Permit

Los Alamos
NATIONAL LABORATORY

Hazardous & Solid Waste Group
Los Alamos National Laboratory
Los Alamos, New Mexico 87545

ESH-19:98-057

March 24, 1998

DOE OVERSIGHT

Ms. Janice Archuleta
New Mexico Environment Department
Hazardous and Radioactive Materials Bureau
2044 Galisteo, P.O. Box 26110
Santa Fe, New Mexico 87502

Dear Ms. Archuleta:

SUBJECT: TRANSMITTAL OF TA-50 SAMPLING AND ANALYSIS PLAN

I have enclosed a copy of the Radioactive Liquid Waste Treatment Facility Sampling and Analysis Plan (SAP) for your information and files. As you probably recall, we discussed this plan during several of your site visits to the Laboratory in 1997. The plan was also referenced in our response to the February 18, 1997, letter from Benito Garcia concerning "Reconfiguration of the Low-level Radioactive Waste Water Treatment Facility". As stated in this response, the Laboratory's RCRA/CWA Working Group was developing an influent and sludge monitoring program to characterize the waste streams received and produced by the Radioactive Liquid Waste Treatment Facility (RLWTF) and demonstrate that applicable RCRA exclusions, such as those contained in 40 CFR 261.3 and 261.4, were being met. The enclosed SAP is a result of that group's effort. Copies of the revised RLWTF Waste Acceptance Criteria and Conceptual Design Report were also transmitted to your office in response to Mr. Garcia's letter on February 10, 1997, and July 2, 1997, respectively.

If you would like to discuss the RLWTF SAP in more detail, or if you have any questions regarding the plan, please feel free to contact me at 667-4882. Thank you.

Sincerely,

Alex A. Puglisi

Alex A. Puglisi
Hazardous & Solid Waste

AP:em

Enclosure: EM-RLW Radioactive Liquid Waste Treatment Facility, Administrative Procedure, Sampling and Analysis Plan

15691

Cy: Barbar Hoditschek
NMED/SWGB
P.O. Box 26110
Santa Fe, NM 87501

Ralph Ford Schmid
NMED/DOE/OB
P.O. Box 26110
Santa Fe, NM 87501

J. Meadows, EM-RLW, MS E518
D. Moss, EM-RLW, MS E518
R. Alexander, EM-RLW, MS E518
P. Schumann, LANL, ESH-19, MS K498
M. Saladen, LANL, ESH-18, MS K497
J. Rochelle, LC/GEN, MS A187
CIC-10, MS A150
ESH-19 Circ File

EM-RLW

RADIOACTIVE LIQUID WASTE TREATMENT FACILITY

ADMINISTRATIVE PROCEDURE

SAMPLING AND ANALYSIS PLAN

Prepared by: Julie Meadows — date: 2/17/98
Julie Meadows, EM-RLW
Waste Management Operations

Reviewed by: Debora Hall — date: 2/23/98
Debora Hall, EM-RLW
Technical Facilities Operator

William David Moss — date: 2/19/98
David Moss, EM-RLW
Operation Supervisor/Assistant Facility Manager

Amy MacDonald — date: 2-17-98
Amy MacDonald, EM-RLW
QA/QC

Mike Saladen — date: 2/24/98
Mike Saladen, ESH-18
NPDES Team Leader

Alex A. Puglisi — date: 2/23/98
Alex Puglisi, ESH-19
RCRA/CWA Coordinator

Approved by: Steve W. Hanson — date: 2-23-98
Steve Hanson, EM-RLW
Group Leader/FMU-84 Facility Manager

EM-RLW

RADIOACTIVE LIQUID WASTE TREATMENT FACILITY

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Administrative Procedure
For
Sampling and Analysis Plan

HISTORY OF REVISIONS

Superseded Document	Date	Superseded By	Date
None		AP-50RLWTF-06, R00	2/98

RADIOACTIVE LIQUID WASTE TREATMENT FACILITY

SAMPLING AND ANALYSIS PLAN

1.0 INTRODUCTION

Environmental Management-Radioactive Liquid Waste (EM-RLW) manages and operates the Radioactive Liquid Waste Treatment Facility (RLWTF) within Facility Management Unit (FMU)-84 at the Los Alamos National Laboratory (LANL). The RLWTF consists of the Main Plant at Technical Area (TA)-50-1 and two pretreatment plants: the Room 60 Pretreatment Plant at TA-50-1 (Pretreatment Plant) and TA-21-257 (DP-257).

The majority of influent received at the Main Plant enters the plant through the Radioactive Liquid Waste Collections System (RLWCS). Small volumes of influent enter the Main Plant in batch loads by way of a tanker truck.

The Main Plant also receives and further treats the effluent from the Pretreatment Plant and DP-257. Volumes of effluent transferred to the Main Plant are variable and depend on the influent volumes treated through these plants. DP-257 operates approximately twice a month; the Pretreatment Plant operates infrequently. The Main Plant operates at least five days a week.

1.1 Federal Regulations

The Main Plant's Mortandad Canyon Outfall, Outfall 051, is regulated under the Clean Water Act by LANL's National Pollutant Discharge Elimination System (NPDES) Permit NM0028355. This permit sets limits for specific constituents in the final effluent discharged from its effluent tanks at TA-50-2 (WM-2) to Outfall 051. The Pretreatment Plant is included in LANL's Hazardous Waste Facility Permit and is a Resource Conservation and Recovery Act (RCRA) treatment facility. The Main Plant and DP-257 operations are not included in the RCRA permit, but both are covered in the 1990 NPDES permit application.

1.2 RCRA Exclusions

Title 40 of the Code of Federal Regulations (CFR) lists exclusions for waste treated through a wastewater treatment plant regulated under the Clean Water Act by an NPDES permit. These exclusions (Attachment 1) are

- §261.4(a)(2), for industrial wastewater discharges that are point source discharges subject to regulation under Section 402 of the Clean Water Act. NOTE: this exclusion applies only to the actual point source discharge. It does not exclude industrial wastewater while it is collected, stored, or treated before discharge, nor does it exclude sludge that is generated by industrial wastewater treatment.
- §261.3(a)(2)(iv)(A-G), for "de minimis" volumes of hazardous waste contained in wastewater subject to regulation under the Clean Water Act.

Definitions for RCRA hazardous waste categories are in Laboratory Implementation Requirement (LIR) 40.00.03.0 "Hazardous and Mixed Waste Requirements for Generators."

1.3 Sampling Operations

As part of routine operations, EM-RLW operation's personnel at the Main Plant collect RCRA operational samples of raw feed and plant sludge generated. Analysis of these samples ensures the RLWTF is operating safely within operational limits and complies with applicable RCRA exclusions.

RLWTF personnel collect NPDES compliance samples of the Main Plant final effluent. Analysis of final effluent samples ensures that the Main Plant effluent meets the NPDES discharge limits. In particular, samples will be used to validate the following:

- Sludge generated during Main Plant and DP-257 operations is not a RCRA waste. NOTE: RCRA listed waste is not accepted for treatment at the RLWTF. Because influent is not a RCRA listed waste, the sludge derived from the treatment of this wastewater will not be classified as a RCRA listed waste. If the sludge exhibits a RCRA hazardous characteristic, the sludge would be classified as a RCRA waste.
- Influent entering the Main Plant and DP-257 is not a RCRA waste and meets applicable RCRA exclusions (Attachment 1).
- Main Plant final effluent meets the discharge limits set by the NPDES Permit No. NM0028355.

Sampling these streams requires carefully collecting adequately sized, representative samples. The sampling method, container type, preservation method, and equipment selected must ensure that chemical and physical properties of the sample are preserved. The appropriate number of quality assurance samples must be analyzed to ensure valid data.

RLWTF sampling operations are described in detail in DOP-50RLWTF-08, "Detailed Operating Procedure (DOP) for Sampling Associated With TA-50 Main Plant Operations".

1.4 Purpose and Scope

This plan helps ensure scientifically valid analytical results that are obtained in accordance with methods required by regulation. The plan applies to all EM-RLW personnel participating in the sampling described in this document. It does not apply to Chemical Science and Technology (CST) analytical and sample management personnel unless otherwise involved in the sampling described in this document. CST personnel perform their operations according to their established procedures. Description of CST analytical and quality assurance/quality control (QA/QC) procedures is beyond the scope of this document.

Personnel collect samples of DP-257 and Pretreatment Plant influent before treatment through these plants. After treatment, the final effluent from these plants is analyzed for radiological components before transfer to the Main Plant for further treatment. Raw feed samples from the Main Plant influent tanks includes these streams; therefore, this document does not include Pretreatment Plant and DP-257 samples. Samples related to the Pretreatment Plant and DP-257 are described in DOP-50RLWTF-01 and DOP-50RLWTF-06, respectively.

This plan does not detail instructions for collecting and handling samples. DOP-50RLWTF-08,

"Detailed Operating Procedure (DOP) for Sampling Associated With TA-50-1 Main Plant Operations," details instructions for all aspects of sampling, including collection, submittal for analysis, and health and safety concerns. DOP-50RLWTF-14, "Packaging and Transportation of Samples," details instructions for packaging and transport of samples according to Department of Transportation (DOT) regulations. These DOPs are available at the Main Plant operator's desk in Room 116C at TA-50-1 and through the EM-RLW Document Control Manager at TA-50-1-6. Personnel are trained according to DOPs and reference controlled copies of DOPs when conducting sampling covered by this plan. Additionally, personnel should be familiar with SOP-50RLWTF-01, "Safe Operating Procedure (SOP) for RLWTF Operations"

2.0 DEFINITIONS,ACRONYMS, AND ABBREVIATIONS

2.1 Definitions

accuracy	The closeness of agreement between an observed value and an accepted reference value.
blank sample	A sample of deionized or distilled water or analyte-free media used to verify the quality of sampling activities by identifying contamination introduced from outside sources.
composite sample	A single sample of combined individual samples collected from the same location at different points in time and combined into a single sample.
final effluent	Treated water flowing from a treatment plant to the final point of discharge (outfall 051) for the plant into Mortandad Canyon.
flow-weighted composite	A sample consisting of a minimum of three grab samples of collection at regular intervals over a normal operating day and combined proportional to flow or a sample continuously collected proportional to flow over a normal day.
grab sample	A single sample collected in less than 15 minutes. {NPDES Permit No. NM0028355} [redacted]
headworks	The point of entry of waste into a wastewater treatment plant. In this document, the influent tanks, where all influent streams to the Main Plant converge.
NPDES compliance sample	Samples taken in compliance with the monitoring requirements following final treatment, prior to or at the point of discharge from the TA-50-1 Treatment Plant.
precision	The agreement among a set of replicate measurements without assumption of knowledge of the true value. Precision is estimated by means of

duplicate/replicate analyses.

raw feed	Wastewater pumped from an influent tank for treatment to be treated through the Main Plant. This water is representative of wastewater treated through the Main Plant.
raw influent	Wastewater entering the Main Plant through the Radioactive Liquid Waste Collection System (RLWCS) before being stored in an influent tank. This stream may not be representative of waste treated through the Main Plant.
RCRA operational samples	Samples routinely collected and analyzed to determine regulatory compliance and ensure that the RLWTF is operating within operational limits. These samples are collected prior to final treatment for NPDES.
reagent grade	Reagents conforming to the current specifications of the Committee on Analytical Reagents of the American Chemical Society (ACS). Synonymous terms for such reagents are analytical reagent grade and ACS reagent grade.
Representative Sample	A sample of a universe or whole (e.g., sludge, lagoon, wastewater) which can be expected to exhibit the average properties of the universe or whole.
Sludge	Any solid, semi-solid, or liquid waste generated from a municipal, commercial, or industrial wastewater treatment plant, water supply treatment plant, or air pollution control facility exclusive of the treated effluent from a wastewater treatment plant. {40 CFR 260.10}
SW-846	The EPA publication intended to provide a unified, up-to-date source of information on sampling and analyses related to compliance with RCRA regulations.
TCLP	Toxicity Characteristic Leaching Procedure, Test Method 1311 in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,"EPA SW-846. TCLP is the test method used to determine if a waste exhibits a RCRA toxic characteristic. SW-846 details the TCLP procedure.
TCLP metals	TCLP metals are the eight RCRA metals analyzed for using the TCLP test method. These metals are Arsenic, Barium, Cadmium, Chromium, Lead, Mercury, Selenium, and Silver.
time-weighted composite	Individual samples collected from the same location at incremental points in time and combined into a single sample.

2.2 Acronyms and Abbreviations

ALARA	as low as reasonably achievable

CFR	Code of Federal Regulations
CLP	contract laboratory program
CST	Chemical Science and Technology (Division)
DOE	U.S. Department of Energy
DOP	Detailed Operating Procedure
DOT	U.S. Department of Transportation
EM-RLW	Environmental Management-Radioactive Liquid Waste
EPA	U.S. Environmental Protection Agency
ESH	Environmental Safety and Health (Division)
ESH-1	Health Physics Operations Group
ESH-18	Water Quality and Hydrology Group
ESH-19	Hazardous and Solid Waste Group
LIR	Laboratory Implementation Requirement
NPDES	National Pollutant Discharge Elimination System
QA	quality assurance
QC	quality control
RCRA	Resource Conservation and Recovery Act
SVOC	Semi-volatile organic compound
TCLP	toxicity characteristic leaching procedure
TTO	Total Toxicity Organics
VOC	Volatile Organic Compounds

3.0 ROLES AND RESPONSIBILITIES

The quality of sampling operations and the safety of sampling personnel depend on personnel fulfilling specific responsibilities. This section describes these personnel and their responsibilities.

3.1 EM-RLW

3.1.1 Group Leader

The EM-RLW Group Leader is ultimately responsible for the quality of the sampling program and ensuring the RLWTF complies with all regulatory requirements. This includes

- Reviewing and approving this plan and any subsequent revisions.
- Providing written authorization for any deviation from this plan, routine sampling procedures, or routine transportation procedures.
- Ensuring adequate staffing and funding resources for EM-RLW sampling operations, analysis of all samples, and quality assurance related to the sampling according to this plan and associated DOPs.
- Ensuring EM-RLW sampling operations do not pose a threat to humans or the environment.

3.1.2 Sampling Plan Manager

The Operations Supervisor typically serves as the Sampling Plan Manager. The Sampling Plan Manager or designee does the following:

- With the EM-RLW Training Coordinator and applicable Environment, Safety, and Health (ESH) personnel, identifies the training requirements for sampling personnel and verifies these people are adequately trained.
- Ensures EM-RLW personnel follow all sampling procedures established by this plan and the associated DOPs.
- Ensures EM-RLW personnel follow DOT regulations when transporting samples.
- Ensures QA records generated by sampling operations are maintained as described in the DOP and this plan.
- Notifies applicable ESH-18 or ESH-19 personnel of any nonconformance identified by audits and assessments of the sampling program.
- Ensures EM-RLW personnel submit samples for analysis according to appropriate analytical methods.

3.1.3 Sampling Personnel

Typically EM-RLW operation's personnel conduct routine sampling operations. These personnel do the following:

- Conduct sampling according to procedures established by this plan, DOP-50RLWTF-16, DOP-50RLWTF-14, and SOP-50RLWTF-01.
- Attend all required training identified by the Sampling Plan Manager and the EM-RLW Training Coordinator.
- Consult with CST-9 and CST-12 analytical personnel to determine the appropriate type, size, and number of samples to comply with the laboratory's analytical and quality assurance/quality control (QA/QC) requirements.
- Safely conduct sampling operations as described in the associated DOPs and the SOP.
- Resolve all safety concerns with the Operations Supervisor, and, if appropriate, ESH-5 and/or ESH-1 personnel.
- Maintain appropriate documentation of sampling and analyses such that data is readily available.
- Report all spills occurring during sampling operations to the site spill coordinator, as required by the Laboratory's *Spill Prevention Control and Countermeasures Plan*, the Group Spill Implementation Plan (GSIP), and the "TA-50-1 Site Emergency Plan."

3.1.4 Training Coordinator

The EM-RLW Training Coordinator does the following:

- With the EM-RLW Sampling Plan Manager and applicable ESH personnel, establishes minimum qualifications for EM-RLW sampling personnel.
- Schedules personnel for training to ensure personnel are adequately trained for the operations they perform and that this training remains current.
- Tracks employee training and qualification in the Employee Development System (EDS) and maintains records in employee training files.

3.2 CST

3.2.1 Analytical Personnel

CST analytical personnel have the capability to perform some of the sample analysis required. CST analytical personnel:

- Provide guidance related to sampling containers, volumes, preservatives, and holding times.
- Analyze samples before the holding time has expired.
- Analyze samples according to the appropriate analytical methodology.

3.2.2 Sample Management Personnel

Sample Management personnel do the following:

- Package samples according to DOT regulations for transport to off-site analytical laboratories .
- Transport samples for analysis according to DOT regulations.
- Deliver samples to meet holding times.
- Notifies EM-RLW personnel if holding times are missed.
- Complete all appropriate forms, including the Chain-of-Custody record, to ensure samples can be adequately tracked.

3.2.3 CST-3

CST-3 provides sampling quality assurance. The sampling QA section does the following:

- Reviews the sampling contract and assures that samples are being analyzed according to the specified level of QA.
- Assist the EM-RLW Sampling Plan Manager in determining the root cause of nonconformances, initiating corrective actions, and verifying that corrective actions have been completed.
- Review analytical data records.

3.3 ESH

3.3.1 ESH-18

Personnel from the Water Quality and Hydrology Group (ESH-18) provide environmental compliance support for issues related to the Clean Water Act, including the following:

- Provides technical support for NPDES permitted outfalls.
- Oversees the NPDES permit.
- Evaluates and reports sampling data used to determine NPDES compliance.
- Acts as the primary interface between the permitted outfall owner and regulatory agencies.

3.3.2 ESH-19

Personnel from the Hazardous and Solid Waste Group (ESH-19) provide environmental compliance support for issues related to RCRA, including the following:

- Oversees LANL's RCRA Permit.
- Evaluates and reports sampling data used to determine RCRA compliance.

3.3.3 ESH-1

Health Physics Operations Group (ESH-1) personnel provide health physics support including the following:

- Provide guidance for working with waste containing radioactive components.
- Recommend personal protective equipment (PPE) for radiological work.
- Perform radiological surveys on filled sample containers before transportation.

3.3.4 ESH-5

Industrial Hygiene and Safety Group (ESH-5) personnel provide industrial hygiene support, including recommending appropriate PPE to be worn by sampling personnel.

3.4 BUS-6

BUS-6 Packaging and Transportation Group personnel provide guidance on DOT requirements.

4.0 SAMPLES

EM-RLW operations personnel routinely collect samples from the Main Plant final effluent stream for NPDES compliance. Additionally they collect raw feed and sludge samples to ensure compliance with RCRA.

4.1 NPDES Compliance Sampling (Main Plant Final Effluent)

As required by its NPDES permit, EM-RLW personnel collect samples of Main Plant final effluent as it discharges from the effluent tanks at TA-50-2 to Mortandad Canyon, Outfall 051. Flow and pH are continuously monitored and recorded during all discharges to the canyon. The flow and pH sample is drawn from a point in the effluent line that is located between the effluent holding tank and the canyon outfall. The following sections list the samples collected for analysis. Sampling associated with facility operations is detailed in DOP-50RLWTF-08.

4.1.1 Weekly Grab Samples

Personnel collect weekly grab samples of Main Plant final effluent on a random basis and submit them for the following analysis:

- Chemical Oxygen Demand
- Total Chromium
- Total Lead
- Total Zinc
- Total Suspended Solids
- Total Copper
- Total Mercury
- Total Cadmium
- Total Iron
- Total Nickel

4.1.2 Monthly Grab Samples

Personnel collect monthly grab samples of Main Plant final effluent on a random basis and submit them for the following analysis:

- Ammonia as Nitrogen (N)
- Total Toxic Organics (TTO) (VOC)
- TTO, Dioxin
- Total Nitrogen
- TTO (SVOC)
- TTO (Pesticides)
- Nitrate-Nitrite (as N)
- Ra-226 and -228

4.1.3 Yearly Grab Samples

Personnel collect yearly grab samples of Main Plant final effluent on a random basis and submit them for the following analysis:

- Total Aluminum
- Total Selenium
- Tritium
- Total Arsenic
- Total Vanadium
- Total Boron
- Total Cobalt

4.2 RCRA

Sampling of the influent and sludge helps ensure the RLWTF meets the applicable RCRA exclusions and the plant is operating within operating limits.

Historically, EM-RLW sampling personnel have taken samples from three sampling points at the facility to ensure compliance with RCRA. These samples were raw influent, raw feed, and sludge.

Currently, samples are taken and analyzed to meet the requirements of RCRA exclusions on only the raw feed and sludge (Subsections 4.2.1 and 4.2.2). The raw feed sample is representative of all the influent treated through the plant, whereas the raw influent only represents the influent entering the Main Plant through the Radioactive Liquid Waste Collections System (RLWCS).

4.2.1 Raw Feed

Since wastewater in the influent tanks (raw feed) is representative of the waste treated through the plant, the influent tanks are considered to be the headworks of the Main Plant. In addition to the influent entering the Main Plant through the RLWCS, the raw feed

includes DP-257 effluent, Pretreatment Plant effluent, and batch waste added directly to the influent tank.

The Main Plant is currently being retrofitted with an influent tank system consisting of four influent tanks. Sample points will be engineered to be representative of the raw feed. Piping configuration and valving on these tanks allows for segregation of waste in the tanks. Each tank of waste is well mixed before entering the plant for treatment and each tank is treated separately in a batch-wise mode.

Operations personnel collect grab samples of raw feed randomly on a weekly basis on a day that the plant is operated. Personnel submit these samples for analysis of VOC, SVOC, and TCLP metals and record this event in the Plant Daily Log. This sample is used for verification that the influent in the headworks of the plant meets the applicable RCRA exclusions, and that the influent is not a RCRA characteristic waste. Details on raw feed and raw feed sample operations can be found in SOP-50RLWTF-01 and DOP-50RLWTF-08.

The Waste Profile system at LANL facilitates that the treatment plant does not receive any RCRA listed waste. Before a waste generator can discharge any waste to the RLWCS, he/she must fill out and submit a Waste Profile Form. Waste Profile Forms containing a listed RCRA waste are not accepted for treatment at the Radioactive Liquid Waste Treatment Facility. Waste acceptance criteria for the RLWTF is located in the LANL document PLAN-WASTEMGMT-002.

4.2.2 DP-257 and Main Plant Sludge

Sludge is transferred from the clariflocculator, collected in sludge holding tanks, and well-mixed before dewatering to less than 27% solids on the vacuum filter. Each time the characteristics of the sludge in a sludge holding tank changes, an operator collects three random grab samples of dewatered sludge from the vacuum filter cake while the filter is operating. The operator collects these samples each time sludge is drawn from the clariflocculator a few days after draining to ensure samples are well-mixed and as representative as possible. EM-RLW uses these samples to determine whether the sludge is a mixed waste. If an analysis indicates the sludge is a RCRA characteristic waste, then EM-RLW will manage the sludge accordingly. Details on sludge and sludge sample operations can be found in SOP-50RLWTF-01 and DOP-50RLWTF-08.

The following analysis is done on sludge samples:

- VOC
- SVOC
- zinc
- percent solids
- radiological constituents
- antimony
- TCLP metals
- nickel
- thallium

4.3 Representative Samples

A single sample may be representative of a homogeneous material because it has a uniform composition throughout the matrix; however, heterogeneous materials vary in composition throughout the matrix. Sampling accuracy is typically achieved by random sampling. Sampling precision is usually achieved by taking an appropriate number of samples from the source.

Representative samples must be obtained to ensure high confidence in analytical data. To ensure samples are representative of the entire waste volume, a sufficient number of samples must be collected. The required number of samples is based on the type of statistical information required and the type of material collected. This plan does not describe representative sampling strategies in detail. The Sampling Plan Manager consults with applicable ESH personnel and CST analytical personnel to determine the appropriate number of samples required to obtain a representative sample and follows their guidance.

4.4 Quality Assurance Samples

QA/QC ensures precision and accuracy, which are essential components of sampling operations. QA principles, practices, and procedures described in this section ensure analytical data is scientifically valid and regulatorily compliant.

4.4.1 Trip Blanks

Trip blanks are not relevant to normal operations at EM-RLW; they are taken on an as needed basis. Trip blanks provide data on contamination acquired during the handling and transporting of samples. Sampling personnel prepare trip blanks in the CST analytical laboratory. Trip blanks are laboratory-grade distilled or deionized water or some other analyte-free media specified by CST analytical personnel placed in containers of the same lot and type as those in which the waste samples will be placed. Personnel label the container with an identification number similar to routine samples, carry the blank throughout the entire sampling process, record the sample on the Chain-of-Custody Record, and transfer the sample to CST personnel in the same manner as routine samples. Personnel also record the event on the Plant Daily Log. Personnel prepare and submit trip blanks for analysis of VOCs, SVOCs, and TCLP metals.

4.4.2 Sampling Blanks

Sampling blanks provide data on potential container, preservative, or media contamination. With the exception of preparing the blank with laboratory-grade distilled or deionized water or analyte-free media specified by CST analytical personnel, sampling personnel handle this blank as a routine sample, adding preservative, if applicable, and labeling the container with a unique identification label similar to routine samples. Personnel record the sample on the Chain-of-Custody Record and record the event in the Plant Daily Log. Personnel prepare and submit field blanks for the analysis of VOCs, SVOCs, and TCLP metals in the raw feed routinely five times a year to validate data obtained for this stream.

4.4.3 Duplicate Samples

Duplicate samples verify the reproducibility of analytical data. Sampling personnel collect duplicate samples at the same time from the same sampling apparatus and preserve and handle duplicates samples identically. This sample is divided into identical containers prepared in the same way and filled to the same volume. CST analytical personnel analyze these samples using the same procedure and instrument. Sampling personnel label duplicate samples with a sequential number similar to other waste samples, record the samples on the Chain-of-Custody, and record the event on the Plant Daily Log. Sampling personnel routinely collect duplicates on the raw feed five times a year to validate data on VOCs, SVOCs, and TCLP metals in this stream.

4.4.4 Replicate Samples

When the analytical laboratory wants to validate data obtained from different analytical methods or analytical laboratories, sampling personnel may collect a replicate sample. Replicate samples are collected from the same source at the same time, and are contained, preserved, and transported in the same manner. Personnel collect two equal volumes of sample in two identical containers. The samples are analyzed by different accepted analytical techniques or by separate laboratories that participate in the EPA Contract Laboratory Program (CLP). Because obtaining accurate replicate samples from heterogeneous or multi-layered samples is often very difficult, sampling personnel must collect these samples with great care to ensure that each portion has the same composition. Sampling personnel record this sample on the Chain-of-Custody Record and on the Plant Daily Log.

4.5 Radiological Screening Samples

Personnel collect a representative sample for radiological screening of any sample stream before the sample is analyzed for nonradioactive components or transported for analysis.

4.6 Analytical Methods

Wastewater samples are analyzed according to the methods in 40 CFR 136 for samples related to NPDES samples. Alternate NPDES analytical methods can be used, but EPAs approval is needed prior to implementation. For samples related to RCRA, wastewater samples are analyzed according to the methods in EPA SW 846. Sludge samples are also analyzed according to EPA SW 846.

5.0 SAMPLE HANDLING AND MANAGEMENT

Personnel carefully collect, handle, and track sampling throughout sample collection, transport, and analysis. Careful sample management ensures samples can be tracked from collection, through analysis and interpretation of analytical results thus enabling EM-RLW and ESH personnel to recreate a sampling event.

General procedures to ensure quality of data are provided in this section. By following the guidelines in this section and detailed instructions in DOP-50RLWTF-08 and SOP-50RLWTF-01,

sampling personnel ensure samples are representative, thus providing scientifically valid and regulatorily compliant analytical data. Details in DOP-50RLWTF-08 include required PPE, sample container types and sizes, volume and type of preservatives.

5.1 Decontamination

Sampling personnel minimize contamination of the collected samples by using sampling containers originally purchased in taped uncontaminated packages. These sample containers are quality-assured EPA standard sample containers that are bought through LANL. To avoid contaminating clean equipment or cross-contaminating samples, sampling personnel use new gloves each time they collect a batch of samples.

Because of radiological concerns, EM-RLW does not decontaminate the composite sampler after each sampling. However, if equipment is not decontaminated this would skew data to a worst-case scenario. If applicable, sampling personnel contact ESH-1 for assistance in radioactive decontamination.

After each sampling event, CST personnel wash the glass composite sample collection bottles in a dishwasher using laboratory-grade detergent and rinsing in distilled water. These cleaned sample containers are maintained in the analytical laboratory.

5.2 Holding Times

The holding time is the maximum time the sample can be held before analysis and begins the date and time the sample is collected. Holding times are specified in 40 CFR 136 for samples related to NPDES, and SW 846 for samples related to RCRA. If sample holding times exceed those specified in 40 CFR 136 and SW 846, the analytical data may be considered invalid. By notifying Sample Management when sampling occurs and/or transferring the sample to the appropriate analytical laboratory immediately after sampling, sampling personnel ensure established sample holding times are not exceeded. To ensure sample holding times are capable of being determined, sampling personnel complete the Chain-of-Custody Record when collecting and relinquishing the samples.

5.3 Sample Preservation and Reagents

The sample must retain the properties of the original waste from the time of sampling to analysis. Except in circumstances where preservation might cause safety problems, personnel preserve samples to ensure samples retain the properties of the original waste. Personnel use the proper volume of recommended preservatives as appropriate following CST guidance and specified in SW-846 or 40 CFR 136. SW-846 specifies preservatives for RCRA samples, and 40 CFR 136 specifies preservatives for NPDES samples. When recommended by CST personnel, sampling personnel will add preservatives to the sample container before collecting the sample. If preservatives are used, special care is taken not to overfill the container, as preservative may be lost or diluted.

Degradation or alteration of the sample through exposure to air, excessive heat or cold, microorganisms, or contaminants from the container must be avoided. Volatilization, loss of acidic gases, and biodegradation can be reduced by storing and transporting the samples at a

reduced temperature, approximately 4°C. Samples are stored in a refrigerator at approximately 4°C.

To avoid freezing aqueous samples, personnel never store samples outside. If frozen, the sample could fracture, resulting in separation of slightly immiscible phases and the release of volatile compounds. Sample fracture can also result in the precipitation of some salts that might not redissolve, resulting in inaccurate analytical results. Further, glass containers may break at freezing temperatures thereby creating a physical hazard.

CST analytical personnel at TA-50-1 ensure preservatives and reagents used for collecting samples are of adequate purity. They also ensure that expiration dates, if any, of preservatives and reagents are not exceeded. Preservatives and sampling equipment, such as pipettes and droppers used for adding sample preservatives are maintained and stored in analytical laboratories. Sample packaging is stored at TA-50-1 in the Main Plant operations area.

5.4 Nonconformances

Sampling personnel are responsible for continual self-assessment to ensure compliance with this plan. EM-RLW sampling plan manager periodically assesses the quality of the sampling process, and sampling events to ensure sampling personnel follow the QA principles and established sample collection procedures.

CST-3, sampling QA section, assesses the analytical process to ensure the required level of QA/QC is being met.

EPA requires LANL to analyze a Discharge Monitoring Report/Quality Assurance (DMR/QA) sample once per year. This sample assures that the analytical methods used are producing accurate data. EPA provides the sample, and EPA knows the composition. LANL analyzes the sample and reports the analysis to EPA. EPA compares LANLs analysis to the known composition and then rates LANLs analytical performance.

If a nonconformance results in a DOE 232.1 occurrence, the EM-RLW Records Manager maintains this documentation in the occurrence files. Otherwise, documentation is retained with sampling records in the Main Plant.

The NPDES permit requires LANL to continue sampling if samples are found out of compliance with NPDES permit limits. Upon receipt of analytical results, any effluent limited parameter found to be out of compliance shall be re-sampled for that noncompliant parameter within 7 days. This will continue until sample results are returned to compliant status.

5.5 Calibration and Maintenance

EM-RLW calibration personnel and CST analytical personnel calibrate the instruments related to sampling and analysis according to the manufacturer's specifications. Personnel use the pH meter and electrode with care to avoid equipment damage, decontaminate the pH electrode between samples, ensure periodic maintenance is performed according to the manufacturer's specifications

or permit requirements, and document calibration and maintenance. EM-RLW calibration personnel routinely calibrate instrumentation used for continuous monitoring of effluent. EM-RLW calibration personnel perform calibrations according to LANL calibration standards and AP-RLWTF-24, "Calibration Plan."

5.6 Discrepancy and Corrective Action Reports

If EM-RLW personnel note significant deficiencies or discrepancies while collecting samples or reviewing the analytical results, they write a report describing the deficiencies or discrepancies and the action required for correction. The report describes how the deficiency or discrepancy was identified, who is responsible for creating and correcting the discrepancy, and contains a recommended schedule for and type of corrective action necessary to ensure that the waste stream is properly characterized. EM-RLW personnel forward a copy of the report to ESH-18 or ESH-19, whichever is appropriate, file the discrepancy and corrective action report with plant records and track implementation of corrective action.

5.7 Sample Labels

Sampling personnel prepare a unique EM-RLW alphanumeric sample label for each sample, and label each sample container at the time of sample collection. Sample labels are necessary to prevent sample misidentification and are used to track samples from collection to analysis and to correlate the analytical results to the original waste stream. This EM-RLW sample number is cross-referenced with the CST Sample Management sample identification number on the Chain-of-Custody Record form. Chain-of-Custody Record form protocol is described in detail in DOP-50RLWTF-08.

5.8 Sample Containers

Sample container size and types vary according to the matrix and nature of the sample to be collected. Wide-mouth containers are generally used for sludge; narrow-mouth containers are used for liquid samples. Sampling personnel consult with the appropriate analytical personnel to determine the number and type of containers required for the sampling effort. Types of sample containers required can also be found in 40 CFR 136. Sampling personnel determine the number of each type of container required by including duplicates and blanks with the number of collection samples. Except when sampling for analysis of VOC, personnel leave an air space equivalent to approximately 1% of the container volume to allow for thermal expansion during shipment. Container types and sizes for each sample type are listed in DOP-50RLWTF-08.

5.9 Sample Volume

Sampling personnel collect a sufficient volume of sample to ensure that all the required analyses can be performed and to provide for any quality control needs, split samples, or repeat laboratory procedures. The required volumes are listed in DOP-50RLWTF-08. Sampling personnel consult CST analytical personnel to ensure adequate sample volumes are collected to ensure enough volume for reanalysis of a sample if necessary.

5.10 Post-Operation Activities

Sampling personnel verify all sample bottles have been correctly labeled and crosscheck label

information for filled sample bottles against information recorded on the Chain-of-Custody Record. Sampling personnel confirm PPE has been disposed of in an approved waste disposal container. Further, sampling personnel maintain custody of filled sample bottles by keeping them in their possession, within view, locked or sealed to prevent tampering, or bring them into an area under lock and key with controlled access. Finally, sampling personnel prepare samples for transport to CST analytical facilities in compliance with applicable DOT regulations.

5.11 Data Review

The Operations Supervisor reviews the raw influent data on a weekly basis to ensure applicable requirements are met. Because of the timeframe required to obtain the analytical data from the laboratories and the need to maintain continuous plant operations, this data cannot be reviewed before the waste is treated through the Main Plant. If a potential noncompliance situation arises, the Operations Supervisor informs ESH-19. ESH-19 personnel also perform periodic reviews of this data to verify that the influent meets the exclusion.

ESH-18 and the Operations Supervisor also review plant effluent data to ensure compliance with the NPDES permit. Effluent data is submitted to ESH-18 so it can be included in monthly Discharge Monitoring Reports (DMRs). DMRs are submitted to EPA and NMED as required by the permit.

Sampling personnel collect ample sample volumes so that the sample may be reanalyzed should a potential noncompliance situation arise.

6.0 TRAINING

To ensure the safety of operations, sampling personnel attend appropriate LANL training courses, as specified in the EM-RLW training matrix, and on-the-job training (OJT) for the type of sampling. OJT also ensures that personnel know the sampling methods and preservation, sample containers, PPE, and strategies that allow for accurate and precise results. OJT includes training on applicable DOPs, the SOP, and this plan.

7.0 RECORDS

Sampling personnel complete and maintain records documenting the sampling and analysis of waste streams to assure quality. Copies of the forms and logs used to document samples and analysis can be found in DOP-50RLWTF-08, Attachments 1 – 6. These records ensure that this plan is properly implemented and that analytical results can be traced back to the waste that was sampled. Personnel retain these records in the Plant Operations Room at TA-50-1. Records pertinent to the waste sampling program, including laboratory records generated by the activities performed, are listed below.

- Request for Analysis Log Sheet/Chain-of-Custody Record
- Analytical reports
- Nonconformance and corrective action reports
- Sampling equipment maintenance and calibration forms

Written requests and approvals for any temporary modifications to sample collection procedures or to shipping papers for samples and reagents are maintained by CST analytical personnel housed at TA-50-1.

Sampling personnel make entries on records with indelible, blue or black ink in a legible, consistent, direct, and succinct manner. Personnel ensure that the records are identifiable, retrievable, and protected against damage, deterioration, and loss. Personnel make changes to records by drawing a single line through the original information so it remains legible, writing the correct information next to the original information, and initialing and dating the change.

7.1 Chain-of-Custody Record

The Chain-of-Custody Record accompanies the sample from the time the sample is collected through the sample transfer and analysis process. Sampling personnel complete the appropriate Chain-of-Custody Record to ensure that they can account for the sample at all times until the sample is relinquished to CST analytical or sample management personnel.

Each time a sample is transferred from the custody of one person to another, the person relinquishing and accepting the sample must complete the required information on the Chain-of-Custody Record. NPDES samples require an NPDES Sample Chain-of-Custody Record. Influent and sludge samples require the EM-RLW Chain-of-Custody Record. This record allows the sample to be tracked throughout the analytical process, assuring control over tracking of the sample and its corresponding analytical results.

More than one sample may be included on one Chain-of-Custody Record. EM-RLW personnel maintain a copy of the signed Chain-of-Custody Record at TA-50-1.

At a minimum, the Chain-of-Custody Record includes the following information:

- Names of the sampling personnel
- date and time of sample collection
- Date and time of the sample shipment/transfer
- Sampling site (station) location
- Unique EM-RLW sample number for each sample being transferred
- Number of containers
- Type of analyses to be performed
- CST Sample Management sample label number
- Signature of person relinquishing the sample(s)
- Date and time the sample(s) is relinquished
- Signature of the person receiving the sample(s)
- Signatures of all persons involved in the chain of possession

Additional information, for example sample description, quantity, and preservation, may also be added to the Chain-of-Custody Record as necessary.

7.2 Shipping Papers

Historical data verifies that influent and final effluent samples qualify as a DOT "limited quantity" of radioactive materials. DOP-50RLWTF-14 contains instructions for packaging and transportation of samples and required documentation in accordance with DOT regulations in 49 CFR Part 173 Subpart I, Radioactive Materials. The Main Plant Operator maintains records of shipping papers associated with Main Plant samples in the plant operations areas.

7.3 Analytical Results Reports

CST analytical personnel generate a report of the analytical results for each sample submitted by the sampling personnel. The following information is included in each report:

- Unique sample number (provided by CST person to whom the sample was transferred)
- Unique sample number assigned by the analytical laboratory
- Date the sample was received by CST analytical personnel
- Date and time of analysis
- Name of the analyst (or initials)
- Analyses requested
- Analytical results for each requested analysis
- Analytical methods used for each analysis
- Data from QC samples (e.g., replicates, matrix spikes, and surrogates) introduced by the analytical laboratory into the sample analysis stream

Additional information, such as problems encountered during analysis that may affect validity of data, is also recorded with the analytical results.

CST submits the analytical reports to the sampling personnel, who compare the reports to Chain-of-Custody Record numbers to identify cross contamination, blank contamination, or other discrepancies. Chain-of-Custody Record numbers are cross-referenced to the analytical transfer number and the analytical transfer number is then correlated with the RLWTF sample number.

7.4 Retention of Records

7.4.1 NPDES

The NPDES permit requires that EM-RLW retains records of monitoring information for a period of at least three years from the date of the sample, measurement, or report. This period may be extended by the request of the State.

These records include the following:

- Calibration and maintenance records.
- Original strip chart recordings for continuous monitoring instrumentation.
- Copies of all reports required by this permit.

7.4.2 RCRA

As a matter of policy, all data related to RCRA must be retained for the life of the facility.

8.0 DISTRIBUTION

Personnel work only from a controlled copy of this document. A controlled copy is maintained for reference with the sampling DOP, DOP-50RLWTF-08, at the operator's desk in TA-50-1-116C. Controlled copies may be obtained through the Document Control Manager located in TA-50-1-6.

9.0 REFERENCES

9.1 Working Documents

EM-RLW; DOP-50RLWTF-14, "Detailed Operating Procedure for Packaging and Transportation of Samples"; most recent revision.

EM-RLW; DOP-50RLWTF-08, "Detailed Operating Procedure for Samples Associated with TA-50-1 Main Plant Operations"; most recent revision.

EM-RLW; SOP-50RLWTF-01, "Safe Operating Procedures for RLWTF Operations"; most recent revision.

9.2 Reference Documents

EM-RLW; AP-50RLWTF-24, "Calibration Plan"; most recent revision.

EM-RLW; Group Spill Implementation Plan (GSIP); most recent revision

FMU-84; AP-FMU84-08, "FMU-84, TA-50 Site Emergency Plan"; most recent revision.

Los Alamos National Laboratory; Laboratory Implementation Requirement (LIR) 40.00.03.0 "Hazardous and Mixed Waste Requirements for Generators"; most recent revision.

Los Alamos National Laboratory; *Spill Prevention Control and Countermeasures Plan;* most recent revision.

New Mexico Environment Department; *Hazardous Waste Facility Permit.* Permit No. NM0890010515-1; issued to Los Alamos National Laboratory November 8, 1989, Santa Fe,.

U.S. Environmental Protection Agency (EPA); National Pollutant Discharge Elimination System Permit. Permit No. NM0028355, issued to the Los Alamos National Laboratory's August 1, 1994, Santa Fe.

U.S. Department of Transportation, *Code of Federal Regulation;* 49 CFR Parts 173 Subpart I, "Radioactive Materials; most recent edition.

U.S. Environmental Protection Agency, *Code of Federal Regulation*; 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutant"; most recent edition.

U.S. Environmental Protection Agency, Office of Solid Waste; *EPA* SW-846, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.* Volumes I and II.; most recent edition

ATTACHMENT 1: APPLICABLE RCRA EXCLUSIONS

261.3 (a) (2) (iv) (A-G)

(iv) It is a mixture of solid waste and one or more hazardous wastes listed in Subpart D of this part and has not been excluded from paragraph (a)(2) of this section under §§260.20 and 260.22 of this chapter; however, the following mixtures of solid wastes and hazardous wastes listed in Subpart D of this part are not hazardous wastes (except by application of paragraph (a)(2) (i) or (ii) of this section) if the generator can demonstrate that the mixture consists of wastewater the discharge of which is subject to regulation under either Section 402 or 307(b) of the Clean Water Act (including wastewater at facilities which have eliminated the discharge of wastewater) and:

(A) One or more of the following solvents listed in § 261.31--carbon tetrachloride, tetrachloroethylene, trichloroethylene—*Provided*, That the maximum total weekly usage of these solvents (other than the amounts that can be demonstrated not to be discharged to wastewater) divided by the average weekly flow of wastewater into the headworks of the facility's wastewater treatment or pretreatment system does not exceed 1 part per million; or

(B) One or more of the following spent solvents listed in § 261.31--methylene chloride, 1,1,1-trichloroethane, chlorobenzene, o-dichlorobenzene, cresols, cresylic acid, nitrobenzene, toluene, methyl ethyl ketone, carbon disulfide, isobutanol, pyridine, spent chlorofluorocarbon solvents—provided that the maximum total weekly usage of these solvents (other than the amounts that can be demonstrated not to be discharged to wastewater) divided by the average weekly flow of wastewater into the headworks of the facility's wastewater treatment or pretreatment system does not exceed 25 parts per million; or

(C) One of the following wastes listed in § 261.32 -- heat exchanger bundle cleaning sludge from the petroleum refining industry (EPA Hazardous Waste No. K050); or

(D) A discarded commercial chemical product, or chemical intermediate listed in § 261.33, arising from *de minimus* losses of these materials from manufacturing operations in which these materials are used as raw materials or are produced in the manufacturing process. For purposes of this paragraph (a)(2)(iv)(D), "*de minimus*" 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 of process equipment, storage tanks or containers; leaks from well-maintained pump packings and seals; sample purgings; relief device discharges; discharges from safety showers and rinsing and cleaning of personal safety equipment; and rinsate from empty containers or from containers that are rendered empty by that rinsing; or

(E) Wastewater from laboratory operations containing toxic (T) wastes listed in Subpart D of this part, *Provided*, That the annualized average flow of laboratory wastewater does not exceed one percent of total wastewater flow into the headworks of the facility's wastewater treatment

or pretreatment system, or provided the wastes, combined annualized average concentration does not exceed on part per million in the headworks of the facility's wastewater treatment or pretreatment facility. Toxics (T) wastes used in laboratories that are demonstrated not to be discharged to wastewater are not to be included in this calculation; or

(F) One or more of the following wastes listed in § 261.32—wastewaters from the production of carbamates and carbamoyl oximes (EPA Hazardous Waste No. K157)—Provided that the maximum weekly usage of formaldehyde, methyl chloride, methylene chloride, and triethylamine (including all amounts that can not be demonstrated to be reacted in the process, destroyed through treatment, or is recovered, i.e., what is discharged or volatilized) divided by the average weekly flow of process wastewater prior to any dilutions into the headworks of the facility's wastewater treatment system does not exceed a total of 5 parts per million by weight; or

(G) Wastewaters derived from the treatment of one or more of the following wastes listed in § 261.32—organic waste (including heavy ends, still bottoms, light ends, spent solvents, filtrates, and decantates) from the production of carbamates and carbamoyl oximes (EPA Hazardous Waste Code No. K156).—Provided, that the maximum concentration of formaldehyde, methyl chloride, methylene chloride, and triethylamine prior to any dilutions into the headworks of the facility's wastewater treatment system does not exceed a total of 5 milligrams per liter.

261.4
Exclusion

(a) *Materials which are not solid wastes.* The following materials are not solid wastes for the purpose of this part:

(2) Industrial wastewater discharges that are point source discharges subject to regulation under Section 402 of the Clean Water Act, as amended.