1   **ATTACHMENT C-4**

2   **RCRA WASTE ANALYSIS**

3   **QUALITY ASSURANCE/QUALITY CONTROL PLAN**

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1   RCRA WASTE ANALYSIS

2   QUALITY ASSURANCE/QUALITY CONTROL PLAN

3   October 2013

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1   **ACRONYMS**

2

3

4   ASTM   American Society for Testing and Materials

5

6   DOT   Department of Transportation

7   DQO   data quality objective

8

9   EDS   Explosive Destruction System

10   EPA   Environmental Protection Agency

11

12   GC/MS   gas chromatograph/mass spectrometer

13

14   JPM E (P)   Joint Project Manager Elimination (Provisional)

15

16   MDL   method detection limit

17

18   NELAP   National Environmental Laboratory Accreditation Program

19   NIOSH   National Institute for Occupational Safety and Health

20

21   OS   original sample

22   OSD   original sample duplicate

23

24   PCD   Pueblo Chemical Depot

25   PQL   practical quantitation limit

26

27   QA   quality assurance

28   QA/QC   quality assurance/quality control

29   QC   quality control

30

31   %RSD   percent relative standard deviation

32   RCRA   Resource Conservation and Recovery Act

33   RPD   relative percent difference

1	TIC	total ion chromatogram
2	TSDF	treatment, storage, and disposal facility
3		
4	VOC	volatile organic compound

1.0 INTRODUCTION

The procedures and methodologies used to characterize Explosive Destruction System (EDS) operation wastes will ensure proper treatment of wastes; safe handling and storage of treatment residues; and safe handling, treatment, or disposal of wastes shipped offsite. This document summarizes quality assurance/quality control (QA/QC) measures to ensure adequate sampling and analysis of waste streams. The Army will sample and analyze liquid and solid wastes for chemical agent concentrations under the U.S. Army Chemical Materials Agency *Programmatic Laboratory and Monitoring Quality Assurance Plan*. This plan addresses only the QA/QC requirements for Resource Conservation and Recovery Act (RCRA) waste sampling and analysis.

A contract laboratory will be used to analyze liquid and solid waste samples for RCRA hazardous waste constituents and characteristics in accordance with *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,* SW-846, current edition, or other EPA-recognized methods, as referenced in the Colorado Hazardous Waste Regulations (6 CCR 1007-3). The contract laboratory selected will be an accredited National Environmental Laboratory Accreditation Program (NELAP) laboratory.

Detailed information on waste characteristics, waste analysis, and waste characterization methodologies for EDS operations is in Section C-2 of this permit modification.

1.1   Purpose

The primary purpose of waste sampling and analysis is to ensure that wastes are properly characterized in compliance with RCRA requirements for general waste analysis 6 CCR 1007-3 § 264.13(b) and (c). Waste sampling and analysis is also performed to ensure the safe management of wastes being stored or treated, proper disposition of treatment residues, and proper characterization of waste for shipment to a permitted hazardous waste treatment, storage, and disposal facility (TSDF).

This plan establishes the requirements that will be followed to ensure RCRA waste sampling and analysis objectives are met, that all data obtained are technically sound, statistically valid, and properly documented. This plan also identifies the tools that will be used to measure the degree of certainty that all objectives have been met.

1   **1.2   QA/QC Objectives**

2

3   Quality Assurance (QA) is a systematic approach to ensure that processes and activities meet quality,

4   safety, technical, and management requirements, and that the data and results compiled for waste analysis

5   are valid and properly documented. QA for the Pueblo Chemical Agent-Destruction Pilot Plant (PCAPP)

6   EDS project waste characterization operations will ensure that waste sampling and laboratory analysis

7   operations are performed in accordance with approved plans and procedures. Quality Control (QC) is the

8   mechanism through which QA achieves its goals. The primary objective of this plan is to control and

9   characterize any errors associated with the collected data. QA activities, such as using standard

10   procedures for locating sample sites and collecting samples, are intended to limit the introduction of

11   errors. QC activities, such as collecting duplicate samples and including blanks in sample sets, are

12   intended to provide the information required to characterize any errors in the data. Other QC activities,

13   such as planning the QC program and auditing ongoing and completed activities, ensure that the specified

14   procedures are followed and that the QA information needed for characterizing errors is obtained.

15

16   The second QA/QC objective is to confirm that waste sampling and analysis has been conducted

17   according to the specifications of the PCAPP EDS Waste Analysis Plan and requirements of this QA/QC

18   Plan. QA/QC activities will include:

19

20   •   Field inspections – performed by the QA Officer or designee, depending on the activity.
21   The inspections will be primarily visual examinations but may include measurements of
22   materials and equipment used, techniques employed, and the final products. The purpose
23   of these inspections is to verify that a specific guideline, specification, or procedure for
24   the activity is successfully completed. Checklists are completed to document results.

25

26   •   Field testing – performed on the site by the QA Officer or designee according to specified
27   procedures.

28

29   •   Laboratory analyses – performed by the contract laboratory on samples of waste. The
30   purpose of laboratory analyses is to determine constituents or characteristics present and
31   their concentration level.

32

33   •   Verifying contract laboratory maintains instruments and performs calibrations.

1 **1.3   Responsibilities and Authority**

2

3   The Joint Project Manager Elimination (Provisional) (JPM E (P)) Site Manager will be responsible for

4   ensuring that appropriate data are provided to Pueblo Chemical Depot (PCD) for submittal to the state and

5   federal regulatory agencies, as stipulated by the PCD RCRA permit. External audits and surveillances,

6   either announced or unannounced, will be conducted as required. Additional external audits or

7   surveillances will be conducted by other qualified organizations as requested by JPM E (P). All

8   documents and data produced by the contract laboratory will be eligible for inspection. The

9   environmental regulatory agencies may also review these data to ensure that the EDS operations

10 personnel are complying with permit requirements as they pertain to waste characterization and treatment

11 operations.

1   **2.0 DATA QUALITY OBJECTIVES**

2

3   Data quality objectives (DQOs) are qualitative and quantitative statements developed by data users to

4   specify the quality of data needed from a particular activity. The Environmental Protection Agency

5   (EPA) provides the basis for developing the DQOs (EPA/240/B-06/001, February 2006).

6

7   The DQOs for waste sampling and analysis will include, but will not be limited to, the following.

8

9   **2.1   Sampling and Analysis Objectives**

10

11   DQOs for waste sampling and associated data analyses are:

12

13   •   Determine if waste samples are representative of the wastes at the time the samples were

14   taken

15

16   •   Determine if treatment residue (neutralent wastes) concentration values meet the

17   treatment level requirements

18

19   •   Ensure waste characterization is adequate for waste acceptance at a permitted hazardous

20   waste TSDF

21

22   •   Ensure laboratory analytical results can be validated.

23

24   **2.2   Data Collection/Sampling Objectives**

25

26   Collected data must be scientifically sound, of known quality, and thoroughly documented. The DQOs

27   for the data assessment are:

28

29   •   Accuracy[1] – The accuracy of an analytical method is represented as the mean of the

30   percent recovery of the target analyte from a given matrix. The quality of the data can be

31   assured through the comparison of individual data values, expressed as percent recovery,

32   to established QC limits. QC limits are derived from analysis of standard matrix QC

33   samples, solvent (nonmatrix), QC samples, or surrogate analytes in the field samples.

---

[1]   When measuring accuracy, the contract laboratory will prepare and analyze matrix spike duplicate samples.

1   •   Precision[2] – The precision will be the agreement between the collected samples

2   (duplicates) for the same parameters, at the same location, and from the same collection

3   device.

4

5   •   Representativeness – The representativeness will address the degree to which the data

6   accurately and precisely represent a real characterization of the population, parameter

7   variation at a sampling point, sampling conditions, and the environmental condition at the

8   time of sampling. The issue of representativeness shall be addressed for the following

9   points:

10

11   -   Based on the waste stream and its volume, an adequate number of samples are

12   collected

13

14   -   The representativeness of selected media has been accurately defined

15

16   -   The sampling and analytical methodologies are appropriate

17

18   -   The environmental conditions at the time of sampling are documented.

19

20   •   Completeness – The completeness shall be defined as the ability of the sampling and

21   analytical methodologies to accurately measure the constituents of concern present in the

22   waste. The goal for completeness is 95 percent.

23

24   •   Comparability – The comparability of the data generated shall be defined as the data that

25   are gathered using standardized sampling methods, standardized analysis methods, and

26   quality-controlled data reduction and validation methods.

27

28   •   Sensitivity – Reflects the ability of the analytical method to detect analytes of interest

29   below the level of concern. This goal is achieved by identifying the level of concern,

30   choosing a method with appropriate detection limit, and ensuring the laboratory analyses

31   calibration standards are at or below the level of concern.

---

[2]   When measuring precision, the contract laboratory will prepare and analyze matrix spike duplicate samples.

1   **3.0 SAMPLING QA/QC**

2

3   Methods used to obtain a representative sample will be consistent with sampling approaches and

4   protocols described in *Test Methods for Evaluating Solid Waste*, *Physical/Chemical Methods*, SW-846,

5   current edition, or other EPA-recognized methods, as referenced in the Colorado Hazardous Waste

6   Regulations (6 CCR 1007-3). The selected sampling methods used to characterize PCAPP EDS waste

7   streams are summarized in Section C-2 of the PCAPP EDS permit modification, and are from: *Test*

8   *Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, current edition, *Annual Book*

9   *of ASTM Standards*, American Society for Testing and Materials (ASTM), current edition; or other

10   EPA-recognized methods, as referenced in the Colorado Hazardous Waste Regulations (6 CCR 1007-3).

11

12   The basic sampling procedure for general RCRA waste analysis will be as follows:[3]

13

14   •   Obtain samples using a precleaned sampler.

15

16   •   Fill sample containers according to laboratory instructions. Sample containers may arrive

17   from laboratory with preservatives already added to container as applicable.

18

19   •   Label sample containers.

20

21   •   Properly clean and decontaminate sample containers and sampling hardware or dispose

22   of sampling equipment as applicable.

23

24   •   Custody-seal all sample containers and place samples in a durable ice-filled cooler or

25   comparable receptacle for transport to the laboratory or immediately place in a

26   refrigerator for storage pending shipment to the laboratory for analysis. Protective

27   material may be placed around the sample containers prior to placement in the cooler or

28   comparable receptacle as necessary.

29

30   •   Complete the chain-of-custody and laboratory request-for-analysis forms.

---

[3]   All samples will be analyzed for and cleared of chemical agent before the samples are transferred to the contract
       laboratory for hazardous waste characterization analyses.

1   •   Review all paperwork and enclose the forms in a leaktight, polyethylene bag taped to the

2   underside of the cooler lid or comparable receptacle.

3

4   •   Seal the coolers or comparable receptacle with tape and mark in accordance with

5   Department of Transportation (DOT) and Army requirements, as applicable.

6

7   •   Transport coolers to the analytical laboratory.

8

9 **3.1**   **Sample Containers, Preservation, Handling, and Management**

10

11 Sample container selection, preservation, handling, and management are critical to sample quality.

12 Considering waste compatibility, durability, volume, and analytical sensitivities, the containers listed in

13 **Tables 1** and **2** are recommended for the EDS waste sampling efforts. **Tables 1** and **2** also provide

14 preservation requirements and holding times that will be followed to preserve sample integrity.

15

16 All sample containers will be labeled with at least the following information:

17

18   •   A unique alphanumeric identifier

19   •   Date and time of collection

20   •   Sample collector's name

21   •   Preservatives used

22   •   Analyses/parameters requested.

23

24 An example of a sample container label is provided in **Figure 1**. Immediately after collection, filled

25 sample containers will be placed on ice (frozen water or dry [CO_2] ice) or ice packs, if necessary, in

26 durable coolers or comparable receptacles for transport to the laboratory or samples may immediately be

27 placed in a refrigerator pending shipment to a laboratory for analysis. If using dry ice, leave enough

28 space to allow gas to escape. RCRA analyses will be conducted at a contract laboratory within the

29 allowable holding times for sample analysis. Coolers or comparable receptacles will be tightly sealed

30 before sample shipment occurs. Samples then will be transported to offsite laboratories to ensure delivery

31 within the allowable holding times for sample analysis. All sample collection, preparation, packaging,

32 transportation, and analysis will conform to the requirements of SW-846, current edition, or other

33 EPA-recognized methods, as referenced in the Colorado Hazardous Waste Regulations (6 CCR 1007-3).

1   Table 1. Requirements for Liquid Samples

2

Parameter	Holding Time[a]	Bottle Type	Preservative	Standard Volume	Minimum Volume
Chemical Agent[b]	N/A N/A	Stainless Steel or Glass jar or bottle with a Teflon-lined cap	4°C 4°C	25 mL 125 mL	2 mL 2 mL
Corrosivity/pH	Analyze immediately	HDPE plastic	4°C	250 mL	50 mL
Ignitability	NS	Glass	None	500 mL	100 mL
TC Metals	180 days	HDPE plastic	pH<2: HNO_3; 4°C	1 L	100 mL
TC Mercury	28 days	HDPE plastic	pH<2: HNO_3; 4°C	500 mL	100 mL
TC Volatile Organics (D-list)	14 days to ZHE, 14 days from ZHE to analysis	Amber glass 40-mL vial with PTFE-lined septum cap	pH<2: HCl; 4°C	2 x 500 mL	500 mL
TC Semivolatile Organics (D-list)	7 days to extract; 40 days to analyze	Amber glass with PTFE-lined cap	4°C	2 x 1 L	1 L

3
4   Notes:
5
6   [a]   Holding times are from the date of collection as referred to in the Federal Register, Vol. 49, No. 209, October 26, 1984, as applicable.
7   [b]   Chemical agent analysis is performed at the PCAPP EDS Mobile Analytical Platform.
8

9 HDPE	=	high-density polyethylene	NS	=	none specified
10 L	=	liter	PTFE	=	polytetrafluoroethylene (Teflon[®])
11 mL	=	milliliter	TC	=	toxicity characteristic
12 N/A	=	not applicable	ZHE	=	zero headspace extraction

13
14   Reference: *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, current edition, or other EPA-recognized
15   methods, as referenced in the Colorado Hazardous Waste Regulations (6 CCR 1007-3).

1   Table 2. Requirements for Solid Samples

2

Parameter	Holding Time[a]	Bottle Type	Preservative	Standard Amount	Minimum Amount
Chemical Agent[b]	N/A	Glass	4°C	50 g	20 g
Ignitability	7 days	Glass	None	100 g	30 g
TC Metals	180 days	HDPE plastic	4°C	100 g	25 g
TC Mercury	28 days	HDPE plastic	4°C	100 g	25 g
TC Volatile Organics (D-list)	TCLP Extract within 7 days, 40 days to analyze	Amber glass	4°C	100 g	25 g
TC Semivolatile Organics (D-list)	TCLP extract within 14 days, preparative extract within 14 days; 40 days to analyze	Amber glass	4°C	100 g	50 g

3
4   Notes:
5
6   [a]   Holding times are from the date of collection as referred to in the Federal Register, Vol. 49, No. 209, October 26, 1984, as applicable.
7   [b]   Chemical agent analysis is performed at the PCAPP EDS Mobile Analytical Platform.
8
9   g   =   gram
10   HDPE   =   high-density polyethylene
11   N/A   =   not applicable
12   TC   =   toxicity characteristic
13   TCLP   =   Toxicity Characteristic Leaching Procedure
14
15   Reference: *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, current edition, or other EPA-recognized
16   methods, as referenced in the Colorado Hazardous Waste Regulations (6 CCR 1007-3).

```
┌─────────────────────────────────────┐
│  ┌───────────────────────────────┐  │
│  │                               │  │
│  │  Sample ID No.: _____  │  │
│  │  _____    │  │
│  │                               │  │
│  │  Sample Description: _____   │  │
│  │  _____    │  │
│  │                               │  │
│  │  Collection Date/Time: _____  │  │
│  │                               │  │
│  │  Preservative: Yes __ Type __ No__  │
│  │                               │  │
│  │  Analysis: _____  │  │
│  │                               │  │
│  │  Sampled By: _____  │  │
│  │                               │  │
│  └───────────────────────────────┘  │
└─────────────────────────────────────┘
                          P-0197-009/label.cdr
                          9/28/12
```

Figure 1. Example of a Sample Container Label

4 The contract laboratory will provide sample containers, labels, and preservatives for parameters of
5 interest.

7 Sampling procedures that will be used at the PCAPP EDS site are designed to ensure that each sample is
8 accounted for at all times. The primary objectives of the sample control procedures are as follows:

10 • Each sample collected for analysis is uniquely identified.

12 • Important and necessary sample constituents are preserved (e.g., refrigerated, capped).

14 • Samples are protected from loss, damage, or tampering.

16 • Any alteration of samples during collection or shipping (e.g., preservation, breakage) is
17   documented.

19 • A record of sample custody and integrity is established.

21 • The correct samples are analyzed and are traceable to the applicable data records (e.g.,
22   chain-of-custody, field records, request for analysis, laboratory ledgers).

1 As part of sample management procedures, personnel collecting the samples will maintain a permanent

2 record of sampling activities. This record will include: the purpose of sampling; date and time of

3 collection; sample number; sampling location, sampling methodology, container description, waste

4 description (metal fragments, rinsewater, etc.); description of process originating the waste; name and

5 address of field contact; number and volume of samples; field observations; destination and transporter;

6 and signature of collector.

7

8 Transportation of samples will be in accordance with DOT, EPA, and Army requirements. Hazardous

9 waste samples will be properly packaged, marked, and labeled. Shipping papers will be prepared as

10 required by DOT regulations, EPA requirements, and Army regulations and guidelines.

11

12 All equipment used to sample waste materials will be disposable or designed for easy decontamination.

13 Contaminated disposable equipment will be managed as hazardous waste, as appropriate. Cleanable

14 equipment will be thoroughly decontaminated prior to reuse. Decontamination solutions will be managed

15 as hazardous waste, as appropriate.

16

17 **3.2   Chain-of-Custody**

18

19 A chain-of-custody record will accompany samples at all times. An example of a chain-of-custody form

20 is included as **Figure 2**. The personnel performing the sampling will be responsible for initiating the

21 chain-of-custody procedures at the time samples are collected. A chain-of-custody record form will be

22 used to document sample collection activities, including sampling site, sample identification, number of

23 samples, and date and time of collection. The form also will document the names of responsible

24 individuals and dates and times of custody transfers.

25

26 Samples will be received at the laboratory by a designated sample custodian. This individual will

27 carefully review received samples and documentation for compliance with applicable sampling and

28 documentation requirements such as type and condition of container, sample preservation, collection date,

29 and chain-of-custody records. After verifying that all samples submitted are listed and that the required

30 information is listed on the form, the sample custodian will sign and date the chain-of-custody form. The

31 sample custodian will then store and secure the samples appropriately (e.g., in locked refrigerator).

32

33 Chain-of-custody documentation for samples will continue throughout the analytical process. After

34 logging in and storing the samples, the sample custodian will distribute sample-receiving logs, which will

35 list sample numbers and analyses to be performed, to designated laboratory personnel. Upon completion

Figure 2. Example Chain-of-Custody Form

1   of analyses, results will be submitted to the laboratory data management section along with QA/QC

2   information. Much of the analytical results will be used to characterize wastes prior to the wastes being

3   sent to a permitted hazardous waste TSDF. All data sheets and laboratory records will be retained as part

4   of the permanent record.

5

6   **3.3   Field QA/QC for RCRA Waste Sampling Analysis**

7

8   The goal of RCRA waste sampling and analysis is to provide representative information regarding the

9   characteristics of wastes generated during EDS operations. The laboratory chosen to perform RCRA

10   analyses will be NELAP-certified, have significant capacity and quality to meet analytical demand,

11   provide timely and complete data packages, and provide technical support in the form of sample kits and

12   shipping supplies. Both field and laboratory blanks will be prepared by the laboratory. For each waste

13   stream sampled, appropriate QA/QC samples will be collected, as shown in **Table 3** and described below.

14

15   Field QC samples may include trip blanks, rinse blanks, and/or duplicate samples. Trip blanks are used to

16   verify that field procedures do not contaminate containers or samplers. They are prepared using

17   analyte-free water when samples are to be analyzed for volatile organic compounds (VOCs). At least one

18   trip blank will be prepared and analyzed for each cooler used for storing and transporting VOC samples.

19

20   Rinse blanks are used to detect cross-contamination resulting from the use of non-dedicated (re-used)

21   sampling equipment. It is anticipated that disposable, one-time use sampling equipment will be used.

22   However, if sampling equipment is re-used, at least one rinse blank will be collected for every 20 samples

23   per parameter group and matrix. This blank will be prepared in the field by rinsing the cleaned sampling

24   equipment with analyte-free water and collecting the rinsate. There will be a minimum of one rinse blank

25   per lab set. If only a few samples are taken, there will still be one rinse blank per 20 samples.

26

27   Duplicates are samples collected at the same time from the same source and are used to measure sample

28   homogeneity and analytical precision. At least one duplicate will be collected per 20 samples. There

29   shall be a minimum of one duplicate per lab set. If only a few samples are taken, there will still be one

30   duplicate. However, if a large number of samples are taken, there will be one duplicate per 20 samples.

31

32   **3.4   Health and Safety Protocols**

33

34   During all sampling activities, strict compliance with industrial hygiene and safety standards will be

35   mandatory. All personnel involved in sampling activities will be trained in the applicable safety

1   Table 3. Sample Quantity Requirements

2

Waste Stream	Parameter/Analysis[a]	Sample[b]	Field Duplicate Sample	Trip Blank[c]
Mustard (HD//HT) Neutralents	H	(1) – 25 mL Stainless steel bottle or 125 mL glass jar or bottle with a Teflon-lined cap	(1) – 25 mL Stainless steel bottle or 125 mL glass jar or bottle with a Teflon-lined cap	
	TC Metals	(1) – 1 L HDPE bottle (1) – 500 mL HDPE bottle	(1) – 1 L HDPE bottle (1) – 500 mL HDPE bottle	
	TC SVOCs	(2) – 1 L Amber glass bottles w/PTFE-lined cap	(2) – 1 L Amber glass bottles w/PTFE-lined cap	
	TC VOCs	(3) – 40 mL Amber glass VOA vials	(3) – 40 mL Amber glass VOA vials	(1) – 40 mL Amber glass VOA vial (deionized water)
	pH	(1) – 250 mL HDPE bottle	(1) – 250 mL HDPE bottle	
	Ignitability	(1) – 500 mL Glass bottle	(1) – 500 mL Glass bottle	
Rinsewaters	TC Metals	(1) – 1 L HDPE bottle (1) – 500 mL HDPE bottle	(1) – 1 L HDPE bottle (1) – 500 mL HDPE bottle	
	TC SVOCs	(2) – 1 L Amber glass bottles w/PTFE-lined cap	(2) – 1 L Amber glass bottles w/PTFE-lined cap	
	TC VOCs	(3) – 40 mL Amber glass VOA vials	(3) – 40 mL Amber glass VOA vials	(1) – 40 mL Amber glass VOA vial (deionized water)
	pH	(1) – 250 mL HDPE bottle	(1) – 250 mL HDPE bottle	
	Ignitability	(1) – 500 mL Glass bottle	(1) – 500 mL Glass bottle	

Table 3. Sample Quantity Requirements (Continued)

Waste Stream	Parameter/Analysis[a]	Sample[b]	Field Duplicate Sample	Trip Blank[c]
Used Decontamination Solution	H	(1) – 25 mL Stainless steel bottle or 125 mL glass jar or bottle with a Teflon-lined cap	(1) – 25 mL Stainless steel bottle or 125 mL glass jar or bottle with a Teflon-lined cap	
	TC Metals	(1) – 1 L HDPE bottle	(1) – 1 L HDPE bottle	
		(1) – 500 mL HDPE bottle	(1) – 500 mL HDPE bottle	
	TC SVOCs	(2) – 1 L Amber glass bottles w/PTFE-lined cap	(2) – 1 L Amber glass bottles w/PTFE-lined cap	
	TC VOCs	(3) – 40 mL Amber glass VOA vials	(3) – 40 mL Amber glass VOA vials	(1) – 40 mL Amber glass VOA vial (deionized water)
	pH	(1) – 250 mL HDPE bottle	(1) – 250 mL HDPE bottle	
	Ignitability	(1) – 500 mL Glass bottle	(1) – 500 mL Glass bottle	

Notes:

[a]   Mustard agents HD/HT are analyzed as H.
[b]   All sample containers should be filled completely.
[c]   One trip blank per cooler containing VOC samples.

H	=	Levinstein mustard
HDPE	=	high-density polyethylene
L	=	liter
mL	=	milliliter
PTFE	=	polytetrafluoroethylene (Teflon®)
SVOC	=	semivolatile organic compound
TC	=	toxicity characteristic
VOA	=	volatile organic analysis
VOC	=	volatile organic compound

1   procedures; the use of all cleaning, decontamination, and sampling equipment; and proper cleaning and

2   decontamination techniques. Sampling personnel will have received Occupational Safety and Health

3   Administration health and safety training for hazardous waste operations. Sampling personnel will be

4   required to wear protective gear as dictated by the Health and Safety Plan. If personnel accidentally

5   contact waste material, decontamination procedures will be performed.

1 **4.0 LABORATORY QA/QC**

2

3 An analytical laboratory must conduct its operations in such a way as to provide reliable information.

4 The QA/QC of data generated by the analytical laboratory will be controlled by a Laboratory Quality

5 Assurance Plan. At a minimum, the contract laboratory used for RCRA analysis will follow the

6 guidelines presented in this section and the laboratory QA/QC plan will document the following:

7

8 • Sample custody and management practices

9

10 • Sample preparation and analytical procedures

11

12 • Instrument maintenance and calibration procedures

13

14 • Internal QA/QC measures including the use of method blanks, spiked samples, and

15 duplicates.

16

17 Internal QA/QC checks are established by submitting QA and QC samples to the analytical laboratory.

18 The frequency of laboratory QC samples is per batch (20 or fewer samples) and is defined by the

19 laboratory's QA/QC plan and procedures. The types of laboratory QC samples are:

20

21 • *Method Blank*. Defined as laboratory grade water taken through the entire analytical

22 procedure to determine if samples are accidentally contaminated by chemicals in the

23 laboratory.

24

25 • *Laboratory Control Samples*. Defined as known matrix spiked with compound(s)

26 representative of the target analytes and is used to document laboratory performance.

27

28 • *Matrix Spike*. Defined as an aliquot of a matrix spiked with a known concentration of the

29 analytes of interest. The matrix spike is subjected to the entire analytical procedure to

30 indicate the appropriateness of the method for the matrix by measuring recovery.

31

32 • *Duplicate*. Defined as a second aliquot taken from the original sample container and

33 analyzed separately to test repeatability of an analysis.

1 **4.1 Preventive Maintenance**

2

3 Preventive maintenance procedures are intended to prevent instrument malfunctions and to detect as early

4 as possible any potential problems with the analytical equipment that might result in inaccurate analyses.

5 Analytical instruments or instrument systems in use at the laboratory will undergo routine preventive

6 maintenance as recommended by the vendor or manufacturer, or if such maintenance is warranted, based

7 on equipment operating experience. All maintenance procedures will be documented.

8

9 Each item of equipment, including reference standards, shall, when appropriate, be labeled, marked, or

10 otherwise identified to indicate its calibration status.

11

12 The laboratory will have documented instructions on the use and operation of all relevant equipment, on

13 the handling and preparation of items, and for calibrating equipment. All instructions, standards,

14 manuals, and reference data relevant to the laboratory's operation will be maintained up to date and will

15 be readily available to the staff.

16

17 Where computers or automated equipment are used for the capture, processing, manipulation, recording,

18 reporting, storage, or retrieval of calibration or test data, the laboratory will ensure the following: (1) the

19 computer software is documented and adequate for use; (2) computer and automated equipment is

20 maintained to ensure proper functioning and provided with the environmental and operating conditions

21 necessary to maintain its integrity; and (3) establish and implement procedures for protecting the integrity

22 of the data.

23

24 Maintenance and repair records will be maintained on all instruments and instrument systems, as required,

25 and will become part of the operating record.

26

27 **4.2 Routine Assessment of Precision, Accuracy, and Comparability of Analytical Data**

28

29 QA for analytical data from collected samples will include evaluation of precision, accuracy, and

30 comparability, which are discussed below.

31

32 Precision

33

34 Precision in reference to laboratory analysis is a measure of mutual agreement among individual

35 measurements of the same property, usually under prescribed similar conditions. Precision is assessed by

1 means of laboratory duplicate/field replicate sample analysis. The laboratory objective for precision is to

2 be within the control limits for the analytical methods as published for the EPA-recognized methods or

3 Army methods, as applicable.

4

5 Precision of the chemical laboratory data will be measured through the use of matrix spike duplicate

6 samples and calculated as the percent relative standard deviation (%RSD). The standard deviation, s, is

7 calculated from the variance, s^2, as follows:

$$s^2 = \frac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{n-1}$$

8 where $\bar{x}$ is the mean value of a variable, x_i is the value of an individual measurement of a variable, n is

9 the number of data points, and s is obtained from:

$$s = \left(s^2\right)^{1/2}$$

10 The %RSD is then:

$$\%RSD = s / \bar{x}$$

11 The standard deviation and %RSD are calculated for every constituent measured.

12

13 <u>Accuracy</u>

14

15 Accuracy means the nearness of a result, or the mean of a set of results, to the true value. Accuracy is

16 assessed by means of reference samples and percent recoveries. The laboratory objective for accuracy is

17 to be within the control limits for the analytical methods as published for the EPA-recognized method or

18 as established by the laboratory.

1   Accuracy of laboratory data will be measured through the use of matrix spike duplicate samples and will

2   be assessed through the calculation of percent recovery from any certified standard that the laboratory

3   analyzes as part of its ongoing QA/QC program. The percent recovery is calculated as follows:

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

4   where SSR is the spiked sample result, SR is the sample result, and SA is the spike added. The laboratory

5   also will be required to run a sufficient number or type of blanks to detect laboratory contamination.

6

7   <u>Comparability</u>

8

9   Comparability is the degree to which one data set can be compared with another. Comparability is

10   achieved by using consistent methods and standards that are traceable to a reliable source. All data will

11   be reported in units consistent with the conventions used for the given analyte and methods employed.

12   Comparability can be enhanced by using:

13

14   •   EPA SW-846 or EPA 600/4-88-039 methods of analysis

15   •   ASTM methods

16   •   National Institute for Occupational Safety and Health (NIOSH) Pocket Guide.

17

18   **4.3   Data Quality and Data Deliverables**

19

20   To ensure hazardous waste characterization data are reliable, the contract laboratory will operate and

21   conduct analyses according to the performance quality requirements identified in SW-846 Chapter 1,

22   Section 4, or other EPA-recognized methods, as referenced in the Colorado Hazardous Waste Regulations

23   (6 CCR 1007-3).

24

25   **4.3.1   Data Quality Assessment and Data Deliverables**

26

27   Data quality assessments will evaluate whether the data generated by the laboratories is consistent with

28   the established DQOs.

29

30   For the RCRA waste characterization, the contract laboratory will furnish a QA Manual (or QA Program

31   Plan) that defines the quality procedures and policies specific to that facility. An Environmental Scientist,

1   Laboratory Manager, or QA Coordinator will be responsible for reviewing the reports from that facility to

2   ensure consistency with RCRA limits and with the furnished QA Manual. An audit of the analytical

3   laboratory may be performed by an Environmental Scientist or assignee at any time during the EDS

4   operations.

5

6   **4.3.1.1 RCRA Characterization Deliverables**

7

8   For the RCRA analysis, data packages will consist of complete data packages with raw data. A request

9   for electronic deliverables may be made as appropriate. Summary reports for the RCRA characterization

10   will include but not be limited to:

11

12   •   Chain-of-custody, Field Sampling Logs, any associated correspondence

13   •   Name and address of laboratory (on letterhead)

14   •   EPA or other approved method used (with title and method number)

15   •   Client delivery order (or job) number

16   •   Sample identification, client, and laboratory number

17   •   Date and time sampled

18   •   Date and time sample received by laboratory

19   •   Date and time sample was extracted/digested

20   •   Dilution factor

21   •   Sample matrix

22   •   Date and time sample was analyzed

23   •   Parameters tested

24   •   Units reported

25   •   Concentration of each parameter found

26   •   Reporting limit or other similar limit for each parameter [practical quantitation limit

27   (PQL)]

28   •   Report date

29   •   Case narrative for each sample batch and any anomalies encountered with samples

30   •   Signature of laboratory supervisor or director (or assignee).

1 **4.3.1.2 QC Deliverables for RCRA Characterization**

2

3 Metals – Toxicity Characteristic Leaching Procedure extraction logs (EPA 1311), method blank results,

4 lab control sample and lab control sample duplicate [% recoveries with calculated relative percent

5 difference (RPD)], matrix spike and matrix spike duplicate (% recoveries with calculated RPD), original

6 sample (OS), and original sample duplicate (OSD) with calculated RPD.

7

8 Organics – extraction logs, method blank results, lab control sample, (% recovery), and matrix spike

9 (% recovery), and surrogates (% recovery).

10

11 Wet Chem – extraction preparation logs, method blank results, lab control sample and lab control sample

12 duplicate (% recoveries with calculated RPD), matrix spike and matrix spike duplicate (% recoveries with

13 calculated RPD), OS, and OSD with calculated RPD.

14

15 Other – For methods in which no spikes can be performed (for example, specific gravity) an original

16 sample and sample duplicate analysis must be performed and reported (OS/OSD with calculated RPD %).

17

18 **4.3.2   Data Qualifiers**

19

20 In cases where results are out-of-control and the laboratory supervisor and the QA Coordinator determine

21 that re-preparation and re-analysis are not possible, then the results for that sample or analytical batch

22 shall be qualified. The qualifiers used for waste analysis results shall be consistent with EPA Contract

23 Laboratory Program data qualifiers that are listed and defined below. Additional notes and explanations

24 may accompany the reports to further describe the occurrence. All non-detectable results will be

25 identified with a less than sign with the detection limit value, when applicable.

26

27 ND: This flag indicates the compound was not detected at or above the method detection limit (MDL).

28

29 U: This flag indicates the compound was detected above the MDL but below the PQL.

30

31 J: This flag indicates an estimated value. This flag is used (1) when estimating a concentration for

32   tentatively identified compounds where a 1:1 response is assumed and (2) when the mass spectral

33   and retention gas chromatograph/mass spectrometer (GC/MS) identification criteria and the result

34   is less than the PQL but greater than zero.

1 N: This flag indicates presumptive evidence of a compound. This flag occurs only on a mass

2 spectral library search. It is applied to all total ion chromatogram (TIC) results.

3

4 B: This flag is used when the analyte is found in the associated method blank as well as in the

5 sample. It indicates probable blank contamination and warns the data user to take appropriate

6 action. This flag is used for TICs as well as positively identified target compounds.

7

8 E: This flag indicates compounds with concentrations exceeding the upper level of the calibration

9 range of the instrument for that analysis. If one or more of the compounds have a response

10 greater than the upper level of the calibration range, the sample or extract shall be diluted and

11 re-analyzed. If the dilution of the extract causes any compounds identified in the first analysis to

12 be below the calibration range in the second analysis, both results are delivered with the flag

13 "DL" attached to the second analysis.

14

15 D: This flag is used for all compounds identified in an analysis at a secondary dilution factor.

16

17 X: Other specific flags may be required to properly define the results. If used, the flags shall be fully

18 described with the description attached to the sample summary package. Use Y and Z if more

19 than one flag is needed.

20

21 R: This flag is used to indicate that the analytical result is rejected. A reason for the data rejection is

22 required.

23

24 **4.4 Corrective Action**

25

26 Corrective action will be initiated by the laboratory QA/QC Officer when required to ensure data quality

27 meets the criteria established in the laboratory QA Plan. Corrective action of QA activities will be

28 initiated in response to performance audits, system audits, comparison studies, or QA program audits.

29

30 Standard laboratory-initiated corrective actions consist of checking instruments, apparatus, obtaining new

31 reagents and/or standards, and calibration verification or recalibration. If standard laboratory-initiated

32 corrective actions do not identify and correct the problem, then instrument custodians, instrument system

33 specialists, method authors, or chemists will troubleshoot and repair or correct the system performance.

1   General Approach to Corrective Action

2

3   For either immediate or long-term corrective actions, steps comprising a closed loop corrective action

4   system are as follows: (1) define the problem; (2) assign responsibility for investigating the problem;

5   (3) investigate and determine the cause of the problem; (4) determine a corrective action to eliminate the

6   problem; (5) assign and accept responsibility for implementing the corrective action; (6) implement the

7   correction and determine its effectiveness; and (7) verify that the corrective action has eliminated the

8   problem.

9

10   Undesirable performance or analytical errors will be identified as a problem in precision or bias. Either

11   the results of replicate measurements were not in close agreement or the results were not in agreement

12   with the expected (target) or reference values. Also, there is a possibility that both situations will occur

13   concurrently. Rules for finding and resolving the causes of these deficiencies are not well established.

14   However, the approach that the analyst takes must be systematic and based on common knowledge and

15   experience of the laboratory personnel. A team effort from the analysts, the immediate supervisor, and

16   the Laboratory Manager will be required. The most obvious causes are to be eliminated first. If the

17   initial investigation does not resolve the problem, the attention is to be directed to the more complex

18   possibilities.

19

20   Obvious simple errors such as the transposition and transcription errors of data, the use of incorrect

21   calculations or calculation errors, incorrect readings and recording of instrument readouts, the use of the

22   wrong analytical procedures, and the lack of attentiveness to details in the laboratory will lead to

23   imprecision or bias. A review and internal audit of the data and a detailed discussion with the analysts

24   concerning how and when they performed specific steps in the laboratory procedures may indicate the

25   cause of, and a corrective action for, the deficiency.

26

27   Bias

28

29   Inexperience of the analyst, instrument instability, variable contamination in the samples, variability of

30   the method blanks, poor reagent quality and control, or fluctuations of the laboratory environment are

31   possible causes of bias. Approaches to resolving these causes are the following: (1) check for obvious

32   and simple errors first; (2) repeat the analysis at the point where sample is first introduced into the

33   analytical procedure; (3) repeat the analysis on a different instrument or use another gas chromatograph

34   column; and (4) have another analyst repeat the analysis.

1 Accuracy

2

3 Incorrect calibrations, losses of analyte during sample preparation or analysis, incorrect calibration

4 standards, stock solutions, innate bias of the analyst, matrix effects on the analyte, instrumental shifts,

5 instrument not calibrated, and contaminations in the sample or standards are possible causes of

6 inaccuracy. Approaches to resolving these causes are the following: (1) check for the obvious and simple

7 errors first; (2) repeat the analysis at the point where sample is first introduced into the analytical

8 procedure; (3) repeat the analysis with new calibration standards; (4) recalibrate the analytical instrument;

9 (5) repeat the analysis on another instrument that is calibrated; (6) have another analyst repeat the

10 analysis; (7) repeat the analysis with fresh or new samples, if possible; and (8) check analytical

11 instrument and detector.

12

13 **4.5   QA Reports to Management**

14

15 QA reports will be generated by the laboratory to document the analytical results and organizational

16 performance. These reports will contain, at a minimum, reports of system or performance audits; reports

17 of required corrective actions implemented; assessment of the generated data precision, accuracy, and

18 comparability; and resolution of previously reported problems. The content, frequency, and recipient of

19 these reports will be established in the laboratory's QA Plan.

1   **5.0 RECORDKEEPING**

2

3   The laboratory performing RCRA analyses will maintain a record system that will include the

4   documentation of all samples received, analyzed, analyses conducted, preparations, QC challenges,

5   maintenance of laboratory equipment, and reports prepared. The PCAPP EDS site operations will

6   maintain documentation on samples collected, chain-of-custody, results, and reports received. All

7   information will become part of the operating record and will be kept until closure.