



Department of Energy

Carlsbad Area Office
P. O. Box 3090
Carlsbad, New Mexico 88221

September 9, 1999

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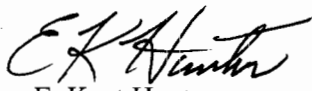
Mr. Greg Lewis
Water and Waste Management Division
New Mexico Environment Department
P. O. Box 26110
Santa Fe, NM 87502-6110

Dear Mr. Lewis:

Please find enclosed the Rocky Flats Environmental Technology Site (RFETS) Non-Mixed Waste Determination for TRU Stabilized Pyrochemical Salts Profile No. RF005.01 Revision 2.1 (GAO-049-99) dated September 1999. This document summarizes the effort performed at RFETS to characterize the waste and make the hazardous waste determination. Based upon the information summarized the waste stream is not hazardous. The waste stream meets the requirements of the Waste Acceptance Criteria for the Waste Isolation Pilot Plant, and the criteria for disposal as non-mixed (non-hazardous) TRU waste at the WIPP. CAO responses to NMED comments on the Non-Mixed Waste Determination for TRU Stabilized Pyrochemical Salts – Profile No. RF005.01 dated June 1999 are also applicable to the enclosed report.

If you have any questions please call me at (505) 234-7456.

Sincerely,


E. Kent Hunter
Assistant Area Manager
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GAO-049-99

September 1999

**Non-Mixed Waste Determination for
TRU Stabilized Pyrochemical Salts –
Profile No. RF005.01**

Revision 2.1

G. A. O'Leary

E. L. D'Amico

A. C. Kercher

J. L. Harrison

V. S. Sendelweck

**Non-Mixed Waste Determination for
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Revision 2

**G. A. O’Leary
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Published September 1999

**Rocky Flats Environmental Technology Site
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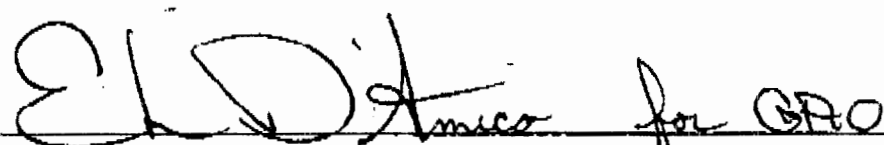
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GAO-049-99
Rev 2
September 1999

Rocky Flats Environmental Technology Site

Non-Mixed Waste Determination for

TRU Stabilized Pyrochemical Salts – Profile No. RF005.01



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9/1/99
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SUMMARY

This document summarizes the efforts performed at the Rocky Flats Environmental Technology Site (RFETS) to characterize the stabilized pyrochemical salts assigned Item Description Codes (IDCs) 454X, 411X, and 433X for the purpose of making a hazardous waste determination. This characterization effort includes a thorough compilation and documentation of acceptable knowledge, physical characterization, and solid waste sampling and analyses. Based upon the information and data summarized in this document, the stabilized pyrochemical salt waste discussed herein is not a hazardous waste (i.e., is non-mixed waste). No hazardous waste codes are assigned as specified in Title 40 of the Code of Federal Regulations, Part 261 (40 CFR 261). The waste meets the requirements for transuranic (TRU) waste as defined by the Waste Acceptance Criteria for the Waste Isolation Pilot Plant, prepared by the Department of Energy (DOE). Consequently, this waste meets the criteria for disposal as non-mixed (nonhazardous) TRU waste at the DOE Waste Isolation Pilot Plant.

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ACRONYMS

ACL	Analytical Chemistry Laboratory
AK	acceptable knowledge
BWR	Backlog Waste Reassessment
BWRBB	Backlog Waste Reassessment Baseline Book
CAO	Carlsbad Area Office
CFR	Code of Federal Regulations
DNFSB	Defense Nuclear Facility Safety Board
DOE	Department of Energy
DCHP	dicesium hexachloroplutonate
DOR	direct oxide reduction
DOT	Department of Transportation
EPA	Environmental Protection Agency
HWA	(New Mexico) Hazardous Waste Act
IDC	Item Description Code
ISSC	Interim Safe Storage Criteria
MSE	molten salt extraction
NRC	Nuclear Regulatory Commission
PRQL	program-required quantitation limit
QA	quality assurance
QAPP	Quality Assurance Program Plan
QC	quality control
RCRA	Resource Conservation and Recovery Act
RFETS	Rocky Flats Environmental Technology Site
RMRS	Rocky Mountain Remediation Services
RTL	regulatory threshold limit
RTR	real-time radiography
TC	Toxicity Characteristic
TCLP	Toxicity Characteristic Leaching Procedure
TIC	tentatively identified compound
TRU	transuranic
TRM	transuranic mixed
VOC	volatile organic compound
WAC	Waste Acceptance Criteria
WEMS	Waste and Environmental Management System

WIPP	Waste Isolation Pilot Plant
W/RT	Waste/Residue Traveler
WSRIC	Waste Stream and Residue Identification and Characterization

Rocky Flats Environmental Technology Site Non-Mixed Waste Determination for TRU Stabilized Pyrochemical Salts – Profile No. RF005.01

1. INTRODUCTION

This document describes the process and information used by the Rocky Flats Environmental Technology Site (RFETS) to determine that the stabilized salt waste assigned Item Description Codes (IDC) 454X, 411X, and 433X¹ are not hazardous waste regulated by either the Resource Conservation and Recovery Act (RCRA) or the New Mexico Hazardous Waste Act (HWA). To be considered a hazardous waste under these statutes, a waste must either be specifically listed as a hazardous waste or exhibit the hazardous characteristics of ignitability, corrosivity, reactivity, or toxicity.

Initially the Defense Nuclear Facility Safety Board (DNFSB) had concerns that a certain segment of the legacy pyrochemical salt IDCs (i.e., salts that have not been stabilized by pyro-oxidation) may have small amounts of unoxidized metals entrained in the salt matrix. As a result, the DNFSB was concerned that the IDCs that contained such metals as calcium, magnesium, or plutonium would have long term on-site storage issues unless such IDCs were shipped to WIPP in a timely manner (e.g., less than five years) or were reprocessed by pyro-oxidation. In light of the DNFSB recommendation 94-1, RFETS pursued a parallel path approach of continued sampling and pyro-oxidation of select IDCs that were originally categorized by the DNFSB as needing processing in the near term. The continued characterization program of legacy pyrochemical salts established the position that further pyro-oxidation of pyrochemical salts is not required because it had been shown that the salts are not reactive, ignitable, or pyrophoric as defined in and by DNFSB definitions. The salts included in Profile No. RF005.01 consist of pyro-oxidized salts only. Salts that will not undergo pyro-oxidation will be included in a separate profile.

Stabilized pyrochemical salt waste has been determined by RFETS to be a non-mixed (nonhazardous) waste. The defense generated waste [Ref. 1], consisting of Direct Oxide Reduction (DOR) Salt CaCl₂ Salt TRU Waste (IDC 454X), Electrorefining Salt TRU Waste (411X), and Scrub Alloy Spent Dicesium Salt TRU Waste (433X) were selected because of the extensive knowledge of this waste as non-mixed waste. The number of drums in the current inventory covered by this Non-Mixed Waste Determination by IDC are: 264 drums of 411X, 278 drums of 454X and 377 drums of 433X. There may be small changes in these numbers of drums due to processing and packaging of some additional salt wastes. Thus, RFETS has focused efforts on this waste for shipment to the Waste Isolation Pilot Plant (WIPP).

The RCRA regulations authorize hazardous waste determinations to be made either by using approved sampling and analysis methods or by applying knowledge of the waste in light of the materials or process used, typically referred to as 'process knowledge.' The WIPP Quality Assurance Program Plan (QAPP) [Ref. 3] refers to 'acceptable knowledge' as applying knowledge of the waste based on the materials or processes used to generate the waste. The term 'acceptable knowledge,' which is referred to throughout this document, is synonymous with 'process knowledge.' RFETS uses the following methods to determine the characterization of the stabilized pyrochemical salt waste (IDCs 454X, 411X, and 433X):

- Acceptable knowledge (AK);

¹ The "X" appended to these IDCs indicates the waste material has undergone salt stabilization prior to packaging. The "X" is not considered part of the waste shipping IDC.

- Visual examination; and
- Solid waste sampling and analysis of randomly selected containers.

RFETS is using AK data to support a non-mixed waste determination for this waste. Sampling and analysis and additional examinations are performed to verify this determination. Sections 2 through 5 of this report describe in detail the actions taken and the conclusions reached by RFETS with respect to this non-mixed determination. RFETS has demonstrated that the stabilized pyrochemical salt waste (IDC 454X, 411X, and 433X) is a non-mixed waste, in accordance with the requirements of 40 CFR 261.

2. WASTE GENERATION PROCESS DESCRIPTION

Pyrochemical salts are byproducts from a variety of pyrochemical plutonium metal purification and production processes conducted in the past at RFETS, including direct oxide reduction (DOR), molten salt extraction (MSE), salt scrub, electrorefining (ER), pyroredox, and vacuum melt processes. These historical processes produced several salt residues that are currently undergoing stabilization to produce IDC 454X, DOR Salt CaCl_2 Salt TRU Waste, IDC 411X, Electrorefining Salt TRU Waste, and IDC 433X, Scrub Alloy Spent Dicesium Salt TRU Waste [Ref. 2, 4, 10, 17]. Descriptions of the historical pyrochemical processes as well as the current stabilization process are provided below.

2.1 Historical Pyrochemistry Operations

In the DOR process, calcined plutonium oxide was placed in a crucible with calcium chloride and calcium metal and heated until molten. The molten material was stirred to initiate and promote the reduction of plutonium oxide to plutonium metal. After the reaction, the metal and salt phases were allowed to separate. The DOR process generated IDC 365 (entitled *salt from bad DOR run*) and IDC 414 (entitled *DOR salt – unoxidized calcium*) [Ref. 4]. This DOR process flow diagram is provided in Appendix A.

In the pyroredox process, impure plutonium metal was placed in a crucible with various salts and heated until molten. While molten, the zinc chloride reacted with the plutonium to form plutonium chloride and zinc metal. After separation and cooling, the salt phase was loaded in a crucible with calcium metal and heated until molten. While molten, the calcium reduced the plutonium chloride to plutonium metal. After the reaction, the metal and salt phases were allowed to separate. The pyroredox process generated molten salt extraction (MSE) salt – Ca, Zn, K (IDC 404) and Gibson salt (IDC 412) [Ref. 4]. The pyroredox process flow diagram is provided in Appendix A.

The vacuum melt process was a distillation process for separating plutonium, americium, magnesium, and zinc [Ref. 10]. This was a research and development process which produced only small volumes of waste, including Zn/Mg alloy metal (IDC 416) [Ref. 4 and 10]. The vacuum melt process flow diagram is provided in Appendix A.

The cleanout of pyrochemical furnace cells generated impure salt from cell cleanout (IDC 413). When this scraped out material was oxidized, the IDC was changed to IDC 426 (reburned 413) [Ref. 4].

In the ER process, non-specification plutonium metal, cast as an anode, was combined with sodium chloride, potassium chloride, and magnesium chloride and heated until molten. The molten mixture was stirred, and a current applied to the anode which flowed through the molten mixture to the cathode. Plutonium ions migrated from the molten anode through the molten salt to the cathode and were reduced to purified metal. After cooling, the salt, anode heel, and purified plutonium metal were separated. The

ER process generated IDCs 363, 364, 411, 473, and 654. [Ref. 4] The ER process flow diagram is provided in Appendix A.

In the calcium chloride– based MSE process, plutonium metal containing unacceptable levels of americium was combined with calcium chloride and dicesium hexachloroplutonate (DCHP). The metal and salts were placed in a crucible and heated until molten. The molten material was then stirred. While the mixture was molten, the DCHP oxidized most of the americium and some of the plutonium, and the oxidized actinides went to the salt phase. After stirring, the salt and metal phases were allowed to separate at the elevated temperature. The MSE process produced IDCs 433, 427, 434, and 435. [Ref. 4] The MSE process flow diagram is provided in Appendix A.

In the salt scrub process, MSE salts were combined with a reducing agent and an alloying agent; the combinations of magnesium and aluminum, calcium and gallium, and calcium and cerium have been used. The crucible and contents were heated until molten and the mixture was stirred. While molten, the plutonium and americium were reduced by the calcium or magnesium and the actinides went into the metal alloy phase. After cooling, the salt and scrub alloy was separated. This process produced IDCs 433, 434, and 435 [Ref. 4]. The salt scrub production process flow diagram is provided in Appendix A.

2.2 Salt Stabilization

To address concerns expressed in the DNFSB recommendation 94-1 concerning potentially reactive/pyrophoric species in salts, RFETS initiated a project for disposal of the pyrochemical salts using a pyro-oxidation process. This process involves the addition of a chemical oxidant such as Na_2CO_3 to a quantity of salt residue containing a known amount of plutonium. The salt and oxidant are melted, stirred, and held at temperatures of approximately 800-900 °C for two hours. The first 105 minutes of the process is conducted under an argon atmosphere to prevent nitride formation. During the last 15 minutes of the process, air is added to the argon stream to oxidize any remaining metallic species in the furnace headspace [Ref. 2, 10 and 17]. Although the results of the 94-1 Residue Characterization Program indicate legacy pyrochemical salts are not reactive or pyrophoric, the Salt Stabilization Program was underway before that determination was made. The stabilization by pyrochemical oxidation continued on those residue salts not sampled by the 94-1 program or for which the entire stream could not be statistically determined to be non-reactive.

The salt residues previously generated at RFETS are categorized into 25 IDCs. These residues have been grouped into four categories for stabilization. Each of the four categories is assigned a separate IDC. This document addresses three of the four categories of stabilized salts produced by the pyro-oxidation process. These categories are assigned IDC 454X, DOR Salt CaCl_2 Salt TRU Waste, IDC 411X, Electrorefining Salt TRU Waste, and IDC 433X, Scrub Alloy Spent Dicesium Salt TRU Waste, and are comprised of feed IDCs as described in Table 2-1 below [Ref. 10]. The process flow diagram for salt stabilization is provided in Appendix A.

Feed IDC	Processed IDC	Feed Salt Description
363	411X	ER Salt – First Run
364	411X	ER Salt – Second Run
411	411X	ER – Final Disposition

**Table 2-1
 Feed Salt and Processed Salt IDCs**

Feed IDC	Processed IDC	Feed Salt Description
473	411X	ER Salt packaged for LANL
654	411X	ER Salt from Pu/Np
413	411X	Impure Salt from Cell Cleanout
426	411X	ER Salt Reburned 413
427	433X	MSE Dicesium Salt
433	433X	Scrub Alloy Spent Dicesium Salt
434	433X	Salt with free calcium containing spent salt
435	433X	Ce/Ca Scrub Alloy Spent Salt
365**	454X or 433X	Salt from Bad DOR Run
404**	454X or 433X	MSE Salt – Ca, Zn, K
412**	454X or 433X	Gibson Salt
414**	454X or 433X	DOR Salt – Unoxidized Calcium
416**	454X or 433X	Zn/Mg Alloy Metal

** When combined with IDC 427, 433, 434, or 435, the output IDC is 433X.

3. CHARACTERIZATION PROCESS

3.1 Hazardous Waste Determination

A waste must exhibit a hazardous characteristic or be listed as a hazardous waste in the regulations to be deemed a hazardous waste under RCRA. The mere presence of particular constituents in a waste does not cause the waste to be hazardous if such constituents do not result from a prescribed use or do not exceed regulatory limits in a representative sampling. The RCRA regulations authorize hazardous waste determinations to be made either by using approved sampling and analysis methods or by applying knowledge of the waste in light of the materials or process used, also known as AK. Additionally, the U.S. Environmental Protection Agency (EPA) and the U.S. Nuclear Regulatory Commission (NRC) have recognized and encouraged the use of AK for radioactive waste (“Joint NRC/EPA Guidance on Testing Requirements for Mixed Radioactive and Hazardous Waste,” 62 Fed. Reg. 62079, 1997).

As discussed before, RFETS initially used the AK characterization process to support a non-mixed waste determination for stabilized pyrochemical salts (IDCs 454X, 411X, and 433X) [Ref. 2, 4, and 17]. Solid waste sampling and analysis and visual examination is performed in accordance with the WIPP QAPP requirements to verify this determination [Ref. 10 and 11].

3.2 Acceptable Knowledge

Acceptable knowledge includes information regarding the physical form of the waste, the base materials composing the waste, the nature of the radioactivity present, and the process generating the waste. This

section of the report specifically addresses the AK documentation used to characterize stabilized pyrochemical salts (IDCs 454X, 411X, and 433X) as a non-mixed waste. Compilation of AK and the conclusions reached following review of AK documentation for each of the categories of hazardous waste are discussed in this section and in Section 4.

3.2.1 Key Acceptable Knowledge Documentation

In October 1989, the Waste and Residue Identification and Characterization (WSRIC) program was implemented to assess the RCRA characterization of waste streams generated at RFETS, including transuranic (TRU) and transuranic mixed (TRM) waste streams. The information collected for the initial WSRIC program is documented in a book created for each building. These books describe in detail the waste streams generated by every process conducted in every building at RFETS, including process descriptions, process flow diagrams, chemical inputs, waste outputs, and waste characterization and rationale. Annual reverification of the books is conducted to assure that accurate and current information is maintained at the point of generation to assure proper waste management. The waste generators are responsible for updating the building books when processes or waste streams are added, deleted, or modified. Files containing the histories of waste streams are also maintained. When waste is packaged, the waste generator is responsible for recording the characterization information for each waste placed in a container. The WSRIC building books and history files are under change control and are maintained as WIPP quality assurance (QA) records [Ref. 1].

In December 1993, the Backlog Waste Reassessment (BWR) program was implemented to assess waste generated prior to approximately 1992 when the Waste/Residue Traveler (documentation that travels with each container) was implemented. Existing characterization documentation, including information from WSRIC, was compiled for the stored inventory of wastes at RFETS, and a review of the information was conducted to assess the RCRA characterization. The information collected by this program is compiled in the Backlog Waste Reassessment Baseline Book (BWRBB). The BWRBB and history files are also under change control and are maintained as WIPP QA records [Ref. 1].

Characterization information is taken from the WSRIC Building Book and recorded on a Waste/Residue Traveler (documentation that travels with each container) for each waste container. This information is then entered into the Waste and Environmental Management System (WEMS) database which tracks and controls the inventory, movement, and various waste management activities for waste containers from initial storage through disposal. The waste custodians are responsible for updating WEMS to incorporate WSRIC and BWR information [Ref. 1].

There are some areas of AK not addressed by the WSRIC or BWR programs because they are not within the scope of these programs. RFETS TRU Waste Acceptable Knowledge Supplemental Information, RF/RMRS-97-018, was initially assembled in 1997 to address these other areas of AK, including defense waste determination, radionuclides, matrix parameter categories, and waste material parameters [Ref. 1].

3.2.2 Description of Acceptable Knowledge Compilation

In an effort to meet the AK requirements in the WIPP QAPP [Ref. 3], summaries of the TRU waste stream information from the WSRIC and BWR programs and the RFETS TRU Waste Acceptable Knowledge Supplemental Information have been developed. The summary of this information is documented in RMRS-WIPP-98-100, Acceptable Knowledge TRU/TRM Waste Stream Summaries [Ref. 2]. The AK waste stream summary for TRU stabilized pyrochemical salts is provided in Appendix B.

Certification audits of the salt stabilization program were conducted at RFETS in September 1998 and April 1999. During these audits, the Carlsbad Area Office (CAO) of the Department of Energy (DOE) performed extensive reviews of the AK documentation. Various observers participated in one or both audits, including the EPA. The overall results from the audits were favorable and certification status for the stabilized salts was granted [Ref. 7].

3.3 Discussion of Acceptable Knowledge Determination for Stabilized Pyrochemical Salts

This section discusses in detail certain key AK documentation for DOR Salt CaCl_2 Salt TRU Waste (IDC 454X), Electrorefining Salt TRU Waste (IDC 411X), and Scrub Alloy Spent Dicesium Salt TRU Waste (IDC 433X) which relates to the non-mixed waste designation.

For waste to be RCRA-listed, it must meet listed waste criteria set forth in 40 CFR 261.31 – 261.33. The salt itself is not a RCRA-listed hazardous waste. AK documentation establishes that no listed waste came in direct contact with the salt during or after the historical pyrochemistry processes or the salt stabilization process [Ref 2 and 4]. Thus, the salts are not characterized as RCRA-listed wastes.

For waste to exhibit the characteristics of ignitability, corrosivity, or reactivity, it must have any of the properties listed in 40 CFR 261.21–261.23. AK documentation demonstrates that the salt does not have any of these properties [Ref. 2 and 4].

To exhibit the characteristic of toxicity, waste levels of certain metals or organics must meet or exceed regulatory thresholds defined in 40 CFR 261.24, based on Toxicity Characteristic Leaching Procedure (TCLP) test results. For RCRA regulatory compliance, data from the analysis of the metal and organic compounds are compared to the toxicity characteristic (TC) levels expressed as total values. These total values are considered the regulatory threshold limit (RTL) values in the WIPP QAPP [Ref. 3, Section 3.3.1]. RTL values are obtained by calculating the weight/weight concentration (in the solid) of a TC analyte that would give the regulatory weight/volume concentration (in the TCLP extract) assuming 100-percent analyte dissolution. AK documentation demonstrates that no organic compounds were used in historical pyrochemistry processes or the salt stabilization process [Ref 2, 4, and 17]. Total metals analyses conducted on the stabilized salts (IDCs 411X, 433X, and 454X) demonstrate that the waste does not exhibit the characteristic of toxicity for metals. Total metals results are summarized in Appendix C.

3.4 Relevant Data and Supporting Information

Physical and chemical characterization of the Electrorefining Salt TRU Waste (IDC 411X), Scrub Alloy Spent Dicesium Salt TRU Waste (IDC 433X), and DOR Salt CaCl_2 Salt TRU Waste (IDC 454X) was undertaken to (a) verify the AK documented for this waste form, and (b) support the determination of the non-mixed status of the waste. Included in this section are those intrusive examinations and analyses conducted to ensure compliance with the WIPP waste acceptance criteria (WAC) [Ref. 3 and 5] and to

confirm the AK RCRA waste determination. These examinations and analyses include physical inspection of the waste (Section 3.4.1) and solid waste sampling and analysis conducted in accordance with the WIPP QAPP (Section 3.4.2). The data generated from these examinations and analyses are maintained in the Waste Records center.

3.4.1 Physical Characterization

For the purposes of this document, “physical” characterization includes visual examination only. Electrorefining Salt TRU Waste (IDC 411X), Scrub Alloy Spent Dicesium Salt TRU Waste (IDC 433X), and DOR Salt CaCl₂ Salt TRU Waste (IDC 454X) are considered “newly generated” waste per the requirements of the WIPP QAPP [Ref. 3] and is therefore visually examined during repackaging following stabilization [Ref. 10 and 11]. Radiography is not required for newly generated waste per the WIPP QAPP [Ref. 3]. In addition, stabilized pyrochemical salts have been exempt from headspace gas sampling and analysis due to the destruction of organic compounds by the pyro-oxidation process [Ref. 9].

3.4.1.1 Program Requirements for Examination of Contents

Newly generated waste in which the matrix parameter category and waste material parameter weights are independently verified by visual examination and documented during waste packaging operations do not require radiography or any other additional visual examination [Ref. 3 and 12].

Newly generated waste at RFETS is segregated and placed into payload containers based upon its physical form. The physical form of the waste contents placed in a payload container is identified by the assigned IDC. Only contents of the same IDC are allowed in one payload container. Contents emplaced into a payload container are documented on the contents log sheet of the W/RT (see Section 3.2.1). Some newly generated waste (e.g., residue repackaging into opaque cans that is subsequently assayed prior to placement into payload containers) will require visual examination to be performed before the waste is placed into payload containers. Electrorefining Salt TRU Waste (IDC 411X), Scrub Alloy Spent Dicesium Salt TRU Waste (IDC 433X), and DOR Salt CaCl₂ Salt TRU Waste (IDC 454X) are examples of this situation.

Visual examination is performed and recorded according to the salt residue stabilization/repack procedure [Ref. 11]. The salt residue stabilization process operators perform visual examination of the stabilized salt to verify the following [Ref. 10 and 11]:

- Matrix parameter category is S3141, chloride salts
- Material is a dry solid and agrees with the assigned IDC
- Material is comprised 100% by weight of waste material parameter “other inorganic material”
- Absence of prohibited materials, such as free liquid, explosives, pyrophoric materials, compressed gases, classified waste, corrosive wastes, reactive wastes, and sealed containers greater than 4 liters
- Waste material does not include plastic, combustible, or other organic material

The visual examination data for the Electrorefining Salt TRU Waste (IDC 411X), Scrub Alloy Spent Dicesium Salt TRU Waste (IDC 433X), and DOR Salt CaCl₂ Salt TRU Waste (IDC 454X) receive an independent technical review. An individual other than the data generator who is qualified to have performed the initial work performs this review [Ref. 11].

3.4.2 Sampling and Analysis

To ensure Electrorefining Salt TRU Waste (IDC 411X), Scrub Alloy Spent Cesium Salt TRU Waste (IDC 433X), and DOR Salt CaCl_2 Salt TRU Waste (IDC 454X) do not exhibit the characteristic of toxicity for metal compounds, sampling and analysis of the stabilized salts is performed [Ref. 10 and 11]. Volatile and semi-volatile organic compound analyses are not performed on the salts because the WIPP QAPP [Ref. 3, Section 5.2.1] specifically excludes salt wastes (summary category S3140) from these analyses due to the high temperature environment of the pyrochemistry operations.

3.4.2.1 Number of Samples and Sampling Frequency

The number of samples and sampling frequency was established using the method for retrievably stored waste described in the WIPP QAPP [Ref. 3, Section 5]. Metals data from preliminary sampling and analysis of IDCs 411X, 433X, and 454X were used to provide an estimate of the mean concentrations and variance for each metal analyte. The metal with the highest coefficient of variance was used to calculate the number of random samples required [Ref. 10 and 14]. The number of samples calculated for IDCs 411X, 433X, and 454X using this method was 5. However, to ensure a sufficient number of samples were collected, this number was increased. The random number generation function of Excel software was utilized to produce the random numbers of the output IDC packages to be sampled [Ref. 10].

Based on the IDC 454X results from the log-transformed data analysis for each analyte, the minimum required sample size is 5 samples. Therefore, the 9 samples of IDC 454X collected and analyzed are sufficient [Ref. 15].

Based on the IDC 411X results from the log-transformed data analysis for each analyte, the minimum required sample size is 5 samples. Therefore, the 8 samples of IDC 411X collected and analyzed are sufficient [Ref. 16].

Based on the IDC 433X results from the log-transformed data analysis for each analyte, the minimum required sample size is 5 samples. Therefore, the 13 samples of IDC 433X collected and analyzed are sufficient [Ref. 18].

3.4.2.2 Sampling and Analysis Procedure

Prior to sampling, the salts are brought to a molten state and stirred for two hours. If a sample is collected of the molten salt, it is collected at the end of the process run immediately after the stirring motor is turned off. A carbon steel cup is lowered into the molten salt to obtain a representative sample. The cup is carbon steel so none of the target analyte metals are introduced into the sample by the sampling tool. The sample also may be collected following the stabilization process by emptying the contents of the crucible into a cake pan and collecting a grab sample [Ref. 10 and 11].

To ensure samples are not cross-contaminated, a new cup is used for each sampling event. The sampling method produces a sample that is representative of the container's contents. The two-hour stirring cycle prior to sampling ensures the homogeneity of the sample. The last four random samples of 433X collected for the salt stabilization process were duplicate sampled to verify the homogeneity of the sampling method. [Ref. 10].

The laboratory procedures used to provide analytical results for metals conform to methods recommended in SW-846 [Ref. 6] and the quality assurance methods, quality controls, and data requirements described in the WIPP QAPP [Ref. 3, Section 15]. Even though SW-846 [Ref. 6] requires sample preservation at 4 °C for mercury, deviation from this preservation requirement is approved by CAO based on the

determination that elevated temperatures used in salt processing preclude the existence of volatile species [Ref. 10 and 13].

3.4.2.3 Summary of Analytical Results

Based on the total metals results from sampling of IDCs 411X, 433X, and 454X, no toxicity characteristic compounds were found to be greater than the regulatory threshold limits. Refer to Appendix C for a summary of the results.

4. DETAILED RCRA ANALYSIS OF STABILIZED SALTS

For a waste to be hazardous under RCRA or the New Mexico HWA, it must be specifically listed as a hazardous waste or exhibit a hazardous characteristic as specified in the applicable regulation. The characteristics are toxicity, corrosivity, ignitability, and reactivity. The mere presence of hazardous constituents does not necessarily cause a waste to be hazardous by definition if the constituents do not result from a prescribed use or do not exceed regulatory limits. RCRA regulations authorize a generator to determine if a waste is hazardous by either applying knowledge of the hazardous nature of the waste in light of the material or the process used or by testing the waste in accordance with approved methods.

4.1 Listed Waste

Waste is potentially subject to RCRA regulations as a hazardous waste if it is a waste listed in 40 CFR 261.31 – 261.33. Based on AK, it is known that Electrorefining Salt TRU Waste (IDC 411X), Scrub Alloy Spent Dicesium Salt TRU Waste (IDC 433X), and DOR Salt CaCl₂ Salt TRU Waste (IDC 454X) are not listed wastes. The F-listing is not applicable because no listed solvents came in direct contact with the salt during or after the historical pyrochemistry processes or the salt stabilization process [Ref 2 ,4 and 17].

4.2 Characteristic Waste

4.2.1 Toxicity

It has been determined that the drums containing IDCs 411X, 433X, and 454X waste do not meet the definition of a toxic characteristic waste. To exhibit the toxicity characteristic, waste levels of certain metals or organics must meet or exceed certain levels that the EPA has identified as hazardous to projected receptors.

As previously stated in Section 3.3, AK documentation states IDCs 411X, 433X, and 454X do not exhibit the characteristic of toxicity for metal or organic compounds [Ref. 2, 4, and 17]. As discussed in Section 3.4.2, total metals analysis was performed on representative samples of IDCs 454X, 411X, and 433X salts to confirm that the waste does not exhibit the characteristic of toxicity. The rationale for performing total metals analysis rather than TCLP is discussed in Section 3.3. As indicated in Appendix C, the analytical results demonstrate that no metals exceed the regulatory threshold limits.

4.2.2 Corrosivity

It has been determined that IDC 411X, 433X, and 454X wastes do not meet the definition of a characteristic waste due to corrosivity. To be corrosive waste under RCRA, a material must possess either of the following properties:

- It is aqueous with a pH less than or equal to 2 or greater than or equal to 12.5. To measure the pH, the EPA prescribes the use of Method 9040 in the definition of corrosivity found at 40 CFR 261.22. This method requires that greater than 20% of the total waste volume is aqueous; or
- It is a liquid as determined by its ability to pass through a certain type of filter and will corrode steel at a rate of 0.25 inches per year.

As determined by visual inspection, this waste is neither aqueous nor liquid. Therefore the waste cannot be corrosive per RCRA definition. This conclusion is substantiated in a letter from the EPA Policy Compendium [Ref. 8]. EPA states that the characteristic of corrosivity as defined in 40 CFR 261.22 is intended to apply only to aqueous or liquid media, "unless and until the EPA promulgates a definition for [corrosive] solids. The agency has no plans to do this at the present time." EPA has not promulgated a definition of solids as of this date.

4.2.3 Ignitability

Based on acceptable knowledge documentation, it has been determined that IDC 411X, 433X, and 454X wastes do not meet the definition of an ignitable waste under RCRA. To be a RCRA waste in this category, a material must possess any of the following properties:

- It is a liquid other than an aqueous solution containing less than 24 percent alcohol and flash point less than 140°F (60°C);
- It is not liquid and is capable of causing fire through friction, absorption of moisture, or spontaneous chemical changes;
- It is an ignitable compressed gas; or
- It is an oxidizer as defined by U.S. Department of Transportation (DOT) regulations.

As discussed above, the waste does not contain liquids. Acceptable knowledge documentation demonstrates that the waste will not ignite through friction, moisture absorption, or chemical changes. There is no compressed gas in the waste. Finally, the waste is not an oxidizer as defined in DOT regulations in 49 CFR 173.1514 [Ref. 2 and 4]. Visual inspection of the waste confirms these conclusions.

4.2.4 Reactivity

It has been determined that IDC 411X, 433X, and 454X wastes do not meet the definition of a reactive waste under RCRA. To be a RCRA waste in this category, a material must possess any one of the following properties:

- It is unstable and can undergo violent change;
- It reacts violently with water;
- It forms potentially explosive mixtures with water;

- It reacts with water to generate toxic gases, vapors, or fumes that are harmful;
- It contains cyanide or sulfide that can generate toxic gases, vapors or fumes;
- It can detonate or explode at standard temperature and pressure; or
- It is a DOT forbidden Class A or B explosive.

IDC 411X, 433X, and 454X wastes are stable and do not undergo violent chemical change. These wastes do not contain cyanides or sulfides and are not capable of detonation or explosive reaction. These wastes do not react violently with water, form potentially explosive mixtures with water, or generate toxic gases, vapors, or fumes when mixed with water [Ref. 2 and 4].

5. CONCLUSION

RFETS has established that the stabilized pyrochemical salt waste (IDCs 411X, 433X, and 454X) discussed above is not a hazardous waste. The waste does not exhibit any hazardous characteristic and is not a listed waste. The determination of whether this waste is hazardous for RCRA purposes was initially made on the basis of acceptable knowledge. Subsequently, visual examinations and solid waste sampling and analysis were conducted to confirm that the AK documentation is correct.

6. REFERENCES

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2. Acceptable Knowledge TRU/TRM Waste Stream Summaries. RMRS-WIPP-98-100, Revision 3. May 20, 1999.
3. *Transuranic Waste Characterization Quality Assurance Program Plan*, Interim Change, CAO-94-1010, November 15, 1996.
4. Backlog Waste Reassessment Baseline Book, Waste Form 34 Pyrochemical Salts. October 26, 1998.
5. *Waste Acceptance Criteria for the Waste Isolation Pilot Plant*, DOE/WIPP-069, Revision 5, April 1996.
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7. I. R. Triay letter to J. Roberson, *Certify Rocky Flats Environmental Technology Site (RFETS)*, CAO:QA:MEB:99-0842 UFC 5822.00, June 3, 1999.
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9. E. K. Hunter letter to J. Legare, *Residue TRU Waste Characterization Methods Evaluation*, CAO:NTP:RAS 98-0617-UFC-5822, February 12, 1998.
10. Salt Residue Stabilization, Building 707 Process Control/Qualification Plan. RS-020-006, Revision 1. April 14, 1999.
11. Salt Residue Stabilization/Repack, Building 707. 4-W84-RS-0114, Revision 3. June 14, 1999.
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14. Salt Residue Stabilization Building 707 Qualification Report. RS-020-017, Revision 2. June 29, 1999.
15. RFETS Interoffice Correspondence from T. R. Gatcliffe to E. L. D'Amico, *Statistical Solid Analysis Data Evaluation Report for Stabilized Pyrochemical Salts Lot 1 (IDC 454X, Profile RF005.01)*, TRG-016-99, April 1, 1999.
16. RFETS Interoffice Correspondence from T. R. Gatcliffe to E.L. D' Amico, *Statistical Solid Analysis Data Evaluation Report for Stabilized Pyrochemical Salts Lot 2 (IDC 411X, Profile RF005.01)*, TRG-032-99, July 12, 1999.
17. Waste Stream and Residue Identification and Characterization, Building 707, Version 6.0, 1999.
18. RFETS Interoffice Correspondence from T. R. Gatcliffe to E. L. D'Amico, *Statistical Solid Analysis Data Evaluation Report for Stabilized Pyrochemical Salts Lot 3 (IDC 433X, Profile RF005.01)* – TRG-040-99, August 31, 1999.

Appendix A
Pyrochemical Salt Process Flow Diagrams

Appendix A

AutoCAD 12, File Name: 707-39

707-V6.0

DOES NOT CONTAIN UCNI
 UNCLASSIFIED CONTROLLED NUCLEAR INFORMATION
 5/25/99
 V8 Sandelmark

Process 39-4
 02/26/99

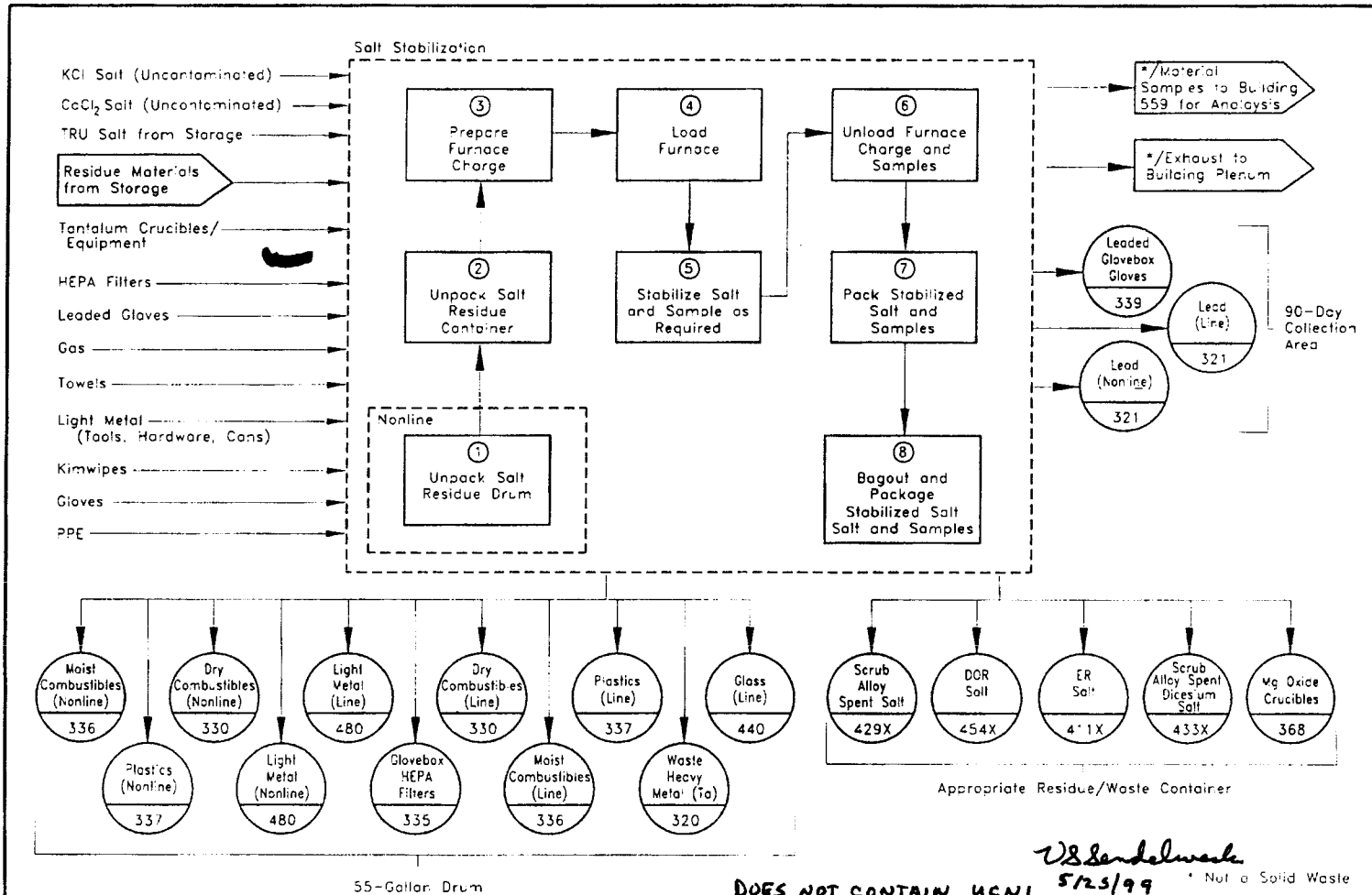


FIGURE 39.1 **DOES NOT CONTAIN UCNI 5/25/99** * Not a Solid Waste
 UNCLASSIFIED CONTROLLED NUCLEAR INFORMATION

Rocky Flats Environmental Technology Site Waste Stream & Residue Identification & Characterization	BUILDING 707/PROCESS 39 SALT STABILIZATION	Task	By	Date
		Designed	J. Schoen	07/03/97
		Drawn	C. Stadl	07/03/97
		Checked	J. Schoen	02/26/99

Appendix A

AutoCAD 10, File Name: f779-30d1 2/21/91

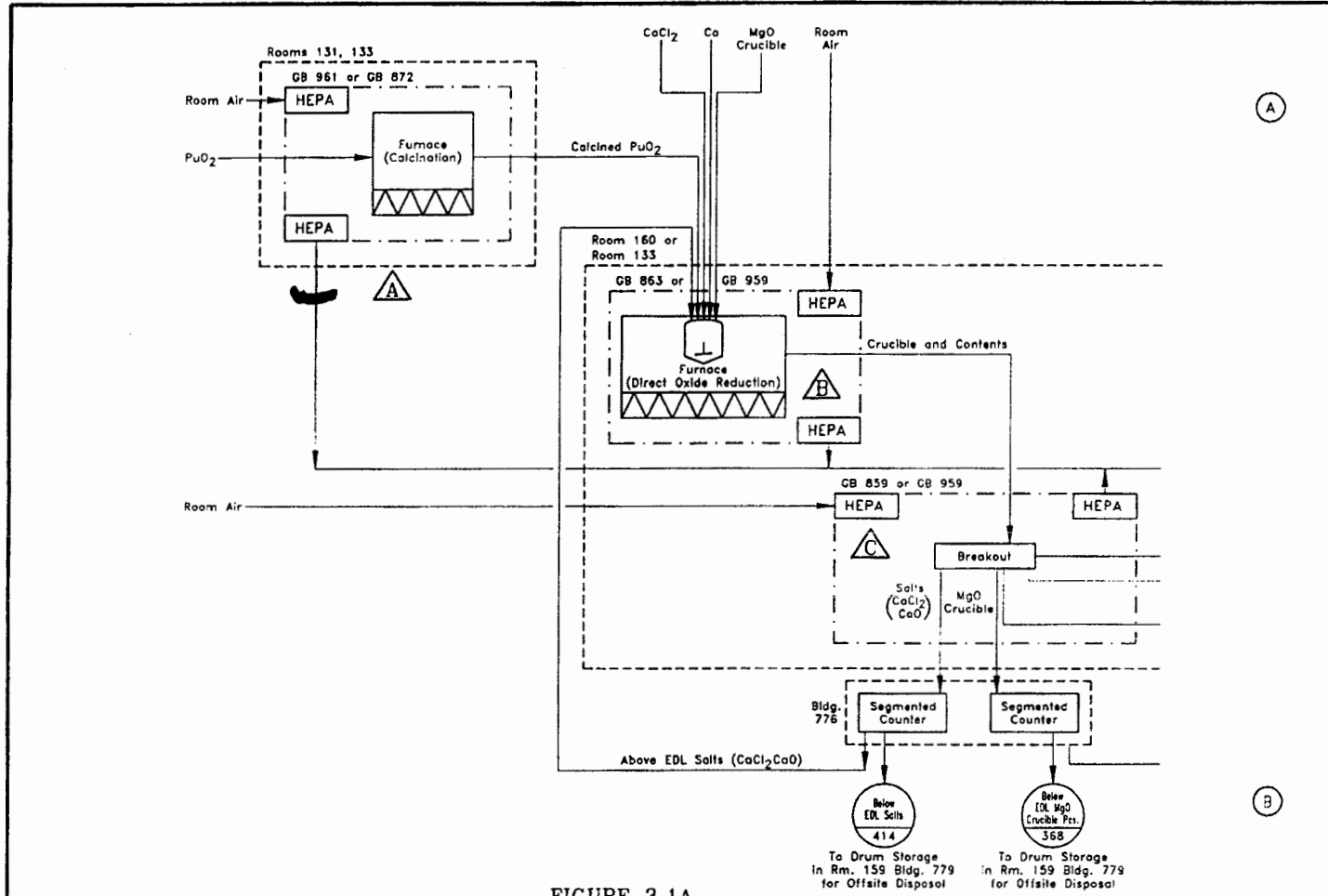


FIGURE 3.1A

Rocky Flats Plant Waste Stream & Residue Identification & Characterization	BUILDING 779/PROCESS 3 DIRECT OXIDE REDUCTION	Task	By	Date
		Designed	R. Foster	2/23/90
		Drawn	E. McDonald	7/30/90
		Checked	R. Medlyn	8/23/90

779-V3.2 Chg. 1

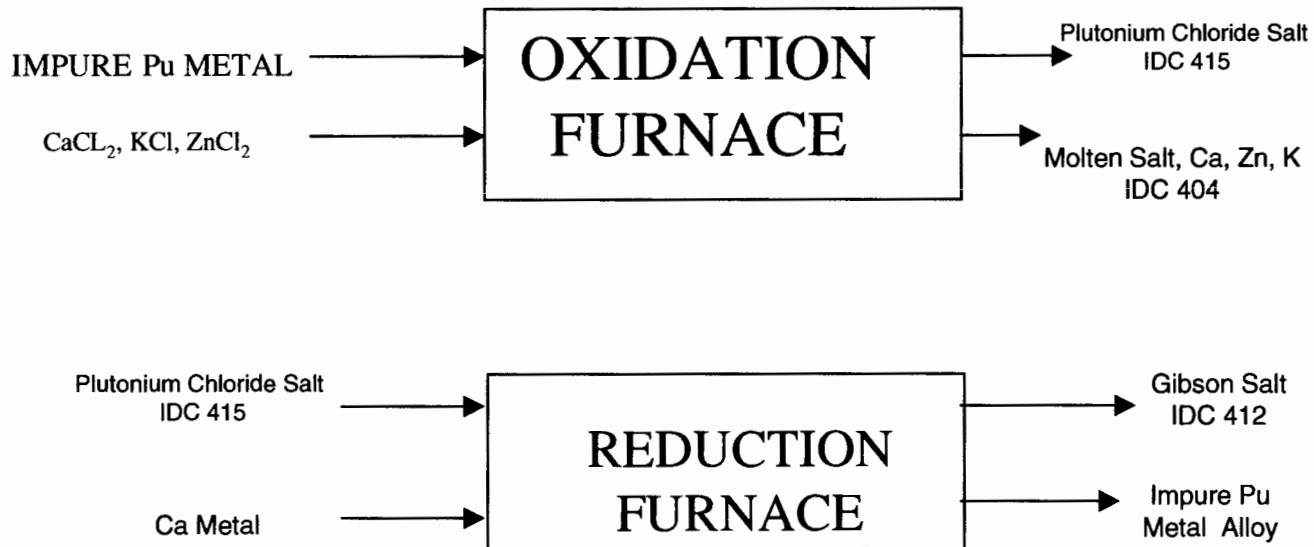
3-2

10/28/91

Appendix A

PYROREDOX PROCESS

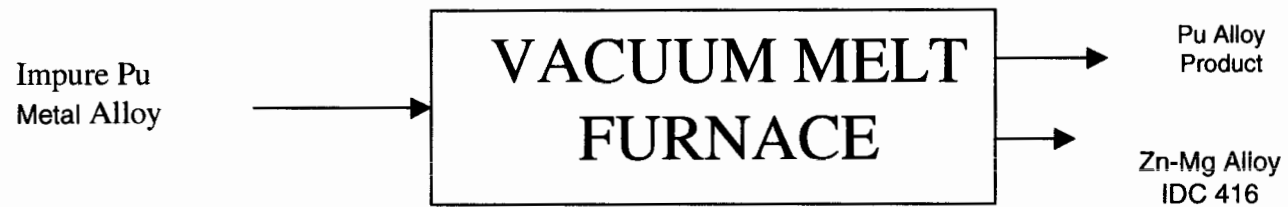
Performed in Buildings 776 and 779



Appendix A

VACUUM MELT PROCESS

Performed in Buildings 776 and 779



Appendix A

AutoCAD 11, File Name: 1779-34c 9/11/91

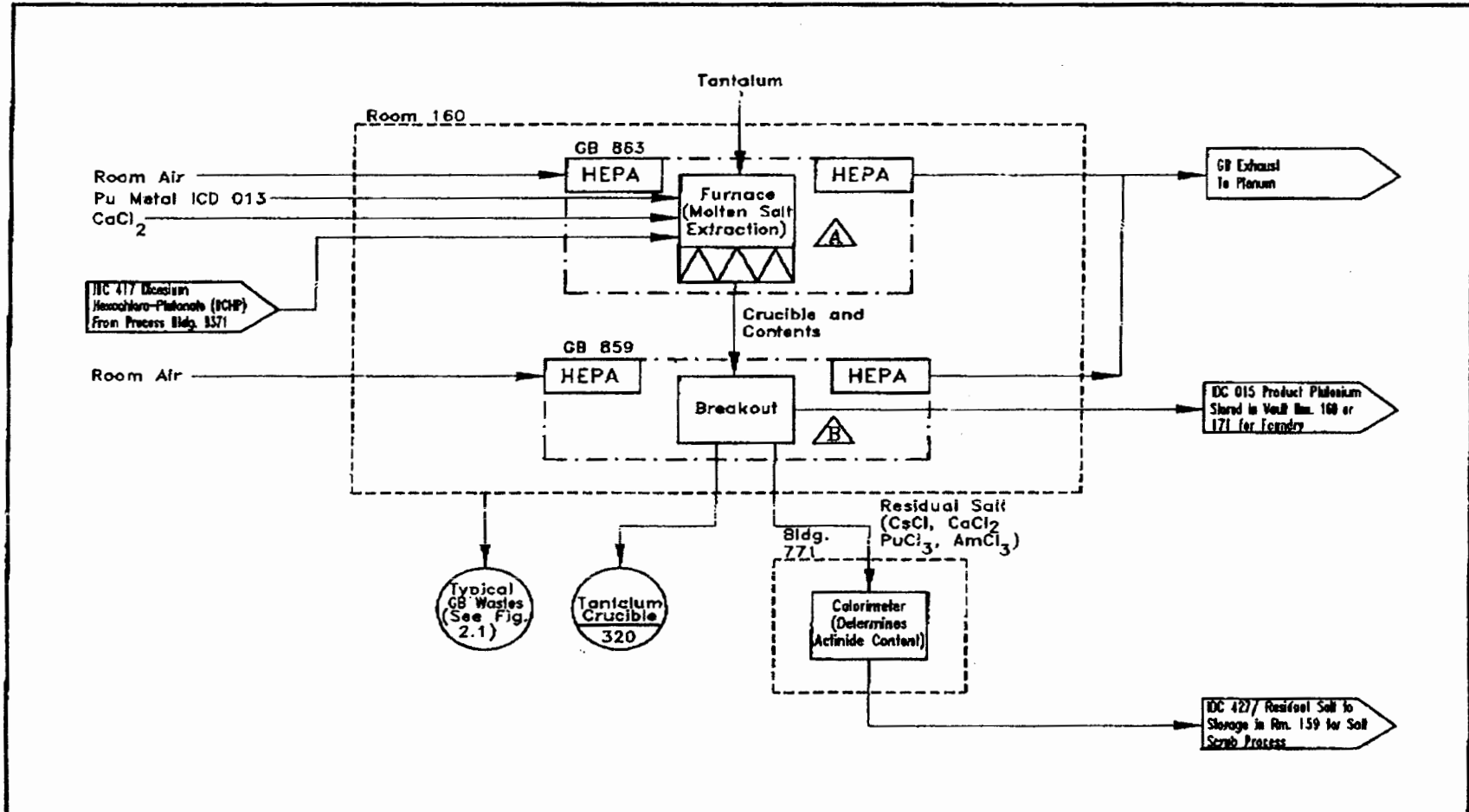


FIGURE 4.1

Rocky Flats Plant Waste Stream & Residue Identification & Characterization	BUILDING 779/PROCESS 4 PYROTECHNICAL TECHNOLOGY-MOLTEN SALT EXTRACTION			Task	By	Date
				Designed	R. Foster	2/23/90
				Drawn	J. Sikora	7/30/90
				Checked	S. Muse	8/1/91

Appendix A

AutoCAD 12, File Name: 779-5 12/19/94

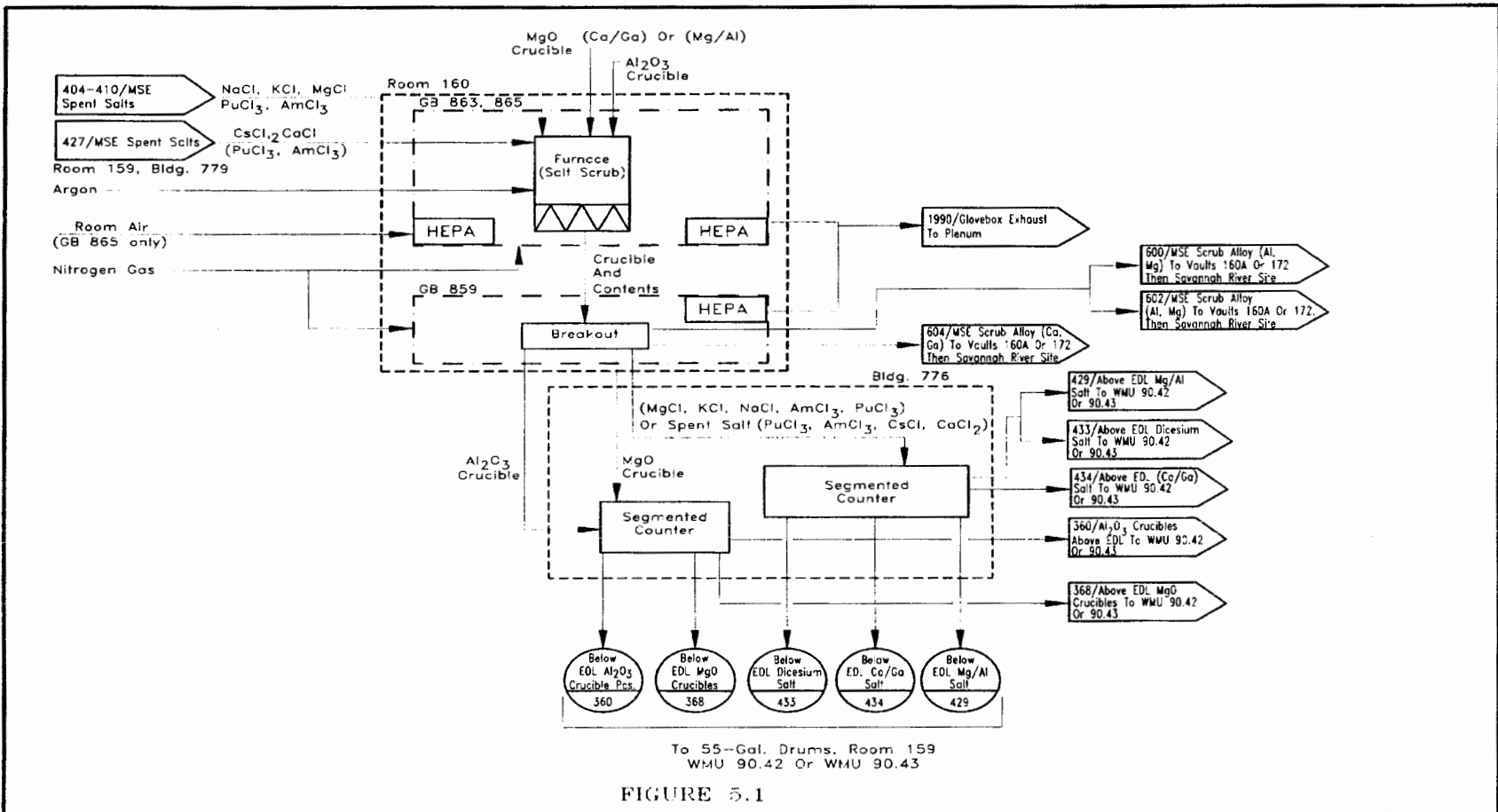


FIGURE 5.1

Rock, Flats Environmental Technology Site Waste Stream & Residue Identification & Characterization	BUILDING 779, PROCESS 5 RTT: NISE SALT SCRUB		Task	By	Date
			Designed	R. Foster	02/23/90
			Drawn	E. McDonald	07/30/90
			Checked	J. Joyce	12/19/94

Appendix B

**TRU Stabilized Pyrochemical Salts
Acceptable Knowledge Waste Stream Summary**

APPENDIX B

5.5 TRU Stabilized Pyrochemical Salts

Profile No. RF005.01

Acceptable Knowledge Waste Stream Summary

Waste Stream: TRU Stabilized Pyrochemical Salts, RF-411X, RF-429X, RF-433X, RF-454X

Generation Building: Building 707^(4,8)

Waste Stream Volume (Current): 785 55-Gallon Drums^(8, 11,12)

Generation Dates (Current): January 1998 – June 1999^(8, 11,12)

Waste Stream Volume (Projected): 615 55-Gallon Drums^(9, 11,12)

Generation Dates (Projected): July 1999 – July 2000^(9, 11)

TRUCON Content Codes^(1,10): RF-124C, RF-124D (pending)

NOTE: RF-124C has been superceded by RF-124D, which is now approved. The AK waste stream summaries will be updated to include these changes.

Transuranic Waste Baseline Inventory Report Information⁽²⁾

WIPP Identification Numbers: RF-TR-0404, RF-TR-0405, RF-TR-0406, RF-TR-0407, RF-TR-0408, RF-TR-0409, RF-TR-0410, RF-TR-0411, RF-TR-0412, RF-TR-0413, RF-TR-0414, RF-TR-0415, RF-TR-0416, RF-TR-0418, RF-TR-0427, RF-TR-0429, RF-TR-0433, RF-TR-0434, RF-TR-0473, RF-TR-0654, RF-MR-0365, RF-MR-0409, RF-MR-0411, RF-MR-0413, RF-MR-0414, RF-MR-0434

NOTE: The mixed residue IDCs have been recharacterized as nonhazardous.⁽⁶⁾

APPENDIX B

Summary Category Group: S3000 Waste Matrix Code Group: S3141

Waste Stream Name: Misc. Pu Recovery Byproducts/TRU, Misc. Pu Recovery Byproducts/TRM

Description from the TWBIR: Generated during plutonium recovery operations such as direct oxide reduction, molten salt extraction, electrorefining, and salt scrub. Includes chunks and powdered mixed salts, a probable presence of magnesium, sodium, and potassium metals.

Waste Stream Description

The following table presents the matrix parameter category and waste material parameters for TRU stabilized pyrochemical salts.⁽²⁾

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IDC	Matrix Parameter Category	Waste Material Parameters	Weight % (Average)
411X	S3141, Chloride Salts	Other Inorganic Materials	100%
429X	S3141, Chloride Salts	Other Inorganic Materials	100%
433X	S3141, Chloride Salts	Other Inorganic Materials	100%
454X	S3141, Chloride Salts	Other Inorganic Materials	100%

APPENDIX B

IDC 411X, Electrorefining Salt: Spent salt from the electrorefining (ER) process in Building 371. Other salts which become IDC 411 for disposal include ER Salt, First Use (IDC 363), ER Salt, Second Use (IDC 364), ER Salt-Final Disposition (IDC 411), impure salt from cell clean-out (IDC 413), reburned IDC 413 (IDC 426), ER Salt Packaged for LANL (IDC 473), and ER Salt from Pu/Np (IDC 654). An "X" may be appended to this IDC to indicate the waste material has undergone salt stabilization prior to packaging. The "X" is not considered part of the waste shipping IDC.^(4,5)

IDC 429X, Scrub Alloy Spent Salt: Spent salt from the molten salt extraction (MSE) scrub alloy process in Building 776. Other salts which become IDC 429 for disposal include MSE, Unknown % Unpulverized (IDC 405), MSE, Unknown % Pulverized (IDC 406), MSE, 8% Unpulverized (IDC 407), MSE, 8% Pulverized (IDC 408), MSE, 30% Unpulverized (IDC 409), MSE, 30% Pulverized (IDC 410),

Pu chloride mixed salt (IDC 415), MSE Salt Packaged for LANL (IDC 418), and scrub alloy spent salt (IDC 429). An "X" may be appended to this IDC to indicate the waste material has undergone salt stabilization prior to packaging. The "X" is not considered part of the waste shipping IDC.^(4,5)

IDC 433X, Scrub Alloy Spent Dicesium Salt: Spent salt from the MSE scrub alloy process that used dicesium salt. Other salts, which become IDC 433X for disposal, include MSE spent dicesium salt (IDC 427), scrub alloy spent dicesium salt (IDC 433), free calcium containing spent salt (IDC 434), and cerium/calcium spent salt (IDC 435). Salts which also may become IDC 433X for disposal include salt from bad DOR run (IDC 365), MSE Salt, Ca, Zn, K (IDC 404), Gibson salt (IDC 412), DOR salt – unoxidized calcium (IDC 414), Zn-Mg alloy metal (IDC 416) and DOR salt oxidized calcium (IDC 454). An "X" may be appended to this IDC to indicate the waste material has undergone salt stabilization prior to packaging. The "X" is not considered part of the waste shipping IDC.^(4,5)

IDC 454X, DOR Salt-Oxidized Calcium: Spent salt from the direct oxide reduction (DOR) process. Other salts which become IDC 454X for disposal include salt from bad DOR run (IDC 365), MSE salt, Ca, Zn, K (IDC 404), Gibson salt (IDC 412), DOR salt-unoxidized calcium (IDC 414), Zn-Mg alloy metal (IDC 416), and DOR salt – oxidized calcium (IDC 454). An "X" may be appended to this IDC to indicate the waste material has undergone salt stabilization prior to packaging. The "X" is not considered part of the waste shipping IDC.^(4,5)

Areas of Operation

TRU stabilized pyrochemical salt wastes are generated in Module A of Building 707.⁽⁴⁾

Generation Processes

Pyrochemical salts are primarily byproducts from a variety of pyrochemical plutonium metal purification and production processes conducted in the past at RFETS.⁽⁶⁾ All pyrochemical salts generated are defense wastes.⁽⁷⁾ The following processes generated pyrochemical salts.⁽⁶⁾

- Molten Salt Extraction
- Electrorefining
- Direct Oxide Reduction

APPENDIX B

- Salt Scrub
- Pyroredox

In addition to these processes, IDC 413 salt was generated by the scraping and cleaning of pyrochemical furnace cells, and IDC 416 metal was generated from research and development salt cleanup projects.⁽⁶⁾

The pyrochemical residues may require stabilization which will be accomplished by oxidation of the materials in furnaces located in Module A of Building 707.⁽⁴⁾ This process will oxidize any potentially pyrophoric or reactive metals present in the salts (i.e., plutonium, cerium, calcium, sodium, magnesium, and potassium).⁽³⁾ The process flow diagram for salt stabilization is provided in the WSRIC Book, 707-39 Figure 1.1.⁽⁴⁾

RCRA Characterization

The following table presents the chemical constituent codes (CCC) and EPA Hazardous Waste Numbers associated with the WSRIC Waste Streams assigned to TRU stabilized pyrochemical salt containers.⁽⁴⁾

IDC	WSRIC Waste Stream	RCRA CCC	Non-RCRA CCC	EPA Hazardous Waste Numbers
411X	707 - 39 - 15	00	00	None
429X	707 - 39 - 17	00	00	None
433X	707 - 39 - 14	00	00	None
454X	707 - 39 - 16	00	00	None

Radionuclides

The following table presents the radionuclides in TRU stabilized pyrochemical salt wastes.⁽⁷⁾

IDC	Radionuclides
411X	WG Pu, Am-241, EU, Np-237
429X	WG Pu, Am-241
433X	WG Pu, Am-241
454X	WG Pu, Am-241

Key: WG Pu weapons-grade plutonium
 Am-241 americium-241
 EU enriched uranium
 Np-237 neptunium-237

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References

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2. DOE 1995. Transuranic Waste Baseline Inventory Report, Revision 2. DOE/CAO-95-1121.
3. RFETS 1998. Salt Residue Stabilization, Building 707 Process Control/Qualification Plan. RS-020-006.
4. RFETS 1999. Waste Stream and Residue Identification and Characterization, Building 707, Version 6.0.
5. RFETS 1998. Solid Radioactive Waste Packaging, 4-D99-WO-1100.
6. RFETS 1998. Backlog Waste Reassessment Baseline Book, Waste Form 34, Pyrochemical Salts.
7. RFETS 1998. RFETS TRU Waste Acceptable Knowledge Supplemental Information, RF/RMRS-97-018, Revision 3.
8. Waste and Environmental Management System (WEMS) database.
9. KH 1999. Kaiser-Hill Interoffice Memorandum from Ken Ferrera to Jerry O'Leary. KPF-006-99. March 15.
10. DOE 1997. TRUPACT-II Content Codes (TRUCON), Revision 11. DOE/WIPP 89-004.
11. RMRS 1999. Interoffice Memorandum from Jeff Harrison to TWCP Records. JLH-001-1999. April 13.
12. RMRS 1999. Interoffice Memorandum from Jeff Harrison to TWCP Records. JLH-005-1999. June 23.

7/2/99

7/2/99

Appendix C
Total Metals Data Summary
IDCs 411X, 433X, and 454X

Appendix C

Total Metals Data Summary IDCs 411X, 433X, and 454X

Eight cans of IDC 411X, 13 cans of IDC 433X, and nine cans of IDC 454X were randomly selected for sampling and analysis for total metals as required in the QAPP. The feed salt IDCs (see Table 2-1) were processed through a pyro-oxidation stabilization treatment prior to sample collection. Samples were collected as described in Section 3.4.2.

The formula used to calculate the 90% upper confidence limit (UCL₉₀) is:

$$UCL_{90} = \bar{x} + \frac{t_{.90, n-1} s}{\sqrt{n}}$$

Where s is the sample standard deviation, \bar{x} is the sample mean, n is the number of samples analyzed for the analyte, and $t_{.90, n-1}$ is the 90th percentile value for a t distribution with $n-1$ degrees of freedom.

The results for the total metals sampling and analysis are summarized in Tables C-1, C-2, and C-3. Statistical calculations were performed on fitted, natural log-transformed values with treatment of less-than-detectable (LTD) values based upon methods discussed and evaluated by Gilliom and Helsel (1986). This methodology is in accordance with the guidance outlined in the QAPP.

Electrorefining Salts TRU Waste (IDC 411X)

Arsenic and selenium were not detected in any of the samples (100% LTD), and barium, cadmium, mercury, and silver were reported with only two detectable measurements each (75% LTD). Distributional assessments are not particularly meaningful with so few actual analyte concentrations and the majority of results reported at the instrument detection limit. UCL₉₀ values for these analytes were calculated based upon conservative estimates that the true values were log-normally distributed (as is commonly found with trace contamination levels) with the largest reported value representing the largest actual value, and all other observations reported as LTD each representing some maximum smaller value which graphically fit the distribution while not exceeding the reported instrument detection limit for that observation.

For chromium and lead, where sufficient detects were present to reliably indicate the probable shape of the underlying probability distribution, less-than-detectable values were graphically and mathematically "fitted" to the most likely distribution values at or below the reported detection limit by the "LR" method described in *Gilliom and Helsel (Estimation of Distributional Parameters for Censored Trace Level Water Quality Data*, Water Resources Research, Vol. 22, No. 2, p. 137, 1986). This normal scores regression method was used in fitting all "non-detects" in the log-transformed data, since none of the analytes with multiple "detects" exhibited near normality in the non-transformed observations.

Log-transformation of numbers near 1.0 yields transformed values near 0.0. Since the instrument detection limit for some analytes are close to 1.0 ppm (mg/Kg) and fitted log-transformed values for other analytes are often centered near 0.0 when concentrations are reported in ppm, estimates of the coefficient

Appendix C

of variation (CV) could potentially be highly inflated by dividing standard deviation by a mean value of approximately zero. To avoid this artificially inflated CV estimate and the resulting over estimation of required sample size, all reported concentrations were converted from parts-per-million (ppm or mg/Kg) to parts-per-billion (ppb or $\mu\text{g}/\text{Kg}$) prior to natural log-transformation.

Near-normality is critical to the validity of the statistical sample size determination and UCL_{90} calculations established by the QAPP. Where the number of detects were too few to reasonably fit the distribution, up to three of the non-detect values were incorporated as upper-bound (conservatively high) estimates for analyte concentrations and the regression on normal scores performed.

All fitted log-transformed analytes passed all mathematical tests for normality, including the Shapiro-Wilk and D'Agostino Omnibus tests which are considered uniformly most powerful in detecting departures from normality (Samuel Kotz & Norman L. Johnson, ed., **Encyclopedia of Statistical Sciences**, John Wiley & Sons, New York, 1982. Vol. 2, PP 315-324.).

Table C-1: Total Metals Summary Data for IDC 411X¹

Analyte	No. of Samples	No. of Samples above MDL	Mean ²	SD ²	UCL_{90} ²	Reg. Level ³
Arsenic	8	0	9.57	0.23	9.69	11.51
Barium	8	2	8.82	0.58	9.11	14.51
Cadmium	8	2	7.98	0.24	8.10	9.90
Chromium	8	6	10.85	1.23	11.47	11.51
Lead	8	5	9.81	1.13	10.38	11.51
Mercury	8	2	3.69	2.25	4.81	8.29
Selenium	8	0	8.38	0.23	8.50	9.90
Silver	8	2	8.32	0.72	8.68	11.51

1. Descriptive statistics (i.e., mean, standard deviation and UCL_{90}) and RTLs are reported as natural log-transformed values in accordance with Section 5.4.1 of the WIPP QAPP.
2. Prior to natural log-transformation, concentrations were converted from parts-per-million (mg/Kg) to parts-per-billion ($\mu\text{g}/\text{Kg}$) to avoid artificial inflation of required sample sizes.
3. Prior to natural log-transformation, the TC values in 40 CFR 261.24 were multiplied by 20 to express as total values as described in Section 3.3.1 of the QAPP, and then were converted from parts-per-million (mg/Kg) to parts-per-billion ($\mu\text{g}/\text{Kg}$).

Scrub Alloy Spent Dicesium Salt TRU Waste (IDC 433X)

Cadmium and mercury were not detected in any of the samples (100% LTD) and arsenic, barium, selenium, and silver were reported as detectable in only 1 or 2 of 13 samples each. Distributional assessments are not particularly meaningful with so few actual analyte concentrations and the preponderance of results reported at instrument detection limit. UCL_{90} values for these analytes were calculated based upon conservative estimates that the true values were log-normally distributed (as is commonly found with trace contamination levels) with the largest reported value representing the largest

Appendix C

actual value, and all other observations reported as LTD each representing some maximum smaller value which graphically fit the distribution while not exceeding the reported instrument detection limit for that observation.

For chromium and lead, where sufficient detects were present to reliably indicate the probable shape of the underlying probability distribution, LTD values were graphically and mathematically "fitted" to the most likely distribution values at or below the reported detection limit by the "LR" method described in *Gilliom and Helsel (Estimation of Distributional Parameters for Censored Trace Level Water Quality Data*, Water Resources Research, Vol. 22, No. 2, p. 137, 1986). This normal scores regression method was used in fitting all "non-detects" in the log-transformed data, since none of the analytes with multiple "detects" exhibited near normality in the non-transformed observations.

Log-transformation of numbers near 1.0 yields transformed values near 0.0. Since the instrument detection limit for some analytes are close to 1.0 ppm (mg/Kg) and fitted log-transformed values for other analytes are often centered near 0.0 when concentrations are reported in ppm, estimates of the coefficient of variation (CV) could potentially be highly inflated by dividing standard deviation by a mean value of approximately zero. To avoid this artificially inflated CV estimate and the resulting over estimation of required sample size, all reported concentrations were converted from parts-per-million (ppm or mg/Kg) to parts-per-billion (ppb or µg/Kg) prior to natural log-transformation.

Near-normality is critical to the validity of the statistical sample size determination and UCL_{90} calculations established by the QAPP. Where the number of detects were too few to reasonably fit the distribution, up to three of the non-detect values were incorporated as upper-bound (conservatively high) estimates for analyte concentrations and the regression on normal scores performed.

All fitted log-transformed analytes passed all mathematical tests for normality, including the Shapiro-Wilk and D'Agostino Omnibus tests which are considered uniformly most powerful in detecting departures from normality (Samuel Kotz & Norman L. Johnson, ed., *Encyclopedia of Statistical Sciences*, John Wiley & Sons, New York, 1982. (Vol. 2, PP 315-324.).

Table C-2: Total Metals Summary Data for IDC 433X¹

Analyte	No. of Samples	No. of Samples above MDL	Mean ²	SD ²	UCL ₉₀ ²	Reg. Level ³
Arsenic	13	1	9.65	0.26	9.75	11.51
Barium	13	2	9.01	0.30	9.12	14.51
Cadmium	13	0	7.79	0.29	7.90	9.90
Chromium	13	9	10.08	1.29	10.57	11.51
Lead	13	5	9.31	2.32	10.18	11.51
Mercury	13	0	4.57	0.025	4.58	8.29
Selenium	13	1	8.40	0.23	8.48	9.90
Silver	13	1	8.60	0.07	8.62	11.51

Appendix C

1. Descriptive statistics (i.e., mean, standard deviation and UCL₉₀) and RTLs are reported as natural log-transformed values in accordance with Section 5.4.1 of the WIPP QAPP.
2. Prior to natural log-transformation, concentrations were converted from parts-per-million (mg/Kg) to parts-per-billion (µg/Kg) to avoid artificial inflation of required sample sizes.
3. Prior to natural log-transformation, the TC values in 40 CFR 261.24 were multiplied by 20 to express as total values as described in Section 3.3.1 of the QAPP, and then were converted from parts-per-million (mg/Kg) to parts-per-billion (µg/Kg).

Direct Oxide Reduction Salt CaCl₂ Salt TRU Waste (IDC 454X)

Arsenic and barium were not detected in any of the samples (100% LTD), and cadmium, mercury, selenium and silver were reported with only one detectable measurement each (89% LTD). Distributional assessments are not particularly meaningful with so few actual analyte concentrations and the majority of results reported at the instrument detection limit. UCL₉₀ values for these analytes were calculated based upon conservative estimates that the true values were log-normally distributed (as is commonly found with trace contamination levels) with the largest reported value representing the largest actual value, and all other observations reported as LTD each representing some maximum smaller value which graphically fit the distribution while not exceeding the reported instrument detection limit for that observation.

Near-normality is critical to the validity of the statistical sample size determination and UCL₉₀ calculations established by the QAPP. Where the number of detects were too few to reasonably fit the distribution, up to three of the non-detect values were incorporated as upper-bound (conservatively high) estimates for analyte concentrations and the regression on normal scores performed.

For chromium and lead, where sufficient detects were present to reliably indicate the probable shape of the underlying probability distribution, less-than-detectable values were graphically and mathematically "fitted" to the most likely distribution values at or below the reported detection limit. Since the natural log-transformation process can yield negative values (representing concentrations less than 1.0 mg/kg), the fitted natural log-normal distribution for cadmium yielded a transformed mean close to zero. As a result, the coefficient of variation is artificially inflated and overestimates the required sample size. This difficulty was avoided by a simple linear transformation of the raw data prior to natural log-transformation. For cadmium, the linear transformation used consisted of multiplying each observation and the associated RTL by 20 prior to further statistical analysis. The fitted natural log-transformation was used because neither the raw, non-transformed data nor the unfitted natural log-transformed data exhibited normality in distribution while the fitted natural log-transformed analytes did pass all applied mathematical tests for normality, including the Shapiro-Wilk and D'Agostino Omnibus tests.

Table C-3: Total Metals Summary Data for IDC 454X¹

Analyte	No. of Samples	No. of Samples above MDL	Mean	SD	UCL ₉₀	Reg. Level ²
Arsenic	9	0	2.91	0.05	2.94	4.61
Barium	9	0	2.22	0.05	2.24	7.60
Cadmium ³	9	1	2.55	1.87	3.42	5.99
Chromium	9	9	3.71	1.18	4.25	4.61
Lead	9	4	2.81	1.27	3.40	4.61

Appendix C

Analyte	No. of Samples	No. of Samples above MDL	Mean	SD	UCL ₉₀	Reg. Level ²
Mercury	9	1	-4.39	2.79	-3.09	1.39
Selenium	9	1	1.00	0.78	1.36	3.00
Silver	9	1	1.00	0.81	1.38	4.61

1. Descriptive statistics (i.e., mean, standard deviation and UCL₉₀) and RTLs are reported as natural log-transformed values in accordance with Section 5.4.1 of the WIPP QAPP.
2. Prior to natural log-transformation, the TC values in 40 CFR 261.24 were multiplied by 20 to express as total values as described in Section 3.3.1 of the QAPP (the total values were not converted to parts-per-billion, as is the case with IDC 411X).
3. Natural log-transformation of each cadmium observation and the associated RTL includes a linear multiplicative transformation by a factor of 20 prior to log-transformation to avoid artificial inflation of the required sample size.

Conclusion of Total Metals Results

The total metal results from sampling and analysis of IDCs 411X, 433X, and 454X confirm that sufficient samples were taken to characterize these IDCs and that no metal analyte exhibits a toxicity characteristic per the applicable QAPP criteria.