component. The mass of each of the separate components are estimated using standard construction estimation techniques.

Estimation values for the densities of materials and the depth of the paint surface can be obtained from many sources. The density values given below are from "The Handbook of Chemistry and Physics" and the experience of the agency. Estimated average paint depth was obtained from the Denver Housing Authority.

Estimated values for ri, rp and dp:

Density of Concrete	2.5 g/cc
Density of Wood	.6 - .8 "
Density of Glass	2.5 "
Density of Steel	7.5 "
Density of Plaster	1.5 "
Density of Gypsum Board	.8 "
Density of Brick	2.0 "
Density of Stone (typical)	2.5 "
Density of Soil (dry)	1.4 "
Density of Pipe	7-8 "
Density of Paint	1.2 "
Average Paint depth	1/100 inch (8.33×10^{-4} ft)

The paint depth value is considered reasonably representative, but if the TCLP sample contains abraded material (such as wood or plaster), the average depth should be measured on-site and the mass of paint plus abraded material calculated.

The estimation of the volume of waste materials and the area covered by the paint surface will take the greatest amount of time. As an example of how this can be done, a typical one story house design was created for calculation purposes (Table 3). Where a number of similar buildings are involved, a single "representative" building could be used. If architectural drawings happen to be available, much of the information can be obtained directly from them.

Lead-painted surfaces are assumed to include all interior walls and ceilings as well as interior trim, windows, doors and kitchen cabinets. It is also assumed that the outside walls of the house and porch were painted with lead based paint.

Many of the volumes for the foundation, framing, and plaster surfaces can be estimated from geometric properties. Density values and the specific weight of water in kg/ft^3 are multiplied by the volume to obtain the weights for each material. The complexity due to the number of items involved is characteristic of residential housing. Federal installation buildings may be somewhat simpler.

Table 3 summarizes the calculation results for the basic structural categories. Area calculations are provided only where it is assumed that lead based paints were applied. The total estimated weight of the house and associated materials was 72,173 kg, which constitutes the debris wastestream. The total painted interior surface area was 2242 ft^2; the outer painted surfaces totalled 1757.1 ft^2. It was assumed that the paint thickness was .01" on average. The estimated weight of paint was 113.1 kg, the total mass of sample containing lead.

TABLE 3. HOUSE ESTIMATION VALUES BY CATEGORY

Category	Volume(ft^3)		Density Factors	Weight
Estimate of Total Debris Mass				
Concrete	648.5	x	2.5 x 28.3 =	45,881 kg
Brick Chimney	59.1	x	2.0 x 28.3 =	3,345
Wood, Framing	653.8	x	.7 x 28.3 =	12,952
Asphalt Roofing	26.0	x	1.5 x 28.3 =	1,104
Steel, Glass, Appliances				1,466
Soil & debris	100	x	1.4 x 28.3 =	3,962
Plaster/Lath	86.3	x	1.5 x 28.3 =	3,663
Sum				72,372 kg

Estimate of Paint Areas and Mass:

Interior	Area (ft^2)
Ceiling	519.6
Walls	1137
Doors	352.4
Windows	132
Cabinets	46
Shelves	55
Totals	2242

paint weight (kg) = paint area(ft^2) x paint depth x paint density

= 1.868 ft^3 x 1.2 x 28.3 = 63.4 kg

Exterior	Area (ft^2)
Siding, Attic	201.6
Siding, Walls	1000
Roof, Underside	200
Porch	355.5
Totals	1757.1

= 1.464 x 1.2 x 28.3 = 49.7 kg

Sum of all building paint = interior + exterior paint mass =
63.4kg + 49.7kg = 113.1 kg

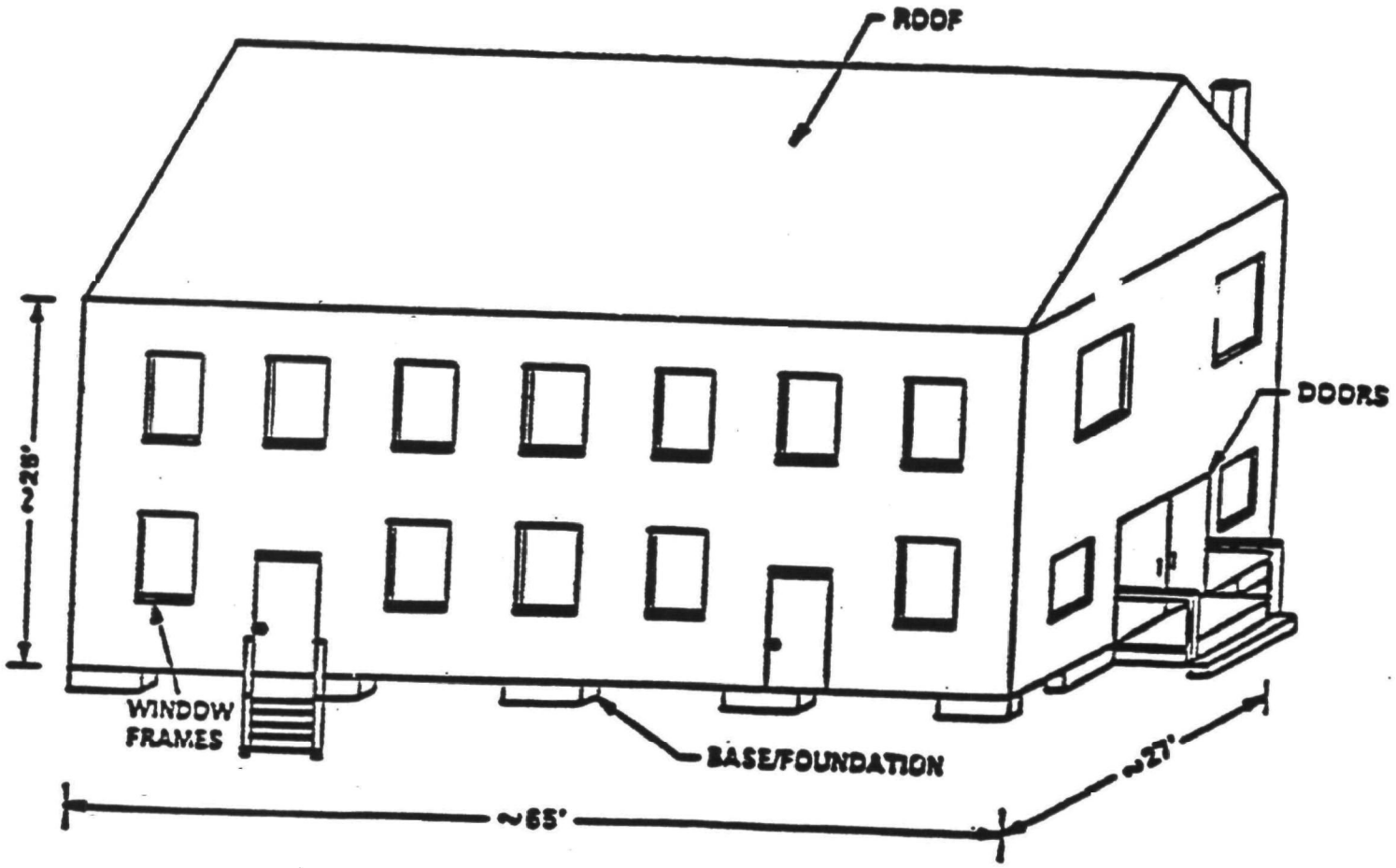
b. Calculations of the TCLP for the Waste:

The ratio of masses, mp/mw = 113.1/72373 = 1.56 X 10^{-3}. This means that for a value of 5 mg/l TCLP to be equalled or exceeded in the debris a corresponding TCLP value of: 5/1.56 x 10^{-3}, or 3200 mg/l Pb, would have to be obtained in the paint sample.

Figure 1
Example Diagram of a Building
(WWII Temporary Barracks Slated for Demolition)

Inside: Partition Walls
Ceiling
Floor

Inner Structure: "Studs"
Support Beams



Appendix XII

Appendix XII
1992 Workshop on Characterizing Heterogeneous Materials

United States
Environmental Protection
Agency

Office of Research and
Development
Washington, DC 20460

EPA/600/R-93/033
March 1993

EPA

Environmental Monitoring Issues

Results of Workshops Held in July 1992 as Part of EPA's Eighth Annual Waste Testing and Quality Assurance Symposium

5. CHARACTERIZING HETEROGENEOUS MATERIALS

5.1. BACKGROUND

In both the RCRA and CERCLA programs, a material often must be characterized to determine if it possesses some property of concern or interest. Types of questions that may require answering include:

- Is the material hazardous?
- Can it be safely or successfully managed using a specified treatment technique?
- How much supplemental fuel must be added to incinerate the waste?

When the material being characterized is homogeneous, conventional sampling and subdividing approaches can be employed. For example, in a truckload of used foundry sand with an average phenol concentration of interest, the material is in the form of relatively fine particles with the phenol uniformly distributed on the surface. Conventional compositing and subsampling can provide representative samples with an average composition very close to that of the entire load. The actual task of obtaining each subsample from the appropriate point in the truck may be very difficult, but the approach used is relatively straightforward.

However, insurmountable problems can develop when faced with a heterogeneous material. Heterogeneity is a relative term and, among other factors, is a function of the objectives of the characterization and the analytical sample size. This workshop was concerned with wastes of such various particle size, waste consistency, or extraordinary concentration gradients that the sampling and analytical objectives could not be met using traditional approaches and standard techniques. This is an important and difficult problem that has been of concern to many organizations involved in waste management.

The workshop goal was to advance the state-of-the-art techniques for characterizing difficult to sample wastes. This covers wastes with a highly variable nature, (e.g. wide ranges of particle size, large concentration gradients, and mixtures of different waste materials (e.g. dredge materials containing sludge, tires, construction debris, bottles, cans, etc.)). These types of materials present challenges that traditional sampling and analysis approaches cannot meet with the level of certainty required for modern waste management decisions.

RCRA has historically taken the position that the sample actually being analyzed does not have to be representative of the material being characterized, but rather that the sum total of the data must represent the property of interest. The problem presented was how to develop practical solutions to characterize these difficult situations. In this session, the participants discussed how to cost-effectively characterize materials that are inherently very heterogeneous and present characterization difficulties.

5.2. SUMMARY OF POSITION STATEMENTS

5.2.1. Regulatory Program Perspective

Mr. Charles Ramsey, EPA's National Enforcement Investigation Center (NEIC), reviewed some issues involved in implementing the RCRA regulatory requirement that a "representative" sample of the waste be collected and analyzed. 40 CFR, Part 260.10 stipulates that a representative sample, '... means a sample of a universe or whole (e.g., waste pile, lagoon, ground water) which can be expected to exhibit the average properties of the universe or whole." This definition has two aspects.

1. What is an average property?
2. How is the universe or whole defined?

The inability to answer these questions in clearly defined scientific terms leads to most of the problems surrounding the characterization of heterogeneous wastes.

Mr. Ramsey Proposed that hazardous waste characteristics are not "averageable" properties. For example, corrosivity is expressed as pH that is a logrithemic function, while ignitibility can be expressed as passing or failing the 60°C ignition test. With these characteristics, the numerical mean is not meaningful; therefore, an average value cannot be obtained.

Using the average value as a measure of hazard with respect to the toxicity and reactivity characteristics may also present potential problems. When the material to be evaluated consists of both a contaminated phase and an inert phase, use of the mean may result in a waste being classified as nonhazardous even though a substantial portion of the waste might be above the regulatory threshold and pose an environmental hazard.

Using "the most likely result" approach (also known as attribute testing) avoids many of the above problems. It requires that a certain percentage (e.g., 50%, 75%, 95%) of the individual sample results fall below the regulatory decision value. This avoids problems associated with determining average values. Use of this approach, however, will require a change in the regulations.

Even when the property of concern is a continuous variable and, therefore, averageable, collecting representative samples of heterogeneous wastes is often extremely difficult. Many heterogeneous wastes are discontinuous in terms of physical/chemical properties and, given the small size of analytical samples relative to the discontinuity, it is not possible to collect (and demonstrate) representative samples of the wastes. Therefore, to ensure that an adequate characterization is conducted, a clear definition is needed of what constitutes the "universe or whole" to be characterized and what constitutes an acceptable level of characterization (i.e., What degree of confidence, that the property is below the regulatory threshold, must be obtained to characterize the waste as nonhazardous.). The universe of the whole must also be defined to determine the period of time and space in which the samples must be collected.

5.2.2. Regulated Community Perspective

An industrial perspective was prepared by Mr. David Reese, Safety-Kleen Corp., and presented by Mr. Gene Klesta, Chemical Waste Management Corp. The presentation focused on the types of samples received at recycle and waste disposal centers and the different types of problems these materials represent relative to heterogeneous wastes found in the field. The industry receives large quantities of containerized wastes from different sources which in the aggregate form a very heterogeneous set.

Industry strongly supports considering the method of management when establishing analytical requirements. The current requirement for full-scale analysis is, in many cases, not necessary and represents a significant financial burden for generators and the waste management industry. The industry recommends that wastes destined for recycling or treatment be exempted from rigorous analysis if the waste management process and end products are thoroughly characterized during operations and prior to disposal.

It is further recommended that the Agency permit the use of methods other than those in SW-846 when conducting acceptance testing of wastes for treatment and recycling. Using proper QA/QC procedures is adequate to ensure that reliable analysis are conducted. Again, process monitoring and end product analysis provide sufficient protection of the environment. The industry will work with the Agency to bring industry-generated methods into the public sector.

The industry position is similar in concept to the Agency's interest in moving towards performance-based methods using sound project QA plans, data quality objectives,and accepted QA/QC procedures as the quality assurance tools to monitor performance.

5.2.3. State-of-Science Perspective

Dr. John Maney, a private consultant, presented a detailed strategy for random and nonrandom sampling of heterogeneous wastes. The presentation, described a unified way of looking at all types of waste based on the source and degree of heterogeneity of the material.

Using flow charts and an extensive set of definitions and examples, the paper presents concrete examples of options available to the field sampling team and the analytical chemist to generate reliable analytical data from complex, heterogeneous materials. However, unless the sample undergoes extensive comminution, Dr. Maney indicated that obtaining an analytical size representative sample of a heterogeneous waste is almost impossible. (See the full paper in Appendix A for details.).

5.3. CHARGE TO THE WORKSHOP PARTICIPANTS

Before breaking into three workgroups, the participants were asked to address three questions:

1. Should EPA change from current practice (average testing) to attribute testing for properties that are not averageable?

2. If so, what is the highest practicable degree of confidence (%) that could be required?

3. Should there be an override that says that if some samples are greater than "X", the waste is hazardous even if the number of such samples are below the percentage established in question 2?

A large part of each workgroup's time was devoted to discussion of the practical definition of attribute testing and how it differs from average property testing. This decreased

the time available to discuss the three issues, so no effort was made to address the third issue within any workgroup.

5.4. RESULTS OF THE WORK GROUP DELIBERATIONS

5.4.1. Should the Agency Change to Attribute Testing for Properties that are Not Averageable?

Approximately 90 percent of the participants believed that attribute testing should be adopted for properties that are not averageable. This approach is more costly than average property testing in those situations where the average property was determined by compositing a series of field samples prior to analysis. There was some discussion that compositing of samples prior to analysis is one way to determine an average property that avoids many of the technical problems associated with averaging the results from a series of discrete sample analyses.

Neither the attribute testing nor the average property testing approach eliminates all the problems associated with the sampling of wastes prior to analysis. The nature of attribute testing can make the sampling in many situations easier. The group agreed that approximately 80 percent of the error in characterizing waste arises from field sampling error that effects both approaches equally.

The Agency should focus its efforts on providing sampling guidance for heterogeneous wastes. Special efforts should be made to provide guidance for specific types of wastes such as tires, telephone poles, construction debris, and military ordinance. Also, knowledge of the process that generated the waste is an important part of the decision to use attribute testing and would be a primary driver in selecting the sampling strategy.

5.4.2. What is the Highest Practicable Degree of Confidence that can be Required?

Several participants felt the answer to this question should take into account the degree of health and ecological risk associated with the material and the amount (mass or volume). Therefore, each site should be considered unique and receive individual consideration. A site-specific degree of confidence should be carefully developed as part of the data quality objective process and be codified in the site-specific QAPjP.

It was generally agreed that a maximum of 10 percent of samples collected should be allowed to fail the regulatory criteria. Sixty-six percent of the participants felt that a 4 to 6 percent failure rate was the most appropriate. All participants felt the Agency should publish clear guidelines on determining if attribute testing should be applied to a specific situation.

5.5. AGENCY FOLLOW TO WORKSHOP

Subsequent to the workshop, Agency staff met to discuss what steps to take to address the issue and the ideas presented during the session. It was decided that quantitative data are needed to determine:

- What the practical effect would be if the attribute testing approach was used instead of the current arithmetic mean.
- How the number of samples needed to achieve a defined degree of confidence would vary across the different types of materials characterized in the RCRA and CERCLA programs.

During the next year, the Agency plans to examine its data in an attempt to answer the above questions. However, the Agency will greatly appreciate receiving any additional information that will help determine if a change in approach will either improve the quality or lower the cost of decision making. If anyone has data that might assist in this effort, EPA will appreciate receiving such information. Such information should be sent to the author at the address indicated in the FORWARD to this report.

CHARACTERIZING HETEROGENEOUS MATERIALS: INTRODUCTION

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BACKGROUND

Throughout the RCRA and CERCLA programs, one is faced with the task of characterizing a material to determine if it possesses some property of concern or interest. Types of questions one may be answering include:

- Is the material hazardous?
- Can it be safely or successfully managed using a specified treatment technique?
- How much supplemental fuel might I have to add to incinerate the waste?

PROBLEM

When the material being characterized is homogeneous, conventional sampling and subdividing approaches can be employed. For example, take the case of a truckload of used foundry sand whose average phenol concentration is of interest. The material is in the form of relatively fine particles. Conventional compositing and subsampling will provide relatively small representative samples whose average composition is very close to that of the whole load. The actual task of obtaining each subsample from the appropriate point in the truck may be very difficult, but the approach used is relatively straightforward.

However, what happens when one is faced with a heterogeneous material? When we speak of heterogeneity, we need to keep in mind that this is a relative term and is a function of the objectives of the characterization and the analytical sample size. The concern of today's workshop is generally with wastes of such various particle size, waste consistency or extraordinary concentration gradients that the sampling and analytical objectives cannot be met using traditional approaches and standard techniques. When it is not possible to obtain a representative sample, what shall we do?

This is an important and difficult problem. It is one that has been of concern to many organizations involved in waste management. For example, in March 1991, the Environmental Protection Agency, the Department of Energy, and ASTM's Committee D-34 conducted a workshop to examine how to characterize heterogeneous wastes that were also contaminated with radioactive materials. A report has recently been issued that describes the results of the workshop[1]. However, many problems and issues remain to be addressed and we have convened today's workshop to address some of the issues.

We would like your help with addressing the following specific issues.

Identify characterization situations that present difficult problems (generically identify both the type of material and situation).

- Develop specific guidance laying out the process to be used to adequately and cost-effectively address the problem. Describe what criteria should be used to guide development of solutions/approaches to specific problem situations. Please be as quantitative as possible on when trade-offs may need to be made and how to select among options.

- If research work needs to be done to develop a solution, please identify what work needs to be done.

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[1] Characterizing Heterogeneous Wastes: Methods and Recommendations, EPA/600/R-92/0, February 1992.

- Since EPA would like to solve monitoring problems through cooperative ventures, what form should a cooperative development program take? How can it be organized? Who might the cooperators be?

If you think of any ideas, information, or suggestions that you feel the Agency should consider when addressing this issue, please send them to us. Send your comments to:

David Friedman (RD-680)
US Environmental Protection Agency
Washington, DC 20460

We will need to receive your comments by August 21, 1992 in order for them to be incorporated into the final conference report.

CHARACTERIZING HETEROGENEOUS MATERIALS: REGULATORY PERSPECTIVE

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INTRODUCTION

Characterization is the process used to determine whether solid waste is also a hazardous waste. Characterization is arrived at by following "testing structure." A testing structure is the particular sampling, analysis, and data interpretation steps that are employed to measure a waste. The purpose of this paper is to examine the testing structure used to measure characteristics (ignitability, corrosivity, reactivity, and toxicity) of heterogeneous solid waste that would make it hazardous waste as defined in 40 CFR part 261.21-24.

To date, there has been little progress in developing a testing structure to characterize heterogeneous waste. EPA regulations state that characterizing waste includes determining the average property of the "universe or whole", 40 CFR part 260.10. The problems with the regulations are that "average" lacks scientific validity and "universe or whole" is not defined. These two items are an integral part of the testing structure.

To address these problems, two areas must be explored: 1) policy, regulations, and guidance issues and 2) scientific concerns. This paper focuses on heterogeneous solid waste because it is a worst case scenario. A testing structure that works for heterogeneous solid waste will work for any solid waste.

POLICY, REGULATIONS, AND GUIDANCE ISSUES

Many problems with characterization of heterogeneous materials under RCRA can be traced to the definition of what type of sample must be collected and analyzed. The required sample is a "representative" sample which is defined in 40 CFR part 260.10. "representative sample means a sample of a universe or whole (e.g., waste pile, lagoon, ground water) which can be expected to exhibit the average properties of the universe or whole." At least two problems arise with this definition: what is the average property and what is the universe or whole?

Average

Does average signify the arithmetic average or the "most likely result" of the target population? To illustrate, if the data points are 5.7, 5.7, 5.8, 5.9, and 0.2, the arithmetic average is 4.7. If the regulatory limit is 5.0, the best point estimate of the average is that it is below the regulatory limit. However, if confidence intervals were required, there would be little confidence in this conclusion.

If average, however, signifies the "most likely result" (the point at which one half of the values are above or below--the median) one would conclude that 80% of the data points are above the regulatory limit. This conclusion is also an expression of a higher degree of confidence than the conclusion with arithmetic average. More of the values agree with this conclusion (4 of 5 are above the limit) than agreed with the conclusion based on arithmetic average (1 of 5 were below the limit).

With the arithmetic average approach, it is possible to have well over half the population above the regulatory limit and still achieve compliance. With the "most likely result" approach, not more than one half of the population can be above the regulatory threshold and be in compliance. The "most likely result" is sometimes referred to as "attribute" testing. An

attribute testing structure enables one to conclude if a waste is or is not hazardous--not the degree of hazard.

For clarification of what is meant by average, the Agency provided Chapter Nine of "Test Methods for the Evaluation of Solid Waste, Physical/Chemical Methods" (SW-846). This guidance suggests that average signifies the arithmetic average.

A problem with this definition can be illustrated by the following scenarios. One of the RCRA characteristics is ignitability which includes analyzing samples for flashpoint. The characteristic is present when the waste flashes at a temperature less that 60° C. In many cases a number is not recorded, only the presence of a flash. The regulations do not state that a waste which flashes at 10° C is any more hazardous than one that flashes at 59° C. This logic fits well with "most likely result" or percentage (which does not require numeric results), but is inconsistent with the arithmetic average. With ignitability testing sometimes the analysis produces numbers and other times it does not. Without numbers, arithmetic averages can not be calculated, but the "most likely result" can be determined. Another characteristic is corrosivity which can be determined by pH. Several pH values cannot be averaged to determine the average property of a waste. First of all they are log values and adding them to derive an arithmetic average is actually a multiplication, thus yielding the wrong information. Furthermore, from chemical principles, the final pH depends on the buffering capacity of the individual components. If a waste in question has pH measurements of 2 and 4 (the regulatory limit under RCRA is 2.5), it is incorrect to conclude that the average pH is 3 and therefore the waste is not hazardous. Arithmetic average is also not scientifically valid for either toxicity or reactivity.

Alternatively, the "most likely result" approach will produce valid results for each RCRA characteristic test. If five samples were randomly selected from a waste and four flashed, then 80% of the samples exceeded the regulatory limit. If "most likely result" is synonymous with greater than 50%, then the conclusion that the waste is hazardous can be made with confidence.

Universe or Whole

The waste population to be characterized is not defined in the regulations. If the "universe or whole" is undefined with respect to both temporal and spatial requirements, it is not possible to design a valid testing structure. This is illustrated through some of the following scenarios.

If there are several piles of waste, must a decision be made for each pile or for the whole of all the piles. What if one pile is above the regulatory limit and the other pile is below? What if several piles are below the regulatory limit, but a few are above? What if the average is below; are any of the piles hazardous?

What if the average property of a pile is below the regulatory limit? Some portions of the pile could be above the limit, and others below. What if the portion of the pile above the limit was loaded into a truck to be hauled to a non-hazardous waste landfill and this was sampled? Is this waste which was not hazardous in the pile now hazardous when in the truck?

What if the waste produced today is above the regulatory limit, but the waste produced yesterday and the waste produced tomorrow is below the regulatory limit, is the waste below the regulatory limit on average or not?

"Universe or whole" must be defined or it is impossible to know where to sample and how to interpret the results.

SCIENTIFIC CONCERNS

While it is always possible to sample, analyze and produce data for any waste, the problem with heterogeneous materials is the confidence that a correct compliance decision was made based upon a valid testing structure. A compliance decision is affected by the purpose of

the measurement, use of proper sampling and analytical methods, and correct interpretation of the results. The variability within heterogeneous materials exacerbates the bias and imprecision of the results. As bias and imprecision increase and the average approaches the regulatory limit, compliance decisions become more difficult. If, however, the proper testing structure was employed, it would dramatically improve the decision making process and increase the confidence in the results.

A proper testing structure should have the following characteristics:

It must be scientifically valid. Basic scientific principles must not be violated. There are instances where the ideal scientific solution can be adjusted for simplicity, politics, and economics, but the solution must remain scientifically valid. Agency regulations, policy, and guidance must never be inconsistent with basic scientific principles ("Safeguarding the Future: Credible Science, Credible Decisions", EPA/600/9-91/050).

It must be consistent. All elements of the testing structure are dependent on each other for their validity. Sample collection must anticipate the analyses and data interpretation to be performed to ensure that the proper samples are collected. Likewise, without detailed knowledge regarding sample collection, data interpretation is meaningless.

Proper Sampling

Proper sampling primarily depends on the regulatory question (e.g., presence, trends, absence, etc.), the universe or whole (population), and the confidence that the correct compliance decision can be made. These three considerations--question, population, confidence--must be definitively addressed before a sampling plan is developed and implemented.

Proper Analysis

Proper analysis for RCRA characteristics is not an issue, because three of the four characteristics have required test methods referenced in the regulations (reactivity methods are only guidance at this time). The correct analytical result is the one obtained by following the prescribed analytical method.

Proper Data Interpretations

Proper data interpretation is important for making correct decisions. Data interpretation ties the analytical results back to the population with some specified degree of confidence to determine if the sampling and analysis answered the question about the waste. Proper data interpretation includes:

Making the proper type of confidence statement. The statement does not need to be statistical; it can be an expression of professional judgment. If statistical statements are made one must recognize that there are many types (e.g., confidence interval of the mean, tolerance limits, prediction intervals, etc.). Not all statistical statements are valid for any particular compliance problem and care must be chosen to select the correct one.

Establishing degree of confidence in the statistical statement. It is possible that one can make a certain statistical statement (or use professional judgment), but if one is not very confident in the statement it serves no purpose proving compliance or non-compliance.

With many types of statistical statements, certain assumptions are made regarding the distribution of the data. If incorrect assumptions are made, the resulting statistical statement and degree of confidence might not be correct. The guidance given in SW-846 makes certain assumptions regarding the distribution of data (normally distributed) which are usually incorrect. By definition, heterogeneous waste (as well as most environmental data) defies these assumptions

to a degree that can make the decision and confidence statements based on arithmetic averag incorrect. The usual indicators of non-normal data are outliers and skewed distributions. Wi the "most likely result" no distribution is assumed (non-parametric) and therefore the problems with heterogeneous waste do not affect the accuracy and confidence of the decision.

CONCLUSIONS

Characterization of heterogeneous waste depends on the development on a valid testing structure. Currently, only portions of the testing structure are in place. Two items that need to be addressed are:

The "most likely results" (percentage) is the valid statistic for making compliance decisions to characterize waste. The current use of arithmetic average is scientifically invalid for the RCRA characteristics and can lead to wrong compliance decisions. There are at least two percentage options that currently exist in the RCRA regulations. One is the empty drum regulation (40 CFR part 261.7) which states that drums with a capac of less than or equal to 110 gallons are considered empty if no more than 3% is left (and the contents have been removed using common practices). Drums with a capacity of greater than 110 gallons are allowed 0.3%. The other percentage option is found in the Land Ban regulations (49 CFR part 268) which requires that no more than 1% of the population can be over the treatment standard.

With this new system, the Agency must decide what percentage of the waste it will allow to be over the regulatory limit and still classify the waste as non-hazardous. To be the most protective of the environment, 0% should be adopted although it is probably an unrealistic standard.

"Universe of whole" must be clearly defined. This is not a scientific issue, but rather a policy issue, The term requires clarity and specificity as to both time and space. Some

examples might be: one day of production, one week of production, one pile, all piles currently on the property, 100,000 Kg, etc. Defining "universe or whole" as one waste stream would ignore the temporal problems. Without knowledge of exactly what the waste population is in terms of both time and space, it impossible to develop a testing structure to characterize it.

HETEROGENEOUS WASTE CHARACTERIZATION: INDUSTRY PERSPECTIVE

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INTRODUCTION

Heterogeneous as defined by Webster in the context of a waste is "consisting of dissimilar or diverse ingredients or constituents". The complexity of this issue is illustrated with a drum of heterogeneous waste in figure 1.

Characterization of heterogeneous wastes presents a number of unique challenges for th industrial community. The principal challenges are: sampling, methodology of analysis, frequency of testing, and controlling/ reducing costs.

SAMPLING TECHNIQUES

Routine sampling techniques consisting of sample segregation, compositing,and particle size reduction are often inappropriate when trying to homogenize a heterogeneous waste. Collecting the sample can become a formidable task when the waste is comprised of multiple large particle size components. SW-846 sampling containers often are not suitable for sample containment and larger vessels are required. Loss of volatiles due to headspace in the vessels affects the validity of the results. Present particle size reduction techniques such as grinding only exacerbates the problem by driving off volatiles.

TESTING REQUIREMENTS AND PROCEDURES

State and Federal regulations often require extensive testing protocols in Waste Analysis Plans (WAPs) and permits. Regulators routinely require the use of SW-846 analytical methods. These methods often are not appropriate for a variety of waste streams (organic solvents, oily wastes, by products of recycling such as distillation bottoms). The use of methods other than SW-846 should be accepted for alternative test procedure approval and not rejected on the sole basis they are not SW-846. Most of the quality assurance/ quality control principals and guidelines can be incorporated into alternative test methods resulting in improved data with only procedural and or hardware modifications. Unfortunately these methods are not considered valid by regulators.

The present sampling and testing requirements fail to consider the volume and frequency of the waste being handled. It is extremely costly to test every fifty-five gallon drum according to SW-846 guidelines, while testing every incoming rail car might be feasible. Current sampling frequencies often overlook one of the key issues facing a heterogeneous hazardous waste handler, that being, the end use of the waste. If small volumes of wastes are going to be mixed with other wastes for processing in terms of recycling, and the facility is permitted to handle the waste, the result will have no impact on the process, thus, sampling and testing need to reflect the use of the waste while providing a safe working environment. The product of the process should become the focus of testing.

Continuing the status quo will drive many companies out of business and create large companies which will monopolize hazardous waste handling while passing the costs on to the generator and ultimately the consumer. This is due to the excessive costs associated with testing every handled waste. Multiple testing of the same waste is redundant and provides no greater assurance of safety, process control, product quality.

CONCLUSIONS: DEVELOPING ALTERNATIVES

Recognize Alternative Test Methods

State and federal regulators must become accepting of industry generated methods of analysis for complex waste streams handled on a daily basis.

Develop New Methods

EPA in conjunction with industry should work together to facilitate the rapid development and approval of new methods. This could be accelerated by using industry developed methods.

Encourage Recycling

Recycling needs to be encouraged by minimizing input testing and focussing on by-products of the process. When a facility is fully permitted to handle wastes, analysis do not affect the processing or end use. This adds unnecessary costs to the recycling process through testing of incoming, in process, and final products.

Account for End Use

Prescribing testing must account for the end use of the waste. Some end uses such as alternative cement kiln fuel present minimal hazards, thus testing should be relevant to the waste destination.

Balance Cost with Severity of Hazard

The health and safety models need to be incorporated into the testing protocol by focussing on hazards and not over testing of materials that pose little or no threat.

Obstacle to Implementation

The largest obstacle for implementation of the above alternatives is the magnitude of the number of individual state regulators with differing view points. The result of negotiating fifty different WAPs in fifty states poses limitations on industries ability to comply fully at all times. It is impossible for a business to operate fifty different ways on the same type of waste stream.

REFERENCE

Characterizing of Heterogeneous Wastes: Methods and Recommendations; EPA 600/R-92/033, Feb. 1992

CHARACTERIZING HETEROGENEOUS MATERIALS: SCIENTIFIC PERSPECTIVE

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INTRODUCTION

A discussion of heterogeneous waste characterization is best started with a brief review of definitions.

Sample: a part or piece taken or shown as representative of a whole group. (Webster Unabridged Dictionary, Second Edition)

Sampling: the act, process or technique of selecting a suitable sample. (Webster's 7th Collegiate Dictionary)

Representative Sample: A sample of a universe or whole (e.g. waste pile, lagoon, ground water) which can be expected to exhibit the average properties of the universe or whole. (CFR 260.10)

Homogeneous: uniform structure or composition throughout. (Webster's 7th Collegiate Dictionary)

After studying the above definitions and applying them to waste characterization, it becomes apparent that sampling of an ideal homogeneous waste will always result in a sample that represents the properties of the waste (assuming that the sampling process itself does not introduce contamination or allows for selective loss of waste components).

Heterogeneous: consisting of dissimilar ingredients or constituents. (Webster's 7th Collegiate Dictionary)

Unfortunately, many of the real-world wastes are not homogeneous, but are heterogeneous, which makes collection of a representative sample a challenging task. Prior to discussing the representativeness of heterogeneous wastes, the following points can be made regarding homogeneity and heterogeneity.

- Homogeneity and heterogeneity are diametric terms.
- Homogeneity and heterogeneity are relative to objectives.
- Homogeneity and heterogeneity are related to spacial and temporal distribution.
- Homogeneity and heterogeneity are sample size dependent.

A determination regarding the homogeneity and heterogeneity of a waste is made by comparing the visual, physical or chemical composition or properties of different samples. The more heterogeneous a waste is, the less homogeneous it is and vise-versa. Thus, when either the homogeneity or heterogeneity of a waste is defined the other is also defined. This relationship should be kept in mind, since for the sake of readability, the remainder of this discussion will often refer to only one term.

Heterogeneity is relative to objectives and perspective. A non-random mixture of fine silver nitrate powder and large crystals of silver nitrate will be considered to be heterogeneous by the analyst performing particle size determination while the analyst performing the TCLP for lead would consider the material homogeneous. Likewise, while the chemical properties of 99.99999% pure uranium are homogeneous, a nuclear chemist may consider it highly heterogeneous on an isotopic level.

The spacial or temporal distribution of dissimilar waste constituents or concentrations gradients can also affect the measured heterogeneity of a waste. If a representative distribu is spread over a greater area or length of time than that represented in a sample, then sample to sample heterogeneity will be greater than when a sample encompasses the distribution of the dissimilar waste components or concentration levels. For example, the measured heterogeneity of an effluent, whose concentration of phenol linearly cycles from 100ppm to 5ppm and back to 100ppm every two minutes, will be a function of what portions, or if the entire cycle is included in sequential samples.

Assuming that the concentration of the target parameter varies with particle size, sample to sample heterogeneity will depend on whether a sample is not only large enough to accommodate all the different sized constituents of a material but will also depend on whether the sample is large enough to accommodate representative amounts of the various sized constituents of a waste. To better understand this concept consider a 2 liter waste container that has one gram nuggets of cadmium randomly distributed through-out an otherwise homogeneous and cadmium free matrix. If one gram samples are collected and analyzed for cadmium, t' sample to sample heterogeneity of the waste will vary dramatically and will depend upon whether the sample did or did not contain a cadmium nugget. However, the heterogeneity will appear to be substantially less if 30 gram samples are collected and analyzed in total for cadmium. Thus the measured heterogeneity of the same waste varies according to the size of the sample. That is why for the following discussion, unless otherwise indicated, the sample size used to determine heterogeneity of a waste will be the analytical sample size. Heterogeneity of a waste will be measured according to the ability of an *unaltered analytical sample* to reproducibly represent the average properties and/or chemical constituents of the waste unit of interest. (The *analytical sample* is the mass/volume of sample submitted to analysis not the field sample, e.g. the 1 to 2 gram sample required by the acid digestion specified in Method 3050 . *Unaltered* means that the sample was not subjected to homogenization or pulverization steps. A *waste unit* is the population or unit of waste that is being subjected to evaluation, e.g. drum, a wastepile or landfill).

Representative Database: a database, generated by the collection and analysis of more than one sample, which together represent the average properties of the universe or whole.

It is often impossible to collect a single sample which is representative of a heterogeneous waste. The average properties of a heterogeneous waste are better represented by collecting a number of waste samples such, that all portions of the waste under study have an equal chance of being sampled. Analyses of these samples can be compiled into a database which should be representative of the waste. It is more correct to think of a representative sample in terms of a representative database, which is closer to the statistical use of the term, sample.

In light of all the attention directed towards heterogeneous waste, it is important to note that from a sampling perspective, the significance of heterogeneity is not in this quality, itself, but in the fact, that heterogeneity hinders or prevents the generation of a representative database. The need for representativeness is the driver behind the interest and studies into heterogeneous waste characterization.

Random: being a member of , consisting of, or relating to a set of elements that have a definite probability of occurring with a specific frequency; being or related to a set whose members have an equal probability of occurring. (Webster's 7th Collegiate Dictionary)

Randomness is a critical factor in the characterization of hazardous waste. Accurate characterization requires that the sampling process reflect the randomness or lack of randomness of the waste. If the waste parameter of interest is distributed in a random fashion a proper randomly designed sampling program should generate a representative set of samples. If the waste parameter is not randomly distributed (refer to discussion of strata in the following section) then a simple random sampling plan may not accurately characterize the waste.

TYPES OF HETEROGENEOUS WASTE

If the average properties of the waste unit of interest is not reproducibly represented in analytical samples, then the material is considered heterogeneous. Such heterogeneous wastes can exist in many forms;

RANDOM HETEROGENEOUS

- Randomly heterogeneous in composition/property.
- Randomly heterogeneous in particle size.
- Randomly heterogeneous in particle size and composition/property.

NON-RANDOM HETEROGENEOUS (STRATIFIED)

- Non-randomly heterogeneous in composition/property.
- Non-randomly heterogeneous in particle size.
- Non-randomly heterogeneous in particle size and composition/property.

EXCESSIVELY NON-RANDOM HETEROGENEOUS (EXCESSIVELY STRATIFIED)

- Excessively non-randomly heterogeneous in composition/property.
- Excessively non-randomly heterogeneous in particle size.
- Excessively non-randomly heterogeneous in particle size and composition/property.

Assuming that all particle sizes in the waste can be accommodated by the analytical sample size and that the analytical method is applicable to all waste constituents, then randomly

and non-randomly heterogeneous waste can be characterized by a database generated from the collection and analysis of random or systematic samples. Regarding non-randomly heterogeneous waste a more precise estimate of mean (representative) concentrations can be obtained with a stratified sampling approach.

Excessively non-random heterogeneous wastes are defined as those wastes that have such dissimilar components, that it is not practical to use traditional sampling approaches to generate a representative database. Nor would the mean concentrations reflected in such a database be a useful predictor of a given subset of the waste that may be subjected to evaluation, handling, storage, treatment or disposal, (i.e. the level of uncertainty is too great).

> **Stratum:** a portion or subgroup having a consistent distribution of the target parameter, and a different distribution than the rest of the waste.

Waste strata can be thought of as different portions of a waste population, which may be separated in time or space or by waste component, and each portion has internally similar concentration distributions of the target parameter, through-out. A landfill may display spatially separated strata, since old cells may contain different wastes than new cells. A wastepipe may discharge temporally separated strata, if night shift production varies from the day shift.

There are wastes which do not display any identifiable temporal or spacial stratification, yet the target compound distribution is excessively erratic. For these wastes it may be helpful to consider a third type of stratification - stratification of the waste by waste component.

> **Component:** A waste component is any identifiable article, discrete unit or constituent of the waste, which is randomly or non-randomly distributed through-out the waste.

Stratification by waste component is easily applied to wastes that contain easily identifiable particles such as large crystals or agglomerates, rods, blocks, gloves, pieces of wood, concrete, etcetera. Strata separated by waste component is a different but key concept. Separating a waste into strata according to waste components is useful when a specific kind of waste component is not randomly distributed through-out the waste and when a contaminant or property of interest is correlated with the waste component. This type of strata is different since the components are not necessarily separated in time or space but are usually intermixed and the properties or composition of the individual components are the basis of stratification. For example, automobile batteries which are mixed in an unrelated waste would be a waste component which could constitute an individual strata if lead was a target parameter. If one were to sequester the batteries they would have a consistent distribution which was different from the rest of the waste. However, if the concentration of the target parameter is similar in different components and the particle size is such that all the components are represented in the chosen sample size, then there is no purpose in stratifying by waste component. Strata by component is an important mechanism for understanding the properties of excessively non-random heterogeneous waste and for designing appropriate sampling and analytical efforts for these types of waste.

Table 1 summarizes mechanisms for segregating strata as well as the discriminating qualities of strata. Since the mechanism and qualities are independent of each other there are a total of 9 possible types of stratified wastes.

TABLE 1. Strata Considerations

Mechanism for Segregating Strata	Discriminating Qualities
Spacial	Composition/Property
Temporal	Particle Size
Component	Composition/Property and Particle Size

The contamination in different strata are often generated by different processes. The different geneses of the contamination will usually result in a different concentration distribution and mean concentrations. Thus, each strata will have their own distribution and mean concentration levels. If a waste having strata of distinctly different concentration distributions is sampled using a simple random sampling approach, the concentration distributions of the different strata may result in a bi-modal or multi-modal distribution. Homogeneous and randomly heterogeneous waste, which are not stratified will display a normal unimodal distribution. See Figure 1.

Non-randomly heterogeneous waste will usually have a limited number of strata which can be identified and sampled individually or sampled as one waste. When different strata are sampled as one population, the properties of the different strata are averaged. If the different strata are similar in composition, then the average concentrations will be a good predictor of composition for subsets of the waste and will often allow the program objectives to be achieved. As the difference in composition between different strata increase, the average values become less useful in predicting composition/properties of individual portions of the waste, which may be handled separately. In this later case, it is advantageous to sample the individual strata separately and if an overall average of waste composition is needed, it is best calculated mathematically using statistical information from each strata.

Excessively non-random heterogeneous waste has numerous strata, each of which contain different distributions of contaminants and/or particle sizes, such that an average value for the

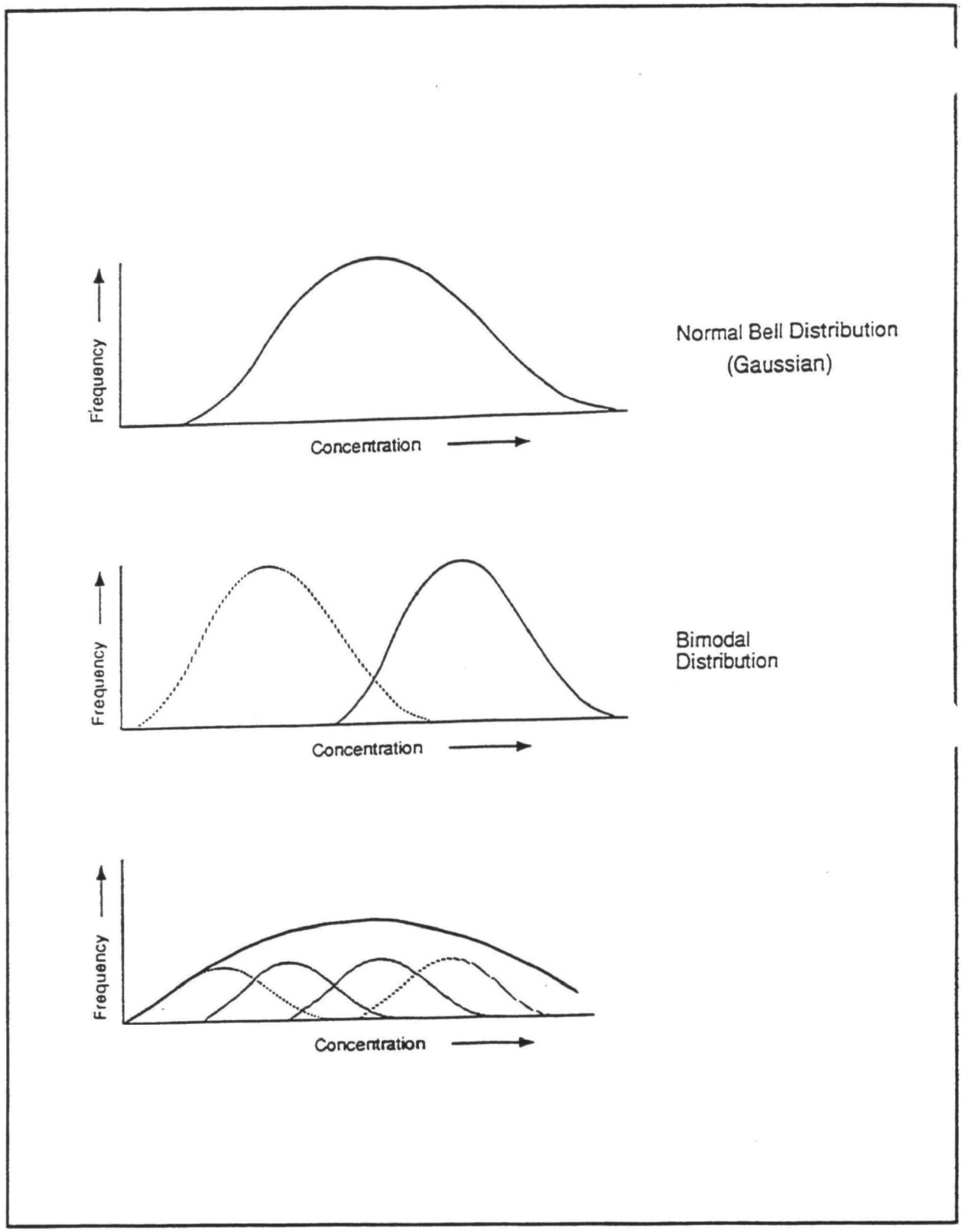

Figure 1: Types of Concentration Distributions

waste would not be useful in predicting the composition or properties of individual portions of the waste (i.e. statistically speaking the variance and standard error of the mean will be large). The line of demarcation between non-random heterogeneous and excessively non-random heterogeneous waste is not hard and fast. A waste can be considered excessively non-random heterogeneous when mean values will not be representative of most portions of the waste and when it is so heterogeneous that the waste cannot be cost-effectively sampled using traditional sampling approaches and meet the project objectives.

A theoretical example of an excessively stratified waste could consists of commingled waste from:

Source A - which generated a waste of varying particle size with a mean antimony concentration of 20ppb and a standard deviation of 7ppb.

Source B - which generated a waste of varying particle size with a mean antimony concentration of 7000ppm and a standard deviation of 5000ppm.

Source C - which generated a waste of varying particle size with a mean antimony concentration of 3% and a standard deviation of 2%.

Source D - which generated a waste of varying particle size with a mean concentration of 81% and a standard deviation of 17%.

To further complicate the above waste, imagine the waste being commingled with other materials of various particle size that may or may not contain various contaminants in addition to antimony.

To improve readability, the remainder of the document will refer to non-random heterogeneous waste as *stratified waste* and excessively non-random heterogeneous waste as *excessively stratified waste.*

Figure 2 presents a graphical depiction of the types of special distriubtions and heterogeneity. Letters are waste constituents which are identical to other constituents of the same letter in terms of the composition, property, or particle size of interest. Different waste constituents are designated by different letters.

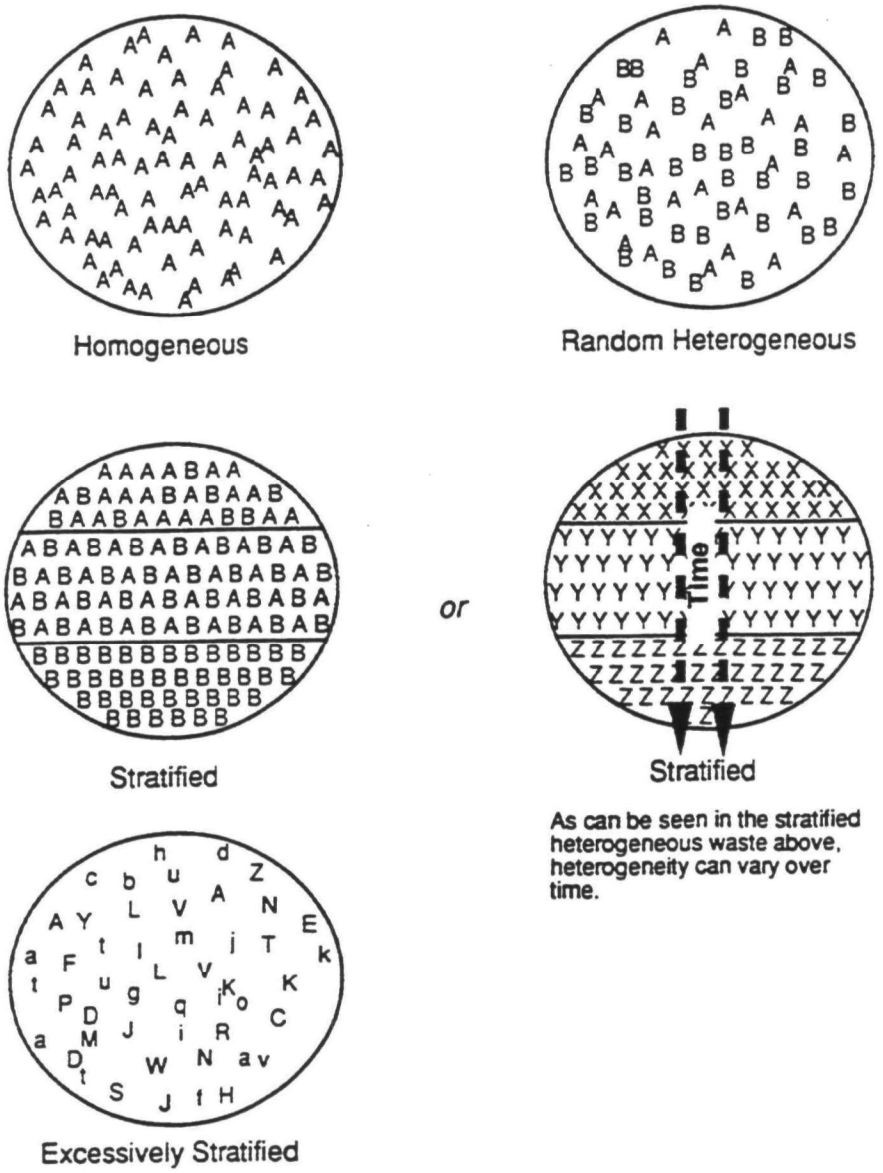

Figure 2: Types of Positional Distributions for a Waste Unit Such as a Landfill

This figure depicts the process by which a waste is classified as a particular waste type. This classification will be based upon knowledge of the waste, observations, or ideally - preliminary sampling of the waste.

If no significant variation is detected between random samples, the waste can be considered homogeneous. The waste is a heterogeneous waste if there is variation between individual samples

If the waste constituents are randomly distributed through-out the waste, the waste would be a randmoly heterogenous waste. If information describes a correlation between waste variation and time or spatial variations or with certain waste components or particle size, then the waste would be classified as a stratified or excessively stratified waste. Wastes that can be cost-effectively sampled to meet project objectives would be classified as stratified wastes. Those wastes which consist of such strata that they cannot be cost effectively sampled are classified as excessively stratified waste

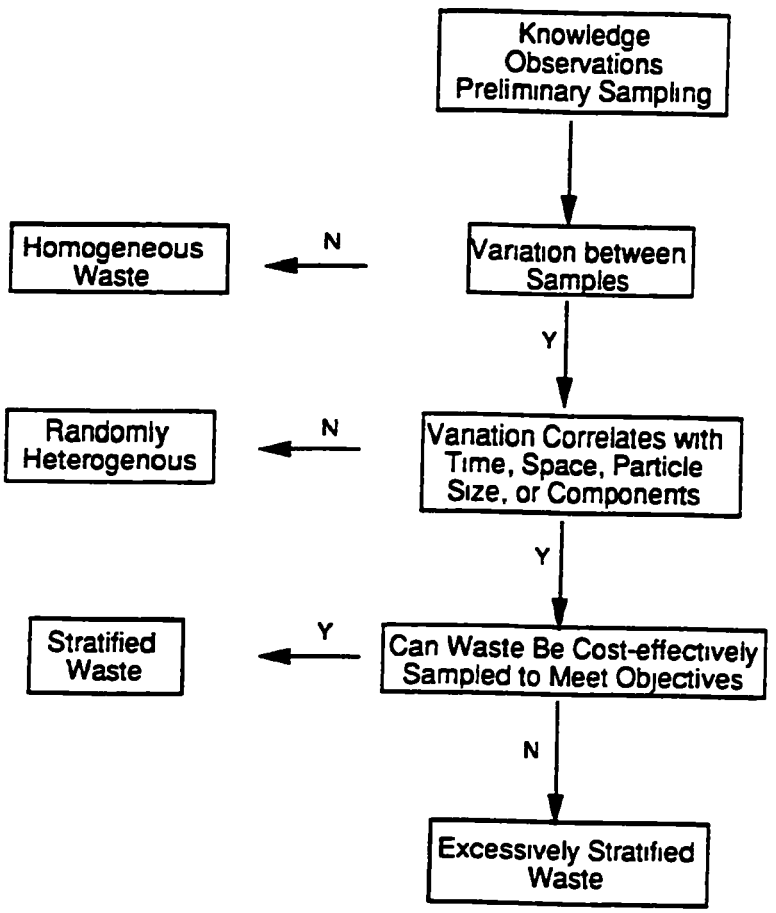

Figure 3: Process for Classifying Wastes

Table 2 details some of the issues and concentration distributions that are pertinent to the different types of heterogeneous wastes. For example, sample size, the number of samples sample locations are not an issue when sampling a homogeneous waste, while they are critical issues when attempting to collect a representative sample of a heterogeneous waste.

PARTICLE SIZE AND SAMPLING THEORY

As a result of a continuing need to improve environmental data, it has been recognized that sampling is presently the greatest contributor to imprecision and inaccuracy. To minimize this error, many have correctly turned to the mining industry, which has a relative wealth of sampling theory and expertise. The theory and applicable expertise of the mining industry has recently been documented by a recognized expert on sampling, Francis F. Pitard, (Pitard, Francis F., "Pierre M. Gy's Sampling Theory and Sampling Practice, CRC Publishers). This theory, which not only applies to field sampling but also to subsampling performed in the laboratory - determines sample mass/volume as a function of the maximum particle size of the material being sampled..

Based on the maximum particle size of a material, sampling theory suggests minimum sample sizes (refer to Table 3.) and particle size reduction of the sample to minimize sampling error. For example, a waste having a maximum particle size of 0.5 inches would require collection and analysis of a minimum sample size of 2 Kilograms to keep the sampling error less than 17%. Since a 2 kilogram sample is too large to be subjected to analysis, it would have to be subjected to particle size reduction prior to analysis.

It would be very costly to routinely use existing sampling theory to minimize sampling error. These increased costs would be driven by the large sample sizes and the requirement to reduce particle size.

The large sample sizes, specified to minimize sampling error, would increase costs since;

- Sample containers and coolers would be larger and more numerous
- Larger samples would be shipped
- Larger sample storage and sample handling areas would be needed
- Large volume of unused samples would increase disposal costs.
- Health and Safety costs associated with exposure to larger samples.

Particle Size Reduction, (PSR), also has a substantial impact on cost because of the manpower and capital investment for the required crushing and pulverizing equipment. In addition the following are reasons for concern if particle size reduction was to be implemented on a large scale;

- Laboratory contamination from fines.
- Damage to instrumentation from fines.
- Difficulty in cleaning PSR equipment to prevent cross-contamination.
- Loss of volatile and labile compounds and elements.
- Generation of a sample which has different properties and thus yields different analytical results than the original sample.
- Increased Health and Safety Costs

Fortunately, the costs of employing large sample sizes and particle size reduction can often be minimized if the waste history and Data Quality Objectives are communicated to and discussed with the lab staff and field personnel.

TABLE 3. SUGGESTED SAMPLE SIZE BASED ON MAXIMUM PARTICLE SIZE ACCORDING TO PIERRE GY'S SAMPLING THEORY

SUBSAMPLE SIZE (grams)	MAXIMUM PARTICLE SIZE (centimeters)
1	.1
2	.13
3	.14
4	.16
5	.17
10	.21
20	.27
30	.31
40	.34
50	.37
75	.42
100	.46*

*The Toxicity Extraction Procedure and the Toxicity Characteristic Leaching Procedure allow samples to contain particles as large as .95 centimeters.

Waters and soils can be contaminated in numerous ways, the most common mechanisms being; direct discharge of the contaminant onto the soil or water and atmospheric fall-out from fires, fugitive emissions, vents and stacks. Soils can also become contaminated from adsorption of groundwater or surface water contaminants while waters can become contaminated by mixing with contaminated waters or by leaching contaminants from contaminated soils or wastes.

Wastes can be contaminated by many mechanisms during generation or by mixing with other wastes or further contaminated by the mechanisms described in the previous paragraph. The different types of contaminants are aqueous liquids, non-aqueous liquids, gases, small to large particles and multi-phased mixtures.

How a contaminant will be dispersed in the matrix will be determined by the mechanism of contamination, the type of contamination, the surface area and adsorptive properties of the sample matrix, gravity, physical manipulation and the physical size of the contaminant at the time of contamination. (Eventually, fate and transport will substantially affect dispersion of contaminants.) The physical size of the contaminant can be referred to as the **contaminant** unit and can be measured in centimeters. Liquids, dissolved contaminants and gases have a contaminant unit on a **molecular** or **atomic level**, (e.g. atoms can be on the order of 1 X 10-8 centimeters in diameter). Solids and suspended solids contaminate on a **microscopic** to **macroscopic** level, (i.e. their Contaminant Unit is equal to their particle size).

Particle size can have an impact on sampling error even when the contaminant unit is on the atomic scale, if matrix particles are of different sizes and have different adsorptive properties. If adsorptive properties are similar for different size particles, then the dramatically larger surface area of smaller particles/volume would result in more atomic scale contaminants being adsorbed to smaller particles. (Thus one could eschew particle size reduction, (PSR), and still employ a smaller subsample size by excluding large particles, if the ease of using a smaller sample size out-weighed the chance of a false-high concentration.)

When the contaminant unit is on a larger, particle scale then adsorptive properties of the matrix will not affect contaminant distribution in the sample. For microscopic and macroscopic particles, an accurate representation in the subsample will be affected by the number of particles and the relative size of the particles to the subsample size. Microscopic particles will have a contaminant unit substantially smaller than the sub-sample size and these small particles should not be discriminated against during subsampling.

When the contaminant unit is macroscopic and approaches the size of the sample, the error in sampling will increase, if large sample sizes or PSR is not employed. If the largest particle size in the sample correlates with the contaminant unit, then the large sample size specified by sampling theory pertains to the waste. As the contaminant unit becomes smaller as

compared to the size of the sample, the greater the opportunity to avoid PSR and to selectively exclude larger particles from the sample without introducing substantial error., (If desired weight of the excluded particles can be used in the calculation of a final concentration. However, this could result in a false low result, since the contamination, if any, associated with the large particles is not accounted for.)

The mechanism of contamination, the type of contaminant and the contaminant unit will impact the complexity and potential error of sampling. These impacts may be better understood by reading the following examples.

Example 1

TYPE OF CONTAMINANT: Particles contaminated with lead

MECHANISM: Direct discharge of particle to soil

CONTAMINANT UNIT: Particles as large as 0.3 centimeters

AVERAGE LEAD CONCENTRATION: 10ppm

HISTORY: A manufacturer of lead-alloy babbitt lining for bearings, stored its machining waste in piles behind it facility. Periodically the machining wastes would be recycled into new babbitt linings. After 50 years of employing this practice, the soil became contaminated with particles of babbitt having a maximum particle size of 0.3 centimeters. The contaminated soil consisted mainly of fine silts with less than 10% consisting of gravel with stones having diameters ranging from 2 to 3 inches. The sampling team uncovered a problem when they referred to the specified "minimum sample size" for 3 inch particles (i.e. 450kg). The problem of large sample size was avoided, since the sampling team was aware of the "Type of Contaminant" and the "Mechanism" of contamination and the "Contaminant Unit". Knowing that lead contaminated particles were discharged directly to the soil, the sampling team was able to discard the large stones with the realization that by discarding the stones, the resulting lead concentrations would be a worst case. (The small lead contaminated particles would preferentially exist in the fine silts as opposed to being adsorbed to the large stones and if lead had dissolved, the dissolved lead would tend to adsorb to the silt which has the much larger surface area.) The sampling team collected approximately 250 mls of soil and delivered them to the laboratory for analysis.

The analyst referred to sampling theory (refer to Table 3.) and determined that the minimum sample size for a sample with a maximum particle size of 0.3 centimeters was 30g. (The small percentage of larger gravel was not used to determine the maximum particle size.)

The analyst split the sample into 30g aliquots and randomly chose one for analysis. Since the analyst did not have particle size reduction equipment and the largest sample volume he could analyze was approximately 10 grams, he split the chosen aliquot in thirds and analyzed all three for lead. After analysis, the concentrations were averaged.

Example 2

TYPE OF CONTAMINANT: Ionic lead dissolved in an aqueous solution (Atomic scale)

MECHANISM: Direct discharge to soil

CONTAMINANT UNIT: Atomic scale, (approximately 1 X 10-8 centimeters for atoms and somewhat larger for ion diameters)

AVERAGE CONCENTRATION: 10 ppm

HISTORY: The above described babbitt manufacturer also employed an acid-treatment process. The aqueous waste was discharged into a tank which allowed the lead contaminated water to leach into the surrounding soils. The surrounding soils consisted of fine silt with 15% of its volume consisting of stones ranging in size from 0.25 to .5 inches. The sampling team collected a 2 Kilogram sample to minimize the sampling error. The samples were sent to the laboratory for analysis. Since the analyst knew that the soil was contaminated by lead on an atomic scale, he discarded the small stones, split the sample and analyzed a 2 gram aliquot. (The analyst knew that the much higher surface area of the silt would have adsorbed orders of magnitude more lead than the stones, so that this approach yielded a worst-case analysis. To check this hypothesis, the analyst performed an acid leach on a few stones and no detectable quantities of lead were measured.)

The above examples show how, with knowledge of the type and mechanism of contamination and the contaminant unit the sampling team and the analyst can decrease the costs of handling large samples and particle size reduction without substantially increasing sampling

error. Without the site history, large sample sizes or particle size reduction would have required or one would risk the introduction of a substantial error into the measurement process. Thus communication with the analyst regarding the type and mechanism of contamination is an important means of controlling costs and minimizing errors.

The above sampling strategies are not ideal since they allow for assumptions and interpretation and these assumptions and interpretations could lead to substantial intentional and unintentional errors. However, the above sampling strategies are more practical, more likely to be implemented and may be a significant improvement over the alternatives. Lastly, when these sampling strategies are implemented by an experienced personnel who are aware of the DQOs, site history, the types and mechanisms of contamination and the contaminant unit, the errors associated with interpretation and assumptions should decrease substantially.

It is important to note, that application of these sampling strategies to wastes is much more difficult than for soils and subsampling of wastes may frequently require a strict application of sampling theory. Wastes are a more difficult media, since the complexities waste generation often preclude knowledge of how a contaminant of interest is dispersed within a waste and whether distribution will be a function of particle size.

EXCESSIVELY STRATIFIED WASTES

The remainder of this discussion will concentrate on excessively stratified wastes, the most difficult wastes to characterize. Compositional or particle size heterogeneity or a combination of both compositional and particle size heterogeneity can be the cause of excessive stratification. This discussion will address each of these causes and will concentrate on sampling issues, since sampling is now recognized as the greatest contributor to imprecision and bias.

However, before addressing the different causes of heterogeneity, it will be useful to consider an issue which should be common to all waste characterization efforts.

The first step in characterizing any heterogeneous waste is to gather all available information on the;

- Need for waste characterization,
- Objectives of waste characterization,
- Pertinent regulations, consent orders, and liabilities,
- Sampling, shipping and laboratory health and safety issues,
- Generation, handling, treatment and storage of the waste,
- Existing analytical data and exacting details on how it was generated,
- Treatment and disposal alternatives.

These types of information will be used in the planning of the sampling and analytical effort. The planning process should be detailed and address the issues defined in EPA's data quality objective (DQO) process. The different disciplines (e.g. sampling, chemistry, engineering, statistics needed to properly understand and exploit the above types of information must be present during the planning process. If enough information is available, the planning process will uncover the existence of excessive stratification which will prevent achievement of objectives. If information is lacking, a preliminary sampling effort would be advisable, and if done properly should detect the existence of excessively stratified wastes.

Excessively stratified waste can not be cost-effectively characterized by traditional methods and this fact usually becomes apparent during the planning process. The following discussion will consider approaches which in effect will lessen the level of stratification and allow for more cost-effective characterization. Some of these approaches will require changes in objectives, waste handling or disposal methods, and some will require compromises, but all approaches will require the above types of information.

Of the following types of excessively stratified waste, the difficulty in characterizing waste increases from those that have strata based solely on particle size, to those which consist of compositional strata to those which have strata of varying composition and particle size. In fact, the difficulty in characterizing waste, which consists of strata of different particle size, is simply a matter of determining that composition is constant across different particle sizes. If the composition is found to be constant across the different particle sizes, the waste should be easily characterized.

The more problematic types of waste, which have enumerable strata of different composition or a combination of different composition and particle size are much more difficult to characterize. The approach to characterizing these wastes usually has to be determined on an individual and unique basis.

Excessive Strata of Different Sized Particles

Wastes having excessive stratification due only to different sized particles will definition have the same composition or property (i.e. homogeneous or randomly heterogeneous) through-out its different strata. The strata can be separated in space or in time. Unless one is attempting to measure particle size, this waste is the simplest of the excessively stratified waste types to characterize. All particles in these types of wastes are usually generated by the same process, (e.g. smelter slag and the previous example of silver nitrate powder and crystals), which is the reason for similar composition across all particle sizes.

The complexity of dealing with these types of wastes is in proving that the waste has similar composition, (i.e. mean levels and concentration distribution of the parameter of interest) across the varying particle sizes. This determination can be made by using knowledge of the waste or by sampling the different sized particles to determine if there are significant compositional differences. If the determination is made using knowledge of the waste, it is advisable to at least perform limited sampling to confirm the determination.

The characterization process is greatly simplified, once a determination has been made that the waste has similar composition or properties across the various particle sizes. The sampling and subsequent analysis can be performed on particles which are readily amenable to the sampling and analytical process and the resulting data can be used to characterize the waste, in its entirety.

It is important to periodically verify the assumption that the different particle sizes are composed of materials having the same concentration levels and distributions of the contaminant of interest. This verification is especially important when there are any changes to the waste generation, storage, treatment or disposal processes. Similarity of composition between particles has to be verified for each parameter of interest. The effect of different particle size must also be considered when measuring properties such as the Toxicity Characteristic Leaching Procedure (TCLP) .

Excessive Strata of Different Composition or Composition and Particle Size

Wastes having excessive stratification due only to composition or property will have similar particle size through-out its different strata. The strata may be separable in space, time or by component or source. Stratifying the waste should simplify the characterization process.

Wastes having excessive stratification due to both composition/property and particle size are usually the most difficult wastes to characterize. The strata can be separated in space, in time, or by component or source.

Figure 4. summarizes an approach to characterizing these types of excessively stratified wastes. If a waste is excessively stratified, traditional methods of sampling will not allow objectives to be cost-effectively achieved. To cost-effectively sample an excessively stratified waste, one must use a non-traditional approach. The non-traditional approach may involve modification to the sampling, sample preparation or analytical phase of the process. If after

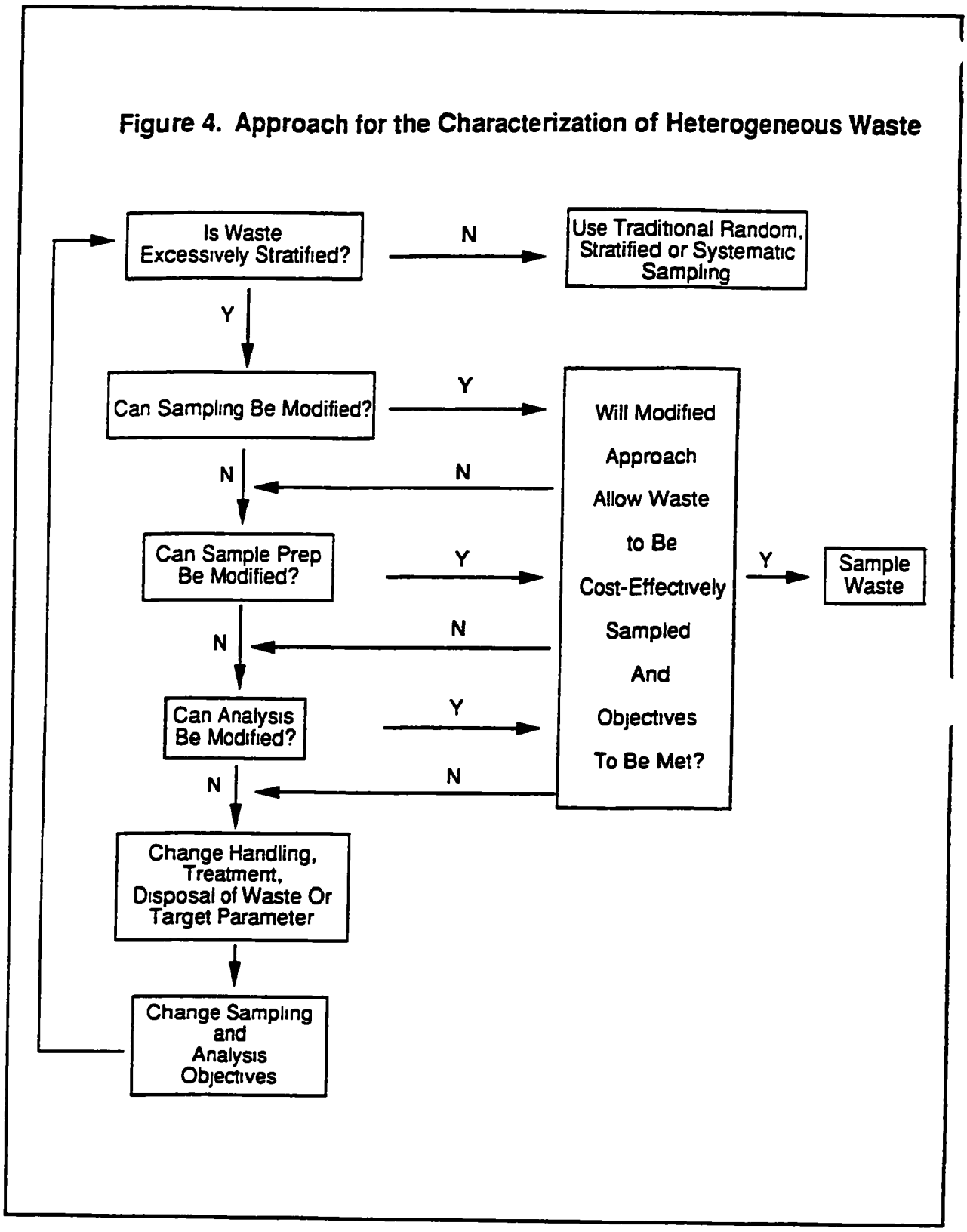

Figure 4: Approach for the Characterization of Heterogeneous Waste

modifying the approach to sampling and analysis the objectives still can not be achieved in a cost-effective manner, then the original plan of waste handling, treatment or disposal has to be examined and changed so the waste can be characterized according to new and achievable objectives.

The following subsections discuss approaches that can be employed to make excessively stratified waste more amenable to a cost-effective sampling approach.

Design of the Sampling Approach

The first efforts to resolve the difficulty in characterizing an excessively stratified waste are usually focused on the sampling aspects of the project. This is a logical place to start and, if a successful sampling approach is designed, the project objectives can be cost effectively achieved.

The difficulty in sampling excessively stratified waste can result from:

1) Various particle sizes and waste consistency which makes sampling difficult and traditional sampling approaches cost prohibitive.

2) Extraordinary concentration gradients between different components or enumerable strata that lead to such excessive variance in the data, that project objectives can not be achieved.

3) Wastes which exhibit both of the above properties.

A strategy for designing a sampling plan for such excessively stratified waste includes the following five steps;

1) Select the target parameters.

2) Determine whether these parameters are correlated with; particle size space time components, or sources.

3) Determine if any waste components or strata can be eliminated from sampling because they do not contribute significantly to the concentration of the target parameter.

4) Determine if small particles in a stratum represent the stratum as well as large more difficult to sample particles. If yes, sample the smaller particles and only track the volume contribution of the larger particles. (See Particle Size and Sampling Theory).

5) Determine if contamination is innate or surface adsorbed. Is the contamination surface adsorbed which would allow the material to be representatively sam by wipe sampling? Can large particles be wiped and smaller particles extracted, leached or digested. Can waste be stratified according to impervious and non-impervious waste and sampled and analyzed accordingly?

To understand how this strategy would work, consider a hypothetical scenario - a storage area containing 4000 drums of waste generated over a 15 year period. The drum contents are excessively stratified and contain a myriad of wastes from process waste; destruction and construction debris such as wood, concrete; lab wastes including broken glassware, paper, empty bottles; etcetera. The appearance of the combined drum contents could best be described as a municipal landfill in drums which appears impossible to characterize.

1) WHAT ARE THE TARGET PARAMETERS?

None of the waste contents are known to be listed waste so the target parameters are the TCLP and other hazardous waste characteristics. In addition, groundwater modeling has indicated that the storage area is the source of a plume contaminated with solvents and beryllium.

2) ARE THE TARGET PARAMETERS CORRELATED WITH AN IDENTIFIABLE STRATA OR SOURCE?

The source of beryllium is traceable to one process, whose waste should be easily identifiable if drum markings are not legible enough to determine the source. The solvents are likewise traceable to a machine shop which would have disposed of its waste in easily identified drums.

Testing will have to be performed to determine if there is any correlation with particle size, space, time or components in the waste.

3) CAN ANY WASTE COMPONENTS OR STRATA BE ELIMINATED?

Historical information indicated that 400 drums of construction debris were generated during construction of a new warehouse. The information indicates that the virgin nature of the materials may make these drums candidates for not sampling or less intensive sampling.

Likewise, the source of beryllium contamination is a beryllium sludge which exists in drums by itself or in drums commingled with shredded packing material and laboratory wastes that were generated during physical testing of the beryllium product. If the materials commingled with the beryllium waste are known not to be a source of contamination, the commingled

material can be discriminated against during sampling and only the beryllium sludge sampled and the volume contribution of the commingled material noted.

4) ARE CONTAMINATION LEVELS CORRELATED WITH PARTICLE SIZE?

Some of the older beryllium sludge has dried and formed a cementaceous aggregate of different particle sizes. Since the sludge is known to be homogeneous within a batch by process knowledge and preliminary sampling data, sampling can be restricted to the more easily sampled - smaller particles sizes.

5) IS CONTAMINATION INNATE OR SURFACE ADSORBED

The waste from the machine shop consists of varied material from fine metallic filings to large chunks of metal and out-of-specification metal product. Since the only contamination in the machine shop is solvents and cutting oils and the waste matrix is impervious, contamination is surface adsorbed in nature. Thus sampling of these wastes will consists of the sampling of fines which will be subjected to extraction, wipe sampling of the large metallic objects and notation of the volume contributions of the different particle sizes.

It is essential that all assumptions , (i.e. any correlations), be verified by at least knowledge of the waste and preferably confirmed by exploratory sampling and analyses.

In the above hypothetical case, the proposed strategy for characterizing the 4000 drums resulted in:

The identification of two large strata that constitute the majority of the waste (i.e. the beryllium sludge and the solvent and cutting oil contaminated machine shop waste).

The elimination of the need to sample 10% of the drums, (i.e. the construction debris), if preliminary testing verifies waste disposal information.

Simplified sampling of the beryllium commingled waste by restricting sampling to the beryllium sludge and not the other commingled materials.

Simplified sampling of the cementaceous beryllium sludge by limiting sampling to the more easily sampled small particles.

Simplifying the sampling of the machine wastes since the source of contamination is surface adsorbed and not innate to the waste materials.

A less expensive and doable sampling design which will result in a more precise estimate of the mean and will generate strata specific information which will be valuable if the strata are eventually treated separately.

The following subsections describe additional strategies that can be employed if the above sampling strategies are not applicable to a waste or if they are applicable but by themselves will not allow the project objectives to be cost-effectively met.

Modification of the Sample Preparation Method

As discussed in the Introduction, heterogeneity is analytical sample size dependent. The greater the particle size and the greater the variance of the concentration of the target parameter, the greater the heterogeneity for a given analytical sample size. To minimize the measured heterogeneity and to accommodate large particle sizes, traditional sample preparatory methods can be altered.

In the laboratory, the term "sample preparation" is commonly meant to include two separate steps; 1) the subsampling of a field sample to generate an analytical sample, and 2) the preparation of the analytical sample for subsequent analysis.

Regarding subsampling, the previously discussed logic for field sampling (refer to Section 3.1.1) is also applicable for the generation of analytical samples. That is, knowledge of concentration distributions within the waste can be used to simplify subsampling by:

1) Eliminating any waste components or strata that do not contribute significantly to the concentration of the target compound;

2) Discriminating against large particles and only select small particles if small particles represent the waste as well as large particles; and.

3) Surface wiping larger particles and extracting or digesting fines if surface contamination is the source of the target parameter.

If the above approaches are not applicable to a field sample, the field sample will have to be subjected to particle size reduction (PSR) prior to subsampling or the sample preparation method will have to be modified to accommodate the entire field sample.

PSR is useful for handling field samples, which have particles too large for proper representation in an analytical subsample. The intent of PSR is to decrease the maximum particle size of the field sample so that the field sample can then be split and or subsampled to gen a representative subsample. The difficulties in applying PSR to waste samples are:

1) Not all materials are easily amenable to PSR (e.g. stainless steel artifacts);

2) Adequate PSR capabilities and capacities do not normally exist in environmental laboratories;

3) PSR can change the properties of material (e.g. leachability)

4) PSR can be a source of cross-contamination; and

5) PSR is often not applicable to volatile and labile compounds.

Modification of sample preparative methods can include the extraction, digestion or leaching of much larger sample masses than specified. The advantage of this approach is that the resulting extract, digestate or leachate are relatively homogeneous which simplifies subsampling. This approach is particularly important for volatile organic compounds which may suffer from substantial losses if subjected to PSR. For volatile organic compound analysis, larger portions of the wastes can be subjected to methanol extraction or possibly the entire field sample could be subjected to heated headspace analysis as one sample or as a series of large aliquots.

Prior to modifying a sample preparatory method, especially a method associated with a property such as the Toxicity Characteristic Leaching Procedure (TCLP), it is advisable to consult the end-user of the data and the pertinent regulator if appropriate.

Modification of Analytical Method

The analytical phase of a sampling and analytical program allows another opportunity to simplify the characterization of an excessively heterogeneous waste. Examples of different classes of analytical methods are ;

Screening methods,
Portable methods,
Field Laboratories methods,
Non-intrusive methods,
Innovative methods, and
Fixed laboratory methods.

Screening, portable and field laboratory methods have the distinct advantage that they allow for the cost-effective analysis of more samples. These methods not only generate more precise data but the greater number of samples make it easier to detect correlations between concentration levels and waste strata or components. Also some screening methods may analyze a larger sample volume than what is traditionally submitted to a fixed laboratory.

Non-intrusive methods can be useful when there are health and safety issues regarding exposure to the waste. These methods may also be used to qualitatively or semi-quantitatively evaluate large volume wastes.

An EPA Document entitled "Characterizing Heterogeneous Wastes EPA 600/R-92/033" has a solid discussion of non-intrusive methods and new methodologies that are being developed for the characterization of heterogeneous wastes.

Modification of the Waste Handling, Treatment Disposal Plan

If the modifications discussed in the previous subsections are not applicable to a given waste or when they are applicable but still do not allow the objectives to be cost-effectively met, then the reasoning behind the original program must be examined. The original need behind the waste characterization objectives has to be examined and an approach for simplifying the characterization process must be devised.

For example, assume that the need behind waste characterization objectives for a ce program was the common requirement to determine if a waste is hazardous prior to waste disposal. An initial attempt to characterize the waste; 1) failed to meet the objective, 2) indicated that the waste was excessively stratified, and 3) proved that portions of the waste are hazardous. After reviewing this preliminary information and the costs to attempt a defensible characterization of the waste, it could be decided that all the waste will be assumed to be hazardous and treated as hazardous waste. Under this scenario, the needs change and now compliance with the land-ban requirements may become the issue. Assume that the waste was incinerated, then the less heterogeneous and more easily sampled incinerator ash would be sampled in lieu of the original excessively stratified waste.

An other example would be a large laboratory operation that generates drums of gas-chromatography (GC) vials containing dissolved standards, solvents and numerous contaminants

extracted from samples of different origins. Although, the vial contents were individually analyzed by GC for certain parameters, the contents were not analyzed for enough parameters to characterize the waste. A quick review of this waste would preclude further and cost prohibitive analyses of every vial. Thus, an alternative approach would have to be devised. An alternative approach could require that the vials be crushed and the vial contents and the solvent used to rinse the broken vials would be collected in a receiving drum. This approach would convert drums which contained hundreds of different vials, each of which could have their own independent concentrations of contaminants, into two relatively homogeneous waste strata, i.e. solvent rinsed glass and contaminated solvent.

CONCLUSIONS

The previous discussion reviewed heterogeneity issues and proposed a single and systematic approach to the characterization of hazardous waste. This approach is based upon the evaluation of wastes in terms of their degree and type of stratification - a factor which drives the degree of difficulty in sampling a particular waste.

This discussion also proposed stratification in an additional dimension than the traditional spacial and temporal dimensions. The stratification of waste according to waste components and sources can simplify the characterization process and provide needed strata information.

This systematic approach and the additional mechanisms for stratifying waste is intended to aid in the characterization of wastes especially those that are excessively stratified.

Appendix XIII

Appendix XIII
Improper Hazardous Waste Characterizations: Financial and Compliance Implications

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QUALITY ASSURANCE IN ENVIRONMENTAL MONITORING

IMPROPER HAZARDOUS WASTE CHARACTERIZATIONS - FINANCIAL AND COMPLIANCE IMPLICATIONS

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IMPROPER HAZARDOUS WASTE CHARACTERIZATIONS---FINANCIAL AND COMPLIANCE IMPLICATIONS

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This paper is *not* a "how to" on when or under what circumstances one is required to make a hazardous waste determination, or a primer reviewing a step by step approach to rendering a proper characterization. Rather, the purpose of this paper is to illustrate why generators of waste should render accurate and well-founded determinations as to whether the material is a hazardous waste pursuant to appropriate federal and/or state requirements. The paper will also review the potential financial and compliance implications of managing nonhazardous waste as hazardous waste in New York State. When waste is classified as hazardous, its management requires the use of a manifest for off-site transportation. Utilizing a manifest results in a number of explicit and implicit protections and liabilities. The implications of using the hazardous waste manifest will be a major portion of the discussion.

The recognition of the uniform hazardous waste manifest among waste generators across the nation is probably second only to the IRS 1040 Form. Since its creation by the Environmental Protection Agency in the late 1970's, the hazardous waste manifest and the vast information network it supports, remains the cornerstone of RCRA's national "cradle to grave" hazardous waste tracking system. We will explain why the manifest possibly has more *responsibilities* than protections associated with its use.

It has been our experience that some clients firmly believe that "when in doubt" waste should be classified as hazardous. These clients assume that transporting the material as a hazardous waste, with an accompanying manifest, is always "safer", affording them some protection that ordinarily would not otherwise exist. This "protective filer" mentality has a number of enforceable regulatory liabilities, as well as financial implications, associated with it which can be burdensome when the facility actually does *not* generate any hazardous waste. These considerations are in addition to the high cost of hazardous waste disposal.

Obviously, this paper supports utilizing the hazardous waste manifest when appropriate, and fosters the concept of proper and judicious hazardous waste characterizations. However, we are opposed to using hazardous waste manifests when solely generating/transporting, nonhazardous industrial waste.

Before we discuss the financial and compliance liabilities associated with transporting manifested nonhazardous waste, we will briefly describe the hazardous waste program.

WHEN IS A WASTE A HAZARDOUS WASTE?

Section 1004(5) of RCRA defines hazardous waste as a "...solid waste, or combination of solid wastes, which because of its quantity, concentration or physical, chemical or infectious characteristics may:

(A) cause or significantly contribute to an increase in mortality or an increase in serious irreversible, or incapacitating reversible illness;
or

(B) pose a substantial present or potential hazard to human health or the environment when improperly treated, stored, transported or disposed of, or otherwise managed."

While this statutory definition is subjective, it clearly states that in order for a material to be a hazardous waste, it must first be a "solid waste", as defined by RCRA. In order for a solid waste to be defined as a hazardous waste, it must meet the following conditions:

- Is not excluded from regulation as a hazardous waste, and;
- Exhibit any of the **characteristics** of a hazardous waste, and/or;
- Be named a hazardous waste and **listed** by regulation as such, or;
- Is a **mixture** containing a characteristic waste/listed hazardous waste and a nonhazardous solid waste, unless the mixture is specifically excluded or no longer exhibits any of the characteristics of hazardous waste. A mixture containing a nonhazardous waste and a listed hazardous waste will remain a listed hazardous waste.

In the preceding section, we provided a general definition of hazardous waste. However, there are two principle mechanisms to determine whether a waste is a hazardous waste. First, one must determine if it is a "listed waste," so named because it is specifically listed as such by the EPA or a State as part of its hazardous waste regulations. Secondly, based on knowledge or laboratory analysis, one must determine if it exhibits any *characteristics* of a hazardous waste: ignitability, corrosivity, reactivity and toxicity.

CHARACTERISTIC WASTE

Ignitability

A solid waste that exhibits any of the following properties is considered a hazardous waste due to its ignitability:

- A liquid, except aqueous solutions containing less then 24 percent alcohol, that has a flash point less than 60°C(140°F);

- A nonliquid capable under normal conditions of spontaneous and sustained combustion;

- An ignitable compressed gas in accordance with Department of Transportation (DOT) regulation;

- An oxidizer per DOT regulation.

EPA's reason for initially including ignitability as a characteristic was to identify waste that could cause fires during transport, storage or disposal.

Corrosivity

A solid waste that exhibits any of the following properties is considered a hazardous waste due to its corrosivity:

- An aqueous material with pH less than or equal to 2.0 or greater than or equal to 12.5;

- A liquid that corrodes steel at a rate greater than 0.25 inch per year at a temperature of 55°C (130°F).

EPA chose pH as an indicator of corrosivity because waste with high or low pH can react dangerously with other waste or cause toxic contaminants to migrate from certain waste. Steel corrosion was chosen because waste capable of corroding steel can escape from its container.

Reactivity

A solid waste that exhibits any of the following properties is considered a hazardous waste due to its reactivity:

- Normally unstable and reacts violently without detonating;

- Reacts violently with water;

- Forms an explosive mixture with water;

- Generates toxic gases, vapors or fumes when mixed with water;

- Contains cyanide or sulfide and generates toxic gases, vapors or fumes at a pH of between 2 and 12.5;

- Capable of detonation if heated under confinement or subjected to strong initiating source;

- Capable of detonation under standard, temperature and pressure;

- Listed by DOT as Class A or B explosive.

Reactivity was chosen as a characteristic to identify unstable waste that can pose a problem at any stage of that waste management cycle.

Toxicity

The toxicity characteristic test is designed to identify waste likely to leach particular toxic constituents into the groundwater as a result of improper management.

To ascertain if a solid waste is hazardous because of the toxicity characteristic, constituents are extracted from the waste in a manner designed to simulate the leaching action which occurs in landfills. The extract is then analyzed to determine if it possesses any hazardous constituents listed in Table 1. If the concentrations of the toxic constituents are equal to or exceed the regulatory levels listed, the waste is classified as hazardous.

Characteristic hazardous wastes are defined by certain physical/chemical criteria which may require a representative waste sample analysis by the generator. The Toxicity subcategory is more likely than the other characteristics to require chemical analysis. Consequently, a waste generator must be very careful in selecting/paying for the correct protocols to characterize his waste for Toxicity.

There are financial implications tied to Toxicity wastes in two areas. First, the prescribed leaching protocol (Toxicity Characteristic Leaching Procedure - TCLP) is an expensive protocol to run. However, it may not be required if the waste is a 100% solid matrix or a solid/water matrix, for which a total constituent analysis has been performed. The extraction is also not required if the waste is a liquid with no solid phase. Thus, an understanding of when the TCLP is needed has a significant influence on the cost of waste characterization.

Secondly, it is important to compare the appropriate Toxicity analytical result with the regulatory level to avoid incorrect hazardous waste determinations (false positives) which will result in the compliance/financial implications that will be discussed below. For example, the total constituent analysis of a 100% solid waste sample would be reduced by a factor of 20, before comparing it to the regulatory level (the 1 to 20 factor is derived from the 1 to 20 dilution factor that is part of the TCLP protocol).

A good source of information on the entire Toxicity category and, in particular, on correctly using the TCLP procedure/interpreting analytical results is: "Technical Assistance Document for Complying with the TC Rule and Implementing the Toxicity Characteristic Leaching Procedure (TCLP)", May 1993, U.S. EPA-Region II.

Table 1

TOXICITY CHARACTERISTIC CONTAMINANTS AND REGULATORY LEVELS

EPA Hazardous Waste Number	Contaminants	Chronic Toxicity Reference Level (mg/l)	Basis[a]	Regulatory Level (mg/l)[c]
D004	Arsenic	0 05	MCL	5.0
D005	Barium	1 0	MCL	100.0
D018	Benzene	0.005	MCL	0.5
D006	Cadmium	0 01	MCL	1 0
D019	Carbon tetrachloride	0 005	MCL	0.5
D020	Chlordane	0 0003	RSD	0.03
D021	Chlorobenzene	1	RfD	100.0
D022	Chloroform	0 06	RSD	6 0
D007	Chromium	0 05	MCL	5 0
D023	o-Cresol	2	RfD	200.0[d]
D024	m-Cresol	2	RfD	200.0[d]
D025	p-Cresol	2	RfD	200.0[d]
D026	Cresol	2	RfD	200.0[d]
D016	2,4-D	0 1	MCL	10 0
D027	1,4-Dichlorobenzene	0 075	MCL	7.5
D028	1,2-Dichloroethane	0 005	MCL	0.5
D029	1,1-Dichloroethylene	0 007	MCL	0 7
D030	2,4-Dinitrotoluene	0 0005	RSD	0 13[b]
D012	Endrin	0 0002	MCL	0.02
D031	Heptachlor (and its hydroxide)	0.00008	RSD	0 008
D032	Hexachlorobenzene	0 0002	RSD	0.13[b]
D033	Hexachloro-1,3-butadiene	0 005	RSD	0.5
D034	Hexachloroethane	0.03	RSD	3 0
D008	Lead	0.05	MCL	5.0
D013	Lindane	0 004	MCL	0 4
D009	Mercury	0 002	MCL	0.2
D014	Methoxychlor	0 1	MCL	10 0
D035	Methyl ethyl ketone	2	RfD	200 0
D036	Nitrobenzene	0.02	RfD	2.0
D037	Pentachlorophenol	1	RfD	100 0
D038	Pyridine	0 04	RfD	5 0[b]
D010	Selenium	0 01	MCL	1 0
D011	Silver	0.05	MCL	5 0
D039	Tetrachloroethylene	0 007	RSD	0 7
D015	Toxaphene	0 005	MCL	0 5
D040	Trichloroethylene	0 005	MCL	0 5
D041	2,4,5-Trichlorophenol	4	RfD	400 0
D042	2,4,6-Trichlorophenol	0 02	RSD	2 0
D017	2,4,5-TP (Silvex)	0.01	MCL	1 0
D043	Vinyl chloride	0.002	MCL	0 2

[a] MCL = Maximum Contaminant Level or National Interim Primary Drinking Water Standard
RSD = Risk-Specific Dose
RfD = Reference Dose

[c] The regulatory level equals the chronic toxicity reference level times a dilution/attenuation factor (DAF) of 100, unless otherwise noted

[d] If o-, m-, and p-cresol concentrations cannot be differentiated, the total cresol (D026) concentration is used Note that D026 was added to the final rule for this purpose, but is not a new constituent.

[b] The quantitation limit (i e , five times the detection limit) is greater than the calculated regulatory level; thus, the quantitation limit becomes the regulatory level.

Source: 55 FR 11804 and 11815-11816
1-24-94

LISTED WASTE (SPECIFIC/NONSPECIFIC)

As we mentioned above, solid waste is considered hazardous waste if it is "listed" as one of the following:

- Nonspecific source waste - These are generic wastes, commonly produced by manufacturing and industrial processes. Examples from this list include spent halogenated solvents used in degreasing and wastewater treatment sludge from electroplating processes.

- Specific source waste - This list consists of wastes from specifically identified industries such as wood preserving, petroleum refining and organic chemical manufacturing. These wastes typically include sludges, still bottoms, wastewaters, spent catalysts and residues; e.g., wastewater treatment sludge from the production of pigments.

- Commercial chemical products - The third list consists of specific unused commercial chemical products or manufacturing chemical intermediates. The key criterion for this category is that the waste is unused, e.g., off-spec or spilled materials. This list includes chemicals such as chloroform and creosote, acids such as sulfuric acid and hydrochloric acid, and pesticides, such as DDT and kepone.

These lists were developed by examining different types of waste and chemical products to ascertain if they:

- Exhibit one of the four characteristics of a hazardous waste(listed above);

- Meet the statutory definition of hazardous waste;

- Are acutely toxic or acutely hazardous;

- Are otherwise toxic.

It should be noted that individual States may designate additional materials as listed hazardous wastes. For example, New York State regulates PCB wastes as listed hazardous wastes (B-codes).

REGULATORY REQUIREMENTS

If a facility produces hazardous waste based on the regulatory criteria discussed above, it may be classified as a hazardous waste generator. Hazardous waste generators are the first link in the "cradle to grave" hazardous waste management system established pursuant to the Resource Conservation and Recovery Act (RCRA). Generators of 100 kilograms of hazardous waste or 1 kilogram of acute hazardous waste per month must comply with certain enforceable generator standards.

The pretransport regulatory requirements for hazardous waste generators include:

- Obtaining an EPA ID number. One way that EPA monitors and tracks generators is assigning each generator a unique identification number. Without this number, the generator is barred from treating, storing, disposing, transporting or offering for transport any hazardous waste to any transporter or treatment, storage or disposal facility;

- Adhering to procedures for handling hazardous waste before transport;

- Manifesting hazardous waste for off-site transportation;

- Maintaining a 24-hour Emergency Contact for each shipment;

- Record keeping and reporting;

- Proper packaging to prevent leakage of hazardous waste during normal transport conditions and in potentially dangerous situations (e.g., when a drum falls out of a truck);

- Identifying the characteristics and dangers associated with the waste being transported through labeling, marking and placarding of the packaged waste;

- Preparing applicable Land Disposal Restriction (Land Ban) shipping notices.

It is important to note that these pretransport regulations only apply to generators shipping waste off-site.

In addition to the requirements outlined above, EPA and authorized states also developed pretransport regulations for accumulation of waste prior to transport. A generator can accumulate hazardous waste on-site for 90 days or less without a permit, as long as the following requirements are met:

- Proper Storage - The waste is properly stored in containers or tanks marked with the words "Hazardous Wastes" and the date on which accumulation began. The waste must also be inspected at least weekly and inspections records maintained.

- Emergency Plan - A contingency plan and emergency procedures to use in an emergency must be developed.

- Personnel Training - Facility personnel must be trained in the proper handling of hazardous waste.

- Preparedness and Prevention Measures - Providing adequate security measures, signage, and communication systems.

The 90-day period allows a generator to collect enough waste to make transportation more cost-effective; that is, instead of paying to haul several small shipments of waste, the generator can accumulate waste until there is enough for one large shipment.

THE HAZARDOUS WASTE MANIFEST

The manifest is the fundamental element of the hazardous waste tracking system. The uniform hazardous waste manifest is the document which accompanies shipments of waste and tracks the material from the generator (the cradle) to the ultimate disposal facility (the grave). The RCRA manifest requires the following information:

- Name and EPA identification number of the generator, transporter(s) and the facility where the waste is to be treated, stored or disposed;

- U.S. DOT description of the waste being transported;

- Quantities of waste being transported; and

- Address of the treatment, storage or disposal facility to which the generator is sending the waste.

- 24-hour emergency contact telephone number.

It is especially important for the generator to prepare the manifest properly, since the generator is responsible for the hazardous waste produced and its ultimate disposition.

Waste Minimization

When Congress passed the Hazardous and Solid Waste Amendments (HSWA) in 1984, it established a framework aimed at eliminating specific forms of waste management such as land disposal, in favor of more technologically advanced, permanent destruction methods, such as incineration.

Among its complex and far reaching provisions, the HSWA contained a statutory provision which initiated waste minimization criteria. Sections 3002(a)(6), regarding the preparation of biennial waste reduction reports, and 3002(b) of HSWA, entitled, "Waste Minimization", require generators of hazardous waste to practice and report on waste minimization activities. It requires generators of hazardous waste to sign a specific certification

on the manifest indicating that they are doing all that is "economically practicable" towards reducing the volume or quantity and toxicity of the hazardous waste generated at a facility. It is a signed certification that generators are in full compliance with HSWA waste minimization criteria.

Section 3002(b) of HSWA, entitled "Waste Minimization", reads as follows:

> "(b) Waste Minimization - Effective September 1, 1985, the manifest required by subsection (a)(5) shall contain a certification by the generator that -
>
> (1) the generator of the hazardous waste has a program in place to reduce the volume or quantity and toxicity of such waste to the degree determined by the generator to be economically practicable; and
>
> (2) the proposed method of treatment, storage, or disposal is that practicable method currently available to the generator which minimizes the present and future threat to human health and the environment."

With regard to the requirement for biennial reporting of waste reduction efforts to regulatory agencies, Section 30021(a)(6) reads as follows:

> "(6) submission of reports to the Administrator (or the State agency in any case in which such agency carries out a permit program pursuant to this subtitle) at least once every two years, setting out -
>
> (A) the quantities and nature of hazardous waste identified or listed under this subtitle that he has generated during the year;
>
> (B) the disposition of all hazardous waste reported under subparagraph
>
> (C) the efforts undertaken during the year to reduce the volume and toxicity of waste generated; and
>
> (D) the changes in volume and toxicity of waste actually achieved during the year in question in comparison with previous years, to the extent such information is available for years prior to enactment of the Hazardous and Solid Waste Amendments of 1984."

In addition to these enforceable waste reduction mandates brought about by the HSWA manifest certification, many states have already passed additional statutes and regulations to address pollution prevention and waste reduction.

For example, in August 1990, New York State passed a law requiring facilities that generate and have the potential to release hazardous wastes and toxic substances into the environment reduce, to the maximum extent possible the volume or quantity and toxicity of wastes, whether emitted into the air, discharged into the waters, or treated and disposed of in a

permitted facility. The waste reduction may be achieved by implementing technically feasible and economically practicable waste reduction technology, process or operation changes. The legislature declared that implementing such measure will help the State achieve an overall reduction in the generation and release of hazardous waste of fifty percent over the next ten (10) years.

This law requires generators of hazardous wastes to prepare, implement and submit a Hazardous Waste Reduction Plan (HWRP) to the New York State Department of Environmental Conservation (NYSDEC). The HWRP, which is reviewed for acceptance by NYSDEC, must be updated biennially and annual status reports must be submitted. Failure to submit an acceptable plan precludes the generator from signing the hazardous waste manifest certification.

The requirements of the Hazardous Waste Reduction Plan include, but are not limited to, the following:

- Quantification of hazardous waste(s)
- Description of hazardous waste source(s) of generation and disposal method(s)
- Indices of hazardous waste generation to production (i.e. output from, or input to, the process generating the waste stream)
- Submission of a hazardous waste generator summary
- Cost estimate(s) for managing each waste
- Evaluation of technical feasibility and economical practicability of implementing waste reduction options
- Listing of technically feasible and economically practicable waste reduction measures and schedule for implementing identified waste reduction measures
- Description of corporation's and facility's waste reduction policy
- Identification of party responsible for implementation of waste reduction plan
- Identification of waste reduction measurement(s)
- Identification of employee training programs
- Estimate of anticipated hazardous waste reduction
- Estimate of anticipated transference of hazardous waste into other environmental media
- Submission of Hazardous Waste Reduction Program Summary (HWRP)

- Biennial updates of HWRP
- Annual status reports

In addition, to the state statute, EPA has published its interim final rule regarding waste minimization program requirements. Although published in the Federal Register as an interim final rule, the guidance puts "additional enforcement teeth" behind the hazardous waste manifest certification requirements mandated by the Hazardous and Solid Waste Amendments.

TAX ASSESSMENT/REGULATORY FEES

In addition to the regulatory/compliance implications discussed above, there is an additional liability when hazardous waste is generated at your facility and properly managed via a manifest, or a nonhazardous waste is accompanied by a manifest. That liability is a special assessment and regulatory fee. In New York State, these assessments and fees are administered by the New York State Departments of Taxation and Finance and Environmental Conservation, respectively. The "bottom line" is that the generation of hazardous waste in New York State can affect your "bottom line." Another good reason to make sure your hazardous waste is in fact, *hazardous*. Let's briefly review the two revenue programs.

As mentioned above, the first tax, entitled, "Special Assessments on Generation, Treatment or Disposal of Hazardous Waste in New York State" is administered by the Department of Taxation and Finance and is self reported by generators and TSDFs within the state on Form TP-550. This self reporting program is managed comparable to other state taxes, that is, it is prepared and reported by the generator and subject to review and audit by the Department of Taxation and Finance. In its simplest form, the Special Assessment, or "waste end assessment" as it is commonly referred to, is calculated by generators based on the tons of hazardous waste generated in New York State that received on site treatment or disposal or that were designated for removal or removed from the site of generation for treatment or disposal or for storage prior to such treatment or disposal during the reporting period. With regard to treatment and/or disposal facilities, these entities are only required to report the tons of hazardous waste received from generators outside New York State for treatment disposal or for storage prior to such treatment or disposal (this avoids double counting.)

In accordance with the Environmental Conservation Law (§27-0923 Special Assessments on Hazardous Wastes Generated) the following assessment rate schedule currently applies:

Category	Assessment Rate (in dollars per Ton)
Tons disposed of in landfill on-site of generation.	$27
Tons designated for removal or removed from the site of generation for disposal in a landfill or designated for removal or removed from the site prior to disposal in a landfill . . .	$27

Tons designated for removal or removed from the site of generation for treatment or disposal (except by landfill or incineration), or for storage prior to such treatment or disposal	$16
Tons designated for removal or removed from the site of generation for incineration or for storage prior to incineration	$ 9
Tons incinerated on the site of generation	$ 2
`Tons received from out-of-state for landfill disposal or for storage prior to such disposal	$27
Tons received from out-of-state for treatment or disposal other than landfill or incineration, or for storage prior to such treatment or disposal	$16
Tons received from out-of-state for incineration or for storage prior to incineration.	$ 9

As can be seen from the table above, the rates are structured to provide an incentive for disposal/treatment of waste by incineration and discourages disposal via landfilling. A deduction may be taken for waste that is reclaimed.

These special assessments are paid quarterly and are due to the Department by the 20th day of the month after the end of each calendar quarter. While the fees may not seem onerous at first glance, it does not take much material to achieve a ton of waste. For instance, a 55 gallon drum of water (the density used to calculate the fee) weighs approximately 459 lbs or approximately .23 of a ton. So one can see how quickly the special assessments can take a bite of your bottom line.

The second financial implication of generating hazardous waste is the Regulatory Fee, which is administered by the New York State Department of Environmental Conservation pursuant to 6 New York Codes, Rules and Regulations Parts 480 through 486 (Revised 1991).

Unlike the "waste end assessment" discussed above, the Hazardous Waste Program Fee prescribed in Part 483 is an invoice prepared and sent by the Department based on data from annual generator reports and manifest documents submitted.

Basically, for generators of hazardous waste, the hazardous waste program fee is currently determined as follows:

- $1000.00 for generators of equal to or greater than 15 tons per year and less than or equal to 100 tons per year of hazardous waste,

- $6,000.00 for generators of greater than 100 tons per year and less than or equal to 500 tons per year of hazardous waste,

- $20,000.00 for generators of greater than 500 tons per year and less than 1,000 tons per year of hazardous waste, and

- $40,000.00 for generators of greater than 1,000 tons per year.

In addition to the above, generators of equal to or greater than 15 tons per year of hazardous *wastewater* are assessed $3,000.00.

The use of a manifest New York State should only accompany shipments of hazardous waste as defined under Part 371. In fact, Part 372.2(b)(6) states ..."Use of a Uniform Hazardous Waste Manifest constitutes a determination by the generator that the solid waste is a hazardous waste in New York and/or the state of generation."

Therefore, the utilization of a manifest accompanying a waste material that is clearly not a hazardous waste material may be interpreted as a violation of New York State hazardous waste regulations and therefore could be enforceable.

SUMMARY

We have just reviewed a number of regulatory and financial requirements that generators of hazardous waste in New York State and elsewhere across the country are obligated to comply with in order to protect human health and environment. Presentations such as this are typically offered to assist hazardous waste generators in achieving and maintaining regulatory compliance... and thereby... ***minimize*** liability from regulatory violations and associated fines.

However, as we stated at the outset, the objective of this presentation is quite the opposite. The focus here is to identify how one's liability is actually increased by utilizing the same regulatory system discussed above (i.e. manifest document etc.) when it simply is not required because the waste is not truly a *hazardous* waste.

Example. Facility A uses a water soluble alkaline powdered product to aid in degreasing engine electric motors and transmission parts prior to rebuilding as part of its scheduled maintenance program. Rather than establish a proper waste characterization program with appropriate data quality objectives and quality assurance/quality control program, the facility manager characterizes the spent solution as a characteristic hazardous waste and ships it off-site via a licensed transporter with a signed manifest. The waste is characterized as corrosive (D002). This practice continues for a number of years, with manifests documenting 10's of thousands of gallons of "hazardous waste" being generated at the facility. (In fact, subsequent analytical data and proper waste characterization determined the material not to be hazardous).

What are the liabilities associated with this scenario?

First, the use of a manifest signifies that the entity is a hazardous waste generator and, as such, is required to comply with appropriate federal and state generator requirements. Most important among these requirements is obtaining an EPA ID number. The ID number ***must*** be

obtained by the generator in order to use a manifest. Now that you have declared generator status, the following enforceable requirements are applicable:

- compliance with specific procedures for handling waste
- record keeping and reporting
- proper labeling, marking and placarding
- develop and implement an emergency plan
- personnel training
- Preparedness and prevention measures
- annual generator report.

The above requirements are enforceable and may be subject to fine if determined not to be satisfactory to government inspectors. However, if the generator manifests wastes as "non-hazardous," the above requirements are not applicable.

Remember, the signature box on the manifest provides for a certification indicating that the generator has a program in place to reduce the volume and toxicity of waste, and that the method of treatment, storage or disposal minimizes present and future threats to human health and the environment. While these are commendable objectives, they are not applicable for a small manufacturing facility that does not generate hazardous waste in the first place.

There are also hazardous waste reduction plan requirements. Hazardous waste manifest documents are utilized to quantity the amounts of hazardous waste being generated at a facility. This process can include an otherwise non hazardous waste generator on the list of *hazardous* waste generators required by state law to prepare a Hazardous Waste Reduction Plan. The use of manifests and the submission of annual generator reports at a facility that does not generate hazardous waste in the first place can be an unnecessary regulatory burden. It costs resources to develop, submit and implement a hazardous waste reduction plan.

Last but not least, there are the "waste end assessments" reportable and payable to the Department of Taxation and Finance in New York State and regulatory fees assessed directly by the Department of Environmental Conservation. While perhaps not perceived as an overwhelming burden, when taken as a whole in consideration with the other prescribed regulatory requirements, improper hazardous waste characterizations can take a bite from your bottom line.

In short, the time, money and resources spent up front in proper waste characterizations including the development and implementation of clear data quality objectives and a quality assurance/quality control program, can go a long way toward reducing environmental compliance and financial liability.

References

Technical Assistance Document for Complying with the TC Rule and Implementing the Toxicity Characteristic Leaching Procedure (TCLP), May 1993, US EPA Reg. II, DCN EPA 902-b-93-001.

Appendix XIV
Region II State TCLP Guidances

Recd 6/9

State of New Jersey
Department of Environmental Protection and Energy
Environmental Regulation
Hazardous Waste Regulation Program
CN 421
Trenton, NJ 08625-0421
Phone# 609-633-1418

Scott A. Weiner
Commissioner

Frank Coolick
Administrator

M E M O R A N D U M

JUN 04 1993

TO: Leon Lazarus, Environmental Scientist
USEPA, Monitoring Management Branch

FROM: Richard Johnson, Supervising Env. Specialist
Bureau of Advisement and Manifest, DEPE

RJ

SUBJECT: Position Coordination for EPA's TCLP
Workshop on June 21, 1993

I. Spike Correction

On November 24, 1992, the Environmental Protection Agency (EPA) removed the TCLP spike recovery correction and required the method of standard additions be used for metals. This change occurred at 40 CFR 261, Subpart D.

The New Jersey Department of Environmental Protection and Energy (DEPE) requires that the test methods described in Appendix II of 40 CFR 261, Subpart D be used for performing the toxicity characteristic leaching procedure. Therefore, since the quality control change has been completed at the federal level, it is effective in New Jersey by reference.

II. Totals Analysis Versus TCLP Extract Analysis

This Bureau recommends following the guidance given in the January 12, 1993 memo from the EPA Office of Solid Waste which explains Section 1.2 of the TCLP. In brief, for hazardous waste classification purposes, this Bureau will accept a total constituent analysis value which has been divided by a factor of twenty, in place of a TCLP value.

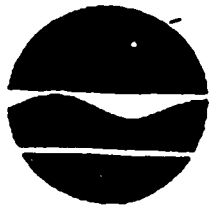
However, our preference is the use of the TCLP instead of a total constituent analysis for hazardous waste classification. The reason for this is that a total constituent analysis such as a priority pollutant analysis does not include all of the TCLP constituents. A laboratory that submits a priority pollutant analysis in place of a TCLP analysis, must be careful to submit any additional analyses which might be needed to compliment any missing analytes from the priority pollutant test. The inclusion of these additional analyses are usually omitted in packages which are submitted to us, which causes unnecessary delays in completing a classification.

III. Status of EPA Land Ban Regulations in New Jersey

The EPA land ban regulations were authorized as part of the EPA Hazardous and Solid Waste Amendments (HSWA). EPA will enforce these requirements until New Jersey adopts this regulation. Therefore, the EPA land ban requirements are enforced at the federal level at this time. The DEPE is planning to adopt an equivalent set of regulations in the future. When this occurs, DEPE personnel will enforce the land ban requirements.

PR75(S1):nb
c: John Barry, Enforcement, SFO
Tom Sherman, BHWE, DEPE
Phil Flax, HWCB, USEPA
Cathy Grimes, BEERA
Henry Hoffman, Laboratory Certification

New York State Department of Environmental Conservation
50 Wolf Road, Albany, New York 12233



Thomas C. Jorlin
Commissioner

MEMORANDUM

TO: Distribution Below

FROM: Norman H. Nosenchuck, Director
Division of Hazardous Substances Regulation

SUBJECT: EPA Revised Disposal Standards for F001-F005 Spent Solvent Wastes

DATE: JAN 2 1 1993

The United States Environmental Protection Agency (EPA) has revised the disposal standards for the regulated hazardous constituents of F001-F005 spent solvent wastes.

These revisions were promulgated as part of the Land Disposal Restrictions (LDRs) for Newly Listed Wastes and Hazardous Debris that were published in the Federal Register on August 18, 1992. Due to the volume and widespread occurrence of spent solvent wastes, it was necessary to prepare this guidance, as our Part 376 standards are now different from the revised 40 CFR 268 standards. The attached table will, for each constituent, identify which agency's regulation is more stringent and must be followed. Also included are the new standards as they were promulgated by the EPA. This unfortunate but unavoidable dual regulation will exist until the revised standards can be adopted and promulgated by New York State. Current projections have this adoption of revisions at least 14 to 18 months away.

Other changes, and certain exclusions included in this final rule, have created some confusion as to which agency's (EPA or DEC) regulation is more stringent and, therefore, the one to be enforced. All newly listed wastes are unique to the federal regulations.

If you have any questions concerning these revisions to the LDRs, please contact Mr. John D. Miccoli, of my staff, at (518) 485-8988.

Attachment

printed on recycled paper

DISTRIBUTION:

R. Becherer, Region 1
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A. Shah, Region 3
C. Van Guilder, Region 4
D. Curtis, Region 5
T. Morgan, Region 6
S. Eidt, Region 7
D. Rollins, Region 8
F. Shattuck, Region 9

bcc: w/att: N. Nosenchuck (2)
M. O'Toole
D. Mafrici
L. Nadler
J. Middelkoop
P. Counterman
R. Haggerty
J. Desai
J. Miccoli

JDM:NHN:gz

F001-F005 SPENT SOLVENT REGULATED HAZARDOUS CONSTITUENTS - DISPOSAL STANDARD TO BE FOLLOWED

CONSTITUENT	WASTEWATER	NONWASTEWATER
Acetone	40 CFR Part 268	Part 376
Benzene	Same in both	Same in Both
n-Butyl alcohol	40 CFR Part 268	40 CFR Part 268
Carbon disulfide	Same in both	Same in both
Carbon tetrachloride	40 CFR Part 268	40 CFR Part 268
Chlorobenzene	40 CFR Part 268	Part 376
Cresol (m-and-p isomers)	40 CFR Part 268	40 CFR Part 268
o-cresol	40 CFR Part 268	40 CFR Part 268
Cyclohexanone	Same in both	Same in both
o-Dichlorobenzene	40 CFR Part 268	Part 376
Ethyl acetate	40 CFR Part 268	Part 376
Ethyl benzene	40 CFR Part 268	Part 376
Ethyl ether	40 CFR Part 268	Part 376
Isobutyl alcohol	40 CFR Part 268	Part 376
Methanol	Same in both	Same in both
Methylene chloride	40 CFR Part 268	Part 376
Methyl ethyl ketone	40 CFR Part 268	Part 376
Methyl isobutyl ketone	40 CFR Part 268	Part 376
Nitrobenzene	40 CFR Part 268	Part 376
Pyridine	40 CFR Part 268	Part 376
Tetrachloroethylene	40 CFR Part 268	Part 376
Toluene	40 CFR Part 268	Part 376
1,1,1-Trichloroethane	40 CFR Part 268	40 CFR Part 268
1,1,2-Trichloroethane	Same in both	Same in both

F001-F005 SPENT SOLVENT REGULATED HAZARDOUS CONSTITUENTS - DISPOSAL STANDARD TO BE FOLLOWED

CONSTITUENT	WASTEWATER	NONWASTEWATER
1,1,2-Trichloro-1,2,2-trifluoromethane	40 CFR Part 268	Part 376
Trichloroethylene	40 CFR Part 268	Part 376
Trichloromono-fluoromethane	40 CFR Part 268	Part 376
Xylenes (total)	40 CFR Part 268	Part 376
2-Nitropropane	Same in both	Same in both
2-Ethoxyethanol	Same in both	Same in both

L. J. Miccoli

New York State Department of Environmental Conservation
50 Wolf Road, Albany, New York 12233

Thomas C. Jorling
Commissioner

MEMORANDUM

TO: Distribution Below

FROM: Norman H. Nosenchuck, Director
Division of Hazardous Substances Regulation

SUBJECT: Revised Toxicity Characteristic Leaching Procedure (TCLP)

DATE: MAY 24 1993

The United States Environmental Protection Agency (EPA) has revised the Toxicity Characteristic Leaching Procedure (TCLP) in a revision to the Toxicity Characteristic (TC) rule.

The revision, promulgated on November 24, 1992, is the result of the EPA's reassessment of the matrix spike correction requirement, promulgated on June 29, 1990 as a final rule technical correction to the TC rule. At that time, EPA's intention was to achieve consistency with the SW-846 Chapter One requirements, proposed on February 8, 1990, which EPA anticipated would be promulgated as a final rule prior to the effective date of the TC rule.

However, to date, EPA has not promulgated the SW-846 Chapter One requirements. Therefore, in response to public comments originally received on the February 8, 1990 proposed changes, the EPA decided not to proceed with the proposed spike recovery correction requirements for Subtitle C analytical methods.

EPA stated in the November 24, 1992 preamble to their revision that authorized states are given the option to adopt this change to the TCLP procedure.

New York State, as did other states authorized for Land Disposal Restrictions (LDR), included the TCLP as a requirement to implement these LDR regulations. As part of our current rulemaking process to update our hazardous waste regulations, we will include the revised TCLP for the TC rule and the LDR regulations.

In the meantime, to eliminate confusion regarding specific in the TCLP testing methods in the interim, DEC will accept the EPA version currently in place as Appendix II to 40 CFR Part 261, designated as Method 1311, as an alternative to the TCLP (Appendix 35) currently found in Part 376 to implement the LDRs.

Attached for your convenience and use, is both a copy of the Federal Register notice of November 24, 1992, and the draft changes to Appendix 35 found in Part 376. The draft Appendix 35, Section 8.0 - Quality Assurance, has brackets [ ] surrounding words or formulas being removed, and underlines all additions.

Please call John D. Miccoli, of my staff, at (518) 485-8988 if you have any questions concerning the possible ramifications of this change to our LDR program.

Attachment

DISTRIBUTION:

R. Becherer, Region 1
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D. Rollins, Region 8
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cc: w/att: E. Sullivan
N.G. Kaul
M. O'Toole
G. Kelly

bcc: w/att: N. Nosenchuck (2)
D. Mafrici
L. Nadler
D. Aldrich
J. Middelkoop
P. Counterman
R. Haggerty
J. Desai
J. Miccoli

JDM:DLA:NHN:gz

Appendix XV

Appendix XV
Risk Assessment for Disposal of Solidified/Stabilized Waste and Contaminated Soil

Texas A&M University

CIVIL

Department of Civil Engineering • Texas A&M University • College Station, TX 77843-3136

Bill Batchelor • Professor • (409) 845-1304 • FAX (409)862-1542 • Email bill-batchelor@tamu.edu

April 11, 1994

FAX MEMORANDUM

To: Leon Lazarus
USEPA
FAX: 908-321-6622

From: Bill Batchelor

Subject: Risk Assessment Paper

Attached is the paper I presented on risk assessment of materials treated by solidification/stabilization. It is now under review for publication, but can be referenced in the interim as:

> Batchelor, B., "A Framework for Risk Assessment of Disposal of Solidified/Stabilized Wastes and Contaminated Soils", Symposium on Treatment and Modeling of Hazardous Waste Processes, 24th Annual Meeting of the Fine Particle Society, Chicago, Illinois, August 24-28, 1993.

I'm looking forward to downloading your TCLP manual from the Clu-In BBS.

Texas A&M University

CIVIL

Department of Civil Engineering • Texas A&M University • College Station, TX 77843-3136

Bill Batchelor • Professor • (409) 845-1304 • FAX (409)862-1542 • Email bill-batchelor@tamu.edu

April 19, 1994

Mr. Leon Lazarus
USEPA
2890 Woodbridge Ave.
Edison, NJ 08837

Dear Leon:

I am enclosing a copy of the risk assessment paper that I previously faxed to you. I hope it is useful to you.

Sincerely,

Bill Batchelor, Ph.D, P.E.

Professor
Environmental & Water Res. Engr.
Civil Engineering Dept.

Director
Institute for Environmental Engineering
Texas Engineering Experiment Station

A Framework for Risk Assessment of Disposal of Solidified/Stabilized Wastes and Contaminated Soils

Bill Batchelor
Civil Engineering
Texas A&M University
College Station, TX 77843

ABSTRACT

Quantitative risk assessment is the tool used at hazardous waste sites to evaluate the need for treatment and to determine which control strategies are most effective. A general approach to conducting quantitative risk assessments at contaminated sites is reviewed and a framework for applying it to evaluating risk associated with ground water contamination by disposal of materials treated by solidification/stabilization is presented. The approach is based on a material balance around the disposal zone and the ground water flowing past it. Two limiting cases are identified that can control the concentration of contaminant leaving the leaching zone. The first case occurs when the flow past the solid is low and the its concentration can approach that in equilibrium with the pore water in the solid. The second case occurs when the flow past the solid is sufficiently large that the concentration of contaminant in the flow is negligible. In this case, the flux from the solid can be predicted by simple leach models. Examples of applying this framework to materials treated by solidification/stabilization are presented.

INTRODUCTION

Risk assessment is becoming increasingly important to the practice of environmental engineering. More and more it is becoming the technique used to determine the level of treatment required and the resources to be allocated to different environmental problems. Its use for environmental decision making is analogous to the use of costs and benefits expressed as dollars in economic decision making. In this way, quantitative estimates of risk can be considered the "currency" of environmental decision making.

Risk assessment is particularly important in remediation of hazardous waste sites. Federal legislation requires decisions to be made on a site-specific basis, rather than on the basis of national standards. The latter approach is more common in protecting water and air quality. Three types of risk assessments are made during the process of developing a plan to remediate a site.[1,2] First a baseline risk assessment is made to evaluate the danger of the site as it exists. This risk assessment is then improved in the process of determining preliminary remediation goals that are used to screen remediation options. Finally, risk assessments are used to evaluate the remedial alternatives in the processes of selecting the most appropriate ones.

Risk assessments are required to evaluate the impact of the site on human health and on the environment. The approaches for assessing human health impacts are much simpler, because they do not need to deal with the complexities of environmental ecosystems. Work is underway

to identify indicators of ecological health that can be easily used in environmental risk assessments.

There is a great deal of uncertainty in quantitative risk assessments. The results of such assessments should not be considered important because they accurately predict quantitative impact on human health, but rather as methods to compare different alternative actions. They are valuable in the relative sense of providing a comparison of the risk at one site to that at another, or the risk of using one technology rather than another.

This paper will present a framework for applying risk assessment to evaluating the effect on human health of disposal of materials treated by solidification/stabilization. It will begin with a review of the steps typically taken in a quantitative risk assessment. It will then develop the approach for applying them to solidification/stabilization. Finally, it will provide example calculations of how the approach could be used. The approaches to risk assessment described will be those promulgated by the U.S. EPA,[1,2] however, the opinions expressed will be those of the author.

STEPS IN RISK ASSESSMENT

Data Collection and Evaluation

The first step in a quantitative risk assessment is to collect the data upon which the analysis will be based. Much of this data would normally be obtained for other purposes, however, some additional data may be required. The types of data required will become clear as the other steps are discussed.

Exposure Assessment

The exposure assessment step involves determining the extent to which the contaminants of concern will be taken up by exposed individuals. This process begins by evaluating the physical setting. This will allow identification of the exposed populations and the pathways by which they will be exposed. Exposed populations could include those that live near a site, those that work at a site, or casual visitors to a site. Adults, children or other groups could be defined as distinct populations for calculating risks. Exposure pathways could include inhalation of contaminants released to the air, drinking of ground water contaminated at the site, consumption of food that was contaminated by material released from the site, or consumption of soil transported from the site by air.

When exposed populations and pathways are identified, the concentrations to which the populations are exposed can be calculated. This often includes the use of transport and fate models to estimate the concentrations of contaminants near the exposed population. These calculations are based on the concentrations of contaminants at the site and the relevant characteristics of the physical setting at the site. For example, a diffusion-reaction model could be used to estimate release of volatile contaminants from soils at a site and an air dispersion model could be used to estimate the concentration of contaminants in air at the boundary of the site where individuals would be exposed. In another case, a vadose zone transport model could be used to predict movement of a contaminant at a site down into the saturated zone and another ground water transport model could be used to predict movement off-site to a drinking water

well. Both models could include descriptions of the processes of advection, dispersion, sorption, and degradation to predict transport and fate.

Good judgment should be used in determining the complexity of the models used to calculate exposure concentrations. A complex model will not necessarily provide better estimates of concentrations, particularly when the model requires more data than is available. Furthermore, complex models generally are more difficult to use. An important factor to consider in choosing an appropriate level of model complexity, is that it is not necessary to model precise conditions in the future. For example, the wind speed and direction will not be known for the future, so exact calculations of concentration at any time in the future are not possible. Rather, it is important to develop a realistic way to evaluate risk that can be used to compare risks at one site to another, and to compare risks associated with one remedial alternative to those associated with another. Consistency across different sites and remedial alternatives is more important than accuracy for a specific set of conditions.

When exposure concentrations are obtained, intakes of the exposed population can be calculated. This requires assumptions about factors that affect the extent of exposure, such as the volumes of air, water or soil consumed per day by an exposed individual. Intakes are expressed as the mass of contaminant taken into an exposed individual per mass of the individual per unit time.

Toxicity Assessment

The toxicity assessment involves determining the relevant toxicological data to be applied and values for exposure periods that are reasonable for the specific case under study. Toxicological data for many compounds have been compiled in a form that is readily applied to risk assessments in a data base called IRIS (Integrated Risk Information System). It is available in electronic format, and is updated regularly.[3]

Toxicity of compounds believed to be carcinogenic is expressed differently than for those compounds that are not believed to be carcinogenic. A proportionality is assumed between the intake of a carcinogen and the probability of developing a fatal cancer over the lifetime for the exposed individual. The proportionality factor is called a slope factor and can be considered the lifetime cancer risk associated with a unit intake of the compound. Therefore, higher slope factors indicate more toxic compounds.

Toxicity of compounds that are not believed to be carcinogenic is expressed as a reference dose. This is the upper limit on intake of the chemical for which adverse effects are not expected. Therefore, lower reference doses indicate compounds that are more toxic.

Risk Characterization

This stage integrates the exposure and toxicological assessments to determine quantitative estimates of risk. First, risk estimates are calculated for each contaminant of concern, each pathway, and each exposed population. The lifetime cancer risk for a given pathway and chemical is calculated for carcinogenic compounds as the product of the calculated intake and the slope

factor. A hazard quotient is calculated for each pathway and each exposed population as the intake divided by the reference dose. Lifetime cancer risks and hazard quotients are summed over all pathways and all chemicals for each exposed population to determine the total lifetime cancer risk and the hazard quotient. Lifetime cancer risks are to be reduced to the range between 10^{-4} and 10^{-6} and hazard quotients are to be reduced to a level below 1.0.[4]

The last step in risk characterization is to assess the uncertainty in the risks. This is an important step because there are many uncertainties associated with risk assessment. It is important that the range of reasonable values of risk be communicated to interested parties along with the value of risk calculated to be the most probable. This will give an important context to the estimates of risk. For example, a hazard index of 0.5, with a confidence interval of 0.4 to 0.6 carries a different meaning from one with the same value but a confidence interval of 0.01 to 50.

There is typically a great deal of variability in contaminant concentrations at a site. Some areas are highly contaminated, while others are lightly contaminated or have no measurable contamination. The physical characteristics of a site that affect the movement of contaminants can be highly variable at different locations at the site. For example, hydraulic conductivities and contaminant distribution factors have been found to have scales of variation on the order of a few meters in aquifers.[5,6] Processes that can affect the fate of contaminant at a site can be highly variable. Weather is highly variable, as can be processes such as biological degradation. Many assumptions must be made to calculate chemical intakes that are known with little certainty. Finally, the response of humans to the toxic compound must often be predicted based on response of laboratory animals to high doses of the chemical. Extrapolating this toxicological data to humans exposed to low doses is a highly uncertain process.

Uncertainty in risk analysis is quantified by repeating calculations using different but reasonable values for the variables. This can take the form of a sensitivity analysis where values of individual variables are changed over their expected ranges and the effect on calculated risk are evaluated. A more extensive procedure is to conduct a Monte Carlo analysis in which probability distributions of all variables are used to estimate the probability distribution of the calculated risks.[7]

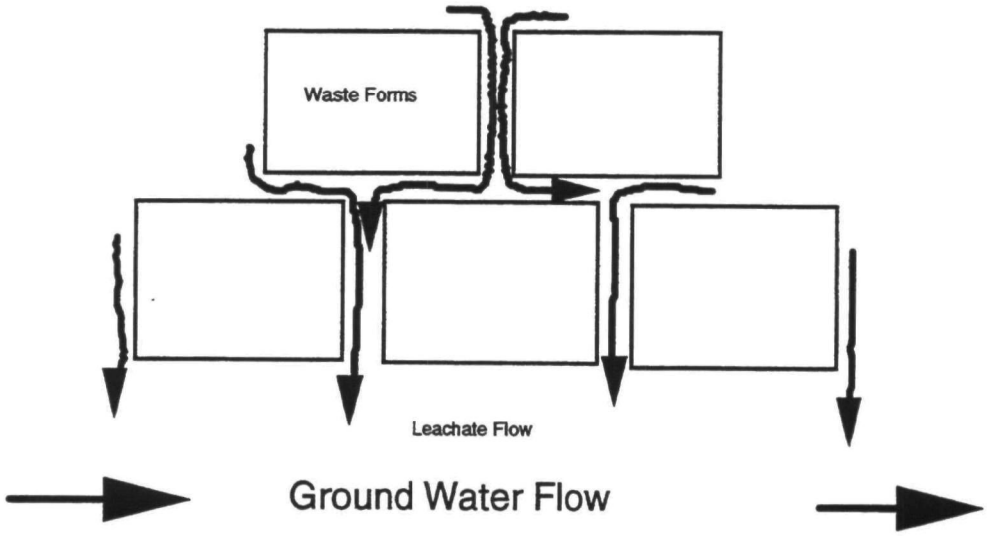
However, there is a problem in the way risks typically are calculated at sites. Guidance from the U.S. EPA requires that conservative assumptions be made in calculating the risk. Therefore, the value of risk determined by this method is not the most probable risk, but a risk that is likely to be somewhat higher than is most probable. It would be better, to make the most reasonable assumptions possible in the basic risk assessment process and then to provide a range of more conservative values of risk on which to base decisions. A stochastic risk assessment could be made so that decisions could be based on whatever level of conservatism desired. This would bring the question of the desirable level of protection into the public discourse, rather than hide it in the model calculations.

RISK ASSESSMENT FOR S/S

Exposure Assessment

The purpose of this step is to estimate quantitative intakes of those chemicals identified as being major risks to human health. First, the physical setting for s/s activities should be defined. S/S could cause short-term adverse health effects during the treatment stage or long-term effects after disposal. The potential exposed populations and pathways by which chemicals could affect them should be identified. Potential pathways of short-term exposure include fugitive dust emissions during transport of the material to be treated and volatile organic emissions during treatment. However, in many cases the long-term risk of exposure through the pathway ground water contamination is of greatest concern. Figure 1 shows a schematic of how leachate could transport chemicals to the ground water underneath a disposal site.

Figure 1 shows in a very simplified manner the way in which leachate could pass by



material treated by s/s and become contaminated with the chemical contained within the treated material. This leachate could then mix with underlying ground water. The procedure described below will provide a method for estimating the risk to a nearby resident who might consume this ground water.

A simple material balance can be used to estimate the concentration of contaminant in the ground water. This balance states that the mass flow of contaminant leaving the zone where the treated material has been disposed plus the mass flow of contaminant in the ground water approaching the mixing zone equals the mass flow of contaminant leaving the mixing zone.

$$Q_l C_l + Q_{gw} C_{gw}^{in} = (Q_{gw} + Q_l) C_{gw}^{out} \tag{1}$$

$$C_{gw}^{out} = \frac{Q_l C_l + Q_{gw} C_{gw}^{in}}{Q_{gw} + Q_l} \tag{2}$$

where: Q_l = flow passing through disposal zone

C_l = concentration of contaminant in leachate leaving disposal zone

Q_{gw} = flow of groundwater approaching mixing zone

C_{gw}^{in} = concentration of contaminant in groundwater approaching mixing zone
C_{gw}^{out} = concentration of contaminant leaving mixing zone

Equation 1 demonstrates the importance of the leachate flow and leachate concentration. The leachate flow can be changed by changing the design for disposal of the treated material. Isolating the treated material within caps and liners will greatly reduce the flow of water passing through the disposal zone. The value of the flow to be used in this risk assessment must be determined based on site-specific factors. Tools such as the Hydraulic Evaluation of Landfill Performance model[8] or other ground water transport models applied to the site could be used to estimate such flows.

Choosing a value for the ground water flow approaching the mixing zone determines the extent to which the contaminant will be diluted in the ground water. Choosing a single value is a great simplification of the ground water transport situation, but it can be suitable for a risk assessment if good judgment is used. Simple mixing models or more complex ground water transport models can be used to facilitate choosing this variable.

Many complex processes including chemical reaction and mass transport could affect the concentration of contaminant in the leachate leaving the disposal zone. However, two limiting cases can be examined to facilitate calculations. The first limiting case is when equilibrium conditions control. This could happen when the leachate flow is relatively low and the leaching rate is relatively high. Under these conditions, contaminants would leach rapidly enough so that the contaminant in leachate leaving the site would be in equilibrium with the contaminant in the waste forms.

$$C_l = C_{eq} \tag{3}$$

The value of the equilibrium concentration could be obtained by conducting a batch leaching test in which the volume of leaching solution is small relative to the mass of treated material and the leaching time is long.

A second limiting case would occur when the concentration of contaminant in the leachate is controlled by leaching kinetics. The concentration of contaminant in the leachate could be calculated for this case by recognizing that the mass flux of contaminant leaving the disposal zone must equal the mass flow of material from the waste forms.

$$Q_l C_l = F_l \tag{4}$$

where: F_l = mass flow of contaminant in leachate leaving disposal zone

A variety of leaching models could be used to calculate the mass flow for Equation 4, but those based on the "infinite bath", "infinite solid" and "rectangular geometry" assumptions are simple and often appropriate.[9] These models assume that the concentration of contaminant in the leachate near the waste form is negligible relative to the concentration of leachable contaminant in the waste form (infinite bath), that leaching has not progressed to the point where the interior of

the solid has been leached (infinite solid) and that leaching has not progressed to the extent that curvature of the waste form is important. The infinite bath assumption will be valid when leachate flow past the solid is high enough to sufficiently dilute the contaminant to concentrations that are low relative to the concentration within the pore water of the solid. The infinite solid assumption will be valid as long as the model is not applied to leaching times for which a large (>20%) fraction of the contaminant has been leached.

These models can be used to describe leaching affected by chemical reactions through the use of an observed diffusivity. The observed diffusivity for a contaminant in a waste form can be determined by leaching tests, or it can be estimated by more complex models that quantify the reactions.[10] These models show that the mass rate of leaching is inversely proportional to the square root of leaching time.

$$F_l = \frac{M_0}{L}\left(\frac{D_{obs}}{\pi t}\right)^{0.5} \tag{5}$$

$$C_l = \frac{M_0}{Q_l L}\left(\frac{D_{obs}}{\pi t}\right)^{0.5} \tag{6}$$

where: M_0 = mass of contaminant in disposal zone
L = ratio of volume of treated material to external surface area exposed to leachate
D_{obs} = observed diffusivity of contaminant in treated material
t = time since leaching began

The mass of contaminant in the disposal zone can be calculated based on characterization of the material before treatment and the amount of material treated. The observed diffusivity can be determined by dynamic leach tests or estimated from results on similar materials.[11] The ratio of the volume of treated material to its external surface area is a measure of the average particle size. If the treated material retains its physical integrity, this variable could be large, maybe several meters. If the material disintegrates over time, it could be as small as 1 to 10 millimeters. Note that the mass rate of leaching depends on time, so that the concentration of contaminant in the ground water leaving the mixing zone will vary over time.

After the concentration in the ground water leaving the mixing zone is determined, it can be used to calculate the chemical intake of an exposed person. The following method can be used to calculate chemical intake from drinking water.[1]

$$I = \frac{C_w I_r E_f E_d}{B_w A_t} \tag{7}$$

where: I = intake of chemical (mg / kg - day)
C_w = concentration of chemical in water(mg/l)
I_r = ingestion rate of water (l / day)
E_f = exposure frequency (days / year)
E_d = exposure duration (years)
B_w = body weight (kg)
A_t = averaging time, period over which exposure is averaged (days)

The concentration in the water would be taken as the concentration calculated in the ground water leaving the mixing zone. Typical values for the other variables are: 2 l/day ingestion rate of water, 365 days/year exposure frequency, 70 years exposure duration, 70 kg body weight, and 70 years averaging time.[1]

Toxicity Assessment

Toxicity assessments are carried out differently for chemicals that are believed to be carcinogenic and those that are not. The potency of suspected carcinogens is expressed as a slope factor, which gives the lifetime cancer risk per unit of chemical intake. Potency of non-carcinogens is expressed in terms of a reference dose. This is the estimate of chemical that is likely to be without an appreciable risk of deleterious effects during a lifetime and is similar to an acceptable daily intake. Values for these variables can be obtained from standard sources.[3]

Risk Characterization

The process of risk characterization combines information on toxicity with that of chemical exposures to determine overall risks at a site. The risk of a chemical believed to be carcinogenic is obtained by multiplying the slope factor for that chemical times its calculated intake to obtain the lifetime cancer risk for that chemical. Summing over all chemicals and all pathways gives the lifetime cancer risk for that site.

The risk of exposure to non-carcinogens is calculated as the non-carcinogenic hazard quotient, which is the ratio of the calculated intake for a chemical to its reference dose. Hazard quotients are summed for all chemicals and all pathways to obtain the hazard index for this site.

There are many uncertainties with any risk assessment and they should be specifically addressed. The extent to which this is done depends on the importance of the risk assessment. Evaluation of uncertainty of the risk assessment can be done qualitatively by describing the uncertainties in each parameter value assumed, and each assumption in the risk assessment procedure. Quantitative evaluation of risk can be done by conducting sensitivity analyses in which different values of parameters are used to determine the range of expected risks. This can be done more completely with Monte Carlo simulations which will provide an estimate of the probability distribution of calculated risks.

EXAMPLE

The following example shows how the calculations could be conducted for a carcinogen and a non-carcinogen. The following general assumptions will be used:

ground water flow approaching mixing zone = 30 m^3/d
concentration of carcinogenic and non-carcinogenic contaminants in ground water approaching mixing zone = 0
mass of carcinogen = $5x10^7$ g
slope factor for carcinogen = 0.05 kg-day/mg
mass of non-carcinogen = $6x10^7$ g

reference dose for non-carcinogen = 0.001 mg/kg-day
size of individual waste form (cube) = 1 m
ingestion rate for water = 2 l/day
exposure frequency = 365 day/year
exposure duration = 70 years
body weight = 70 kg
averaging time = 365 days/year x 70 years = 25,550 days

The following assumptions will be made for the case of equilibrium-controlled conditions:
leachate flow = 0.5 m^3/d
equilibrium concentration of carcinogen = 1.0 g/m^3
equilibrium concentration of non-carcinogen = 0.1 g/m^3

The following assumptions will be made for the case of kinetic-controlled conditions:
leachate flow = 20 m^3/d
time after placement (leaching time) = 100 d
observed diffusivity of carcinogen = 10^{-13} m^2/d
observed diffusivity of non-carcinogen = 10^{-15} m^2/d
ratio of waste form volume to external surface area exposed to leaching = length of cube/6 = 0.167 m (i.e. all of external area of waste form is exposed to leachate flow)

Carcinogen with equilibrium control

$$C_{gw}^{out} = \frac{Q_l C_l + Q_{gw} C_{gw}^{in}}{Q_{gw} + Q_l} = \frac{(0.5)(1.0)+(30)(0)}{30+0.5} = 0.0164 \text{ mg/l}$$

$$I = \frac{C_w I_r E_f E_d}{B_w A_t} = \frac{(0.0164)(2)(365)(70)}{(70)(25,550)} = 4.69x10^{-4} \text{ mg/kg-day}$$

Risk = Intake x Slope Factor $= (4.69x10^{-4})(0.05) = 2.35x10^{-5}$

Carcinogen with kinetic control

$$C_l = \frac{M_0}{Q_l L}\left(\frac{D_{obs}}{\pi t}\right)^{0.5} = \frac{5x10^7}{(20)(0.167)}\left(\frac{10^{-13}}{(3.1416)(100)}\right)^{0.5} = 0.267 \text{ mg/l}$$

$$C_{gw}^{out} = \frac{Q_l C_l + Q_{gw} C_{gw}^{in}}{Q_{gw} + Q_l} = \frac{(20)(0.267)+(30)(0)}{30+20} = 0.107 \text{ mg/l}$$

$$I = \frac{C_w I_r E_f E_d}{B_w A_t} = \frac{(0.107)(2)(365)(70)}{(70)(25,550)} = 3.05x10^{-3} \text{ mg/kg-day}$$

Risk = Intake x Slope Factor $= (3.05x10^{-3})(0.05) = 1.53x10^{-4}$

Non-carcinogen with equilibrium control

$$C_{gw}^{out} = \frac{Q_l C_l + Q_{gw} C_{gw}^{in}}{Q_{gw} + Q_l} = \frac{(0.5)(0.1)+(30)(0)}{30+0.5} = 1.64x10^{-3}\ \text{mg/l}$$

$$I = \frac{C_w I_r E_f E_d}{B_w A_t} = \frac{(0.00164)(2)(365)(70)}{(70)(25,550)} = 4.68x10^{-5}\ \text{mg/kg-day}$$

$$\text{Hazard Quotient} = \frac{\text{I}}{\text{Reference Dose}} = \frac{4.68x10^{-5}}{10^{-3}} = 0.0468$$

Non-carcinogen with kinetic control

$$C_l = \frac{M_0}{Q_l L}\left(\frac{D_{obs}}{\pi t}\right)^{0.5} = \frac{6x10^{7}}{(20)(0.167)}\left(\frac{10^{-15}}{(3.1416)(100)}\right)^{0.5} = 0.0321\ \text{mg/l}$$

$$C_{gw}^{out} = \frac{Q_l C_l + Q_{gw} C_{gw}^{in}}{Q_{gw} + Q_l} = \frac{(20)(0.0321)+(30)(0)}{30+20} = 0.0128\ \text{mg/l}$$

$$I = \frac{C_w I_r E_f E_d}{B_w A_t} = \frac{(0.0128)(2)(365)(70)}{(70)(25,550)} = 3.66x10^{-4}\ \text{mg/kg-day}$$

$$\text{Hazard Quotient} = \frac{\text{I}}{\text{Reference Dose}} = \frac{3.66x10^{-4}}{10^{-3}} = 0.366$$

SUMMARY

Quantitative risk assessment can be used as an environmental currency to evaluate the relative risks associated with different environmental problems and the relative effectiveness of different control strategies. A standard approach has been proposed for use at sites contaminated by hazardous wastes and this approach has been applied to provide a framework for evaluating risks associated with disposal of materials treated by solidification/stabilization. This framework is intended to provide a flexible approach for conducting such assessments. More complex or simpler approaches may be appropriate for any given site. In all cases, a risk assessment must be conducted by those who understand the physical, chemical and biological factors that affect the actual risk at a site.

The framework for risk assessment proposed here considers the pathway of ground water contamination and is based on a material balance around the disposal zone and the ground water flowing past it. Two limiting cases are identified as controlling the concentration of contaminant leaving the leaching zone. In one case, the concentration approaches the concentration that would be in equilibrium with the contaminant in the solid. This case would be applicable to cases in which the flow past the solid is low. The second limiting case would occur when the flux of contaminant leaving the solid can be approximated by leaching models that assume negligible concentrations in the leachate. This case would be applicable to conditions of high flow past the solid.

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Appendix XVI

Appendix XVI
Barbara Metzger's 1992 Speech on Environmental Data Use - Meeting the Customer's Need

Environmental Data Use - Meeting the Customer's Need

Introduction:

Good morning. When Llew Williams asked me to be the kickoff speaker for this conference, I wasn't sure whether I should be flattered or intimidated. For those of you who know me, I think you realize that I chose to be flattered with the invitation. On a more serious note, I am really pleased to have the opportunity to address an audience that is interested in making quality assurance work. As an Environmental Services Division Director at EPA, I'm often called upon to champion QA, but most of the time it's standing up for it because it's the "in thing", TQM and all, and fighting for the need and the costs of appropriate quality assurance practices. In today's climate of static or shrinking resources to collect adequate environmental information to support hazardous waste site cleanup or remediation decisions, competition for available resources is intense, and getting more so. It is imperative that we, the quality assurance professionals, are able to support the appropriateness and the effectiveness of the quality assurance practices we put in place to provide useful information, not just qualified data, for our respective program decisionmakers' use. Quality assurance for quality assurance's sake is no longer a viable option. Quality assurance practices must, in TQM terms, truly meet the customer's needs to be appropriate.

As most of you are aware, this isn't as easy as it sounds. When I ask the customer what he needs, he may be able to tell me what compounds he is concerned about, but not much more. Detection level, precision, and accuracy are words without meaning to the discussion at hand.

As we look at the different phases of any environmental data collection effort, we can see the importance of incorporating quality assurance considerations if we are to provide our customers with useful information.

There are five generic phases of any environmental data collection effort:

TABLE I

1. Planning
2. Sample Collection
3. Laboratory Analysis
4. Data Review/Validation
5. Data Assessment

For each of these generic phases, there are corresponding quality assurance activities:

TABLE 2

Env.Data Collection Activity	QA Activity
1. Planning	1. Data Quality Objectives (DQO's), Quality Assurance Project Plans (QAPP's)
2. Sample Collection	2. Standard Operating Procedures (SOP's), Audits
3. Laboratory Analysis	3. SOP's, Contract Specs, Audits
4. Data Review/Validation	4. SOP's
5. Data Assessment	5. DQO's

At EPA, we have put a great deal of effort in quality assurance for Superfund. Unfortunately, when Superfund started, Quality Assurance was not an integral component of EPA engineers' culture. Quality Assurance was a laboratory thing. Quickly, Superfund RPM's found that contractors were fallible. Data coming back - when it finally did arrive - couldn't be used. It failed the quality assurance criteria, whatever they were. At the outset of Superfund, over 65% of the data was outright unusable, 25% due to sampling problems, and 75% due to laboratory performance. My Regional Administrator's image in Niagara Falls was certainly not that of the great American Eagle, but more like Ben Franklin's choice for the symbol of America, the turkey. Once a month for 6 months, Chris Daggett had to go to a public meeting in Niagara Falls for the infamous Love Canal site and tell the good Sister Margeen, the affected public, Lew Steele, and housewife, now activist, Lois Gibbs that EPA, the organization that was supposed to correct the problems caused by toxic/hazardousl wastes, could not tell the assembled crowd what was in the samples taken over eight months ago, and six months ago, etc. Somebody in the Region had to get religion, that is Quality Assurance Religion, and start practicing it fast. A cadre of chemists were pulled together (an EPA euphemism for stealing resources from other programs), and EPA was immersed in the Stage 4 QA activities of Data Review/Validation. There were no contracts available to review other contractor's data; it was an EPA function. It seemed every available resource had to be thrown into the Data Validation/Data Review process if the customer's needs for data were to be met. This is not to say that other phases were ignored, but they sure didn't get the attention in resources or priority that would lead to a better, more balanced, program.

Simply put, the Regional Administrator didn't enjoy hanging out there in Niagara Falls, or anywhere else for that matter, and it was QA's job to ensure that this wouldn't happen again. Recently, QAMS and others have openly questioned if it isn't time to start paying more attention to the remaining QA activities in other phases of the data collection process. I agree.

Parenthetically, I will use Region II examples during this presentation partly because I know us best, but also because I recognize that most of what occurs in Region II happens in other regions to a greater or lesser extent. We're in that 95% confidence interval of who does what most of the time.

Planning Phase:

As I mentioned, we do expend effort and attention, albeit insufficient, on the planning phase of our environmental data collection activities. Currently, our planning phase QA is carried out through the requirement for, and review of, Quality Assurance Project Plans (QAPP's) or Sampling and Analysis Plans (SAP's). Although we feel confident that our guidance and training for consistent planning requirements meet our objectives for development of relevant plans; reviews are conducted by knowledgeable staff professionals to assure that appropriate QA requirements are incorporated. We have not been able to participate effectively in scoping meetings held by the RPM and the contractor who prepares the QAPP or SAP. Resources are the issue. "Prevention is the cure" is not a concept readily accepted if resources are required for implementation. These meetings, if they were held, would help us to understand the purpose of the data collection activity and identify what questions the generated data, converted into useful information, are supposed to help answer. Lack of understanding the reason for collecting data sometimes leads to a false sense of security. In more than one case where the Quality Assurance Project Plan was acceptable, the resulting data did not meet the informational needs of the decision maker.

Region II has a dioxin site in the Ironbound section of Newark, New Jersey. The site was essentially cleaned, along with the adjacent streets and markets. The collected dioxin from the cleanup was, and still is, securely stored on site. The site is on the banks of the Passaic River. Many of you may recall the headlines on this site. Many discussions were held with the Superfund program staff and biological/technical staff to appropriately design a study to define the magnitude, extent, and impact of the dioxin contamination on the Passaic River/Newark Bay ecosystem. Field procedures and analytical methods were specified to assure that biological impact criteria levels would be measurable, etc. The QAPP was approved, and the field sampling and laboratory analyses were conducted in accordance with the plan, yielding data within biological criteria levels. Unfortunately, the critical, most demanding data users were not part of the staff designing the study. The bioaccumulation concept was not considered in the planning phase, and when the risk assessment derived concentrations were calculated, it was discovered that the analytical detection levels were orders of

magnitude above those necessary to make an appropriate ecological assessment. The study had to be redone to meet the needs of the data user.

As you can readily see from this example, an excellent QAPP yielded good quality data that did not meet the information requirements of the decision makers. " The operation was a success, but the patient died."

Let's try an analogy: You've gotten a new job that requires you to drive to work and consequently, you need a new car. You're moving your family, and with the night school class, you just don't know when you're going to find time to shop for the car. Good old mom comes to the rescue. She'll shop around, and when you come to dinner next week, she'll have it all scoped out. Sure enough, next week, even before she served any food, she proudly announces that she's decided that you should buy the new little Cadillac convertible. It's powerful, and quick, and sporty, absolutely gorgeous, and, or course, it's a Cadillac.

You then ask, "But Mom, doesn't that cost an awful lot, and does it have room for two kid seats (I maybe forgot to tell you it's going to be twins), and can it pull a trailer with all of the camping stuff, and does it have two air bags, and does it get good gas mileage, and what about reliability, and how about safety?" Well, Mom doesn't actually have any answers to these questions. You then start yelling and screaming about how you need the car and she ought to know what you need, and at least she ought to know not to rely on company ads..."

Finally, at that point your brother, Quincy Adams Ogilvy, or "QAO," interrupts. "Hold it, hold it," he says, "It's not her fault. How could she know what you need, or what is important to you, or even when you need the information. You didn't tell her any of that. What you both really need is a Quality Assurance Plan to ensure that your needs are stated clearly up front and that her methodologies will satisfy them. You need to tell her, up front, that you need an inexpensive, efficient, safe, car that can pull a trailer, etc. You also need to agree that Consumer Reports is enough, or that test drives are needed, or that a chart comparing different models is required, or whatever." And so forth.

I think I've made my point up 'til now without discussing the term Data Quality Objectives (DQO's). What is the purpose of collecting the data? What questions have to be answered? What are the user needs? Everyone agrees that DQO's are necessary, yes, even of utmost importance. But what are DQO's? If I asked each of you to write down your definition of DQO, I would probably have as many definitions as there are people in this room. Most would be somewhat similar, but they would range from the rigorously statistical definition on one end of the spectrum to the general conceptual definition on the other end of the spectrum. If DQO's are as important as we feel they are (and I agree with that assessment), then we in the business of QA need to develop a definition that is comprehensible and consistent across agency and organizational boundaries, one that is user

friendly (ie someone other than a statistician can understand and use), and a definition that allows the information user sufficient understanding so that they can appropriately participate in the development of the DQO's. Unless they are active participants, we are likely to continue to have repetition of the example scenario which I have shared.

We must develop a mechanism which assists the customer in clarifying and communicating his needs.

Sample Collection Phase:

Field sampling phase activities can only be quality assured by a combination of appropriate, planned and documented field procedures or SOP's which are designed to meet all the informational requirements of the data user. But as the saying goes "The best laid plans of mice and men.....". On site audits are essential to provide us with the assurance that the sampling plan and field procedures described, are, in reality, being followed. Not all deviations from the QAPP or SAP occur as an attempt to circumvent or shortcut the planned procedures. Anyone who has been in the field recognizes that Murphy's law is always in operation on any field sampling project. For example, how often does the field sampling plan identify the conditions under which the sampling event is aborted. It should. The field team should know whether one well not recovering within the specified time after evacuation in the QAPP is a valid criteria for aborting the sampling event. Maybe the water bearing layer will never permit the well to recover within the specified timeframe. The point I wish to make here is that the plan should not be followed just because it is "the approved plan". The sampling team must understand the rationale, the "why", of the requirements of the sampling and analysis plan, so that they can appropriately modify the plan when circumstances require such modification, and annotate the plan.....without compromising the information requirements of the data user.

As QA auditors, if we observe field practices which will compromise data quality, we must immediately inform the data user, not wait and write a report qualifying or rejecting the data several weeks or months down the road. In a recent audit, a PRP field contractor was observed by our QA staff auditors to grossly violate the planned protocol for VOA sampling. We notified the RPM and the PRP immediately, and a very grateful PRP was able to immediately resample while the contractor was on site, and only pay to have appropriate samples analyzed, thus realizing great cost savings over having to remobilize, resample, and reanalyze in the future.

Laboratory Analysis Phase:

Here we go again with laboratory analysis. How many times can it be said. You have to make sure the method you select will give you what you need! If you need low ppb level organic data, then you better be using the appropriate low level organic methods. Familiarity with the concepts of detection limits and quantitation limits and Contract Required Detection Limits, ...etc, is extremely important. I am familiar with a case in Region II where the project manager for a treatability study needed detection levels below the 1-5 ppb concentration of concern who requested low level analyses. He was told by the "helpful" QA staff that the available methods were good for concentrations in the 10-25 ppb range. He had his kazillion samples, oops! an exaggeration (over 3000 in reality), booked and analyzed and the data validated. Beaucoup dollars and months later, upon receipt of the validated data, the manager called the quality assurance officer totally devastated. The data were all below detection levels in the 10-25 ppb range. The quality assurance officer was pretty satisfied with the lab's performance. "What was this guy's problem?" The project manager told the QA person that he really needed information in the 1-5 ppb range, and this data was useless to him. Further discussion revealed that the project manager thought that the method he had indicated as acceptable would really get down to the 1-5 ppb level if the laboratory did everything right. Lesson learned..... Not all project managers have a good feel for analytical chemistry. Just as I barely passed organic chemistry because the professor called it a "black art", project managers look at analytical chemistry as a very black art.

We cannot assume that the requestor is familiar with the options available to him. In Region II, the QA staff and the laboratory chemists handle the majority of advice and decisions regarding what analytical protocols are used. We have let the project managers "off the hook", and now the QA folks are responsible.

Care needs to be taken in choosing the appropriate analytical method, and the chemists who will perform the analyses. The laboratory doing the non-routine method should "play" with the method and the sample matrix to be analyzed to be sure that the method chosen will yield the results anticipated. Too often, we send the entire set of site samples off to a laboratory with only blind faith that all will go well. As I discussed in the field sampling phase, we should know when to abort the mission.

As with field sampling, there must also be audits to verify that the laboratories are, in fact, following their stated SOP's.

Data Review/Validation Phase:

The principal purpose of data validation is to assure that the number on the paper really represents what is in the sample. How precise and accurate is the data,. etc.? As I indicated earlier in this presentation, EPA spends an inordinate proportion of time and effort on this phase of the process. We've developed SOP's on how to validate organic, inorganic, and dioxin data sets; we've given training courses to our support contractors and to any other interested parties, especially for validation of RCRA data sets.

Data validation produces for the customers a data set that indicates the quality of the data, ranging from an indication of "the data is exactly what it is shown to be" to "the data is so flawed as to render it useless". The user understands these two ends of the spectrum, but if the data is qualified with conventional flags like "the actual value is known to be greater than (or less than) the reported value" or "the presence of the target analyte is verified, but not quantified", the user becomes confused and unsure of the effect of the qualifier on the use of the data. As a result he generally either disregards the data or ignores the qualifiers. The latter approach seriously compromises Records of Decision (ROD's) when qualifiers are deleted from backup documents. Similarly, if the qualified data is disregarded, potentially useful information is lost.

Clearly, the data review process that only produces a product that eliminates constructive use of the data is not meeting the needs of the customer.

Data Assessment Phase:

Data Assessment is the phase that remedies the uselessness of the product resulting from data review/validation. We've probably spent less than 1% of our effort on this phase. These activities incorporate the sum total of all environmental data and qualifying information into the basis for the decision which must be made by the data user. The conversion of data into useful information is critical to the decision maker's success. He needs the QA staff to relate all the qualifying information to data usability; to advise on what the qualifiers mean; and to establish a measure of confidence for the data to be used as the basis for decisionmaking. As the QA experts, we must help to "close the loop" for the decision makers, and provide them with the quality framework within which they can interpret the environmental data collected.

It is our obligation to inform them of any data limitations, and the risks associated with using specific data sets.

How can we best accomplish these tasks? What mechanisms are there to involve the users in this phase of the process? Did we provide the customer what he identified as necessary to make the most informed decision? If we did all the "right" QA activities in each phase of the project, and are unable to use these activities to provide useful information to the data user, then we have failed. On the other hand,if we build user focus and customer involvement into each phase of the project, we are assured at every milestone of the project that we are on the "right" road.

There are two endings to this presentation - the first and less kinder and gentler is the message - COMMUNICATE, COMMUNICATE, COMMUNICATE ..... At almost every step of the way there is a crying need for communication. Without meaningful communication between the usere and supplier, there will never be other than accidental success.

As a second, more formal ending, I hope I have been able to stimulate your thinking on how appropriate QA might be developed through active customer involvement; how we might better build a " user focus" into quality assurance activities during every phase of any environmental data collection effort. Your active participation in the upcoming sessions is important if we are to develop a consensus approach which will yield useful information, not just qualified data, to our customers, the decision makers of our respective agencies. Thank you.