LA-UR- 99-864

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Title:	Preparation of the First Shipment of Transuranic Waste by the Los Alamos National Laboratory: A Rest Stop on the Road to WIPP RECEIVED MAY 0 3 1999 OSTI
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Submitted to:	To be presented at the WM '99 Conference in Tucson, AZ. Febraury 28 - March 3, 1999



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Preparation of the First Shipments of Transuranic Waste by the Los Alamos National Laboratory: A Rest Stop on the Road to the WIPP

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ABSTRACT

The Los Alamos National Laboratory (LANL) achieved a national milestone on the road to shipping transuranic (TRU) waste to the Waste Isolation Pilot Plant (WIPP) when it received certification authority on September 12, 1997. Since that time, LANL has been characterizing a non-mixed TRU waste stream and preparing shipments of this TRU waste for disposal in the WIPP.

The paper describes the TRU waste identified as waste stream TA-55-43 Lot No. 01 from LANL Technical Area-55 and the process used to determine that it does not contain hazardous waste regulated by the Resource Conservation Recovery Act (RCRA) or the New Mexico Hazardous Waste Act (HWA). The non-mixed determination is based on the acceptable knowledge (AK) characterization process, which clearly shows that the waste does not exhibit any RCRA characteristics nor meet any RCRA listing descriptions.

LANL has certified TRU waste from waste stream TA-55-43 Lot No. 01 and is prepared to certify additional quantities of TRU waste from other non-mixed TRU waste streams. Assembly and preparation of AK on the processes that generated TRU waste is recognized as a necessary part of the process for having waste ready for shipment to the WIPP.

INTRODUCTION

LANL characterized waste stream TA-55-43 by assembling an AK Summary Report, conducting headspace gas analysis and sampling (HGAS), and completing real-time radiography (RTR) examination of 36 parent drums in Lot No. 01. The U.S. Department of Energy (DOE) Carlsbad Area Office (CAO) approved the Waste Stream Profile Form

submitted by LANL, allowing LANL to proceed with certification of the waste. At that point, cooperative negotiations with New Mexico Environment Department (NMED) were started to determine whether NMED concurred with the non-mixed designation of the waste stream. This period of negotiation provided a pause in waste certification activities, faciously referred to as a "rest stop" on the road to the WIPP.

During this period, LANL was first asked to provide more detail of the characterization and certification process. Second, LANL was asked to prepare a crosswalk showing how the acceptable knowledge requirements in the CAO Transuranic Waste Characterization Quality Assurance Project Plan (QAPP) compare to those in the draft WIPP RCRA permit(1). Third, LANL was asked numerous questions as part of a two-week audit of its program by NMED staff. These questions led LANL to write and issue a Waste Determination Report(2), describing more fully the waste stream as it relates to the RCRA requirements. Fourth, LANL was asked to conduct a confirmatory sampling and analysis campaign for NMED (to be discussed in a related paper by Stan Kosiewicz). All of these factors led to a decision by the NMED on December 2, 1998 that waste stream TA-55-43, Lot No. 01 was non-mixed(3).

PROCESS FOR CHARACTERIZATION AND CERTIFICATION OF A WASTE STREAM

Since this waste was the first waste stream certified for disposal at the WIPP, communicating and familiarizing NMED with the processes used for characterization and verification of AK was important. LANL prepared a flow diagram tying together the process of characterizing and certifying waste stream TA-55-43, Lot No. 01. This process flow included the references to records and other documents that LANL used during the characterization and certification steps. This reference list allowed NMED to more easily read through the large stack of records that support characterization and verification of the contents of the waste stream. See Figure 1 for the process flow.

ACCEPTABLE KNOWLEDGE AVAILABLE IN THE SUMMARY REPORT

LANL prepared an AK Summary Report for Combustible /Noncombustible, Metallic, and HEPA Filter Waste Resulting from ²³⁸Pu Fabrication Activities (4), which addressed all of the AK requirements contained in the CAO Transuranic Waste Characterization Quality Assurance Program Plan (QAPP). The report covers AK information for five waste streams generated at TA-55 during operations to fabricate various heat sources using feedstock ²³⁸Pu supplied by the Savannah River Site (SRS). The ²³⁸Pu feedstock itself does not contain quantities of RCRA-regulated constituents above regulatory threshold limits, as known from process knowledge at SRS and as confirmed by chemical analysis. No RCRA-regulated chemicals were used during ²³⁸Pu fabrication activities at TA-55, and all ²³⁸Pu activities were physically separated from other plutonium processing activities. Most of the waste generated from the ²³⁸Pu fabrication activities is thus non-mixed waste.

The potential existed, however, for the presence of toxicity characteristic compounds due to off-gassing and the decomposition of the waste itself. In TRU wastes, the decomposition process is accelerated by radiolysis, the reaction of energetic particles produced by the decay of transuranic elements with the material in the waste. For example, radiolysis leads to the well-known generation of hydrogen in TRU wastes. Experimental studies have demonstrated that radiolysis also can lead to the generation of several organic compounds through decomposition of waste materials, particularly polyvinyl chloride plastic(5). The breakdown of off-gas products in the gas phase through radioloysis is also possible. Many different organic compounds have been observed to form through radiolytic decomposition of the original waste materials, such as acetone detected as the compound produced in highest concentration from all waste material tested(5). Compounds formed through radiolytic decomposition would not be noted in the process knowledge for the waste generation process.

ACCEPTABLE KNOWLEDGE CROSSWALK

Surprisingly, the extensive requirements in the QAPP for the AK Summary Report on combustible/noncombustible, metallic, and HEPA filter waste resulting from ²³⁸Pu fabrication activities (covering waste streams TA-55-43, TA-55-44, TA-55-45, TA-55-46, and TA-55-47) were not sufficient for the auditors from the NMED to declare the waste non-mixed. LANL was asked to prepare a matrix detailing the AK requirements in the NMED draft WIPP RCRA permit as well as those in the QAPP. Using this approach, LANL was able to show how the requirements for AK which concerned NMED were met through the CAO QAPP (see Figure 2 for an example of a couple of pages of that crosswalk).

AUDIT QUESTIONS

NMED sent two staff members to LANL to conduct an independent audit of the LANL TRU Waste Certification Program. The audit produced a list of questions to which LANL was asked to respond. The types of questions were twofold: (1) technical questions on the content of the waste and request for more information regarding the waste stream, and (2) specific questions on possible discrepancies in records and documents.

Some of the questions regarding the waste stream were:

- Please identify and provide information on the generation processes for the paper waste, rags and scrap metal.
- Please identify the composition of the metal valves, tools, cans, motors, and pumps. Also, please describe the "miscellaneous similar items."
- What is the composition of the graphite crucibles, is it just graphite or are there other compounds used to fabricate them? Please provide information.
- Explain how visual inspection can identify all pyrophorics?

- Table III-3 indicates high levels of Ca in the feedstock; what is the final physical state of this Ca after the feedstock is processed at TA-55. Is it calcium metal? Is it pyrophoric?
- Please identify the wastes generated from the maintenance and repair of equipment inside and outside of the glovebox, but in the same room. Please be specific to the maintenance or repair process, location, equipment, a list of the chemical used, frequency of the maintenance or repair and management of wastes generated.
- Rags generated during the cladding and decontamination stage or the heat source fabrication process and the maintenance and repair operations are also of concern. The rags in the fabrication process are of concern due to the possibility of heavy metals accumulation during the decontamination step. Other rags used during maintenance are of concern because of the lack of information on the maintenance performed. Why have no analyses been performed on these rags?

LANL responded to all questions and then prepared a document to supplement the AK Summary Report, called the Waste Determination Report(2).

WASTE DETERMINATION REPORT

The Waste Determination Report(2) was written to provide supplemental information on this first waste stream slated for characterization and certification for disposal in the WIPP. This publication showed how LANL determined that the waste did not contain hazardous waste regulated by RCRA or HWA. For a waste to be hazardous under these acts, it must be specifically listed as hazardous or exhibit a hazardous characteristic as specified in Title 40 of the Code of Federal Regulations (40 CFR) Part 261.

For this reason the Waste Determination Report was organized to discuss the (1) process description, (2) waste stream description, (3) characterization process, and (4) detailed RCRA analysis.

Process Description

Supplemental information in the process description included stating that all processing was conducted in a series of gloveboxes that were physically separated from other TA-55 operations. Also, this waste resulted not only from general processing activities but also from maintenance activities.

The additional information in this section detailed material types of containers – for example, stainless-steel transport container, platinum boat, aluminum-oxide tube, graphite-element heating furnace, stainless-steel jar, T-111 alloy (90% tantalum, 8% tungsten, 2% hafnium) container.

This section also detailed the use of rags. For example, the clad heat source was decontaminated by placing it in an acid bath consisting of mixtures of nitric and hydrofluoric acid in water. Each time a heat source was removed from the bath, it was placed on a rag that was damp with water. The rag was rubbed over the cladding to remove any plutonium oxide on the external surface of the heat sources. The heat of the source caused the acid solution and water in the rag to evaporate rapidly.

Waste Stream Description

The waste in Lot No. 01 is debris resulting from cleaning, repairs, and normal every-day operations during the fabrication of the heat sources and fuel recovery. Supplemental information provided on the waste stream description included a full listing of the waste items and a detailed description of the maintenance activities that generate much of that waste. The waste categories were each discussed paragraph by paragraph.

Characterization Process – Hazardous Waste Determination

A waste must exhibit a hazardous characteristic or be specifically listed as hazardous to be deemed hazardous waste under RCRA. An important point is that the mere presence of particular constituents in a waste does not mean that the waste is hazardous. A waste may be hazardous if it possesses a certain hazardous quality defined in the regulation regardless of constituent concentrations (e.g., an aqueous waste with a pH of 1). A waste may be hazardous if it contains specific hazardous constituents at high enough concentrations and mobility as identified in the toxicity characteristic, or a waste may be hazardous if the source of generation meets a listing description as provided in the regulations, regardless of constituent concentrations. Therefore, it is a characteristic, a concentration of constituent, or a source of a constituent that causes a waste to be hazardous, not the mere presence of a constituent.

Acceptable Knowledge – Knowledge of the Process

Feedstock – The process feedstock was plutonium oxide that originated from SRS. Savannah River provided sufficient information to ensure that no material used to manufacture the feedstock would produce a Listed waste. Savannah River also provided information that indicated that the feedstock did not have any hazardous characteristic (i.e., it was not ignitable, corrosive, or reactive and it did not contain constituents at levels capable of producing a toxicity characteristic waste).

Concerning metals that have the potential to produce a toxicity characteristic, SRS conducted totals analysis of the feedstock for cadmium, lead, and chromium. Typical results were 10 parts per million (ppm) for cadmium and lead and 100 ppm for chromium. The feedstock was not analyzed for arsenic, mercury, and selenium because the metals and their oxides vaporize at or below 685°C and the feedstock purification process reached 740°C.

Waste Management Practices – The high level of process knowledge is substantiated by the QA practices, conduct of operations, and training provided to generators of waste in Lot No. 01 and the generator's waste characterization practices. Activities at TA-55 must conform to a QA Program that identifies the quality of data necessary to meet the specific data quality objectives associated with the CAO TRU Waste Characterization Program. TA-55 waste management activities are described and applied to the TRU Waste Certification Program through a TRU Waste Interface Document(6), which details the procedures used by TA-55 personnel, including the packaging procedure and the procedures detailing how the waste items are recorded. The Certification Program assesses the activities of the TA-55 personnel to ensure that these activities are being accomplished in accordance with the approved procedures and that all the necessary information is being recorded on the proper forms. Corrective action is taken when conditions adverse to quality are identified. The cause of any adverse condition that affects compliance is identified promptly and corrective action taken to preclude its recurrence.

Relevant Data and Supporting Information

Real-Time Radiography – RTR was performed on every drum in Lot No. 01. The composition of many materials inside the drum can be determined by varying the x-ray energy. For example, less-dense materials such as paper and plastic are clearly distinguishable from metals. Lead can be picked out easily from other metals because of its high density. A liquid is detected by movement of the liquid surface as the drum is rotated. The outlines of items can be seen, which allows RTR to be used to identify items not allowed in waste sent to the WIPP, such as compressed-gas cylinders and sealed containers greater than 4 liters. RTR support the determination that the waste is not hazardous because the process verified that no lead items are present. Use of this technique, in conjunction with repackaging, supports the determination that no corrosive waste is present by demonstrating that insufficient liquids are present.

Visual Examination – A visual examination was performed on five of the original 36 drums to verify the RTR interpretations as well as the volumes estimated for each waste type. Visual examination supports the determination that the waste is not hazardous by verifying and substantiating the identification and inventorying of waste items by the generators. This supports the conclusions based on process knowledge.

Headspace Gas Analysis – Before being repackaged, the drums underwent headspace gas analysis to determine the identity and quantity of volatile organic compounds (VOCs), hydrogen, and methane. Results of this analysis showed that some of the drums contained small detectable quantities of the following organic compounds: methanol, acetone, methylene chloride, methyl ethyl ketone, cyclohexane, benzene, toluene, and methane. Because no solvents were used in the process, the presence of these constituents are attributed to (1) off-gassing of constituents in the tape and plastic bags used to package waste items and (2) radiolysis of the plastic, rubber, tape, Tygon, and similar items in the waste. Radioloytic decomposition of waste by alpha particles produced minor quantities of VOCs. Calculations indicated that the average waste stream concentrations were below the program-required quantitation limit at the 90% and 95% upper confidence limit. Pursuant to NMED criteria, these constituents are not required to be identified as listed hazardous wastes.

Repackaging – During the repackaging process, all the drums from Lot No. 01 are opened, and the waste items are removed. The labels on each item are recorded and traced to generator records, and to date, all generator records have correlated with items found in the containers. During repackaging, all waste generated from filtering (1) acid solutions used in the decontamination step and (2) solutions used in analyzing the feedstock and the seasoned fuel pellets are removed. Also, the containerized ashes from thermal decomposition of rags and waste from a plutonium-pellet dissolution process are removed. The repackaging process is described in a related paper by David Yeamans(7).

Repackaging confirms the determination that the waste in Lot No. 01 is not hazardous by verifying and substantiating the identification and inventory of the items by the generators.

Gamma Spectroscopy – A gamma spectroscopy analysis is performed on all repackaged drums holding waste in Lot No. 01. The main purpose of this analysis is to verify that the isotopic composition of the waste matches the known isotopic composition of ²³⁸Pu heat source material. The analysis also identifies and reports the isotopes of interest to the WIPP. Because the heat source development and fuel recovery processes involved only ²³⁸Pu, this information supports knowledge of process characterization. The knowledge that only ²³⁸Pu is present allows calculation of metal constituent concentrations to establish that the concentrations are below toxicity characteristic levels.

Neutron Radioassay – A passive/active neutron radioassay is performed on every repackaged drum to determine the waste's ²³⁸Pu content. This assay measures both neutron emissions emitted by the radionuclides (passive) and neutron emissions that result from exposure to a neutron beam (active). The resulting information is used to measure the total alpha activity, total decay heat, and fissile gram equivalent to report to the WIPP. This activity supports the determination that no hazardous waste is present in Lot No. 01 by establishing the amount of the ²³⁸Pu present. Based on the amount of ²³⁸Pu in the waste and the metal constituent levels known to correlate to the plutonium levels, LANL confirmed that metal constituent levels are below the concentrations that could cause the waste to exhibit the toxicity characteristic, even if all the RCRA-regulated metals were to leach from the plutonium. Knowing the exact amount of ²³⁸Pu present allows accurate calculations of the metal constituents in the waste.

Confirmation Activities – WIPP is not accepting mixed waste for disposal until it receives its RCRA Part B Permit. WIPP has examined information provided on waste in Lot No. 01 and concurred with LANL's determination that the waste is not mixed. WIPP's analysis of the characterization of the waste is based on review of the AK Summary Report, supporting documentation, and headspace gas analysis. The

determination that no characteristics or listings apply to the waste is supported by approval of the waste stream profile form.

The original TRUCON codes assigned to the drums of Lot No. 01 waste indicated legacy waste. Because the waste was repackaged, a new TRUCON code has been assigned to the repackaged waste, which is in accordance with the TRUPACT-II Safety Analysis Report(8) that governs shipments in the TRUPACT shipping container. The repackaged drums are loaded into standard waste boxes that meet the Nuclear Regulatory Commission approved TRUCON code LA 125A. Characterization and certification information is entered into the WIPP Waste Information System (WWIS) for approval by the DOE CAO prior to development of a payload assembly. Once the containers are certified and approved by the WIPP, the payload assemblies can be prepared and entered into WWIS for approval.

Detailed RCRA Analysis - Listed Waste and Characteristic Waste

As part of the detailed RCRA analysis, Listed waste was discussed as well as characteristic waste. The discussion of characteristic waste included toxicity, corrosivity, ignitability, and reactivity.

Based on process knowledge, it is known that no listings apply to the waste contained in the drums. The F Listing is not applicable because no solvents fitting a listing description were used in the process. No constituents were detected in headspace analysis at or above the program-required quantitation limit.

Toxicity – To exhibit the toxicity characteristic, waste levels of certain metals or organics must meet or exceed certain levels in an extract generated using a test method known as the Toxicity Characteristic Leaching Procedure (TCLP). In lieu of the TCLP, the total-metals analysis can be used to estimate a worst-case situation. In contrast to the TCLP, which leaches only the amount of contaminants available to be released from the particular matrix, the total metals analysis assumes that all of the metals or organics present in the waste would leach from it. If the concentration calculated by assuming leaching of all the prescribed contaminants in the waste indicated that insufficient amounts of contaminants exist to exceed the toxicity limits, then one can safely conclude that if only the mobile fraction contained in the waste were leached, it could never meet the toxicity criteria.

With regard to the metals that may exist in this waste stream, calculations using data from the feed material, the intermediate material, and the waste have been performed that indicate no metals are present above toxicity levels established by RCRA. Although several of these metals are present as impurities in the feed material, chromium has been determined to be the metals of greatest significance because of its relative concentration with regard to the regulatory limits. To calculate the concentration of chromium potentially present in the waste material, analytical data from the feedstock were used to establish the highest concentration of chromium in the ²³⁸Pu. A mass-balance calculation was then performed to determine the ratio of chromium to Pu in the feedstock. Considering that the amount of Pu allowable in the waste stream is restricted pursuant to the WIPP requirements and that the Cr-to-Pu ratio does not increase during processing, the highest concentration of Cr in the waste was determined based on the Pu content. The Pu was measured in each drum using nondestructive assay testing. The level of Cr potentially present was then compared with the concentration necessary to fail the TCLP. No concentrations were found to provide sufficient amounts of Cr to fail the TCLP even if all the Cr present were leached.

Corrosivity – To be a 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 Section 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 inch per year.

As has been determined by RTR, none of these drums contain 20% by volume aqueous waste. Because no liquids were observed by visual inspection during repackaging, no liquid existed that could pass or be collectable through the prescribed filter; hence, the waste does not meet the criteria of a liquid. A determination of such a substance's ability to corrode steel would then be inappropriate if it were not liquid by definition. Consequently, it would be impossible to test the pH of the waste or its ability to corrode steel pursuant to the required methodologies. If the material in the waste stream does not satisfy the regulatory criteria for the waste form, required tests cannot be performed nor can the waste fail the test for corrosivity.

Process knowledge and visual inspection indicate that no aqueous material or liquid was discarded in this waste stream(2). Also, should vapor be generated by radiolysis of plastics, any subsequent condensation of such vapor to a liquid phase and accumulation in a representative sample would likely be insufficient to meet either the aqueous or the liquid requirements necessary to test for corrosivity.

Ignitability – 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% alcohol and a flash point less than 140°F (60°C);
- It is not liquid and is capable of causing fire through friction, adsorption 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.

The waste does not contain liquids. Nothing in the waste stream is a solid that has qualities likely to ignite through friction, moisture adsorption, or chemical changes. As determined through RTR, no compressed gas cylinders exist in the waste and no DOT oxidizers are used in the process or exist in the waste. Visual inspection upon repackaging confirmed these conclusions.

Reactivity – 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 generated toxic gases, vapors, or fumes that are harmful;
- It contains cyanide or sulfide that can generated toxic gases, vapor, or fumes;
- It can detonate or explode at standard temperature and pressure; or
- It is a DOT forbidden, or Class A or B explosive.

No material was used or generated in these processes that, under the conditions specified above, could react violently, form harmful gases, contain cyanide or sulfide, or meet the DOT explosive definitions prescribed.

SAMPLING AND ANALYSIS PLAN

Since mass balance calculations were available for heavy metal concentrations in the waste, based on chemical analyses of the process input materials, the evidence for the non-mixed classification of the waste was strong. However, given the high probability that a lawsuit would be filed challenging the waste classification, the NMED requested and DOE agreed to conduct confirmatory sampling and analysis of the waste. Confirmatory sampling and analysis differs considerably from sampling to determine hazardous constituent contents. Confirmatory sampling requires acquisition of fewer samples because the existing information on the waste is used to estimate the statistical variability of the analysis parameters. In this case, statistical calculations showed that only two samples were required to confirm the RCRA-metal concentrations calculated for the waste stream lot. However, because the waste was debris containing five distinct types of materials, LANL chose to collect two confirmatory samples for each material type, and the number of required samples increased to 11 (including rust-colored powder sample and one duplicate). This Sampling and Analysis Plan(9) is the subject of a paper to be given later in this session.

NEXT REST STOP ON THE ROAD TO THE WIPP

It appears that we have pulled out from the rest stop and are again starting to move down the road to the WIPP. However, there may be additional rest stops along the way... the hearing determination by Judge Penn, the lawsuit against the EPA, possible activist stoppages of the TRUPACT-II trucks. All of this uncertainty means that the road to the WIPP may include additional rest stops, but if we continue making progress, the road becomes shorter and shorter each time.

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⁽AK Denotes Acceptable Knowledge, TWCP Denotes Transuranic Waste Characterization/Certification Program)

Figure 1. Process for Characterization and Certification of Waste Stream TA-55-43, Lot No. 01 (page 1 of 2)



Figure 1. Process for Characterization and Certification of Waste Stream TA-55-43, Lot No. 01 (page 2 of 2)

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