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Department of Energy
Carlsbad Area Office
P. O. Box 3090
Carlsbad, New Mexico 88221
June 22, 1999

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

Dear Mr. Lewis:

Please find enclosed the Rocky Flats Environmental Technology Sites' (RFETS) *Non-Mixed Waste Determination for TRU Stabilized Pyrochemical Salts Profile No. RF005.01* (GAO-029-99) dated June 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 a non-mixed (non-hazardous) TRU waste at the WIPP.

The references contained in section 6 of the non-mixed waste determination that have not been previously provided are enclosed. A specific listing of the documentation is also enclosed.

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

Sincerely,

E. Kent Hunter, Assistant Manager
Office of Waste Disposal Operations

Enclosure

cc: w/o enclosures:
Peter Maggiore, NMED
Richard Mertz, NMED
Susan McMichael, NMED
Dan Ruge, DOE –HQ
Ines Triay, CAO

Refer to: CAO/OWDO:MRB 99-0948 / UFC 5822

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List of Enclosures for Letter 99-0948 / UFC 5822

The following is a list of the references that are enclosed. The list is reproduced from GAO-29-99 June 1999, section 6.0 References.

1. RFETS TRU Waste Acceptable Knowledge Supplemental Information. RF/RMRS-97-018, Revision 4, May 10, 1999 - Enclosed.

2. Acceptable Knowledge TRU/TRM Waste Stream Summaries, RMRS-WIPP-98-100, Revision 3 May 20, 1999. The applicable summary is included as Appendix B of the GAO-029-99.

3. *Transuranic Waste Characterization Quality Assurance Program Plan* – Interim Change, CAO 94-1010, November 15, 1996. The Quality Assurance Program Plan with interim changes is currently available at NMED.

4. Backlog Waste Reassessment Baseline Book, Waste Form 34 Pyrochemical Salts, October 26, 1998 – Enclosed

5. *Waste Acceptance Criteria for the Waste Isolation Pilot Plant*, DOE/WIPP-069, Revision 5, April 1996. – Currently available at NMED.

6. *Test Methods for Evaluating Solid Waste*, EPA SW-846, Third Edition, November 1986 – EPA documentation currently available at NMED.

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, 1998 – Enclosed

8. Environmental Protection Agency Policy Compendium, 9443.1987(17), August 18, 1987. EPA documentation currently available at NMED

9. E. K. Hunter letter to J. Legare, *Residue TRU Waste Characterization Methods Evaluation,* CAO:NTP:RAS 98-0617-UFC-5822, February 12, 1998. – Enclosed

10. Salt Residue Stabilization, Building 707 Process Control/Qualification Plan. RS-020-006, Revision 1. April 14, 1999 – Enclosed

11. Salt Residue Stabilization/Repack, Building 707.4-W84-RS-0114, Revision 3. June 14, 1999. – Enclosed

12. Rocky Flats Environmental Technology Site Transuranic (TRU/TRM) Waste Sampling Plan. 95-WP/SAP-001, Revision 3. February 26, 1999. - Enclosed

13. E. K. Hunter letter to J. A. Legare, *RFFO Exemption Request for Preserving Samples of Salt Waste,* CAO:QA:RAS 98-0958 UFC 5822, July 14, 1998. – Enclosed

14. Salt Residue Stabilization Building 707 Qualification Report. RS-020-017, Revision 1, April 8, 1999 – Enclosed

15. RFETS Interoffice Correspondence from T. R. Gatliffe 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. – Enclosed

GAO-029-99

June 1999

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

G. A. O'Leary
E. L. D'Amico
A. C. Kercher
J. L. Harrison

Reviewed For Classification/UCNI
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Date 18 JUN 99 (UNY)

GAO-029-99

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Published June 1999

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Prepared for the
U.S. Department of Energy

GAO-029-99
June 1999

Rocky Flats Environmental Technology Site

Non-Mixed Waste Determination for

TRU Stabilized Pyrochemical Salts – Profile No. RF005.01

G. A. O'Leary, Manager, RMRS TRU/TRM Waste Projects — Date 6/18/99

A. D. Rodgers, Div. Mgr., Kaiser Hill Waste and Remediation Operations — Date 6/18/99

J. A. Legare, Asst. Manager, DOE-RFFO Environmental Compliance — Date 6/18/99

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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
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 Code (IDC) 454X[1] is 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 $CaCl_2$ Salt TRU Waste (IDC 454X) was selected because of the extensive knowledge of this waste as non-mixed waste. 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 (IDC 454X):

- Acceptable knowledge (AK);
- Visual examination; and
- Waste solid sampling and analysis of randomly selected containers.

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

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) 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), 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 [Ref. 2, 4, 10]. Descriptions of the historical pyrochemical processes as well as the current stabilization process are provided below.

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 historical 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 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].

To address concerns expressed in the DNFSB recommendation 94-1 concerning potentially reactive/pyrophoric species in salts, RFETS initiated a project for stabilization 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 and 10]. 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 only one of the four categories of stabilized salts produced by the pyro-oxidation process. This category is assigned IDC 454X, DOR Salt $CaCl_2$ Salt TRU Waste, and is

comprised of feed IDCs 365, 414, 404, 412, and 416 as described above [Ref. 10]. The process flow diagram for salt stabilization is provided in Appendix A.

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 (IDC 454X) [Ref. 2 and 4]. Solid waste sampling and analysis was 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 report specifically addresses the AK documentation used to characterize stabilized pyrochemical salts (IDC 454X) as a non-mixed waste. Compilation of AK and the conclusions reached following review of AK 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. 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, including EPA personnel, participated in one or both audits. The overall results from the audits were favorable and certification status for the stabilized salts was received [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) 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 demonstrates 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 and 4]. Total metals analysis conducted on the stabilized salts (IDC 454X) demonstrates 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 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 NQA-1 waste records center.

3.4.1 Physical Characterization

For the purposes of this document, "physical" characterization includes visual examination only. DOR Salt $CaCl_2$ Salt TRU Waste (IDC 454X) is 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 WAPP [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. DOR Salt $CaCl_2$ Salt TRU Waste (IDC 454X) is an example 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 DOR Salt $CaCl_2$ 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 DOR Salt $CaCl_2$ Salt TRU Waste (IDC 454X) does 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 IDC 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 IDC 454X using this method was 5. However, to ensure a sufficient number of samples were collected, this number was increased to 10 samples.

An estimate of the output IDC population is used to determine the random sample numbers. Initially, the estimate for IDC 454X was 900 cans; however, this proved to be an overestimation. 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]. Because the last random number was 828, and the number of drums of IDC 454X to be generated was less than 828, only 9 of the 10 samples were collected. Based on the results from the non-transformed data, the minimum required sample size is 8 samples, and based on 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 is sufficient [Ref. 15].

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 three random samples collected for the salt stabilization process are 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 based on a 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 IDC 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 DOR Salt $CaCl_2$ Salt TRU Waste (IDC 454X) is not a listed waste. 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 and 4].

4.2 Characteristic Waste

4.2.1 Toxicity

Based on AK and confirmed by sampling and analysis, it has been determined that the drums containing IDC 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 in an extract generated using the TCLP method. Results are then compared with levels that the EPA has identified as hazardous to projected receptors.

AK documentation demonstrates IDC 454X does not exhibit the characteristic of toxicity for metal or organic compounds [Ref. 2 and 4]. As discussed in Section 3.4.2, total metals analysis was performed on representative samples of IDC 454X salt 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

Based on examination and knowledge of the waste, it has been determined that IDC 454X waste does 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 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 testing, visual examination, and knowledge of the waste, it has been determined that IDC 454X waste does 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 then 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

Based on testing and knowledge of the waste and the generation process, it has been determined that IDC 454X waste does 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 454X waste is stable and does not undergo violent chemical change. The waste does not contain cyanides or sulfides and is not capable of detonation or explosive reaction. The waste does 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 (IDC 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

1. RFETS TRU Waste Acceptable Knowledge Supplemental Information. RF/RMRS-97-018, Revision 4. May 10, 1999.
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.
6. *Test Methods for Evaluating Solid Waste*, EPA SW-846, Third Edition, November 1986.
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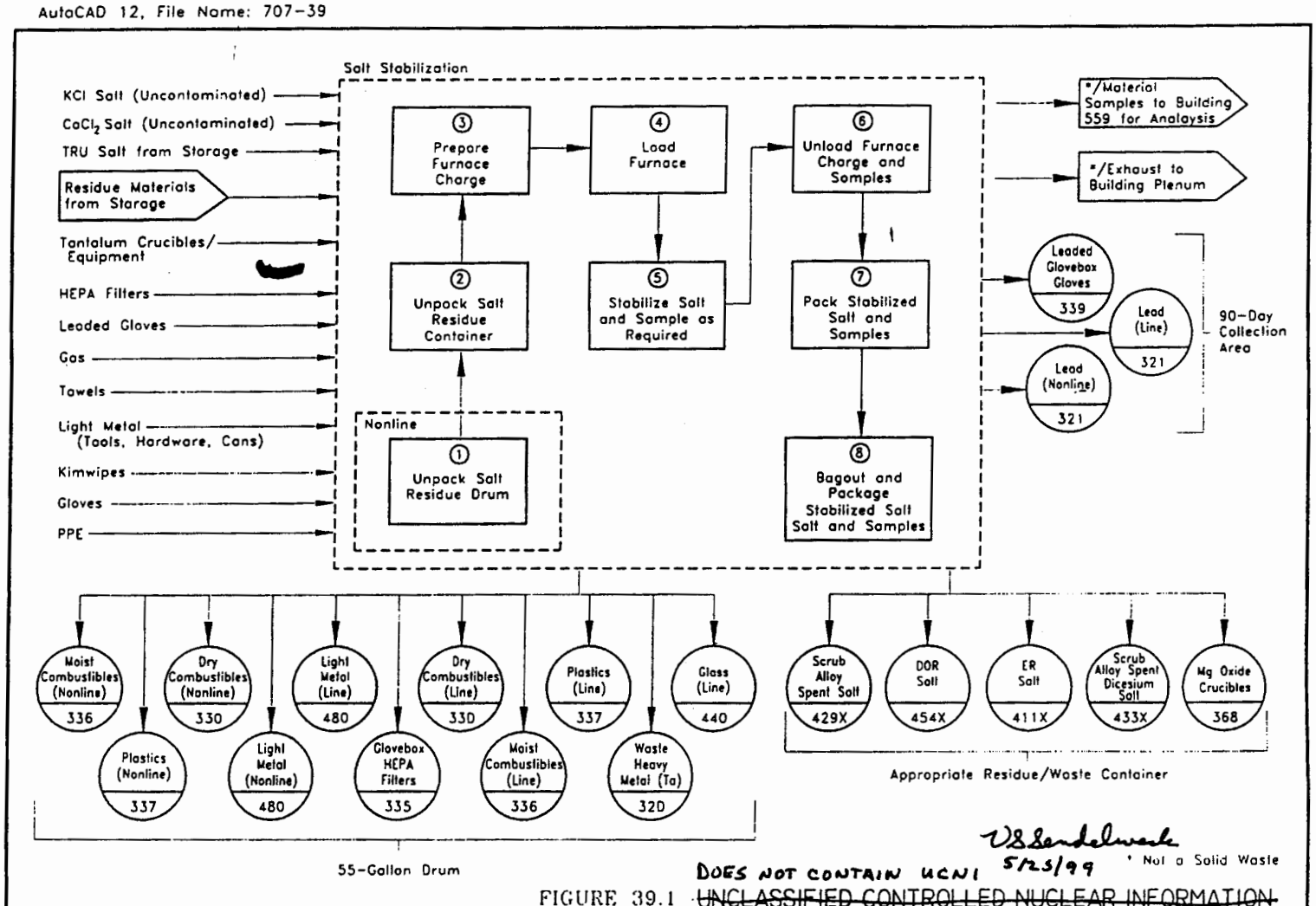
8. Environmental Protection Agency Policy Compendium, 9443.1987(17), August 18, 1987.
9. E. K. Hunter letter to J. Legare, *Residue TRU Waste Characterization Methods Evaluation*, CAO:NTP:RAS 98-0617-UFC-5822, February 12, 1998.
10. Salt Residue Stabilization, Building 707 Process Control/Qualification 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.
12. Rocky Flats Environmental Technology Site Transuranic (TRU/TRM) Waste Sampling Plan. 95-WP/SAP-001, Revision 3. February 26, 1999.
13. E. K. Hunter letter to J. A. Legare, *RFFO Exemption Request for Preserving Samples of Salt Waste*, CAO:QA:RAS 98-0958 UFC 5822, July 14, 1998.
14. Salt Residue Stabilization Building 707 Qualification Report. RS-020-017, Revision 1. April 8, 1999.
15. RFETS Interoffice Correspondence from T. R. Gatliffe 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.

Appendix A

Pyrochemical Salt Process Flow Diagrams

- **Building 707, Process 39, Figure 39.1 – Salt Stabilization**
- **Building 779, Process 3, Figure 3.1 – Direct Oxide Reduction**

Appendix A



DOES NOT CONTAIN UCNI V.S. Sendelweck 5/25/99

~~UNCLASSIFIED CONTROLLED NUCLEAR INFORMATION~~

FIGURE 39.1

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. Stadt 07/03/97 Checked: J. Schoen 02/26/99

707-V6.0 Process 39-4 02/26/99

Appendix A

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

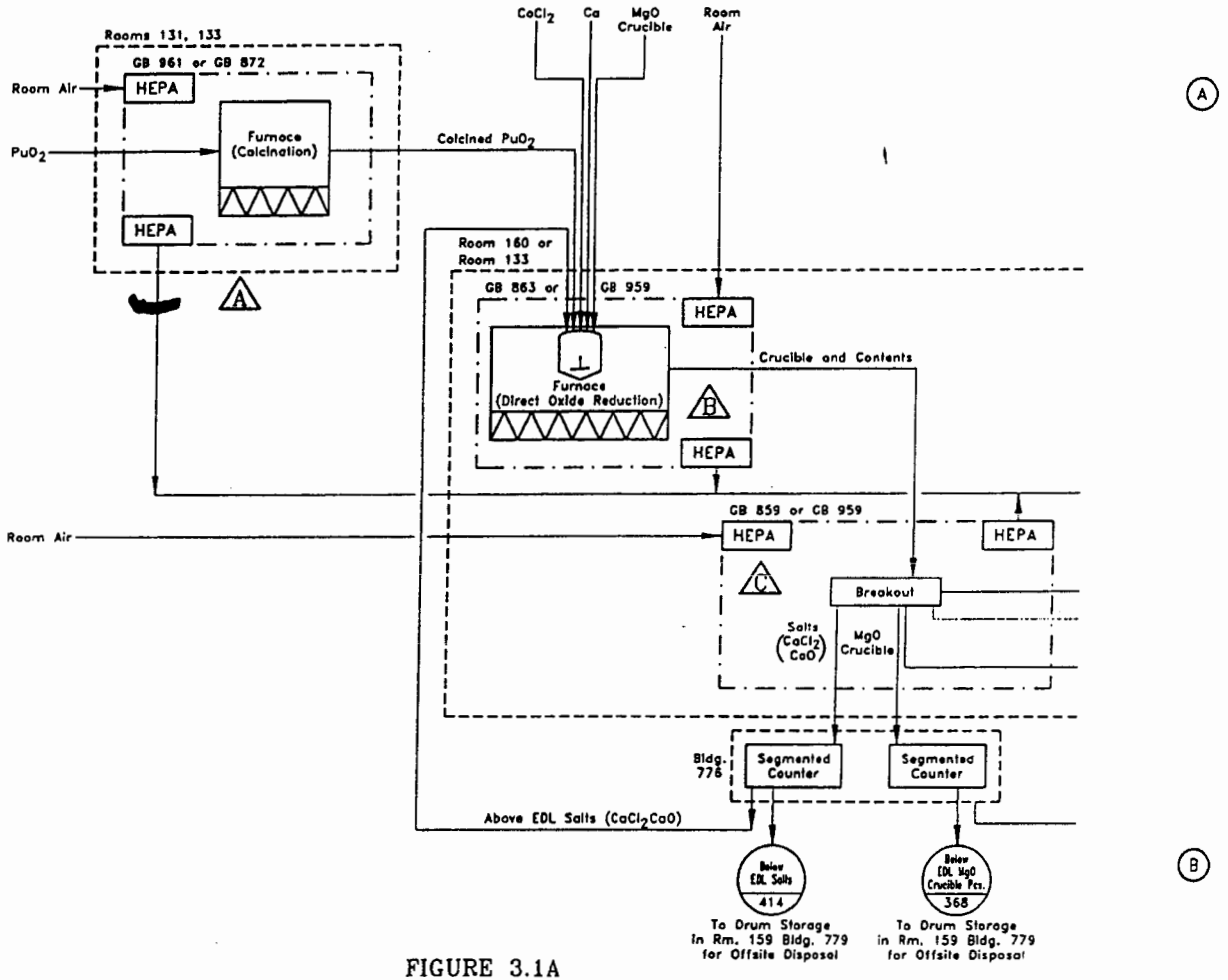


FIGURE 3.1A

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

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): 651 55-Gallon Drums [8, 11]

Generation Dates (Current): January 1998 – April 1999 [8, 11]

Waste Stream Volume (Projected): 749 55-Gallon Drums [9, 11]

Generation Dates (Projected): May 1999 – July 2000 [9, 11]

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

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]

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]

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%

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

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(IDC 364), 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]

NOTE: The feed IDCs for IDC 411X have changed and also include IDCs 413 and 426. This AK waste stream summary will be updated to add these feed IDCs to IDC 411X and remove them from IDC 454X.

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), and MSE Salt Packaged for LANL (IDC 418). 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]

NOTE: The feed IDCs for IDC 429X have changed and also includes IDC 415. This AK waste stream summary will be updated to add this feed IDC to IDC 429X and remove it from IDC 454X.

IDC 433X, Scrub Alloy Spent Dicesium Salt: Spent salt from the MSE scrub alloy process that used dicesium salt. Other salts which become IDC 433 for disposal include MSE spent dicesium salt (IDC 427), free calcium containing spent salt (IDC 434), and cerium/calcium spent salt (IDC 435). 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]

NOTE: The feed IDCs for IDC 433X have changed and also could include IDCs 365, 404, 412, 414, and 416. This AK waste stream summary will be updated to add these feed IDCs to IDC 433X. These feed IDCs are still included in IDC 454X.

IDC 454X, DOR Salt-Oxidized Calcium: Spent salt from the direct oxide reduction (DOR) process. Other salts which become IDC 454 for disposal include salt from bad DOR run (IDC 365), MSE salt, Ca, Zn, K (IDC 404), Gibson salt (IDC 412), impure salt from cell clean-out (IDC 413), DOR salt-unoxidized calcium (IDC 414), Pu chloride mixed salt (IDC 415), Zn-Mg alloy metal (IDC 416), and reburned IDC 413 (IDC 426). 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]

NOTE: The feed IDCs for IDC 454X have changed and do not include IDCs 413, 415, or 426. This AK waste stream summary will be updated to remove these feed IDCs from IDC 454X and add IDCs 413 and 426 to IDC 411X and IDC 415 to 429X.

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

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- Electrorefining
- Direct Oxide Reduction
- 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, EU, Np-237

Key:

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

References

1. DOE 1996. TRUPACT-II Content Codes (TRUCON), Revision 10. DOE/WIPP 89-004.
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.

Appendix B

4. RFETS 1998. 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.

Appendix C

Total Metals Data Summary

Appendix C

Total Metals Data Summary

IDC 454X Waste (DOR Salt $CaCl_2$ Salt TRU Waste)

Nine cans of stabilized pyrochemical salts (IDC 454X) were randomly selected for sampling and analysis for total metals as required in the QAPP. This waste stream was 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, is the sample mean, n is the number of samples analyzed for the analyte, and 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 the accompanying table. 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.

Arsenic and barium were reported as 100% LTD and cadmium, mercury, selenium and silver were reported with only one detectable measurement each. Distributional assessments are not particularly meaningful with so few actual analyte concentrations and the preponderance of results reported at the 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 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. 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.

Appendix C

Conclusion of Total Metals Results

The total metal results from the nine samples confirm that sufficient samples were taken to characterize the waste stream and that no metal analyte exhibits a toxicity characteristic per the applicable QAPP criteria.

Table D-1: Total Metals Summary Data[1]

Analyte	No. of Samples	No. of Samples above MDL	Mean (mg/kg)	SD (mg/kg)	UCL_{90} (mg/kg)	Reg. Level (mg/kg)
Arsenic	9	0	2.91	0.05	2.94	4.61
Barium	9	0	2.22	0.05	2.24	7.60
Cadmium	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
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] Statistical calculations (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.