Contract 003CT0008-8L
Project No. 819592.01
June 2001

Response to Request for Information Pursuant to the New Mexico Hazardous Waste Act and the Resource Conservation and Recovery Act, Los Alamos National Laboratory EPA ID No. 0890010515

Prepared by:

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Response to Request for Information Pursuant to the New Mexico Hazardous Waste Act and the Resource Conservation and Recovery Act Los Alamos National Laboratory EPA ID NM 0890010515

Introduction

The following information supplements the April 16 and May 15, 2001, 60-day responses by Los Alamos National Laboratory (LANL) to a Request for Information (RI) sent by the New Mexico Environment Department (NMED) on February 12, 2001. The full title of the RI is "Request for Information Pursuant to the New Mexico Hazardous Waste Act and the Resource Conservation and Recovery Act, Los Alamos National Laboratory, EPA ID NM 0890010515," officially received by LANL on February 16, 2001.

LANL has continued efforts to collect historical and other process-generation documentation from internal waste-generating organizations to supplement the information presented in the previous responses. As stated in those responses, proposed dates for submittal of supplemental data collected for the 60-day response are May 15, June 15, and July 15, 2001. This document comprises the June 15, 2001, response.

This document consists of supplemental responses, where additional information has been obtained for the 19 information requests contained in the RI. The submittal includes appendices, as referenced in the individual responses to the numbered information requests. NMED's original information requests are included in this document as italicized text for ease of review.

Information Requests and Responses

1. *Please identify each radionuclide waste or waste stream, including mixed and non-mixed wastes, that is currently or has been at any time generated, treated, stored, disposed of, otherwise managed at, or transported to the LANL Facility, and that meets the statutory definition of "hazardous waste" in section 1004(5) of RCRA, 42 U.S.C. § 6903(5). (Please note that the statutory definition is broader than the regulatory definition.)*

As noted in the discussion included with the Introduction of the April 16, 2001, response to the RI, because source, special nuclear, and/or by-product materials are exempt from the definition of solid waste, they cannot meet the statutory definition of "hazardous waste." However, as discussed, LANL is providing information regarding the management of such material that is not subject to the Resource Conservation and Recovery Act (RCRA).

Materials discharged from outfalls subject to the National Pollutant Discharge Elimination System (NPDES) are also included even though they are not subject to RCRA. This information has been provided through the Laboratory's NPDES permit applications and associated information submittals. The most recent application was the "Los Alamos National Laboratory NPDES Permit Re-Application, Permit No. NM0028355," submitted to the U.S. Environmental Protection Agency (EPA), the NMED Surface Water Quality Bureau, and the NMED Hazardous and Radioactive Materials Bureau on May 4, 1998. The application contains information for all outfalls permitted at LANL, including radioactive liquid waste streams treated for discharge from the Radioactive Liquid Waste Treatment Facility (RLWTF) at Technical Area (TA) 50 (NPDES Outfall 051). This information includes waste stream information for discharges to the RLWTF, treatment process descriptions, and the facility's sampling and analysis plan with monitoring results. Supplemental information for the application (Appendix N of that application) includes discussions of the RLWTF waste acceptance criteria and sample results to support the facility's RCRA exclusions [New Mexico Administrative Code, Title 20, Chapter 4, Part 1 (20.4.1 NMAC), §261.3(a)(2)(iv)(A-G)]. Influent waste stream information for the permit application was also developed from the LANL Waste Stream Characterization Program, an inventory and generator interview project conducted from 1991 to 1997. The Laboratory's NPDES Permit (No. NM0028355) became effective on February 1, 2001.

Additional waste management information collected for Request No. 1 since the May 15, 2001, submittal is presented below.

Information on radionuclide wastes and waste streams recently generated at Key and Non-Key Facilities at LANL is presented and described in the "SWEIS Yearbook—1999" (LA-UR-00-5520; http://lib-www.lanl.gov/pubs/LA-UR-00-5520.htm). The Annual Yearbook compares operational data with the projections of the "Site-Wide Environmental Impact Statement for Continued Operation of the Los Alamos National Laboratory" (SWEIS) for the level of operations selected by the Record of Decision, issued in September 1999. (Copies of the SWEIS and the annual yearbooks have been provided to the NMED.) The fifteen (15) Key Facilities at LANL include:

- The Plutonium Complex at TA-55,
- The Tritium Facilities at TA-16 and TA-21,
- The Chemical and Metallurgy Research (CMR) Building at TA-3,
- The Pajarito Site at TA-18,
- The Sigma Complex at TA-3,
- The Materials Science Laboratory at TA-3,
- The Target Fabrication Facility at TA-35,
- The Machine Shops at TA-3,
- The High Explosives (HE) Processing Facilities at TA-8, TA-9, TA-11, TA-16, TA-22, TA-28, and TA-37,
- The HE Testing Facilities at TA-14, TA-15, TA-36, TA-39, and TA-40,

- The Los Alamos Neutron Science Center at TA-53,
- The Health Research Laboratory at TA-43,
- The Radiochemistry Facility at TA-48,
- The RLWTF at TA-50, and
- The Solid Radioactive and Chemical Waste Facilities at TA-50 and TA-54.

Non-Key Facilities at LANL, as defined in the "SWEIS Yearbook—1999," comprise the remainder of LANL. Wastes generated at these Key and Non-Key Facilities at LANL are discussed further in the response to Request No. 6. The waste-generating organizations in the Key Facilities have also provided additional current and historical waste management information included in this response.

Information on treatment of radioactive and mixed wastes at LANL and on recycling of mixed low-level (MLL) waste is presented in the response to Request No. 8.

Supplemental information regarding storage of radioactive waste at LANL is presented in the response to Request No. 9.

Gian Bacigalupa (Hazardous and Solid Waste Group [ESH-19] Technical Staff Member [TSM]) collected information used to prepare this response. His address is P.O. Box 1663, Los Alamos, NM 87545.

Documents providing information used to prepare the response to Request No. 1 are identified in the text above.

Any additional waste management information that can be identified regarding generation, treatment, storage, disposal, recycling, and transportation of radioactive and mixed waste will be produced in a later supplement to the RI, as provided by Instruction No. 5 on page 3 of NMED's February 12, 2001, letter.

2. *Please identify each radionuclide waste or waste stream, including mixed and non-mixed wastes, that is currently or has been at any time generated, treated, stored, disposed of, otherwise managed at, or transported to the LANL Facility, and that meets the following criteria: a) LANL claims the waste to be exempt from regulation as a solid waste under section 1004(27) of RCRA, 42 U.S.C. § 6903(27), because such waste meets the definition of source, special nuclear, or by-product material as defined by the Atomic Energy Act, 42 U.S.C. §§ 2011 et seq.; and b) the waste would meet the statutory definition of "hazardous waste" in section 1004(5) of RCRA, 42 U.S.C. § 6903(5), but for such exemption.*

There has been no additional waste management information identified or collected for Request No. 2 since the April 16, 2001, submittal. Any additional waste management information that can be identified regarding the generation, treatment, storage, disposal, recycling, and transportation of Atomic Energy Act (AEA)-exempt radioactive

waste at LANL will be provided in a later supplement to the RI, as provided by Instruction No. 5 on page 3 of NMED's February 12, 2001, letter.

3. *For each waste and waste stream identified in response to Request #1 and #2, please provide a detailed description of the radioactive, chemical, and physical properties of the waste. Include in your response a description of all radionuclides, all radioactive decay chains, and the half-lives of both the radionuclides and their daughter products.*

Information on the radioactive, chemical, and physical properties of the non-liquid wastes identified in the response to Request No. 1 was provided in Appendix C of the April 15, 2001, response. Sources of information regarding the radioactive, chemical, and physical properties of the radioactive liquid wastes are referenced herein in the NPDES portion of the response to Request No. 1. Information that can be used to determine radioactive decay chains, half-lives, and daughter products can be found in the reference cited in the response to Request No. 3 in the April 16, 2001, response.

Gian Bacigalupa (ESH-19 TSM) collected information used to prepare this response. His address is P.O. Box 1663, Los Alamos, NM 87545.

Documents providing information used to prepare the response to Request No. 3 are referenced in the text above.

Any additional waste management information that can be identified regarding the descriptions of radioactive, chemical, and physical properties of radioactive and mixed waste at LANL will be provided in a later supplement to the RI, as provided by Instruction No. 5 on page 3 of NMED's February 12, 2001, letter.

4. *For each waste and waste stream identified in response to Request #1 and #2, please state whether or not the waste exhibits any of the characteristics of a hazardous waste under 40 C.F.R. pt. 261, subpt. C:*

   a. *Ignitability under 40 C.F.R. § 261.21;*

   b. *Corrosivity under 40 C.F.R. § 261.22;*

   c. *Reactivity under 40 C.F.R. § 261.23;*

   d. *Toxicity under 40 C.F.R. § 261.24.*

Available information regarding the characteristics of the wastes identified in the responses to Request Nos. 1 and 2 is provided in the documents discussed or referenced in the response to Request No. 3. Wastes identified in the pyrolization treatment discussion in the response to Request No. 8 had the characteristics of ignitability (D001) and reactivity (D003). The corrosive (D002) waste treated by

elementary neutralization at TA-55, Building 4 (PF-4), Room 420 is discussed in the response to Request No. 8.

5. *For each waste and waste stream identified in response to Request #1 and #2, please state whether or not the waste contains any hazardous constituents listed under 40 C.F.R. pt. 261, Appendix VIII and name the specific constituent or constituents.*

Any available information regarding the hazardous constituents of the wastes identified in the responses to Request Nos. 1 and 2 is provided in the documents discussed or referenced in the response to Request No. 3 and in Appendix A of this response.

6. *For each waste and waste stream identified in response to Request #1 and #2, please provide a detailed description of the generation of the waste, including the location of its generation, the date of its generation, the process or processes by which it was generated, and the volume of waste that was generated.*

Additional waste management information collected for Request No. 6 since the May 15, 2001, submittal is presented below.

According to the "SWEIS Yearbook—1999," the Key Facilities at LANL (discussed in the response to Request No. 1) contribute more than 90% of all radioactive liquid waste generated and more than 90% of the radioactive non-liquid waste generated. The capabilities or activities that may lead to waste generation at each Key Facility location and the volumes of radioactive and mixed waste generated are presented in that report for operations conducted in 1999. The volumes of radioactive and mixed waste generated during operations conducted in 1999 at the Non-Key Facilities at LANL are also presented. A similar report is available for operations conducted in 1998, and will be published for subsequent years. The non-liquid wastes generated are typically sent to TA-54 for subsequent storage and/or disposal. Liquid wastes may be likewise sent to TA-54 or they may be treated and discharged from a NPDES-permitted outfall.

Radioactive waste generated at the Plutonium Complex Key Facility is primarily from operations conducted in PF-4 at TA-55. This facility opened in April 1978, and continues to operate. Radioactive and mixed wastes are generated as a result of plutonium-processing and related activities. Mixed waste-generating information for TA-55 was provided in the April 15, 2001, response to Request No. 6.

Radioactive waste generated at the CMR Building Key Facility is primarily from operations conducted in Wings 2, 4, 5, 7, and 9. This facility was constructed at TA-3 in 1952 to house analytical chemistry, plutonium and uranium chemistry and metallurgy, and some engineering and support functions. Radioactive and mixed wastes are generated as a result of research and analysis, uranium processing, and weapon components fabrication activities. Mixed waste-generating processes for the CMR Building are described in Appendix B, the Waste Analysis Plan, of the "Los

Alamos National Laboratory General Part B Permit Application," Revision 1.0, submitted to the NMED in October 1998.

Gian Bacigalupa (ESH-19 TSM) collected information used to prepare this response. His address is P.O. Box 1663, Los Alamos, NM 87545.

Documents providing information used to prepare the response to Request No. 6 are identified in the text above.

Any additional waste stream descriptions that can be identified for radioactive and mixed waste at LANL will be provided in a later supplement to the RI, as provided by Instruction No. 5 on page 3 of NMED's February 12, 2001, letter.

7. *For each waste and waste stream identified in response to Request #1 and #2 that was transported to the LANL Facility from elsewhere, please state the origin of the waste, the volume of the waste transported to the LANL Facility, broken down by shipment if possible, and the date or dates the waste was received at the LANL Facility.*

Additional waste management information collected for Request No. 7 since the May 15, 2001, submittal is presented below.

In May 1996, LANL received a shipment of three drums of waste from the U.S. Department of Energy (DOE) Pantex Facility in Texas. The waste consisted of plastic materials, personal protective equipment, and tools contaminated with transuranic (TRU) radionuclides. The waste was documented under general LANL waste transport and management procedures. Further description of the waste content and radionuclide information is included in Appendix C of the April 16, 2001, response.

Gian Bacigalupa (ESH-19 TSM) collected information used to prepare this response. His address is P.O. Box 1663, Los Alamos, NM 87545.

Documents providing information used to prepare the response to Request No. 7 are identified in the text above.

Any additional waste management information regarding the transportation of radioactive and mixed waste to LANL that can be identified will be provided in a later supplement to the RI, as provided by Instruction No. 5 on page 3 of NMED's February 12, 2001, letter.

8. *For each waste and waste stream identified in response to Request #1 and #2 that was treated at the LANL Facility, please provide a detailed description of the treatment, including the method or process of treatment, the effectiveness of the treatment in reducing the hazardous properties of the waste, and the volume of waste treated.*

Additional waste management information collected for Request No. 8 since the May 15, 2001, submittal is presented below.

The following information is described using the categories presented in the April 16, 2001, response for Request No. 8. For this submittal, these categories include:

- Non-RCRA radioactive waste treatment
- Treatment that is not subject to RCRA interim status or permitting requirements
- Mixed waste treatment

An environmental pilot treatment study for the remediation of uranium-contaminated soil was conducted at a former weapons research site at TA-33. Radioactive soil was chemically treated in a containerized vat leach process by use of sodium bicarbonate leach solution. The effectiveness of this treatment process and the volume of waste treated are discussed in the interim report provided in Appendix B of this response.

Liquid radioactive waste streams may be treated in facilities subject to Section 402 of the Clean Water Act. Information regarding the treatment processes is provided in the Laboratory's NPDES permit applications and associated information submittals. The most recent application was the "Los Alamos National Laboratory NPDES Permit Re-Application, Permit No. NM0028355," submitted to the EPA, the NMED Surface Water Quality Bureau, and the NMED Hazardous and Radioactive Materials Bureau on May 4, 1998. Information regarding the effectiveness of treatment and the volume of the waste treated can be obtained from "Radioactive Liquid Waste Annual Reports," which are submitted to the NMED Water Quality Bureau.

HE-contaminated tuballoy generated at the Machine Shops and at the HE Processing Key Facilities was treated prior to 1980 at the TA-16 Burn Ground. The materials were placed on concrete pads, surrounded by combustible materials, and burned to destroy the HE. The remaining noncombustible material was then collected and containerized in drums for disposal. Although the volume of this treated waste is unknown, the memorandum provided in Appendix A herein provides a typical snapshot of the quantities of materials handled in the 1970 timeframe at the TA-16 Burn Ground.

The "Compliance Order Site Treatment Plan FFC ACT" (STP), originally submitted to the NMED in 1995, addresses treatment of MLL and mixed TRU (MTRU) wastes at LANL. Annual updates to the STP Background Volume and revisions to the STP Compliance Plan Volume are submitted to the NMED STP Manager and the NMED Bureau Chief in March of each year.

At the Plutonium Complex Key Facility, treatment of TRU waste by cementation is conducted, on occasion, if an item is amenable to cementation technology and it contains radiological material that is below the discard ceiling, based on DOE Order M474.1, or is approved for discard by DOE. A description of the cementation process

for MTRU waste at TA-55 is provided in the "Technical Area 55 Part B Permit Application: Building 4, Container Storage Areas 1-11, Storage Tank Systems, Cementation Treatment Unit; Container Storage Pad; Building 185, Container Storage Area," Revision 0.0, submitted to the NMED in June 1996. The TRU waste cementation process is similar to that for MTRU waste. Effectiveness of treatment by cementation is discussed in "Waste-Form Development for Conversion to Portland Cement at Los Alamos National Laboratory Technical Area 55" (LA-13125). A copy of this document is provided herein as Appendix C. Currently (as of May 30, 2001), there are 2.28 cubic meters (m^3) of cemented waste in storage at TA-55. Treatment by elementary neutralization is conducted at TA-55, PF-4, Room 420. Acid and caustic liquid waste is piped to the RLWTF at TA-50 for further treatment. The amount of this liquid waste is included in the biennial report required by 20.4.1 NMAC, §262.41, and submitted to the NMED Hazardous Waste Bureau.

In May of 1994, a 90-day emergency permit (Permit Number 0890010515-EP1) was issued by the NMED to allow processing (pyrolization) of plutonium-impregnated nitrated cheesecloth (a mixed waste) at TA-55, PF-4. The nitrated cheesecloth (cellulose) processed was hazardous because it had the characteristics of ignitability (D001) and reactivity (D003). Plutonium-impregnated non-nitrated cheesecloth (a radioactive waste) was also processed. In July and August 1994, these materials were subjected to thermal decomposition in the Inert Atmosphere Decomposition Unit (IADU) as the first step to recover plutonium from the cheesecloth. The IADU was operated at a process temperature of 800 °C to ensure proper deactivation of the hazardous components of the nitrated wastes. The volume of nitrated cheesecloth treated was 3,841 grams, and the volume of non-nitrated cheesecloth treated was 27,638 grams. As a result of treatment, 6,630 grams of nonhazardous residue were generated; this residue was stored in process residue cans. As required in the emergency permit, a final report on this treatment process was submitted to the NMED. A copy of this report, submitted in October 1994, is included herein as Appendix D.

The STP updates also report, in text and tabular formats, the estimated inventory of covered waste that has been recycled during the fiscal year. Table 2-4 in the Fiscal Year (FY) 1997 update lists the MLL waste off-site shipments to recycling, and the text (Section 2.2.1) discusses where the material was recycled. In the FY 1998 through FY 2000 updates, Table 3.2 lists MLL waste inventory by date shipped, destination, waste type, volume, and the number of items recycled. Also in these updates, Section 3.2 discusses off-site recycling and Section 3.5 discusses on-site recycling.

In addition to the information on recycling listed in the STP annual updates, the Laboratory organizations at the Key Facilities that utilize radioactive materials were queried to determine if any of the resultant wastes had been recycled without going through the Facility and Waste Operations Solid Waste Operations Group at TA-54. The Materials Science and Technology Group indicated that they had independently shipped approximately 7,000 pounds of scrap metal contaminated with pyridine (D038)

and tritium to Scientific Ecology Group, Inc., in Oak Ridge, TN, for metal recycling on November 10, 1995.

The Laboratory generally focuses on waste reduction during the recovery process. However, some waste items, such as contaminated gloveboxes, can be reused for other operations.

Gian Bacigalupa (ESH-19 TSM) and Paul Schumann (Environmental Restoration Project Team Leader) collected information used to prepare this response. Their address is P.O. Box 1663, Los Alamos, NM 87545.

Documents providing information used to prepare the response to Request No. 8 are identified in the text above.

Any additional waste management information that can be identified regarding treatment of radioactive and mixed waste will be provided in a later supplement to the RI, as provided by Instruction No. 5 on page 3 of NMED's February 12, 2001, letter.

9. *For each waste and waste steam identified in response to Request #1 and #2 that was stored at the LANL Facility, please state the location of such storage at the LANL Facility, the method of storage, the volume of waste stored, and the dates during which each volume of such waste was stored at each such location.*

Additional waste management information collected for Request No. 9 since the May 15, 2001, submittal is presented below.

Non-liquid radioactive and mixed wastes generated at the Key and Non-Key Facilities at LANL are typically transported to TA-54 for subsequent storage and/or disposal. Radioactive liquid wastes may likewise be sent to TA-54, or they may be piped or transported to the RLWTF at TA-50 for treatment prior to effluent discharge through NPDES Outfall 051 when discharge criteria are met. Sludges from this treatment process are dewatered, solidified, and transported to TA-54 for storage and/or disposal. Evaporator solids are sent off site for additional treatment.

Radioactive wastes at the Plutonium Complex Key Facility are stored at TA-55 in the following locations: the container storage pad west of PF-4; Building 185; and PF-4. These wastes are stored in various containers, including 1-liter polyethylene containers, U.S. Department of Transportation (DOT)-certified containers, standard waste boxes (SWB), and Strong Tight IP1 containers (see the Code of Federal Regulations, Title 29, §§173.24, 173.410, and 173.411 for container definitions). The total volume of radioactive wastes currently (as of May 30, 2001) in storage at TA-55 is 175 m^3. Low-level waste accounts for 120 m^3 of the total volume, and TRU and MTRU waste accounts for 55 m^3. A small volume of slag waste, originally received from off site as samples for analysis, is included in this current total storage volume.

Radioactive wastes at the CMR Building Key Facility are stored in the vaults, in the filter towers of each wing, and in the yard west of Wing 9. These wastes are stored in various containers, including 1-liter polyethylene containers, DOT-certified containers, SWBs, and Strong Tight IP1 containers. The total volume of radioactive wastes currently (as of May 30, 2001) in storage at the CMR Building is 353 m^3. Low-level waste comprises 333 m^3 of the total volume, and TRU waste comprises 20 m^3. A small volume of slag waste, originally received from off site as samples for analysis, is included in this current total storage volume.

Gian Bacigalupa (ESH-19 TSM) collected the information that was used to prepare this response. His address is P.O. Box 1663, Los Alamos, NM 87545.

Any additional waste management information regarding storage of radioactive waste at LANL that can be identified will be provided in a later supplement to the RI, as provided by Instruction No. 5 on page 3 of NMED's February 12, 2001, letter.

10. *For each waste and waste stream identified in response to Request #1 and #2 that was disposed of at the LANL Facility, please provide a detailed description of the disposal, including the method of disposal, the location of disposal, the dates of disposal, and the volume of waste disposed of at each such location.*

There has been no additional waste management information identified or collected for Request No. 10 since the May 15, 2001, submittal. Any additional waste management information regarding disposal of radioactive waste and mixed waste at LANL that can be identified will be provided in a later supplement to the RI, as provided by Instruction No. 5 on page 3 of NMED's February 12, 2001, letter.

11. *For each waste and waste stream identified in response to Request #2, please state the basis for LANL's claim that the waste is exempt from regulation as a solid waste under RCRA because such waste is source, special nuclear, or by-product material as defined by the Atomic Energy Act.*

There has been no additional waste management information identified or collected for Request No. 11 since the April 16, 2001, submittal. Any additional waste management information regarding generation, treatment, storage, disposal, recycling, and transportation of AEA-exempt radioactive waste at LANL that can be identified will be provided in a later supplement to the RI, as provided by Instruction No. 5 on page 3 of NMED's February 12, 2001, letter. Further discussion of the basis for the AEA exemption as source, special nuclear, or by-product material will be included, as such information is applicable.

12. *For each Site listed in Part 1 of Attachment A, please identify each waste or waste stream that is currently or has been at any time disposed of at the Site.*

There has been no additional information identified or collected for Request No. 12 since the April 16, 2001, submittal. Any additional wastes and waste streams that can be identified will be provided in a later supplement to the RI, as provided by Instruction No. 5 on page 3 of NMED's February 12, 2001, letter.

13. *For each waste and waste stream identified in response to Request #12, please provide a detailed description of the radioactive, chemical, and physical properties of the waste. Include in your response a description of all radionuclides, all radioactive decay chains, and the half-lives of both the radionuclides and their daughter products.*

There has been no additional information identified or collected for Request No. 13 since the April 16, 2001, submittal.

14. *For each waste and waste stream identified in response to Request #12, please state whether or not the waste is a listed hazardous waste under 40 C.F.R. pt. 261, subpt. D and indicate the specific listing or listings.*

There has been no additional information identified or collected for Request No. 14 since the April 16, 2001, submittal. Any additional information regarding listed hazardous wastes and waste streams that can be identified will be provided in a later supplement to the RI, as provided by Instruction No. 5 on page 3 of NMED's February 12, 2001, letter.

15. *For each waste and waste stream identified in response to Request #12, please state whether or not the waste meets any of the characteristics of a hazardous waste under 40 C.F.R. pt. 261, subpt. C:*

    a. *Ignitability under 40 C.F.R. § 261.21;*

    b. *Corrosivity under 40 C.F.R. § 261.22;*

    c. *Reactivity under 40 C.F.R. § 261.23;*

    d. *Toxicity under 40 C.F.R. § 261.24.*

There has been no additional information identified or collected for Request No. 15 since the April 16, 2001, submittal. Any additional information regarding characteristic hazardous wastes and waste streams that can be identified will be provided in a later supplement to the RI, as provided by Instruction No. 5 on page 3 of NMED's February 12, 2001, letter.

16. *For each waste and waste stream identified in response to Request #12, please state whether or not the waste contains any hazardous constituents listed under*

40 C.F.R. pt. 261, Appendix VIII and name the specific constituent or constituents.

There has been no additional information identified or collected for Request No. 16 since the April 16, 2001, submittal. Any additional information regarding whether or not the wastes or waste streams contain any hazardous constituents listed in 20.4.1 NMAC, Subpart II, Part 261, Appendix VIII, that can be identified will be provided in a later supplement to the RI, as provided by Instruction No. 5 on page 3 of NMED's February 12, 2001, letter.

17. *For each waste and waste steam identified in response to Request #12, please provide a detailed description of the disposal, including the method of disposal, the location of disposal, the dates of disposal, and the volume of waste disposed of at each such location.*

There has been no additional information identified or collected for Request No. 17 since the April 16, 2001, submittal. Any additional information regarding a detailed description, method, location, date and volume of waste disposal that can be identified will be provided in a later supplement to the RI, as provided by Instruction No. 5 on page 3 of NMED's February 12, 2001, letter.

20. *For each Request #1 through #19, inclusive, identify each and every person who provided information that was used to prepare the response. Identify each such person by name, title or job description, employer, and current or last known address.*

As discussed in the April 16, 2001, response, numerous sources of information were used to prepare this response. A responsible individual (or individuals) who collected data and prepared the response has been identified for the appropriate portion of the response for each numbered request. If necessary, these individuals can provide further details regarding the preparation of this response. Title or job description, employer, and current or last known addresses for these individuals are also provided in each numbered request, in accordance with Instruction No. 7 of the RI.

21. *For each Request #1 through #19, inclusive, identify each and every document that provided information that was used to prepare your response. Identify each such document by type of document, title or description, author, and date.*

Each and every document that provided information used to prepare responses are identified in the corresponding responses to the numbered request, in accordance with Instruction No. 7 of the RI. The document type, title or description, author, and document date are also provided in the numbered request, per Instruction No. 7.

ATTACHMENT A

PART 1

PRS Name	*TA*	*SWMU Number*
MDA-A	*21*	*21-014*
MDA-B	*21*	*21-015*
MDA-C	*50*	*50-009*
MDA-D	*33*	*33-003(a)-99*
MDA-E	*33*	*33-001(a)-99*
MDA-F	*6*	*6-007(a)-99*
MDA-K	*33*	*33-002(a)-99*
MDA-M	*9*	*9-013*
MDA-N	*15*	*15-007(a)*
MDA-P	*16*	*16-018*
MDA-Q	*8*	*8-006(a)*
MDA-R	*16*	*16-019*
MDA-S	*11*	*11-009*
MDA-T	*21*	*21-016(a)-99*
MDA-U	*21*	*21-017(a)-99*
MDA-V	*21*	*21-018(a)-99*
MDA-W	*35*	*35-001*
MDA-X	*35*	*35-002*
MDA-Y	*39*	*39-001(b)*
MDA-Z	*15*	*15-007(b)*
MDA-AA	*36*	*36-001*
MDA-AB	*49*	*49-001(a-g)*
90's Line	*16*	*16-008(a)*
Firing Sites	*39*	*39-004(a-e), 39-008*
Firing Sites	*15*	*15-004(f); 15-006(a, c, d); 15-008(a)*
Townsite PRS's	*0, 1*	*0-010(b), 1-001(a-w), 1-002, 1-003(a-e)*
Outfall	*21*	*21-011(k)*
Surface Impoundments	*35*	*35-003(d, r), 35-010(a-e)*
Outfalls	*46*	*46-004(g, h, m, q, s, u, v, x, y, z, a2, b2, c2)*
Bayo Canyon Sites	*10*	*10-003(a-o), 10-007*
Fish Ladder	*16*	*16-003(o)*

APPENDIX A

MEMORANDUM FROM J.B. PANOWSKI AND P.G. SALGADO TO JESSE ARGON, DATED JULY 28, 1971

Hard copies of this document were provided to
the New Mexico Environment Department.

ATTACHMENT 1

Memorandum from J.B. Panowski and P.G. Salgado to Jesse Aragon, dated July 28, 1971

OFFICE MEMORANDUM

H. F. Schulte

DATE July 28, 1971

VIA : Jesse Aragon

J. B. Panowski and P. G. Salgado

AUG 7 '71 AM

H-6 FILE

GROUP GMX-3 EFFLUENTS

GMX-3

Reference : Memorandum from Harry F. Schulte to Group Leaders, dated April 12, 1971, subject "Effluents from AEC Facilities"

In response to the reference memorandum, we have surveyed effluent materials from Group GMX-3 operations. In accordance with the agreement reached in our meeting on May 20, 1971, we are reporting these effluents (1) as best estimates from specific buildings, which are large users of solvents and explosives or users of materials of special concern and (2) as an itemized breakdown of material issued to individual buildings.

GMX-3 has a large number of buildings and auxiliary facilities. So that you will not have to reduce the information from a formidable number of "Survey of Effluent Stream" forms, we are providing the following information:

Table 1 Numbers of GMX-3 buildings that emit no or negligible effluent

Table 2 Numbers of GMX-3 buildings that emit explosives, solvents, or gases in moderate amounts

Table 3 Numbers of GMX-3 buildings that emit large amounts of explosives, solvents, gases, or materials of particular concern

Table 4 Numbers of GMX-3 structures that are associated with the Burning Ground and disposal facilities

Table 5 Numbers of GMX-3 buildings that are the responsibility of agencies other than GMX-3

Appendix A Forms for buildings listed under Items 3 and 4, above

Appendix B Quantities of "materials of concern" supplied to GMX-3 buildings including those listed under Item 2, above

In assessing the effluent from GMX-3, the data on the forms should be given credence over the list of materials delivered. In these cases, records of usage at the buildings indicate that the material delivered, in excess of that used, is still on hand. In cases where there are no forms, the effluent may be considered to be equal to those materials delivered.

As shown in Appendix B, the GMX-3 effluents of major concern are solvents and high explosives because of the quantity of material involved. Approximately 3600 gallons of various solvents are delivered to our operating buildings over a six-month period. These solvents leave the buildings by various routes, but we feel that virtually all this material either evaporates into the atmosphere within a short distance, perhaps half a mile, of the site boundaries or is burned as part of our disposal activities. With respect to waste explosives, we burn approximately 50,000 pounds during a six-month period and estimate that another 125 pounds is carried a short distance downstream in our sump effluent.

In an attempt to get some perspective for the scope of the GMX-3 effluent problem, we have compared the site solvent and HE effluent with that of an automobile as shown below.

EFFLUENT COMPARISONS

A. GMX-3

	Disposal (6 months)	Rate (lbs/hr, 24-hr day)
Solvents	3,570 gal ($\approx$ 7 lbs/gal)	5.7
HE	49,400 lbs	11.3
	Total	17.0
Products	Hydrocarbons, C, CO_2, N_2, H_2O, NO_x	

B. One automobile (15 miles/gal: Gasoline at 0.7 g/cm³ or 5.9 lbs/gal)

	Disposal (one hour)	Rate (lbs/hr)
Gasoline	4 gal	23.6
Products	Hydrocarbons, C, CO, CO_2, N_2, NO_x, SO_2, H_2O, $Pb(C_2H_5)_4$	

C. Summary

GMX-3	17.0 lbs/hr
One automobile	23.6 lbs/hr

This information is the best available at this time and we are sending it to you with the thought that such interim information may be useful in outlining the scope of the overall problem. We expect to have additional quantitative information after analysis of liquid and soil samples from our effluent streams. These data will be forwarded to you when available.

J. B. Papowski

P. G. Salgado

JBP/PGS/sf

Enclosures: As cited above

cc: E. H. Eyster
R. W. Drake
D. D. Meyer
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H. S. Jordan
Jesse Aragon
C. R. West
GMX-3 Reading File
File (2)

TABLE 1

GMX-3 BUILDINGS FROM WHICH
POLLUTION EFFLUENTS ARE EFFECTIVELY NEGLIGIBLE

TA-16- 7	TA-16-209	TA-16-343	TA-16-520	TA-37- 7
10	220	344	525	8
27	221	345	TA-11- 1	9
54	223	360	2	10
58	224	400	3	11
59	225	411	4	12
61	226	413	6	13
65	261	414	24	14
73	263	415	25	15
88	277	417	30	16
89	278	435	36	17
90	281	437	TA-28- 1	18
91	283	462	2	19
92	285	463	3	20
93	286	476	4	21
99	300	477	5	22
164	303	478	TA-37- 1	23
191	304	489	2	24
200	305	515	3	25
203	308	516	4	26
206	319	517	5	27
207	341	518	6	
208	342	519		

TABLE 2

GMX-3 BUILDINGS THAT EMIT SMALL OR MODERATE AMOUNTS
OF EXPLOSIVES, SOLVENTS, GASES,
OR OTHER MATERIALS OF PARTICULAR CONCERN
(No forms are provided.)

TA-16- 7	TA-16-267	TA-16-307	TA-16-450
92	280*	308*	460
260	301*	410	
265	302*	430*	

* Effluent from building consists of small quantities of solvents and HE that are carried to the sump and reported as burned.

TABLE 3

GMX-3 BUILDINGS THAT EMIT LARGE QUANTITIES OF EXPLOSIVES, SOLVENTS, GASES, OR OTHER MATERIALS OF PARTICULAR CONCERN
(Forms are provided.)

TA-16-193	TA-16-306
202	340
222	370

TABLE 4

GMX-3 BURNING GROUND AND DISPOSAL STRUCTURES

TA-16-386	TA-16-389	TA-16-394
387	390	399
388	392	401
		412

TABLE 5

BUILDINGS ASSOCIATED WITH GMX-3 FOR WHICH POLLUTION EFFLUENTS WILL BE REPORTED BY OTHER AGENCIES

TA-16- 16	TA-16-531	TA-16-540
180	532	542
192	533	560
195	534	
210	535	
530		

APPENDIX A

Completed Forms "Survey of Effluent Streams"

LOS ALAMOS SCIENTIFIC LABORATORY

Survey of Effluent Streams

Do not w
in this s

Group GMX-3 Tech Area 16 Building 193

Accounting Number(s) _____

Nature of Effluent Streams: Gas ____ Liquid X Solid ____ Other ____

(Fill out one sheet for each effluent stream)

Effluent Treatment: Type: None

Discharge Point: (Identify and show sketch on reverse if more than one per building)

Stack ____ Industrial Sewer ____

Sanitary Sewer X Storm Sewer ____

Trash Container Type: ____

Other: ____

Disposal Method: Atmospheric Dispersal ____ Sewage Plant ____

Waste Process Plant ____ Chemical Dump ____

Sewage Lagoon ____ Surface of Ground ____

Contaminated Burial Area ____ Septic Tank ____

Community Landfill ____ Other ____

Volume of Effluent: Known ____ Estimated ____

Contaminants (see list)	Estimated Discharge
Detergent (250 lbs/mo)	Awaiting analysis
Bleach - Purex (13 gal/mo)	" "
Aerosol - OT (1/2 lb/10 gal of water once a month)	" "

* See below

Monitoring Equipment on Effluent Stream: No X Yes ____

Describe ____

Remarks There is no detectable HE in the wash water (< 1 ppm)

* Ammonium Phosphate Dibasic (16 lbs/10 gal of water once a month) - Awaiting analysis

LOS ALAMOS SCIENTIFIC LABORATORY

Survey of Effluent Streams

Group GMX-3 Tech Area 16 Building 202, Rm 114

Do not write in this space

Accounting Number(s) _____

Nature of Effluent Streams: Gas X Liquid ___ Solid ___ Other ___

(Fill out one sheet for each effluent stream)

Effluent Treatment: Type: None

Discharge Point: (Identify and show sketch on reverse if more than one per building)

Stack _____ Industrial Sewer _____

Sanitary Sewer _____ Storm Sewer _____

Trash Container Type: _____

Other: Exhaust duct

Disposal Method: Atmospheric Dispersal X Sewage Plant _____

Waste Process Plant _____ Chemical Dump _____

Sewage Lagoon _____ Surface of Ground _____

Contaminated Burial Area _____ Septic Tank _____

Community Landfill _____ Other _____

Volume of Effluent: Known _____ Estimated 500 ft^3/yr

Contaminants (see list)	Estimated Discharge
Brazing alloy(Easy Flo)	1 lb/yr
Acetylene	500 ft^3/yr

Monitoring Equipment on Effluent Stream: No X Yes _____

Describe _____

Remarks One lb/yr of alloy is used. Only a small fraction of the alloy is volatile. The acelylene is burned and the products of combustion are emitted to the atmosphere.

LOS ALAMOS SCIENTIFIC LABORATORY

Survey of Effluent Streams

Group GMX-3 Tech Area 16 Building 202 Room 114

Do not write in this space

Accounting Number(s)

Nature of Effluent Streams: Gas X Liquid ___ Solid ___ Other ___

(Fill out one sheet for each effluent stream)

Effluent Treatment: Type: None

Discharge Point: (Identify and show sketch on reverse if more than one per building)

Stack ___ Industrial Sewer ___

Sanitary Sewer ___ Storm Sewer ___

Trash Container Type:

Other: Charging Refrigeration Unit

Disposal Method: Atmospheric Dispersal X Sewage Plant ___

Waste Process Plant ___ Chemical Dump ___

Sewage Lagoon ___ Surface of Ground ___

Contaminated Burial Area ___ Septic Tank ___

Community Landfill ___ Other ___

Volume of Effluent: Known ___ Estimated 30 lb/yr

Contaminants (see list)	Estimated Discharge
Freon	30 lb/yr

Monitoring Equipment on Effluent Stream: No X Yes ___

Describe

Remarks This is the amount of Freon that must be added to the refrigeration unit to replace that which leaks and evaporates.

LOS ALAMOS SCIENTIFIC LABORATORY

Survey of Effluent Streams

Group GMX-3 Tech Area 16 Building 202 Room 114

Do not w
in this s

Accounting Number(s) _____

Nature of Effluent Streams: Gas ____ Liquid X Solid ____ Other ____

(Fill out one sheet for each effluent stream)

Effluent Treatment: Type: None

Discharge Point: (Identify and show sketch on reverse if more than one per building)

Stack ____ Industrial Sewer ____

Sanitary Sewer ____ Storm Sewer ____

Trash Container Type: Wiping rags

Other: ____

Disposal Method: Atmospheric Dispersal X Sewage Plant ____

Waste Process Plant ____ Chemical Dump ____

Sewage Lagoon ____ Surface of Ground ____

Contaminated Burial Area ____ Septic Tank ____

Community Landfill ____ Other ____

Volume of Effluent: Known 1 gal/mo Estimated ____

Contaminants (see list)	Estimated Discharge
Chlorothene	1 gal/mo
Petroleum Distillate	0.2 gal/mo
Muriatic Acid	0.1 gal/yr

Monitoring Equipment on Effluent Stream: No X Yes ____

Describe ____

Remarks These materials are used to wipe and clean surfaces.

LOS ALAMOS SCIENTIFIC LABORATORY

Survey of Effluent Streams

Group GMX-3 Tech Area TA-16 Building 222

Do not wr in this sp

Accounting Number(s)

Nature of Effluent Streams: Gas ____ Liquid X Solid ____ Other ____

(Fill out one sheet for each effluent stream)

Effluent Treatment: Type: None

Discharge Point: (Identify and show sketch on reverse if more than one per building)

Stack ____ Industrial Sewer X

Sanitary Sewer ____ Storm Sewer ____

Trash Container Type: ____

Other: ____

Disposal Method: Atmospheric Dispersal ____ Sewage Plant ____

Waste Process Plant ____ Chemical Dump ____

Sewage Lagoon ____ Surface of Ground X

Contaminated Burial Area ____ Septic Tank ____

Community Landfill ____ Other ____

Volume of Effluent: Known ____ Estimated 40 gal/wk

Contaminants (see list)	Estimated Discharge Diluted (Undiluted)
Acetic acid	5 gal/wk (approx 500 lb/yr)
Hydroquinone	7 lb/wk (approx 90 lb/yr)
Sodium Sulfite and Sodium Metaborate	40 lb/wk (approx 500 lb/yr)

* See below

Monitoring Equipment on Effluent Stream: No X Yes ____

Describe ____

Remarks Effluent is fixer (20 gal/wk) and developer (20 gal/wk) film processing solutions. The discharge, about once every three months, is accompanied by voluminous quantities of water.

* Potassium Bromide 1-1/2 lb/wk (approx 20 lb/yr)

LOS ALAMOS SCIENTIFIC LABORATORY

Survey of Effluent Streams

Group GMX-3 Tech Area 16 Building 222

Do not wri in this sec

Accounting Number(s)

Nature of Effluent Streams: Gas X Liquid ___ Solid ___ Other ___

(Fill out one sheet for each effluent stream)

Effluent Treatment: Type: None

Discharge Point: (Identify and show sketch on reverse if more than one per building)

Stack ___ Industrial Sewer ___

Sanitary Sewer ___ Storm Sewer ___

Trash Container Type: ___

Other: Coolant leakage

Disposal Method: Atmospheric Dispersal X Sewage Plant ___

Waste Process Plant ___ Chemical Dump ___

Sewage Lagoon ___ Surface of Ground ___

Contaminated Burial Area ___ Septic Tank ___

Community Landfill ___ Other ___

Volume of Effluent: Known ___ Estimated 150 lb/yr

Contaminants (see list)	Estimated Discharge
Sulfur Hexafluoride	

Monitoring Equipment on Effluent Stream: No X Yes ___

Describe ___

Remarks This material is used as a coolant in radiograph equipment. It is replaced as it leaks.

LOS ALAMOS SCIENTIFIC LABORATORY

Survey of Effluent Streams

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Group GMX-3 Tech Area 16 Building 306

Accounting Number(s)

Nature of Effluent Streams: Gas ____ Liquid (1) ____ Solid ____ Other (2) Solvent Vapor

(Fill out one sheet for each effluent stream)

Effluent Treatment: Type: (1) Sump, (2) None.

Discharge Point: (Identify and show sketch on reverse if more than one per building)

Stack ____ Industrial Sewer ____

Sanitary Sewer ____ Storm Sewer X (1)

Trash Container Type: ____

Other: Hoods (2)

Disposal Method: Atmospheric Dispersal X (2) Sewage Plant ____

Waste Process Plant ____ Chemical Dump ____

Sewage Lagoon ____ Surface of Ground X (1)

Contaminated Burial Area ____ Septic Tank ____

Community Landfill ____ Other Sump (1)

Volume of Effluent: Known ____ Estimated 1000 gal/day

Contaminants (see list)	Estimated Discharge /6 mo (gallons)
Acetone	220
Chlorothene	55 Methylchloroform (TCA?)
* See below	

Monitoring Equipment on Effluent Stream: No ____ Yes ____

Describe ____

Remarks Most of the indicated discharge is water.

* Methylene chloride	10

LOS ALAMOS SCIENTIFIC LABORATORY

Survey of Effluent Streams

Group GMX-3 Tech Area 16 Building 340

Do not write in this space

Accounting Number(s)

Nature of Effluent Streams: Gas ____ Liquid X Solid ____ Other ____

(Fill out one sheet for each effluent stream)

Effluent Treatment: Type: Sump

Discharge Point: (Identify and show sketch on reverse if more than one per building)

Stack ____ Industrial Sewer X

Sanitary Sewer ____ Storm Sewer ____

Trash Container Type: ____

Other: ____

Disposal Method: Atmospheric Dispersal ____ Sewage Plant ____

Waste Process Plant ____ Chemical Dump ____

Sewage Lagoon ____ Surface of Ground X

Contaminated Burial Area ____ Septic Tank ____

Community Landfill ____ Other ____

Volume of Effluent: Known ____ Estimated X

Contaminants (see list)	Estimated Discharge/6 mo (gallons) **
H Cl	4
Acetone	700
n-Butyl acetate	120

* See below

Monitoring Equipment on Effluent Stream: No X Yes ____

Describe ____

Remarks These solvents will, in a short time, evaporate into the atmosphere, although, perhaps 90% of them leave as liquids.

* Ammonium sulfate	500
Methyl ethyl ketone	750
Toluene	10
Methanol	110

** With about 442,000 gallons of water

LOS ALAMOS SCIENTIFIC LABORATORY

Survey of Effluent Streams

Group GMX-3 Tech Area 16 Building 370

Do not wr
in this spa

Accounting Number(s)

Nature of Effluent Streams: Gas ____ Liquid X Solid ____ Other ____

(Fill out one sheet for each effluent stream)

Effluent Treatment: Type: None

Discharge Point: (Identify and show sketch on reverse if more than one per building)

Stack ____ Industrial Sewer ____

Sanitary Sewer ____ Storm Sewer ____

Trash Container Type: ____

Other: Metal chips to salvage

Disposal Method: Atmospheric Dispersal ____ Sewage Plant ____

Waste Process Plant ____ Chemical Dump ____

Sewage Lagoon ____ Surface of Ground ____

Contaminated Burial Area ____ Septic Tank ____

Community Landfill ____ Other Burn

Volume of Effluent: Known 1 gal/mo Estimated ____

Contaminants (see list)	Estimated Discharge
Kerosene	1 gal/mo

Monitoring Equipment on Effluent Stream: No X Yes ____

Describe ____

Remarks ____

LOS ALAMOS SCIENTIFIC LABORATORY

Survey of Effluent Streams

Do not wr
in this spa

Group GMX-3 Tech Area 16 Building 370

Accounting Number(s) _____

Nature of Effluent Streams: Gas ____ Liquid X Solid ____ Other ____

(Fill out one sheet for each effluent stream)

Effluent Treatment: Type: None

Discharge Point: (Identify and show sketch on reverse if more than one per building)

Stack ____ Industrial Sewer ____

Sanitary Sewer ____ Storm Sewer X

Trash Container Type: ____

Other: ____

Disposal Method: Atmospheric Dispersal ____ Sewage Plant ____

Waste Process Plant ____ Chemical Dump ____

Sewage Lagoon ____ Surface of Ground X

Contaminated Burial Area ____ Septic Tank ____

Community Landfill ____ Other ____

Volume of Effluent: Known ____ Estimated 15 gal/mo

Contaminants (see list)	Estimated Discharge
Trichloroethylene	15 gal/mo

Monitoring Equipment on Effluent Stream: No X Yes ____

Describe ____

Remarks ____

LOS ALAMOS SCIENTIFIC LABORATORY

Survey of Effluent Streams

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in this sp

Group GMX-3 Tech Area 16 Building 370

Accounting Number(s)

Nature of Effluent Streams: Gas X Liquid Solid Other

(Fill out one sheet for each effluent stream)

Effluent Treatment: Type: None

Discharge Point: (Identify and show sketch on reverse if more than one per building)

Stack X Industrial Sewer

Sanitary Sewer Storm Sewer

Trash Container Type:

Other:

Disposal Method: Atmospheric Dispersal X Sewage Plant

Waste Process Plant Chemical Dump

Sewage Lagoon Surface of Ground

Contaminated Burial Area Septic Tank

Community Landfill Other

Volume of Effluent: Known Estimated 3840 ft^3/yr

Contaminants (see list)	Estimated Discharge
Acetylene	3840 ft^3/yr

Monitoring Equipment on Effluent Stream: No X Yes

Describe

Remarks The acetylene is burned.

LOS ALAMOS SCIENTIFIC LABORATORY

Survey of Effluent Streams

Group GMX-3 Tech Area 16 Building Burning Ground Area (1) Do not write in this space

Accounting Number(s)

Nature of Effluent Streams: Gas X Liquid X Solid X Other Solvent Vapor

(Fill out one sheet for each effluent stream)

Effluent Treatment: Type: Combustible material is burned.

Discharge Point: (Identify and show sketch on reverse if more than one per building)

Stack _____ Industrial Sewer _____

Sanitary Sewer _____ Storm Sewer _____

Trash Container Type: _____

Other: Burning pits and filter vessels (1)

Disposal Method: Atmospheric Dispersal X Sewage Plant _____

Waste Process Plant _____ Chemical Dump _____

Sewage Lagoon _____ Surface of Ground _____

Contaminated Burial Area _____ Septic Tank _____

Community Landfill _____ Other Restricted Landfill

Volume of Effluent: Known _____ Estimated _____

Contaminants (see list)	Estimated Discharge (lbs/6 mo)
Explosives	49,500 (2)
Barium nitrate	3,000
~~HF contaminated oil and solvents~~	~~3,000 (2)~~
Uranium alloys	54 (3)
Kerosene	165 (4)

Monitoring Equipment on Effluent Stream: No X Yes X (3)

Describe (1) TA-16-412, 387, 388, 389, 392, 399, 401, 406

(2) A detailed listing of material burns, quantities burned, and smoke densities is attached.

(3) Uranium alloy burns monitored and documented. Residues are monitored, collected, and buried at Mesita del Buey.

Remarks

(4) This material is burned and the products of combustion are evolved.

APPENDIX B

Materials of Concern Supplied to S-Site Buildings

LISTING OF MISCELLANEOUS MATERIALS VERSUS BUILDINGS
GROUP GMX-3
(November 1970 through April 1971)

	Quantity (lbs)				
Building TA-16-	193	222	340	450	460
Material					
Ammonium Phosphate	75				
Ammonium Sulfate			500		
Diethylene Triamine				1.1	1.1
Hydroquinone		45.5			
Chlorine Bleach	3				
Phosphate Dibasic	96				
Detergents	1500				

LISTING OF SOLVENTS VERSUS BUILDINGS
GROUP GMX-3
(November 1970 through April 1971)

	Quantity (gal)						
Building TA-16-	7	202	260	306	340	460	Unidentified
Material							
Met-l-it	1	2					
Freon-PCA Solvent	55			2			
Dimethyl Sulfoxide			94				
Acetone				220	[illegible]	139	

MATERIALS DISPOSED OF BY BURNING AT
GROUP GMX-3 DISPOSAL AREAS
(November 1970 through April 1971)

Materials	Quantity (lbs) Burning Ground	Quantity (lbs) Incinerator
Explosives	49,500	
Barium Nitrate	3,000	
Oil and Solvents (HE contaminated)	3,000	
Uranium Alloys	54	
Trash	7,500	143,000

LISTING OF SOLVENTS VERSUS BUILDINGS
GROUP GMX-3
(November 1970 through April 1971)

	Quantity (gal)						
Building TA-16-	7	340	370	389	410	460	Unidentified
Material							
Ethyl Acetate		10.9					
Methanol		110					
Methyl Ethyl Ketone		750					
Toluene		110					
Trichloroethylene			90				55
Kerosene				165			
Ethylene Glycol					10		
Carbon Tetrachloride						4	
Chlorobenzene						0.2	
n-Hexane						0.5	55
Methylcyclohexane						1.1	
Ethanol							55
Petroleum Distillate	1.2						

LISTING OF METALS AND METAL SALTS VERSUS BUILDINGS
GROUP GMX-3
(November 1970 through April 1971)

	Quantity (lbs)				
Building TA-16-	193	202	222	370	460
Material					
Nickel (Foil)	1.4				
E-Z-Flo Brazing Alloy		0.5		0.25	
Cupric Oxalate					1
Cupric Oxide					3
Sodium Bichromate					2
Sodium Sulfite - Sodium Metaborate			260		
Magnesium Perchlorate			10		0.25
Potassium Bromide					1
Potassium Hydroxide					6

LISTING OF GASES AND ACIDS VERSUS BUILDINGS
GROUP GMX-3
(November 1970 through April 1971)

	Quantity (lbs)					
Building TA-16-	202	222	340	370	450	460
Material						
Acetylene	17			130		16
Carbon Dioxide					100	
Freon 13	15					
Acetic Acid (Glacial)		270				10
Hydrochloric (Muriatic) Acid	0.4	36	33			24
Hydrobromic Acid						0.07

MATERIALS DISPOSED OF BURNING AT
GROUP GMX-3 DISPOSAL AREAS
(November 1970 through April 1971)

Materials	Quantity (lbs) Burning Ground	Quantity (lbs) Incinerator
Explosives	49,500	
Barium Nitrate	3,000	
Oil and Solvents (HE contaminated)	3,000	
Uranium Alloys	54	
Trash	7,500	143,000

LISTING OF SOLVENTS VERSUS BUILDINGS
GROUP LMX-3
(November 1970 through April 1971)

	Quantity (gal)						
Building TA-16-	7	340	370	389	410	460	Unidentified
Material							
Ethyl Acetate		10.9					
Methanol		110					
Methyl Ethyl Ketone		750					
Toluene		110					
Trichloroethylene			90				55
Kerosene				165			
Ethylene Glycol					10		
Carbon Tetrachloride						4	
Chlorobenzene						0.2	
n-Hexane						0.5	55
Methylcyclohexane						1.1	
Ethanol							55
Petroleum Distillate	1.2						

LISTING OF SOLVENTS VERSUS BUILDINGS
GROUP GMX-3
(November 1970 through April 1971)

	Quantity (gal)						
Building TA-16-	7	202	260	306	340	460	Unidentified
Material							
Met-l-it	1	2					
Freon-PCA Solvent	55			2			
Dimethyl Sulfoxide			94				
Acetone				220	700	139	
Chlorothene		6		55			165
Methylene Chloride				55	72		
n-Butyl Acetate					330		
Chloroform					3	12	
1, 2-Dichloroethane					54.6	0.66	
Dimethylformamide					110	3.3	
Isopropyl Alcohol					72		
n-Octane							1.3
1, 1, 2, 2, Tetrachloroethane							0.99

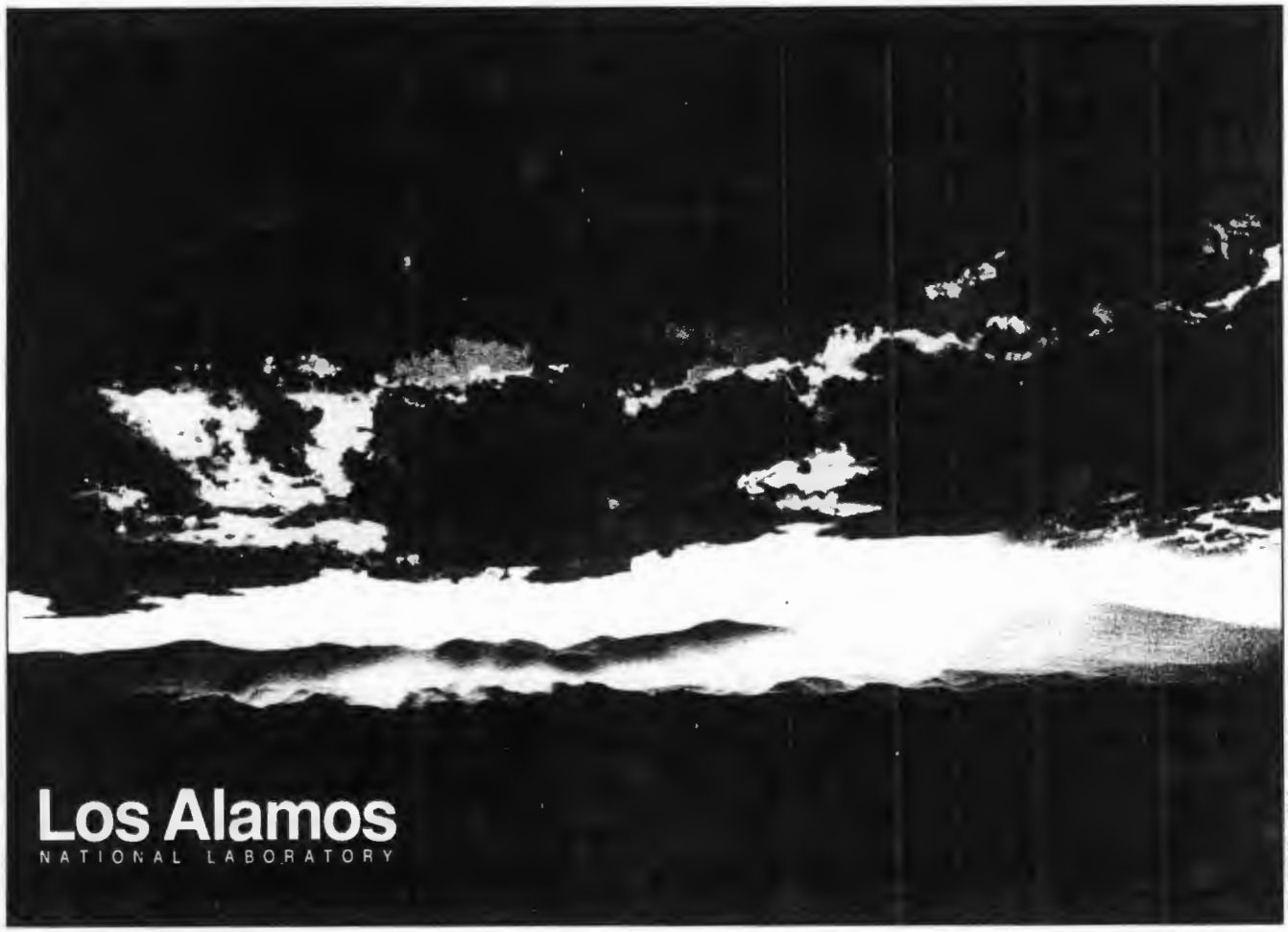
APPENDIX B

INTERIM REPORT ON CONTAINERIZED VAT LEACHING AT TA-33

Hard copies of this document were provided to the New Mexico Environment Department.

Interim Report on Containerized Vat Leaching at TA-33

W. R. J. R. Turney
N. P. Lu
C. F. V. Mason
M. C. Duff
D. E. Dry



Photograph: by Chris J. Lindberg

LA-UR-96-3900

Interim Report on Containerized Vat Leaching at TA-33

LA-UR-96-3900

W. R. J. R. Turney*, N. P. Lu*, C. F. V. Mason*, M. C. Duff*, and D. E. Dry**

Abstract

An environmental pilot treatment study for the remediation of uranium-contaminated soil by use of a two-step, zero-discharge, 100% recycle system was conducted at a former weapons research site. In the first step, following excavation, the soil was sorted by use of the ThemoNuclean Services segmented gate system (SGS) into two different streams: One stream with radioactivity greater than 30 pCi/g, the other steam with radioactivity less than 30 pCi/g . Following the sorting, the soil with radioactivities less than 30 pCi/g was returned to the excavation site as it met regulatory soil release guidelines. The portion of soil with radioactivities greater than 30 pCi/g was chemically treated in a second step containerized vat leach process by use of sodium-bicarbonate leach solution. The results of the second step are reported here.

Objective

The two objectives of the pilot treatment study were: 1) to chemically leach uranium from the SGS segregated soil, by use of the containerized vat leach (CVL) system so that the activity concentrations of soil produced a total dose of less than 15 mrem/yr. (as an approximation the total activity from uranium must be less than 114 pCi/g) and thereby permitting the soil to be returned to the excavation site, and 2) to remove uranium from the leach solution by use of ion exchange resin. (Soil sorting criteria for the SGS was to divert the soil with greater than 30 pCi/g to the CVL system for treatment. This decision was made based on SGS operations at TA-33.) Following treatment the resins were to be either disposed of in an approved low-level radioactive landfill, or would be recycled by removal of the uranium from the resins.

Introduction

The site selected for this pilot study is situated at Technical Area 33 (TA-33) near Building 16 (TA-33-16) at an area known as Area 6 developed in 1948 for initiator experiments. TA-33-16 housed a military air gun for the firing of uranium projectiles as part of nuclear weapons research. Another gun was stationed and fired from a concrete pad

*CST-7
**CST-11

adjacent to TA-33-16. The projectiles were fired at targets that were placed in front of two catch boxes and located less than 50 feet south of TA-33-16. Experiments at Area 6 were discontinued in 1955.

The two catch boxes, constructed of timbers, were five feet wide, ten feet deep, and ten feet in height and were located less than 50 feet south of TA-33-16 (Figure 1). The boxes were filled with soil, wood chips, and vermiculite. The targets were constructed, in part, of uranium, beryllium, and polonium-210 (alpha emitter, $t_{1/2}$ = 138 days). The projectiles often cracked open, contaminating the concrete and asphalt pads adjacent to TA-33-16, and the surrounding area, with the uranium, beryllium, and polonium-210. Initially the projectiles were constructed of tuballoy (natural) uranium. About ten percent of the projectiles were alloyed with one to two percent molybdenum to increase their compressive strength. Occasionally boron carbide was used as it is an effective neutron reflector. Titanium was alloyed either with the uranium or with the outer steel casings to decrease the weight in some instances (Mason, 1996). In 1994 the timbers of the catch boxes were disintegrating but still in place (Figure 2).

An environmental investigation, conducted in 1994 by ICF Kaiser, Engineers, at the direction of the Field Unit Three of the Los Alamos National Laboratory (LANL) Environmental Restoration (ER) program determined the existence of uranium-contaminated soil within the catch boxes (Turney, *et al.*, 1996). The exterior faces of the boxes were

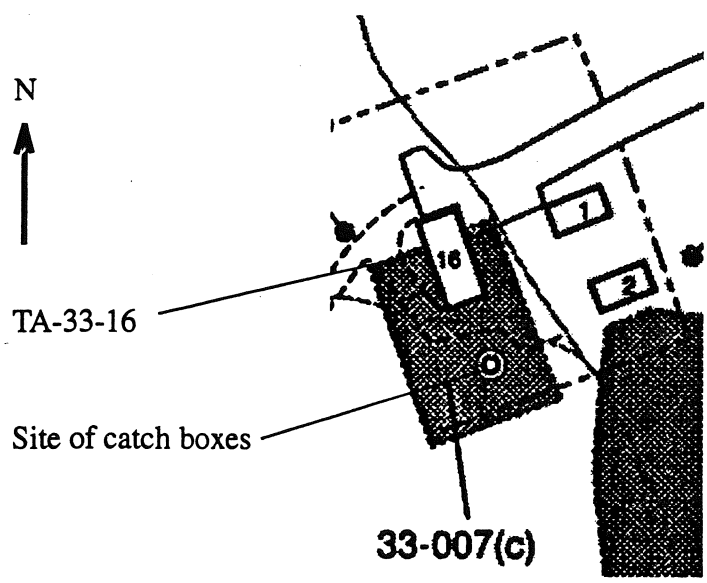


Figure 1. TA-33 Area 6 Site. Projectiles were shot from building 16 into catch boxes less than 50 feet south of the building. The area around building 16 is designated as SWMU 33-007(c) (DOE 1992).

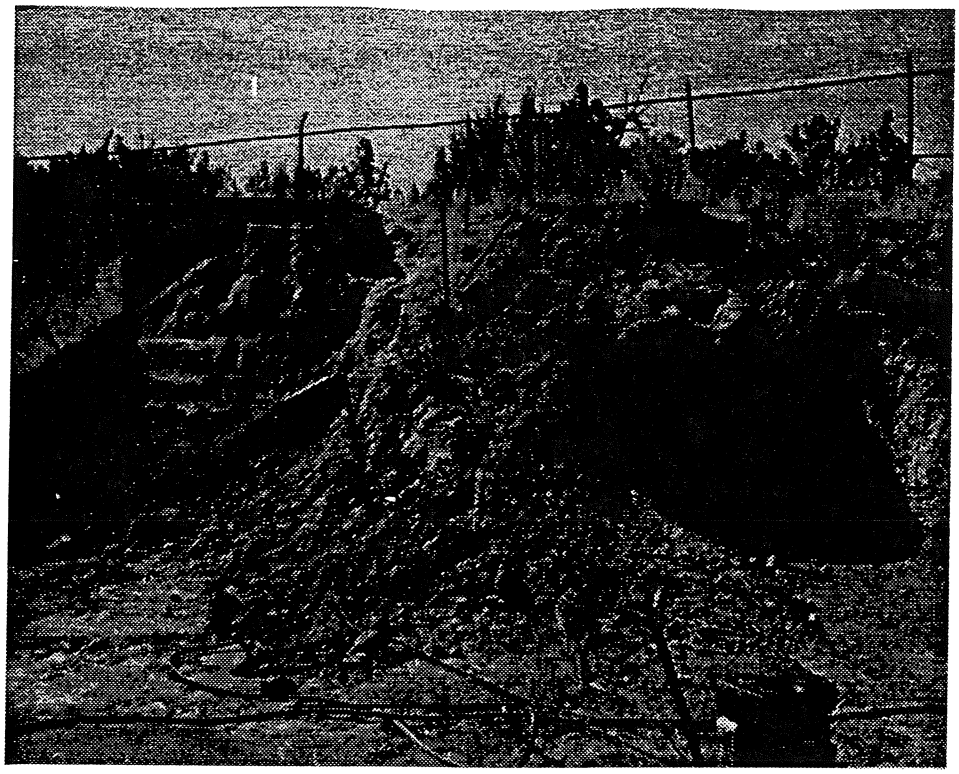


Figure 2. Remains of catch boxes at TA-33 Area 6 Site, 1995 (looking south from Building 16).

dismantled with a backhoe and trenches were excavated into the soil within the eastern and western catch-boxes. The western catch-box contained oxidized uranium in a "pocket" approximately 25 cm in circumference. Its location within the trench was one meter inward from the toe of the trench and 60 cm in elevation above the base of the catch box. Radioactivity in this contaminated zone, measured in the field, was as high as 13,000 cpm using a direct reading beta/gamma instrument. Another larger "pocket" of oxidized uranium was located between the two catch boxes. Radioactivity in this slightly larger contaminated zone was as high as 135,000 cpm beta/gamma.

Eight soil samples were collected from the excavated areas and analyzed for uranium. Two of the samples contained elevated uranium concentrations (216 mg/kg and 6,770 mg/kg). The remaining samples contained concentrations ranging between 2 to 6 mg/kg uranium (indicating background concentrations of uranium in the soil (Longmire, *et al.*, 1996)).

The excavation work in these catch-boxes indicated the uranium contamination was heterogeneously distributed. Due to extent of uranium found within the now former catch-boxes, the trenches were backfilled in 1994 and tarps were placed over the soil. The catch boxes were covered with high density polyethylene (HDPE) in 1995 as part of an Interim Action to prevent erosion of soil.

Area 6 was identified as Solid Waste Management Unit (SWMU) 33-007 in the Hazardous and Solid Waste Amendments (HSWA) Permit Table A and B and is now referred to as SWMU 33-007(c) (DOE 1992).

Preliminary environmental remediation sampling revealed the presence of uranium in the catch boxes. However, no beryllium, lead, or high explosives were detected in the catch boxes. Thus it was selected for a pilot-scale study of chemical leach removal of uranium.

CLEANUP REQUIREMENTS

Preliminary numerical modeling calculations were completed to determine the reduction of uranium concentration in the soil to meet regulatory requirements of concern. 10 CFR 834.305 states authorized limits for radionuclides in soil shall be derived using approved models for deriving soil criteria and ALARA (DOE, 1993). RESRAD (a DOE approved computer model "Residual Radioactive Material") was developed by the Argonne National Laboratory for the Department of Energy (DOE) for deriving soil criteria when conducting decontamination efforts. RESRAD was run by Field Unit Three personnel to meet the following requirements:

- Current DOE Order 5400.5 dose limit of 25 mrem/yr. (for a single source)
- Proposed EPA dose limit of 15 mrem/yr.

The calculations assumed recreational scenarios for a trail hiker and picnicker exposed for typical recreational time periods. The area of contamination was assumed to be 1,500 square meters and thickness of area was set at 3 meters. The highest concentration of uranium found in the catch boxes (6,770 mg/kg, 4,800 pCi/g) was used as the initial soil uranium concentration.

Results from the calculations indicate that a person hiking will receive an exposure of 198 mrem/yr., or picnicking by the pile (with the above dimensions) will result in an exposure of 639 mrem/yr. In order for the trail hiker not to exceed a dose limit of 15 mrem/yr., the uranium concentration of the soil must be reduced by a factor of 13 (to 369 pCi/g). In order for the picnicker not to exceed a dose limit of 15 mrem/yr., the uranium concentration of the soil must be reduced by a factor of 43 (to 114 pCi/g).

The following activities will produce a total dose of 15 mrem/yr. if the three isotopes are present in natural abundances:

$^{234}U = 58.6$ pCi/g
$^{235}U = 1.56$ pCi/g
$^{238}U = 54.1$ pCi/g

As a first approximation, the sum of the activities of the three isotopes needs to be less than 114 pCi/g. In order to meet the most conservative dose limit and scenario, the combined ThermoNuclean and containerized vat leach procedures must be capable of detecting and removing uranium contamination to as low as 114 pCi/g.

SOIL TREATMENT METHODS

The contents of the catch boxes were excavated and a total of 353 metric tons (~204 yd^3) of uranium-contaminated soil was processed use of the ThemoNuclean Services segmented gate system (SGS). (ThermoNuclean is a subsidiary of ThermoNutech, formerly ThermoAnalytical/Eberline). Of 353 metric tons 8.9 metric tons were diverted as being above 30 pCi/g ; this resulted in an initial waste volume reduction of 97.5%. The containerized vat leach system (CVL) system was used to leach uranium from the nine tons of diverted soil.

CONTAINERIZED VAT LEACH SYSTEM

The CVL process involves leaching uranium contaminated soils with 0.5 *M* $NaHCO_3$ (pH~8.3) to selectively dissolve the uranium.

Carbonate (CO_3^{2-}) and bicarbonate (HCO_3^-) leach has historically performed well to recover uranium (U) from ore (Merritt, 1971). More than one-third of the uranium mills operated in the United States have employed carbonate leaching circuits at one time or another. The use of carbonate heap leach requires the use of an integrated and closed circuit process, wherein the leach solutions are recycled and the reagents are reused.

Carbonate salt leach solution has two important roles. The formation of highly soluble anionic carbonate uranyl species, including uranyl dicarbonate ($UO_2(CO_3)_2^{2-}$) and uranyl tricarbonate ($UO_2(CO_3)_3^{4-}$), allows for high concentrations of uranium in a leachate solution. Secondly, carbonate salts are nearly selective for dissolution of uranium from uranium contaminated soils. Compounds of iron, aluminum, titanium, etc., are nearly insoluble in carbonate solution and are largely separated from the uranium during leaching. Oxidized uranium minerals are readily soluble in carbonate solutions. Other advantages of the carbonate leaching process include high solubility, the purity of the solution produced, the relative ease with which a uranium product can be precipitated directly from the leachate solution, and the relatively noncorrosive and safe handling characteristics of carbonate solutions.

From the chemistry of uranium, it is known that oxidized U (VI) in the form of uranyl ion (UO_2^{2+}) will be easily solubilized to a carbonate complex whereas U (IV) will be unaffected by the presence of carbonate. The stages of solubilization have been documented by Grenthe (1992).

$$UO_2^{2+} + CO_3^{2-} = UO_2(CO_3)^0 \quad (1)$$
$$UO_2^{2+} + 2HCO_3^- = UO_2(CO_3)_2^{2-} + 2H^+ \quad (2)$$
$$UO_2(CO_3)_2^{2-} + CO_3^{2-} = UO_2(CO_3)_3^{4-} \quad (3)$$
$$UO_2(CO_3)_2^{2-} + HCO_3^- = UO_2(CO_3)_3^{4-} + H^+ \quad (4)$$

The overall reaction for the dissolution of uranium oxide in bicarbonate solution to uranyl tricarbonate is as follows:

$$UO_2^{2+} + 3HCO_3^- = UO_2(CO_3)_3^{2-} + 3H^+ \quad (5)$$

The CVL process involves application of the bicarbonate solution to uranium-contaminated soil. This dissolves (leaches) uranium in the form of uranyl carbonate ion. The amount of uranium that is leached is primarily a function of the characteristics of the soil and the uranium speciation.

The bicarbonate solution is continually recycled through the soil and the soluble uranium removed onto ion exchange resins.

The overall leach process schematic is shown in Figure 3. Figure 3a is a photo of the leach process in at TA-33. The soil is leached in commercially available inexpensive containers manufactured by Rinchem Company, Inc. (Rinchem constructed the bags pursuant to design specifications from LANL). The bags are reusable.

The Containerized Vat Leach containers measure 0.91 m (1.0 yd.) in height, 0.91 m (1.0 yd.) in length, and 0.91 m (1.0 yd.) in depth for a volume capacity of 0.76 m^3 (1 yd^3). The containers are constructed of a woven polyethylene material and have a 2.95 metric

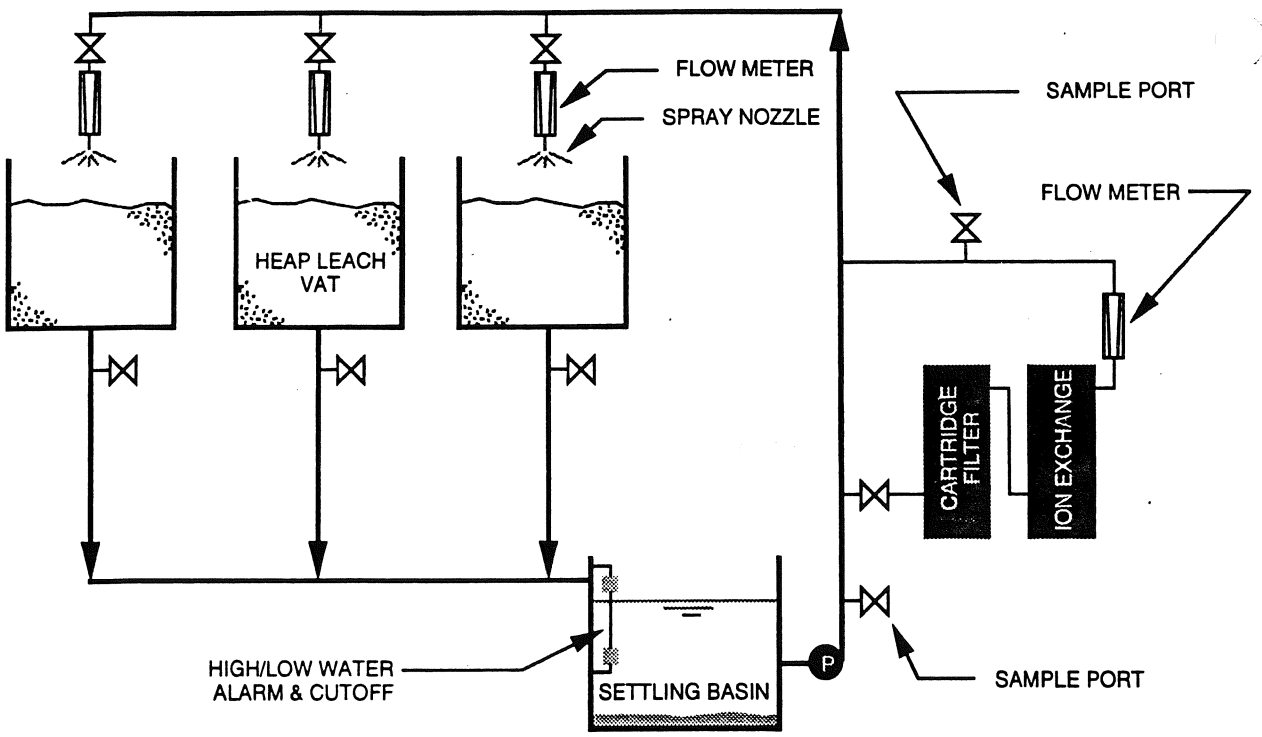


Figure 3. Schematic of the containerized vat leach (CVL) process (modifed from Mason, *et al.*, 1995).

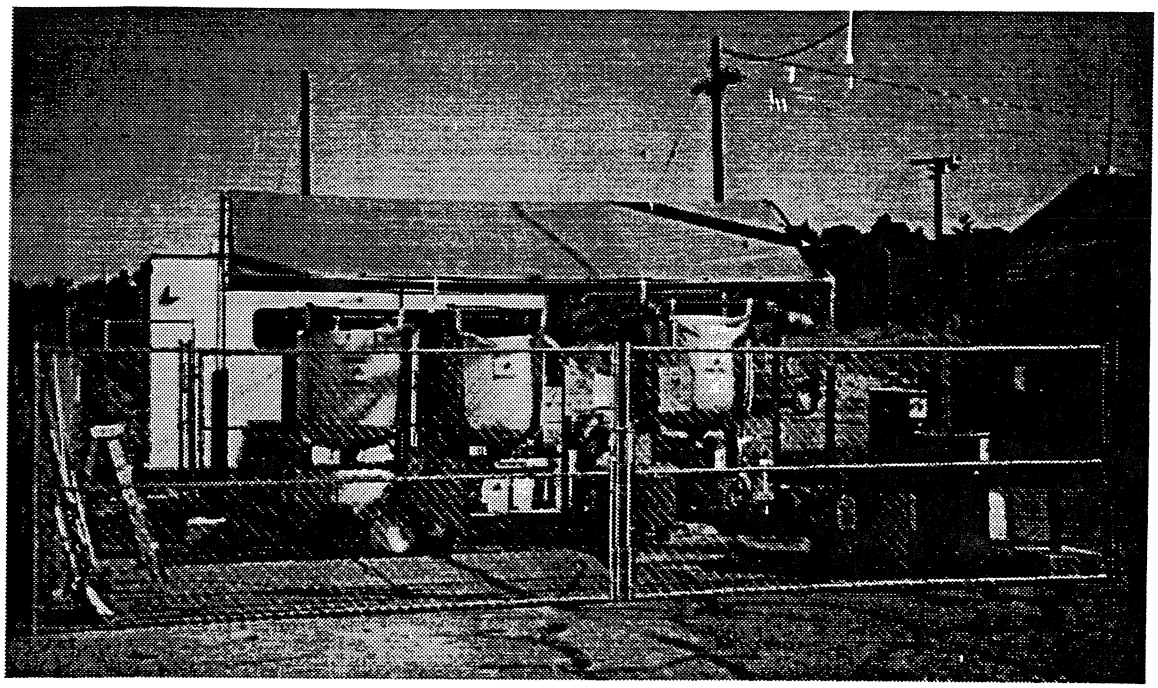


Figure 3a. Containerized vat leach (CVL) process at TA-33. August 14, 1996.

ton (6,500 lb.) capacity. The containers have a plastic liner. An elephant trunk 15 cm (6 in.) and 15 cm in diameter and 15 cm in depth is sown into the bottom of the container allowing liquids introduced to the container to drain out the bottom center of the bag. The elephant trunk also acts as a filter, as shown in Figure 4.

The CVL containers, once filled with soil, are hung from steel racks measuring 54 inches square and 94 inches in height. A platform (constructed of plywood) rests four inches from the bottom of the metal rack. The platform is for the leach solution reservoir that is placed under the CVL container. (See Figure 5.)

The soil was directly deposited in these bags after sorting by the segmented gate system (Figure 6). The soil was then treated with a solution of sodium bicarbonate distributed over the top of the bag with a drip irrigation system. The sodium bicarbonate solution was made by dissolving 8.640 kg of reagent grade sodium bicarbonate in 55 gallons of tap water (directly). After passing through the bag, and a 10 μm elephant trunk filter at the bottom of the CVL, the leach solution is pumped from the reservoir through two filters, a 10 μm filter and a 1 μm filter, before being recycled back to the top of the bag. Latterly, the leach solution was also being passed through an ion exchange column to remove the uranium solubilized as a carbonate complex.

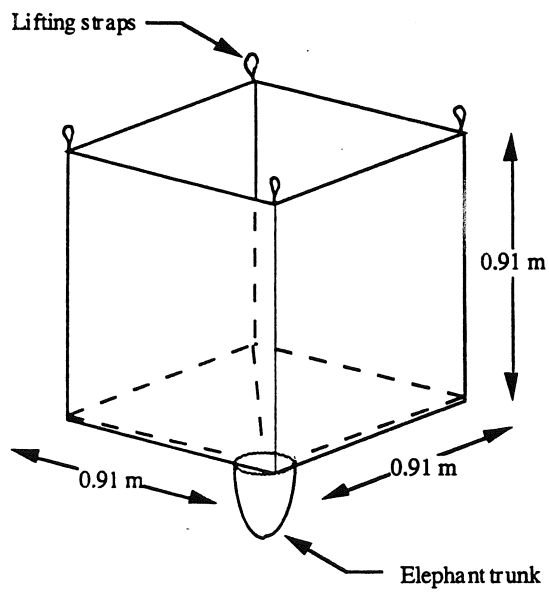


Figure 4. Details of containerized vat leach container. Container has a capacity of one yd^3 and is rated to hold 6,500 pounds of material. Leach solution applied to the soil exits the container through the porous elephant trunk.

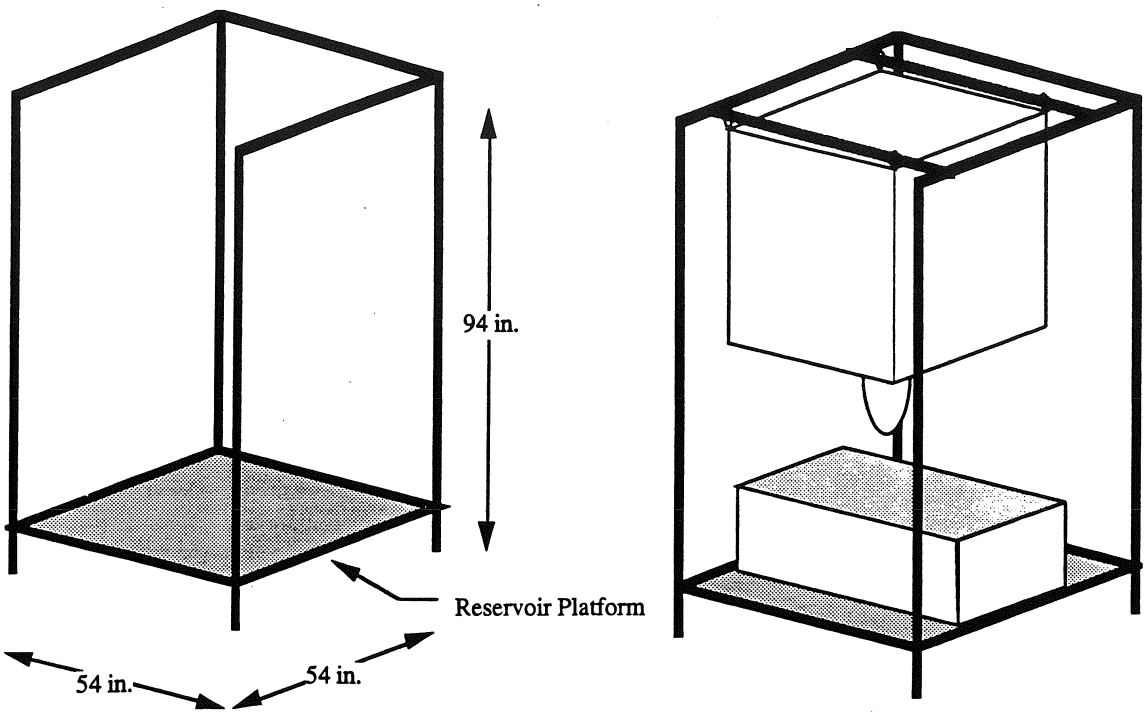


Figure 5. Diagrams of (1) metal rack on which containerized vat leach container is hung, and (2) CVL container hanging on rack with reservoir underneath.

Figure 6. Soil above 30 pCi/g was diverted by the segemented gate system (SGS) and diverted directly to the containerized vat leach (CVL) bags.

ANALYTICAL METHODS

•Alkalinity

Alkalinity analysis of leach samples was completed by use of a HACH Alkalinity Test Kit, model number 24443. The Test Kit is portable and allowed for alkalinity analysis to be conducted in the field.

•Gamma Spectroscopy

Gamma spectroscopy was used for determination of uranium concentrations in solid and liquid samples. The gamma counters utilized are generally high-purity germanium detectors with resolution of ~1-2 keV FWHM at 1000 keV. The counters are energy and efficiency calibrated using multi-line NIST traceable standards. Several geometry standards are used to allow for a variety of sample types and amounts. The counting data generated is analyzed by two computer analysis codes, RAYGUN and SPECANL.

The data provided for the determination of ^{235}U was based on the 185.7 keV emission of ^{235}U. It has been determined that any potential contribution by the 186.1 keV ^{226}Ra would be less than two percent in any worst case. The ^{238}U activity was based on the 1001 keV emission of ^{234m}Pa. It is understood that any uranium separation would disturb the equilibrium between this isotope's parent (^{234}Th, $t_{1/2}$ = 24 day) and ^{238}U.

•Inductively Coupled Plasma - Atomic Emission Spectra

Uranium concentration in the aqueous solutions were analyzed using a Varian Liberty 200 Inductively Coupled Plasma atomic emission spectrophotometer (ICP-AES). Triplicate measurements at one uranium absorbance line (409 nm) were taken for each sample. In some cases, other elements (Ca, Al, U, Fe, Mg, K, Si, and Pb) were also measured to observe the extent of other metals that may have gone into solution during the leach process. (As the leach solution was comprised of 0.5 *M* $NaHCO_3$ ([Na^+] ~ 11,500 ppm) and was thus "swamped" with sodium, sodium was not analyzed for).

•X-Ray Diffraction

Solid samples of uranium, both before and after leaching, were submitted for x-ray diffraction analysis to identify the mineralogical speciation of uranium. X-ray diffraction analysis on the solid mineral phases was performed with a computer-controlled INEL CPS-120 X-Ray Powder Diffractometer (INEL, Inc., Stratham, NY).

•Scanning Electron Microscope

In order to examine surface effects, scanning electron microscope (SEM) photographs were taken, pre- and post-leach. The SEM is an ISI Model SX-40 (International Scientific Instruments, TOPCON Tokyo Optical Company, Japan) with secondary electron and

back-scattered electron detection. It has been equipped with a Tracor Northern Model 5400 X-ray microanalysis unit. With geologic materials, the maximum magnification is about 6000 x.

•Kinetic Phosphorescence Analysis

Automatic KPA-11 by Chemcheck Instruments, Inc., Richland, WA, was used for aqueous uranium analysis by kinetic phosphorimetry (kinetic phosphorimetry analysis - KPA). When KPA was used, uranium concentration was determined based on concentration-intensity curves determined from calibration standards at regular intervals. Calibration standards were prepared by diluting stock uranium solution in a bicarbonate/carbonate solution of an appropriate concentration to minimize matrix effects.

•pH

Aqueous solution pH measurement was accomplished by use of an Orion Model 270A pH meter.

•Soil Preparation for KPA and pH Analysis

Schoepite ($UO_3 \cdot 2H_2O$) recovered from the site was ground to pass a 50 mesh sieve (0.3 mm). Approximately 0.5 g of ground mineral sample was added to 15 mL quantities of 0.5 *M* $NaHCO_3$ and the mineral-solution mixtures were shaken on a reciprocating mechanical shaker (at 200 rpm) at constant temperature (25°C). Upon sampling, the mixtures were centrifuged at 5,000 rpm for 10 min. and the supernatant was decanted. 0.5 *M* $NaHCO_3$ solution was added and the solution + remaining mineral sample was equilibrated on the mechanical shaker (200 rpm) until the next sampling period. Several leachate solution samples were taken over a period of 420 min. and care was taken to determine the amount of carryover from one equilibration to another. The dissolved uranium concentration in the leachate solutions was determined by KPA. Solution pH values were measured for each of the leaching solutions.

ION EXCHANGE EXPERIMENTS

Treatment of these liquors is important to reduce the volumes of the waste, and to recover and to recycle the bicarbonate reagent. Anion-exchange resins and some chelating resins are effective absorbers for removing U from the bicarbonate leach liquors of TA-33 site soil. In site scaleup experiment, U was successfully removed from 210 liters of U-bicarbonate leach liquors using an anion-exchange column system of Ionac™SR-3 resin.

RESULTS

Table 1 shows the characterization of soil from a site at TA-33 that is similar to that studied in the pilot treatment study.

Eight containers, each containing approximately 1 ton of contaminated soil were processed. Table 2 shows the initial amounts and radioactivity supplied for CVL treatment by the SGS system.

Uranium solids present in the soils were also analyzed. The soil contained several easily observed yellow solids. Many of these friable solids were large in size (up to 4 cm in diameter) and occasionally contained small gray to black striations throughout them. Most of the uranium solids were bright yellow in color. A large intact uranium solid was separated from the uranium-contaminated soil, ground to a fine powder and characterized by XRD. Figure 7 shows the XRD powder pattern for the mineral sample – identified as

Table 1. Characterization of representative contaminated soils.

pH = 6.9	Equilibrium Geochemical Modeling of Leach solutions
Gravel = 46.4%	2 to 4 times undersaturated w.r.t. schoepite
Sand = 38.3%	Major uranium species: $UO_2(CO_3)_2^{2-}$, $UO_2(CO_3)_3^{4-}$
Silt = 9.3%	Moisture content = 9.8%
Clay = 6.0%	
Organic Matter = 1.6%	
X-ray-diffraction: schoepite – $UO_3 \cdot 2H_2O$	
Neutron activation analysis – 17,141 mg U / kg	

Table 2. Summary of Totals of Soils Processed by ThermoNucleans's Segmented Gate System (soils diverted had a minimum activity of 30 pCi/g).

Date	Total Mass processed (kg)	Mass Diverted to CVL (kg)	Total Activity Diverted to CVL (pCi)
4/25/96	45,151	240	4.99 E+07
4/26/96	23,927	93	3.95 E+06
5/1/96	43,529	108	4.53 E+06
5/2/96	43,799	1,007	1.34 E+08
5/3/96	41,637	3,826	7.76 E+08
5/6/96	67,997	3,101	5.33 E+08
5/7/96	85,923	485	1.08 E+07
5/8/96	22,170	647	3.47 E+07
5/9/96	7,976	216	1.28 E+07
TOTALS	383,109	9,723	1.56 E+09

schoepite ($UO_3 \cdot 2H_2O$). The narrowness of peaks and the similarities to characterized schoepite indicate the solid is fairly pure.

In 0.5 *M* $NaHCO_3$ solution, schoepite was fairly soluble with respect to uranium dissolution. Schoepite is more soluble than other minerals such as autunite ($Ca(UO_2)_2(PO_4)_2 \cdot 10H_2O$) and soddyite ($(UO_2)_2SiO_4 \cdot 2H_2O$) partially because of the simplicity of its structure. In soil minerals, phosphate groups (in autunite) and silicate groups (in soddyite) often exist as repeating units or "sheets" of tetrahedral group linkages. These linkages are not highly prone to dissolution and when present, are known to influence mineral weathering rates. Hence, uranium (VI) solids such as schoepite, which do not have phosphate and silicate tetrahedrals in their structures, are likely to be more soluble than minerals which have phosphate and silicate groups in their structures. Figure 8 shows uranium dissolution for schoepite mineral sample in 0.5 *M* $NaHCO_3$ with respect to time.

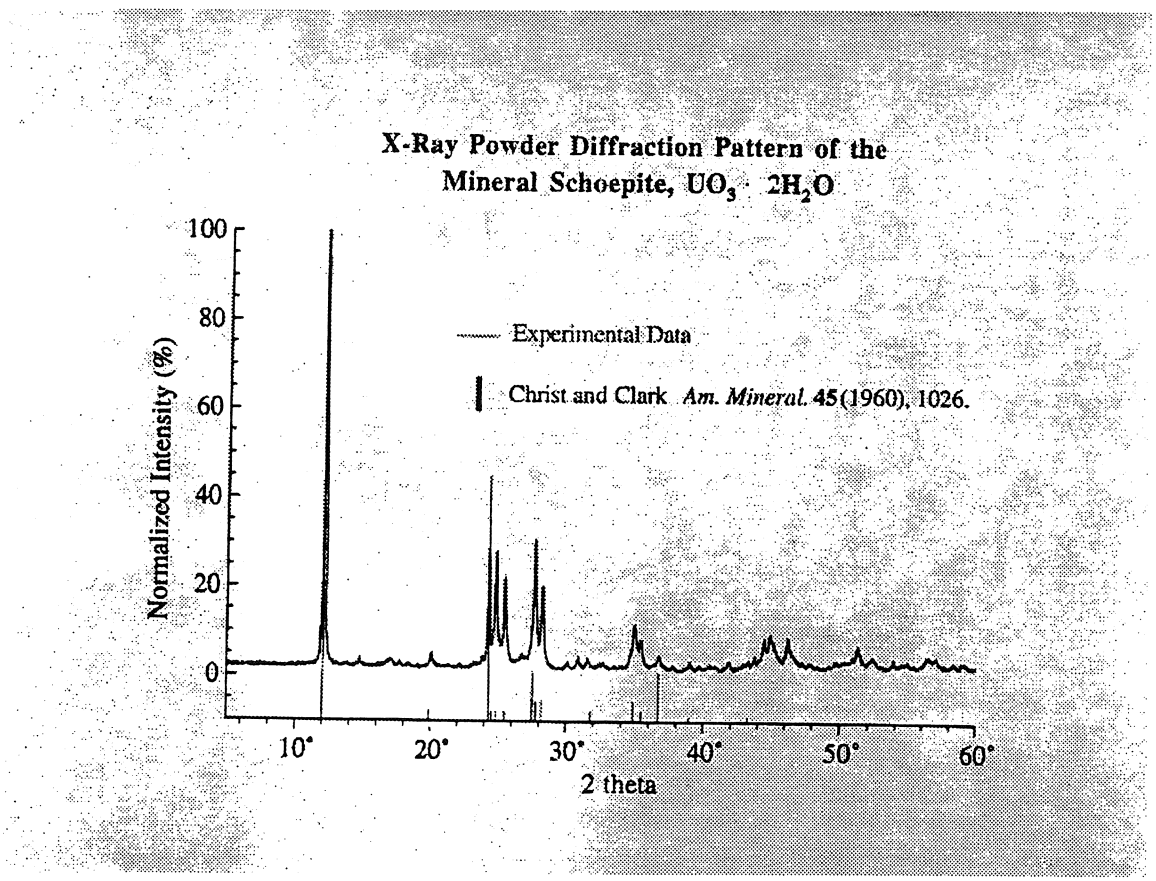


Figure 7. The XRD powder pattern for the mineral sample – identified as schoepite ($UO_3 \cdot 2H_2O$). The narrowness of peaks and the similarities to characterized schoepite indicate the solid is fairly pure.

Figure 9 is an SEM backscattering electron (BSE) image of TA-33 schoepite. The mineral is fairly crystalline as evidenced by the presence of numerous platy-shaped formations. Note the black striations that are not holes or cracks, but true representations of the particle surface. The striations are dark, low electron density portions which *may* be thick layers of water molecules (waters of hydration) lying between layers of uranium and oxygen atoms.

Figure 10 is an SEM photograph of the schoepite after leaching with 0.5 *M* $NaHCO_3$. The dispersion of the schoepite platy-shaped particles can be seen as a result of the leaching action of bicarbonate. The edges of the particles are also smoother and some platy-shaped particles have small depressions or pits, suggesting chemical leach proceeded.

Results for CVL_3 Container

The first containerized vat leach container processed was denoted as CVL_3 and was processed singularly (later bags were processed in tandem) Samples were taken at regular intervals, the leach solution analyzed for pH and uranium (by KPA). These results are shown in Table 3.

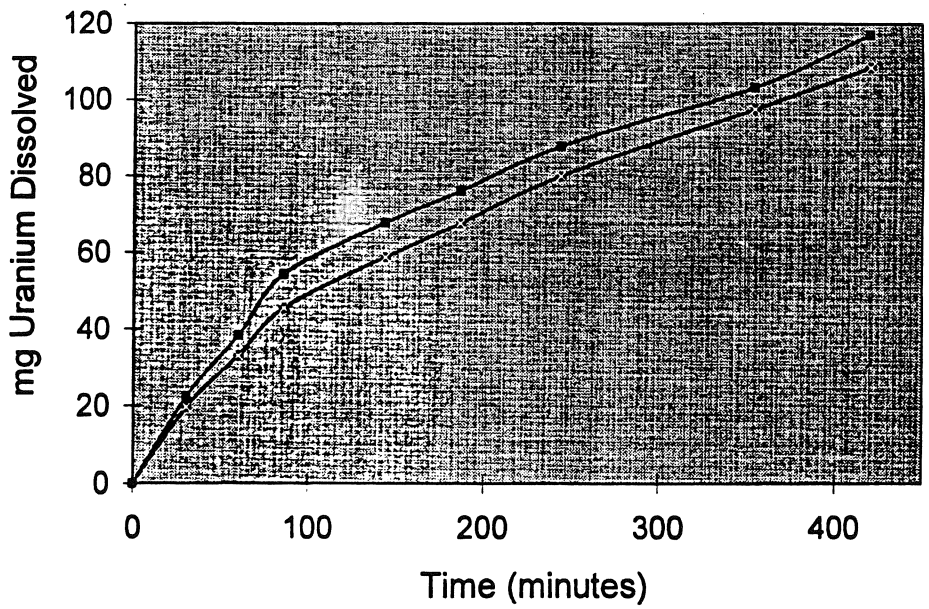


Figure 8. Uranium dissolution for (schoepite, $UO_3 \cdot 2H_2O$) mineral sample in 0.5 *M* $NaHCO_3$ with respect to time.

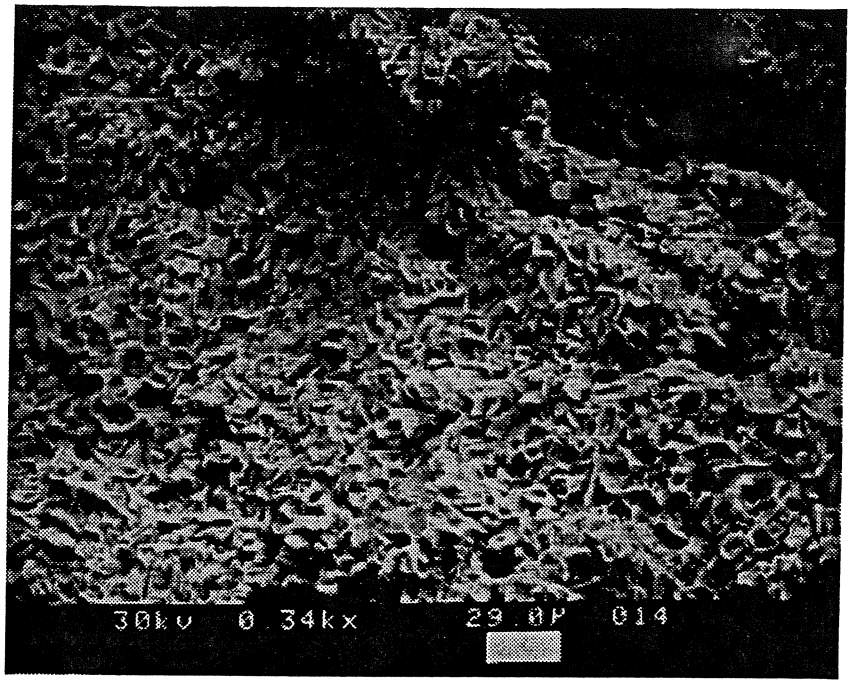


Figure 9. An SEM back-scattering electron (BSE) image of TA-33 schoepite ($UO_3 \cdot 2H_2O$). The mineral is fairly crystalline as evidenced by the presence of numerous platy-shaped formations. Note the black striations that are not holes or cracks, but true representations of the particle surface. The striations are dark, low electron density portions which *may* be thick layers of water molecules (waters of hydration) lying between layers of uranium and oxygen atoms.

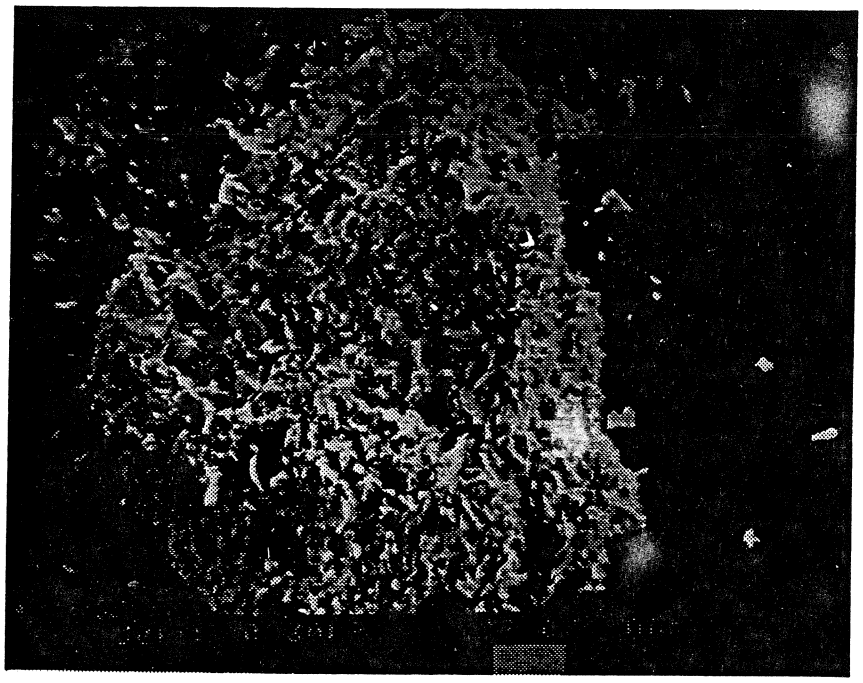


Figure 10. An SEM photograph of the schoepite after leaching with 0.5 *M* $NaHCO_3$. The dispersion of the schoepite platy-shaped particles can be seen as a result of the leaching action of bicarbonate. The edges of the particles are also smoother and some platy-shaped particles have small depressions or pits, suggesting chemical leach proceded.

When the majority of the uranium had been removed, the soil itself was sampled for residual uranium. The soil was analyzed by use of gamma spectroscopy. These results are shown in Table 4. Nine samples were taken: top , middle and bottom center, top, middle and bottom side, and corner top, middle, and bottom of the CVL. The results of the soil analysis post leach indicted uranium concentrations of ~46 p/Ci from ^{234}U, ~2.02 pCi/g from ^{235}U, and ~46 pCi/g from ^{238}U, for a total uranium activity of 94 pCi/g.

Following the leach of CVL_3 solid pieces of schoepite 6 mm in diameter or larger were observed in the top 50 mm of the soil in the CVL. It was decided that future soils to be leached in the CVLs would be sieved first, sieving out the larger than 1 mm fraction. The

Table 3. Leach results from CVL bag No. 3 (denoted CVL3).

Sample Identificaiton No.	Time (days)	pH	Alkalinity (mg/L)	mg U / L (KPA)
CVL3-051496_02	0	8.83	42,060	3.71
CVL3-051496_10	3	8.79	41,960	136.23
CVL3-051496_16	6	8.67	42,110	188.52
CVL3-051496_22	9	8.59	42,010	281.06
CVL3-051496_28	12	9.01	41,890	333.10
CVL3-051496_34	15	9.01	42,170	443.58
CVL3-051496_40	18	8.77	41,990	578.21
CVL3-051496_46	21	8.70	41,790	484.29

Table 4. Post Leach Analyses for uranium from CVL_3 (all concentrations are in pCi/g).

Identification number	Position	^{234}U	^{235}U	^{238}U	TotalActivity
CVL_3_01	top-center*	***20	0.90	***21	42
CVL_3_02	top-corner*	***51	2.25	***52	105
CVL_3_03	top-side center*	***37	1.64	***38	77
CVL_3_04	middle-center*	***59	2.62	***60	122
CVL_3_05	middle-corner*	***57	2.52	***58	128
CVL_3_06	middle-side center*	***56	2.47	***57	115
CVL_3_07	bottom-center*	***56	2.49	***57	115
CVL_3_08	bottom-corner*	***46	2.04	***47	95
CVL_3_09	bottom-side center*	***49	2.17	***50	101
CVL_3_PA1	top-center**	45.5	2.02	45.7	93.22
CVL_3_PA2	bottom-center**	42.1	1.85	42.9	86.85

*Analyzed by CST-11

**Analyzed by Paragon Analytical, Ft. Collins, Colorado.

***^{234}U and ^{238}U were not directlyl analyzed for, and calculated concentrations are shown.

remaining less than 6 mm fraction of the soil would be chemically leached in the CVLs. CVLs that were leached concurrently with CVL_3 were also sieved post leach, removing the larger than 6 mm fraction, and thus a large amount of the remaining radioactivity in the soil.

Results from all the CVL Containers

Besides CVL_3, of the six CVL containers leached to completion to date, two (CVL_1 and CVL6) met the DOE criteria for release (114 pCi/g). Both of the containers were pre-sieved, sieving out pieces of shoepite that were larger than 6 mm in diameter, and were both leached for 15 days each. There was a 97 percent and 90 percent reduction, respectively, of uranium in the soils in the containers by the pre-sieve and leach process. CVL_4 nearly met the criteria (117 pCi/g) after 18 days of leach, resulting in a 67 percent reduction of uranium-contamination. CVL_4 was not pre-sieved. CVL_2 and CVL_5 were both above the release criteria (171 pCi/g and 123 pCi/g, respectively). Reduction of uranium contamination was 86 percent and 91 percent, respectively, in these two containers. CVL_2 was leached for 14 days, and was sieved for shoepite larger than 6 mm following the leach process. CVL_5 was leached for ten days and was sieved following leach. Percent uranium reduction in CVL_2 and CVL_5 was 86 percent and 91 percent, respectively. The other three CVL containers (CVL_7, CVL_8, and CVL_9) were all pre-sieved and are currently still undergoing the leach process. These results are shown in Table 6.

Filters and Sludge from CVL 3 Bag.

Table 5 shows the weight of solids collected on the elephant filter, the sludge at the bottom of the leach collector container, and the two filters - 10 µm and 1 µm.

ION EXCHANGE FOR URANIUM REMOVAL FROM LEACH SOLUTIONS

The initial sodium bicarbonate leach liquors collected from vat leach bag CVL_3 had pH 9.82 and contained 1,292 ppm uranium, 130 ppm Si, 76 ppm Mg, 85 ppm K, 19.8 ppm Ca, 5 ppm Pb, and <0.1 ppm of Al and Fe (Table 7). During the leaching process, the

Table 5. Weights and Activity Found on Filters and Sludge.

Filter Type	Weight (g)	Activity (pCi)
elephant filter	57	0.5 E6
sludge	385	15.0 E6
10 µm filter	245	6.0 E6
1 µm filter	136	1.0 E6

Table 6. Results of pre and post leach gamma analysis on CVL contianers.

ID No.	Pre- or Post	~U234 (pCi/g)	~U235 (pCi/g)	~U238 (pCi/g)	~Total Activity (pCi/g)	Sieve Status	Leach Time (days)	% Uranium Reduction
CVL1_PRE*	Pre-	970	26.55	970	1,967			
CVL1_POST*	Post-	32	1.34	32	65	Pre-sieved	15	97
CVL2_PRE*	Pre-	621	20.33	621	1,262			
CVL2_POST	Post-	83	4.66	83	171	Post-sieved	14	86
CVL3_PRE	Pre-							
CVL3_POST*	Post-	42, 46	1.85, 2.02	42, 46	86, 94			
CVL3_POST**			2.17			Post-sieved	21	
CVL4_PRE*	Pre-	172	5.19	172	349			
CVL4_POST*	Post-	57	2.86	57	117	Post-sieved	18	67
CVL5_PRE*	Pre-	685	17.81	685	1388			
CVL5_POST*	Post-	61	1.02	61	123	Post-sieved	10	91
CVL6_PRE*	Pre-	390	11.38	390	791			
CVL6_POST*	Post-	40	1.81	40	82	Pre-sieved	15	90
CVL7_PRE*	Pre-	777	30.36	777	1584		in progress	
CVL7_POST	Post-					Pre-sieved (started 8/12/96)		
CVL8_PRE*	Pre-	2261	51.37	2261	4573		in progress	
CVL8_POST	Post-					Pre-sieved (started 8/14/96)		
CVL9_PRE	Pre-	3825	83.75	3825	7734		in progress	
CVL9_POST	Post-					Pre-sieved (started 8/16/96)		

* Analysis completed by Paragon Analytic

** Analysis completed by CST-11

liquors were continuously passed through a filter system including one of 10 μm deionization system filter column and one 1 μm filter column. The concentration of U, Ca, Mg, K and Si were reduced by filtration. This filtered solution was used in the ion exchange studies including batch and column experiments.

Plastic columns, capacity ~ 3 L, were used to construct a multi-column system. Four columns were installed in series (Figure 11). At the bottom of each column, a filter pare (20 μm mesh) and a mesh screen (<0.5 mm mesh) were placed on top of a porous plane to support the resin beads. About 2 L of anion-exchange resin, Ionac™ SR-3 (resin bead size of 200 μm to 500 μm) were placed into each column. The filtered uranium-bicarbonate leach liquors were introduced into the columns at a flow rate of 1,700 ml/h. After about 210 liters of the leach liquors were passed through the column system, the experiment was terminated.

Uranium, and other selected ion, concentrations in the solutions of pre- and post-ion exchange column were determined by KPA and are shown in Table 7. The percentage of uranium absorbed by Ionac™ RS-3 was calculated using equation (6).

$$\% \text{ removal of U} = [(U_i - U_p)/U_i]*100\% \qquad (6)$$

Generally, after 210 liters of uranium-bicarbonate leach liquors were passed through 4 columns of Ionac™ SR-3 resin, about 95.3% of uranium in the leach liquors was re-

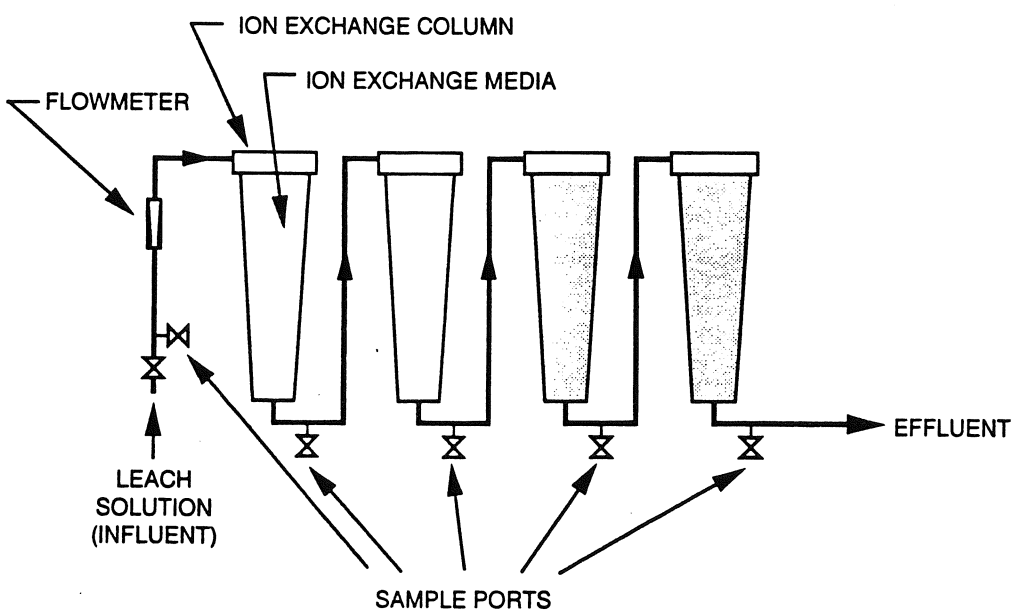


Figure 11. Ion exchange system schematic.

Table 7. The concentrations of selected elements in the bicarbonate leach liquors from vat leach bag CVL-3

Elements	Pre-filtration (ppm)	Post-filtration (ppm)
U	1292.6	553.3
Ca	19.8	9.5
Mg	76.3	56.7
K	84.6	59.8
Al	0.064	0.061
Fe	0.013	0.012
Pb	4.9	4.5
Si	130.4	106.9

moved by the resin. The site scaleup column test shows that the Ionac™ SR-3 is a effective sorbent to remove uranium from relative large amounts of the bicarbonate leach waste stream of the soil. Lu (Lu, *et al*, 1996) describes in detail the results and analysis of the entire ion exchange experiments.

EXPERIMENTS AND ANALYSIS IN PROGRESS

Several experiments and sample analysis are currently underway concerning the containerized vat leach process:

1. Expansion of Leach Time

CVL_7, CVL_8, and CVL_9 continue to be leached at TA-33. Projected total time of leach will be about three months. At the conclusion of three months the efficiency of the leach process will be evaluated. A determination of the leach process to remediate the soil to uraniun concentrations that are as low as resonably achieable (ALARA). It will also be determined if those final uranium concentrations are approaching background uranium concentrations of uncontaimanted soils.

2. Surface Dissolution Analysis

Investigations of surface dissolution rates and surface areas continue. These results should give a more precise evaluation of the leach time needed for dissolution of solid uranium minerals (*i.e.* schoepitea and uraninite) in soils. The observation of the large pieces of remaining schopeite in the leached CVL containers and the subsequent sieving was a qualitative measure. The surface dissolution experiments should yield quantitative data.

3. Transport Modeling

Nine soil cores were recovered from CVL_6. The cores were taken from the center of the CVL (one core) , the sides of the CVL (four cores), and the corners of the CVL (four cores). The cores were subdivided into five segments each (for a total of 45 segements) for uranium analysis. This analysis should yield information relating to transport phenomena within the CVL, of both leach solution and solubilized uranium.

Addition of sodium chloride (NaCl) to the 0.5 *M* $NaHCO_3$ leach solution is planned. Chloride analysis of effluent from the CVL will yield leach solution residence time, and hydraulic characteristics of the leach solution flow through a CVL.

4. Oxidation State of Uranium Minerals in Soil

As observed in the SEM photographs, not all the oxidized uranium solids in the soil was necessarily in the U(VI) state, but in the less oxidized U(IV) state. Other phases and constituency of uranium minerals may exist in the soil. XRD and SEM will be used to further evaluate those solids.

To determine if any improvement of chemical leach characteristics may be gained by use of a solid phase oxidant, sodium peroxide (Na_2O_2) was added to leached CVL_5 soil and will be releached and reanalyzed.

SUMMARY

Of the six CVL containers leached to completion to date, three met the DOE criteria for release (114 pCi/g). Both of the containers were pre-sieved, sieving out pieces of shoepite that were larger than 6 mm in diameter, and were both leached for 15 days each. There was a 97 percent and 90 percent reduction, respectively, of uranium in the soils in the containers by the pre-sieve and leach process. CVL_4 nearly met the criteria (117 pCi/g) after 18 days of leach, resulting in a 67 percent reduction of uranium-contamination. CVL_4 was not pre-sieved. CVL_2 and CVL_5 were both above the release criteria (171 pCi/g and 123 pCi/g, respectively). Reduction of uranium contamination was 86 percent and 91 percent, respectively, in these two containers. CVL_2 was leached for 14 days, and was sieved for shoepite larger than 6 mm following the leach process. CVL_5 was leached for ten days and was sieved following leach. Percent uranium reduction in CVL_2 and CVL_5 was 86 percent and 91 percent, respectively. The other three CVL containers (CVL_7, CVL_8, and CVL_9) were all pre-sieved and are currently still undergoing the leach process.

Ion exchange resin was used to treat about 100 L of leach solution, removing about 50 percent of the uranium from the leach solution. Ionac™ SR-3 ion exchange resin was used to treat about 180 L of leach solution, resulting in 95.3 percent recovery of uranium from the leach solution onto the resin.

RECOMMENDATIONS

When working with soils containing solid pieces of uranium-oxides, it is recommended that the soils be pre-sieved, separating out the larger than 6 mm diameter fraction. This physical treatment will result in a stream of material of that will contain large radioactivity from particles of uranium-oxide larger than 6 mm. The smaller than 6 mm fraction, although greatly reduced in radioactivity but still greater than the release criteria, may then be treated chemically to reduce the uranium-contamination to less than the release criteria dictated by the RESRAD release criteria model.

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GLOSSARY

Al	aluminum
ALARA	As Low As Reasonably Achievable
alkalinity	measure of capacity to neutralize strong acid
autunite	$Ca(UO_2)_2(PO_4)_2 \cdot 10H_2O$
BSE	back-scattered electron
Ca	calcium
carbonate	CO_3^{2-}
$Ca(UO_2)_2(PO_4)_2 \cdot 10H_2O$	autunite
cm	centimeter
CO_3^{2-}	carbonate
CVL	Containerized Vat Leach
DOE	Department of Energy
ER	Environmental Restoration
Fe	iron
HCO_3^-	bicarbonate ion
H_2CO_3	carbonic acid
HDPE	high density polyethylene
HSWA	Hazardous and Solid Waste Amendments
ICP-AES	inductively coupled plasma atomic emission spectra
ICP-MS	inductively coupled plasma mass spectrometry
K	potassium
K_d	hydraulic conductivity
KPA	kinetic phosphorimetry analysis
L	liter
LANL	Los Alamos National Laboratory

m	meter
M	molar
Mg	magnesium
mL	milliliter
nm	nanometer
NaCl	sodium chloride
$NaHCO_3$	sodium bicarbonate
Na_2O_2	sodium peroxide
Pb	lead
pH	$-\log_{10}[H^+]$
ppm (aqueous)	parts per million = mg/L
ppm (solids)	parts per million = mg/kg
schoepite	$UO_3 \cdot H_2O$
SGS	Segmented Gate System
Si	silica
soddyite	$(UO_2)_2SiO_4 \cdot 2H_2O$
SWMU	Solid Waste Management Unit
TA-33	Technical Area 33
TA-33-16	Technical Area 33, Building 16
^{235}U, ^{234}U, and ^{238}U	isotopes of U comprising "natural uranium"
U^{4+}	tetravalent uranium
U^{6+}	hexavalent uranium
U(IV)	tetravalent uranium
U(VI)	hexavalent uranium
UO_2	uraninite
$(UO_2)_2SiO_4 \cdot 2H_2O$	soddyite
$UO_3 \cdot H_2O$	schoepite
uraninite	UO_2
XRD	x-ray diffraction analysis
yd^3	cubic yard
µm	micron ($=10^{-6}$ m)

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APPENDIX C

WASTE-FORM DEVELOPMENT FOR CONVERSION TO PORTLAND CEMENT AT LOS ALAMOS NATIONAL LABORATORY TECHNICAL AREA 55

LA-13125

Hard copies of this document were provided to
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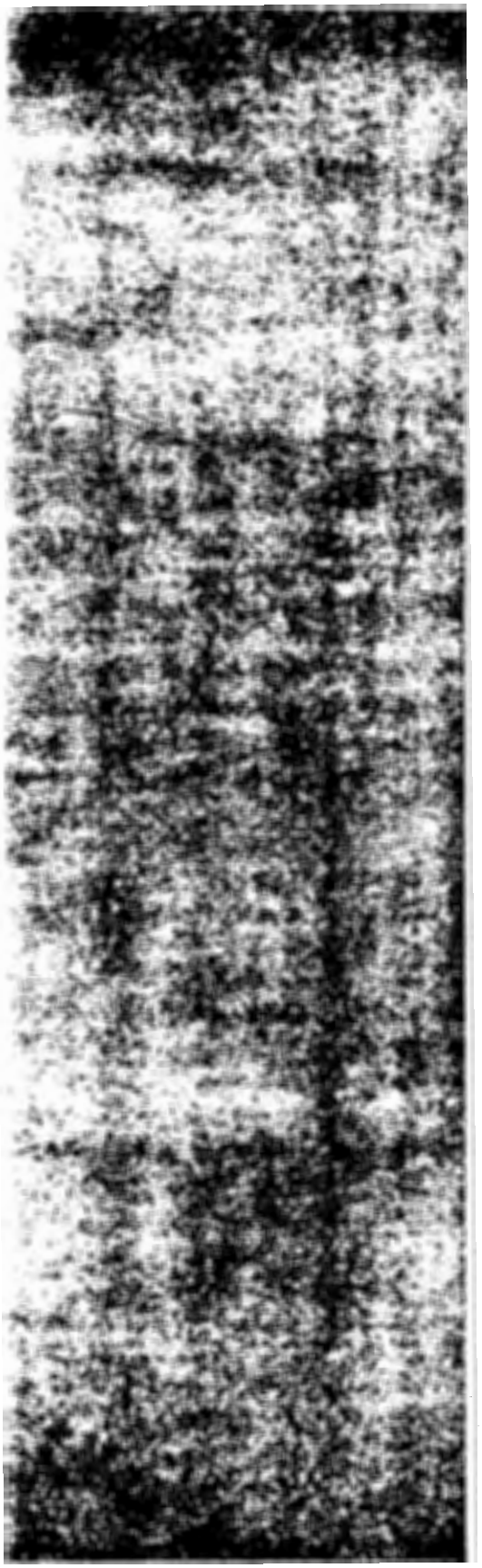
LA-13125



Waste-Form Development for Conversion to Portland Cement at Los Alamos National Laboratory (LANL) Technical Area 55 (TA-55)

Los Alamos
NATIONAL LABORATORY

Los Alamos National Laboratory is operated by the University of California for the United States Department of Energy under contract W-7405-ENG-36.

WASTE-FORM DEVELOPMENT FOR CONVERSION TO PORTLAND CEMENT AT LOS ALAMOS NATIONAL LABORATORY (LANL) TECHNICAL AREA 55 (TA-55)

by

G. W. Veazey, A. R. Schake, P. D. Shalek, D. A. Romero, and C. A. Smith

ABSTRACT

The process used at TA-55 to cement transuranic (TRU) waste has experienced several problems with the gypsum-based cement currently being used. Specifically, the waste form could not reliably pass the Waste Isolation Pilot Plant (WIPP) prohibition for free liquid and the Environmental Protection Agency (EPA)-Toxicity Characteristic Leaching Procedure (TCLP) standard for chromium. This report describes the project to develop a Portland cement-based waste form that ensures compliance to these standards, as well as other performance standards consisting of homogeneous mixing, moderate hydration temperature, timely initial set, and structural durability. Testing was conducted using the two most common waste streams requiring cementation as of February 1994, lean residue (LR)- and oxalate filtrate (OX)-based evaporator bottoms (EV). A formulation with a pH of 10.3 to 12.1 and a minimum cement-to-liquid (C/L) ratio* of 0.80 kg/l for OX-based EV and 0.94 kg/l for LR-based EV was found to pass the performance standards chosen for this project. The implementation of the Portland process should result in a yearly cost savings for raw materials of approximately $27,000 over the gypsum process.

INTRODUCTION

TA-55 houses a variety of operations related to plutonium processing. The intended destination of the TRU wastes generated from these operations is the WIPP repository. Accordingly, these wastes must be in compliance with the WIPP-Waste Acceptance Criteria (WAC).[1] The majority of the wastes at TA-55 meet the WIPP-WAC in their initial state. However, particulates# and free liquids (aqueous and nonpolar organic) require further treatment to meet the WIPP criteria designed to reduce respirability and dispersibility of these wastes. The purpose of the TA-55 cementation operation is to convert these particulate and liquid wastes to a solid, cohesive form that meets the WIPP-WAC.

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* The C/L ratio is used in this report to express the relationship between the cement weight and total liquid volume. The liquid includes the water and ionic components used to prepare the surrogate waste and the NaOH solution used for pH adjustment. In order to remain historically consistent with TA-55 operations, this ratio is used instead of the dimensionless water-to-cement ratio commonly used in the cement industry.

This work was performed under Revision 4 of the WIPP-WAC, which required the immobilization of particulates. Since then, Revision 5 of the WIPP-WAC has been issued. Revision 5 does not require the immobilization of particulates although it does not prohibit their immobilization.

Initially, Portland cement was used as the fixation agent in the TA-55 process, but in 1983 a gypsum/polymer-based cement named Envirostone* was substituted. Envirostone's setting reaction involves the hydration of hemihydrated calcium sulfate ($CaSO_4 \cdot 1/2H_2O$) to the dihydrated form ($CaSO_4 \cdot 2H_2O$). The polymer component serves to reduce permeability within the solidified waste form.[2] There were three reasons for the conversion to Envirostone. First, Envirostone was more compatible with TA-55's nonpolar organic liquid waste. Second, while Portland's setting reaction required an alkaline pH, Envirostone's was compatible with an acidic pH. Since the EV waste is highly acidic, less caustic solution was therefore needed with Envirostone for pH adjustment. Third, the lower pH required for Envirostone reduced the ammonia-based fumes that had previously obscured visibility for the process operator.#

Envirostone served TA-55's cementation needs well until the cementation operation came under the jurisdiction of the more stringent WIPP-WAC and the EPA-Resource Conservation and Recovery Act (RCRA) regulations concerning characteristic toxicity.[3] It then became apparent that Envirostone exhibited several deficiencies that outweighed the benefits of its use. These deficiencies related to free-liquid generation in violation of the WIPP-WAC restriction on such and failure to meet the EPA-RCRA standard for leaching resistance for chromium. Portland cement was found to be a superior performer in both of these areas.[4,5] This report will review the investigation into the performance of Portland cement and describe the development of a Portland cement waste form to address the problems associated with Envirostone.

RATIONALE FOR PURSUING PORTLAND CONVERSION

Radiolytic Free-Liquid Generation: The first inadequacy with Envirostone became apparent in 1989 with the discovery that Envirostone cemented waste forms were generating free liquid several weeks after cementation.[6,7] This liquid generation resulted in the decertification of the TA-55 cementation process to produce waste forms acceptable for WIPP. A significant effort was subsequently initiated to find the cause of and arrest the phenomenon.[7-12] Several mechanisms capable of producing liquid were investigated and discarded.[9,12,13] These included the occurrence of a polymerization reaction having H_2O as a by-product and reversal of the calcium sulfate hydration reaction+. Polymerization was eliminated when Envirostone without the polymer component (plaster of Paris) was found to likewise generate free liquid. Dehydration was eliminated when x-ray powder diffraction analysis found no change in the $CaSO_4$ molecule following free-liquid generation.

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* Envirostone is a trademark of the United States Gypsum Company.

\# The NH_4 content of the waste streams has decreased since the previous Portland operation to the extent that fuming should no longer be excessive at a high pH.

\+ The type of polymerization reaction taking place in Envirostone was unknown because the identity of the polymer component was proprietary. However, if the reaction was a condensation-type polymerization, H_2O would be generated as a by-product. If the cement had already hardened when the polymerization took place, the H_2O may have be seen as free liquid.

The free-liquid mechanism was ultimately shown to be irradiation-induced when it was found that cemented surrogate-waste samples subjected to gamma irradiation produced free liquid, while unirradiated samples did not.[7,12,13] A phenomenon that is compatible with this finding was radiolysis of the water in the pores of the cement structure to produce H_2.[14-16] It was conceivable the H_2 built up sufficient internal pressure to force residual liquid to the surface. This mechanism was investigated at Hanford[17-20] and LANL for its potential to generate free liquid in cemented waste forms. Hanford workers showed that free-liquid generation is related to the waste form's compressive strength and permeability.[19] Work was also done at Hanford to develop a computer model to predict the occurrence of radiolytic free liquid based on a waste form's permeability, gas generation rate, and internal pressurization.[20] Work at LANL with actual waste showed that increasing either the C/L ratio or mixing time reduced the occurrence and volume of free liquid,[9,11,12] presumably because of a resulting reduction in pore size and therefore in permeability.[21] Very good results were achieved in the LANL full-scale operation by extending the mix time into the setting stage until the mixer began to lose its ability to generate a surface vortex.[9,10,22] However, because a few waste forms still generated free liquid,[11,22] the extended-mix technique was not considered to have entirely solved the problem.

A study was conducted to investigate the differences between Portland and Envirostone cements in their susceptibility to radiolytic free-liquid generation.[23] The study was also intended to determine if the Portland waste form would generate free liquid when produced under conditions conducive to high permeability, namely a low C/L ratio and low-energy mixing.* A 590 rad/min ^{60}Co gamma source was used to simulate the radiolytic effects of alpha irradiation in the actual waste. In this study, the Envirostone sample generated liquid after 2.5×10^6 rads in 3 days. The testing was continued to 8.1×10^7 rads (95 days) with the Portland sample producing no free liquid during this time. These results demonstrated the superiority of the Portland waste form in suppressing radiolytic free-liquid generation, even when produced under substandard conditions.

Leaching Resistance: In 1992, the Envirostone waste form was found to be unable to meet the nonhazardous limit for chromium as defined by the EPA. This inadequacy was first indicated in leaching studies using surrogate EV waste[25] and later confirmed with actual EV waste studies.[26] Both tests also showed Portland waste forms had at least an order of magnitude higher leaching resistance for total chromium (Cr^{+3}/Cr^{+6}) in the TCLP. Portland cement also showed superior leaching resistance for cadmium and lead, two other metals found to have elevated concentrations in the EV waste. The graphical comparisons of TCLP performance in the surrogate- and actual waste tests using Envirostone and Portland cement are shown in Figures 1 and 2.

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* The ability of Portland waste forms to suppress radiolytic free-liquid generation completely had previously been questioned after the discovery in 1992 of free liquid in some Portland drums produced before the conversion to Envirostone in 1983.[24] However, the quality of these waste forms was considered poor because very low-energy (manual) mixing was used to produce them. In addition, no records were available that documented the C/L ratio used to produce these waste forms. Substandard conditions may have resulted in excessive bleed-water formation and/or an increase in permeability that allowed radiolytic free-liquid generation.

Waste Form Performance Standards: The following standards were devised to define satisfactory performance of the final waste form. These include the regulatory-based standards to meet the EPA TCLP limits to produce a non-mixed waste form and the WIPP-WAC restrictions on free liquid and particulates. Operational-based standards were included to ensure homogeneous mixing, timely setting for efficient process throughput, and an operating temperature low enough to prevent thermal damage of the drum packaging.

1) Adequate mixability

To ensure that the recommendations of this work will provide consistent results, the mixing must be thorough enough to yield a homogeneous paste. Therefore, the viscosity of the cement paste must not be above the maximum viscosity at which the mixing equipment can provide thorough mixing. This limiting viscosity was defined as the maximum viscosity at which center-to-edge mixing of the cement paste could still occur.* Dilution with water was necessary for samples with viscosities above this limit.

2) No free liquid at ≥24 hours following cementation

The requirement for absence of free liquid was necessary to meet the WIPP-WAC restriction on free liquid.[1] The 24-hour limit was considered desirable to ensure a reasonable throughput efficiency. The free liquid could be due to initial bleed water not reabsorbed or delayed generation of free liquid resulting from radiolysis. Adequate performance related to radiolysis would be defined by no generation of free liquid during a 10^8-rad exposure to a ^{60}Co gamma source.

3) Moderate hydration temperature

The objective of this standard was to prevent the failure of the vinyl drum bag as a result of excessive heat from the hydration reaction of the cement. The conservative estimate by the bag manufacturer for the failure temperature was 145°F.[28] Since the temperature of the cement monolith reached during hydration in a drum-scale sample would be higher than in the bench-scale tests, an assumption had to be made on what temperature ceiling to select for the bench-scale tests. Using the industry standard of an increase of 15°F per 100 lb of Portland cement,[29] the assumption was made that an increase of as much as 45°F could be seen on scale-up to the drum scale. This led to the range of acceptable temperatures being set at ≤100°F for the bench-scale experiments. All temperatures were taken at the center of the waste form. The actual full-scale temperature increase would be determined in a full-scale test.

4) Penetration resistance of ≥500 psi at 24 hours following cementation

To ensure a reasonable throughput efficiency, it was considered desirable that indication of a successful setting reaction should be seen within 24 hours after the start of cement addition. The condition used to indicate a successful set was 500 psi penetration resistance. This indicator was adopted from the American Society for Testing and Materials (ASTM) standard for determining the initial set of a cement sample.[30]

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* Center-to-edge mixing is defined as rotational movement of the cement surface extending to the point of contact with the wall of the mixing container.

5) Sustained compressive strength of ≥ 500 psi at 28 days following cementation

Minimum compressive strength was not needed to meet WIPP or EPA requirements. It was incorporated as a means to ensure the waste form was cohesive enough to resist breakdown to a particulate size prohibited by the Revision 4 WIPP-WAC. A minimum compressive strength of 500 psi was adopted for this study from the Nuclear Regulatory Commission's use of this level of strength as the best indicator of general physical durability for cemented waste.[31] The compressive strength data were collected according to the ASTM standard test method for compressive strength determination.[32] An additional requirement was included after the start of this work that there be no significant decline in compressive strength during the 28-day monitoring period. This was done after an expansive phase phenomenon was discovered that resulted in complete strength failure in a sample after satisfactory early development (see below). The objective was to identify and reject any samples undergoing this phenomenon that still had a 28-day compressive strength above 500 psi, but would have failed at a later time.

6) Pass EPA leaching standards for RCRA metals

The EPA has jurisdiction over the on-site storage of mixed waste. Since mixed waste is much more costly to store than nonhazardous TRU waste, it is advantageous for a waste form be classified as nonhazardous TRU (non-mixed) waste. In the case of TA-55 cemented waste, the major obstacle to achieving non-mixed status is meeting the chromium concentration limits in the TCLP extractant. Performance in this area was determined by TCLP testing on selected samples according to the EPA procedure.[33]

EXPERIMENTAL EVALUATION

The goal of this project was to develop a simple cement formulation using only Type I/II Portland cement. Type I/II is the most readily available type of Portland cement, and its sole use would simplify the procurement of the raw materials. Another advantage to a one-component formulation would be the prevention of segregation problems that can occur with multicomponent formulations in vibrating silos such as the one at TA-55. Other cement types and additives would be investigated only if required to address significant problems in areas such as slow setting, inadequate leaching resistance, or excessive temperature rise. This project was conducted according to the LANL/TA-55 Quality Management Plan: Documents and Records Control Section, procedure number SS-TA-55-110-0-03.1-1.

Surrogate preparation: The surrogates of the LR- and OX-based waste streams were prepared to approximate the median concentrations found in the waste stream characterization study listed in Appendix 1. The OX-based surrogate was prepared with selected RCRA metals to evaluate the TCLP performance of the cemented waste form. The LR-based surrogate was prepared without RCRA metals. The concentrations of the LR- and OX-based surrogates are listed in Table 1.

Test Conditions: It was not the intention of this project to establish the failure boundary of all process parameters. Rather, it was to define a set of target parameters whose performance had been shown to be successful. To that end, this project varied the process parameters of pH, C/L ratio,

and water dilution to collect data regarding their influence on waste form performance. The equipment used for preparation of the samples and data acquisition is described in Appendix 2. Bench-scale testing was completed with LR-based surrogate before OX testing was begun. Conditions found to be unsatisfactory in the LR tests were not included in the OX tests. The pHs evaluated were 8.5, 9.6, 10.5, and 12.1 for the tests involving LR surrogate. The tests with OX-based surrogate were performed at a more limited range of 9.5, 10.5 and 11.5 as a result of pHs 8.5 and 12.1 being eliminated in the LR tests (see below). All pH adjustment was achieved using 10 molar NaOH solution. The pH, C/L ratio, and dilution values for the bench-scale tests are listed in Table 2.

A full-scale test was performed under conditions similar to a specific bench-scale sample (LR16) to ascertain how scale-up would affect bench-scale results. A full-scale test was necessary because there are several areas in which bench-scale testing may not predict performance on the full scale. Bench-scale samples often do not show bleed water when full-scale samples would.[34] Also, larger-scale samples exhibit a higher temperature rise during hydration that can produce cracks in the cement monolith. This damage can compromise performance in compressive strength development and leaching resistance.[34] In our case, the temperature was also a concern because of its potential to cause thermal damage to the vinyl drum bag.

Table 1: Composition of Surrogates
(in g/l except as noted)[++]

Ion	LR-based	OX-based	Ion	LR-based	OX-based
Fe	9.03	8.56	SO_4	1.55	1.00
Ca	58.39	16.88	C_2O_4	3.2	33.8
K	18.51	6.38	F	6.0	1.7
Mg	54.77	21.40	Cd	0	0.0028
Na	6.85	26.98	Cr	0	2.45
Al	4.75	1.75	Pb	0	0.056
NH_4	0.028	0.09	Ag	0	0.0014
H^+ (M)	1.40	4.55	Ba	0	0.0355
Cl	1.02	0.27	Ni	0	1.205
NO_3	630	398			

[++]Based on quantity of chemicals used to prepare surrogate instead of analysis.

RESULTS AND DISCUSSION

All bench-scale samples were evaluated for mixability, bleed-water generation, initial set time, setting-temperature characteristics, and compressive strength. Evaluations of TCLP performance and radiolytic free-liquid generation were conducted only on OX samples. The performance in each category is discussed below. The results of the full-scale test follow those of the bench-scale tests.

The LR samples were used for the dilution study in which the pH and water dilution values were varied concurrently with each sample (see mixability section). Consequently, pH and water dilution must be considered together when determining their influence on LR samples in the following performance categories. The OX tests were conducted with only one of these process parameters being varied at one time. Subsequently, the OX tests are more useful in determining the individual influence of pH and water dilution.

Table 2: Parameters and Results

Sample No.	pH	C/L Ratio (kg/l)	Waste / NaOH / H_2O (vol%)[a]	Initial Set Time (hr.)	Compressive Strength (psi) 7-day	14-day	28-day
LR15	8.5	0.80	58.1 / 28.9 / 13.0	58	450	0	0
LR11	9.6	0.94	67.3 / 32.7 / 0	26	1200	1550	1775
LR18	9.6	0.80	45.0 / 32.0 / 23.2	28	650	825	750[e]
LR16	10.5	0.80	41.4 / 27.8 / 30.8	20	750	700	850
LR14	12.1	0.80	35.1 / 30.6 / 34.3	18	688	925	1075
OX1	9.5	0.94	57.9 / 42.1 / 0	30.6	687	775	975
OX2	9.5	0.94	57.2 / 42.8 / 0	33	650	825[d]	950
OX9	9.5	0.94	56.3 / 38.7 / 5	ND[b]	875	1100	1250
OX12	9.5	0.94	51.4 / 38.6 / 10	24.6	1525	1000	1250
OX19	9.5	0.94	51.4 / 38.6 / 10	24.2	600	875	1000
OX16	9.5	0.80	51.4 / 38.6 / 10	29.8	375	450	650[e]
OX5	10.5	0.99	62.3 / 37.7 / 0	ND[b]	975	1150	1325
OX6	10.5	0.94	55.2 / 39.8 / 5	23.1	800	2125	1300
OX17	10.5	0.94	55.2 / 39.8 / 5	26.7	700	825	1100[e]
OX4	10.5	0.94	52.3 / 37.7 / 10	21.8	725	925	1150
OX10	10.5	0.94	54.2 / 35.8 / 10	25.4	750	850	1100
OX13	10.5	0.94	54.2 / 35.8 / 10	17.7	800	1000	1150
OX18	10.5	0.94	54.2 / 35.8 / 10	22.3	875[c]	1000	1150[e]
OX3	10.5	0.84	58.1 / 41.9 / 0	<24	650[c]	800	1100
OX14	10.5	0.80	54.2 / 35.8 / 10	26.9	300	500	700
OX7	11.5	0.94	56.0 / 39.0 / 5	26.8	850	1100	1275
OX8	11.5	0.94	53.1 / 36.9 / 10	22.6	825	950	1275
OX11	11.5	0.94	53.1 / 36.9 / 10	24.6	950	1025	1150
OX15	11.5	0.80	53.1 / 36.9 / 10	28.4	375	525	675
OX20[f]	4	1.80	79.1 / 20.9 / 0	<24	1000	925	800
OX21[g]	4	1.80	79.1 / 20.9 / 0	<24	1200	1175	1000

[a] Vol% of total liquid volume; [b] Not determined; [c] Determined at 9 days; [d] Determined at 15 days; [e] Determined at 29 days; [f] Envirostone short mix; [g] Envirostone long mix

Bench-Scale Experiments

Mixability. The mixability limit was defined as the paste viscosity above which the mixing equipment lost its ability to achieve center-to-edge mixing. It was found that all pH-adjusted LR samples exceeded this limit after the addition of the cement required for a C/L ratio of 0.80 kg/l. Besides the dilution provided by the NaOH solution used for pH adjustment, additional dilution of these samples with water was required to meet the mixability limit. As a result of increased salt precipitation, samples at a higher pH exhibited a higher viscosity and therefore required more dilution. To quantitatively establish the effect of dilution on waste loading as the pH was increased, the 0.80 kg/l LR samples were prepared at the same viscosity. In this dilution study the viscosity was established at what was necessary to attain a 0.1-watt power demand by the mixer while mixing the pH-adjusted, precemented surrogate at 250 rpm. This viscosity was found to result in a paste that was as viscous as possible without exceeding the mixability limit at 0.80 kg/l . The water, NaOH solution, and waste values as a percentage of the total liquid volume of each LR sample before concentration are reported in Table 2.

The same type of study was not conducted on OX samples. Instead, the water dilutions in the OX samples were fixed at 0%, 5%, and 10%. Although the viscosity of the OX samples with 0% dilution was considered excessive, no difficulty in mixing was observed in any sample at 5% or 10% dilution. This lower dilution requirement for the OX samples is due to the lower salt content of the OX-based surrogate, as reflected in the lower total dissolved solids (TDS) value of the OX waste stream reported in Appendix 1. All dilution and waste percentages for the OX samples are reported in Table 2.

Free Liquid from Bleed-Water Generation. Although several samples initially generated bleed water, all bleed water was reabsorbed before 24 hours in all LR and OX samples.

Initial Set Time. The LR tests showed a definite relationship between each sample's initial set time and the pH and dilution values. This can be seen in Figure 3a, which shows the initial set time decreasing as the pH and dilution values increase. The samples prepared at pH 8.5 and pH 9.6 had initial set times exceeding 24 hours. Because the pH 8.5 sample greatly exceeded the 24-hour limit, it was eliminated from further consideration.

The OX samples presented in Figure 3b show a somewhat different effect of pH on initial set time in that the lowest initial set time occurred at pH 10.5. Nevertheless, the results were consistent with the LR tests in that no sample prepared at a pH $\leq$9.6 met the 24-hour standard. This finding further cast doubt on the acceptability of the samples prepared in the pH 9.6 region. Figure 3b also shows a definite relationship between C/L ratio and initial set time in that the set time increased as the C/L ratio decreased. This increase was enough to prevent all 0.80 kg/l OX samples from meeting the 24-hour standard. Dilution was also found to effect initial set time in that the initial set time decreased as the dilution increased. This decrease is presumably a result of the decrease in the concentrations of set-retarding constituents of the waste. Figure 3c shows this relationship between dilution and initial set time for the 0.94 kg/l OX samples at each pH. This relationship should also have had an increasingly significant effect on the initial set times of the LR samples as the pH was increased.

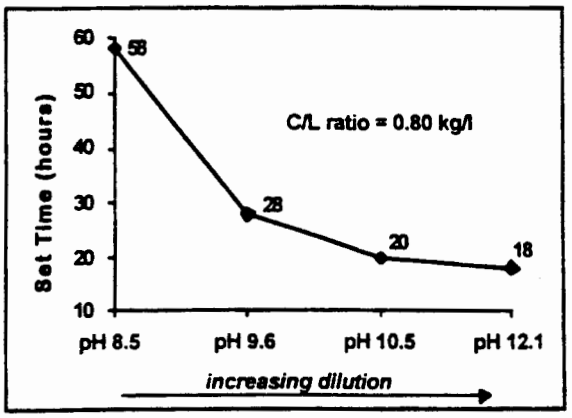

Fig. 3a. Effect of pH/dilution on initial set time for LR samples. (see Table 2 for dilution values)

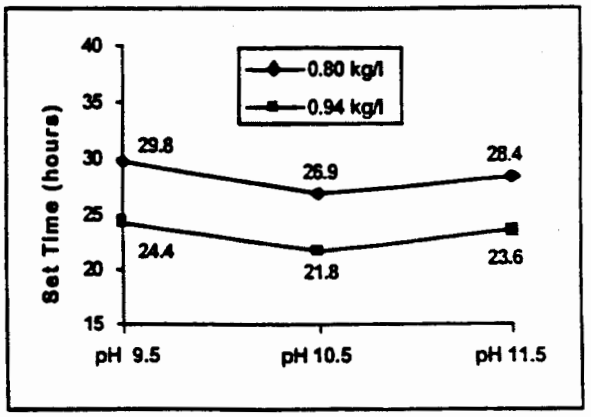

Fig. 3b. Effect of pH and C/L ratio on initial set time for OX samples. (avg. of samples w/ 10% water dilution).

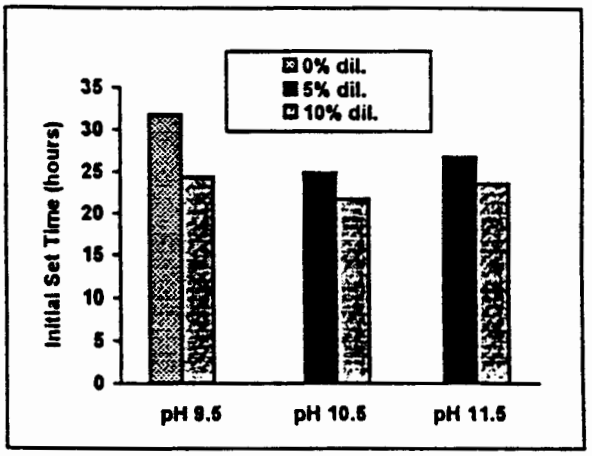

Fig. 3c. Effect of water dilution on initial set time for OX samples (avg. of samples w/ C/L ratio of 0.94 kg/l).

Temperature. The LR tests showed a definite relationship between the pH and dilution values of each sample and the maximum hydration temperature and the elapsed time to maximum temperature. As the pH and dilution values increased, the maximum temperature increased and the elapsed time generally decreased. This can be seen in Figure 4a. The maximum temperature of 107°F reached by the LR14 sample exceeded the 100°F temperature ceiling, thus causing a failure of this sample to meet the bench-scale temperature standard.

The OX samples presented in Figure 4b showed no significant change in temperature behavior as the pH was varied. The temperature profile of all OX pHs looked similar, with a peak temperature

of approximately 860° F at 24 hours. Since the water dilution values of these OX samples were held relatively constant, this may indicate that the changing temperature profiles of the LR samples in Figure 4a were significantly influenced by the changing dilutions of these samples. However, differences in chemical composition of the two EV types can not be ruled out as being a factor.

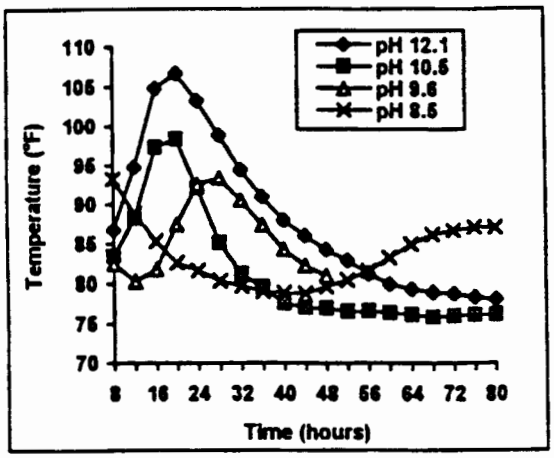

Fig. 4a. Temperature profile for LR samples LR14, LR15, LR16, & LR18. (C/L ratio = 0.80 kg/l, see Table 2 for dilution values).

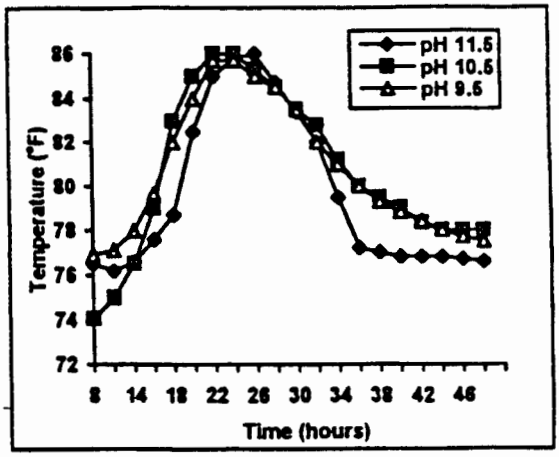

Fig. 4b. Temperature profile for OX samples OX4, OX8, & OX19. (C/L ratio = 0.94 kg/l, water dilution = 10%).

Compressive Strength. As expected, the compressive strength increased as the C/L ratio was increased. This relationship is shown for selected LR and OX samples in Figures 5a and 5b, respectively. The relationship of pH to compressive strength is shown for selected LR and OX samples in Figures 6a and 6b, respectively. In the LR samples a higher pH resulted in a higher 28-day compressive strength. However, this trend is likely to have been promoted by the increased dilution at the higher pHs of the LR samples. This statement is supported by the fact that no consistent trend was seen in OX samples at the same pH with a constant dilution percentage (see Figure 6b and Table 2). Table 2 also shows that the average 28-day compressive strength values for the OX samples with 0% water dilution (OX1 and OX2) were less than those at 5% and 10%. This was expected because of a higher ionic concentration in the 0% samples. However, the 5% average values in all pH categories were greater than those at 10%. The reason for this apparent anomaly is not known although it may be a statistical artifact resulting from the small number of samples.

The compressive strength of each sample generally increased with time, but this was not always the case. An exception occurred in the pH 8.5 LR sample. A phenomenon was observed in this sample that allowed satisfactory early strength development, but resulted in cube expansion and complete structural failure (compressive strength = 0 psi) at the 14- and 28-day strength determinations (see Figure 6a). It is possible that the LR sample prepared at pH 9.6 and 0.80 kg/l also exhibited some effects of this phenomenon (see Figure 6a). Although the pH 9.6 sample still had a compressive strength above 500 psi at 28 days, the compressive strength at 28 days was lower than at 14 days. This may indicate that the waste form would have self destructed at a later time.

This concern, along with the marginal initial-set-time performance of samples at this pH, prompted the removal of pH 9.6 from further consideration. The OX samples did not experience this delayed expansion phenomenon at the tested pHs of 9.5, 10.5, and 11.5. The explanation for this phenomenon in the LR samples is not certain. Since this occurred only in the LR samples with the lowest dilutions, the effect may be linked to a high concentration of one or more of the ions that are not as concentrated in the OX surrogate.

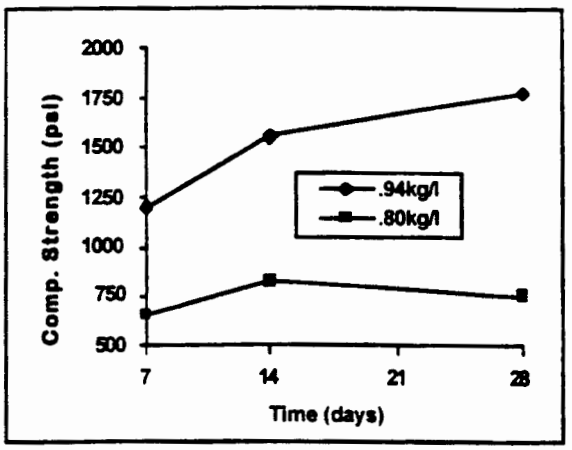

Fig. 5a. Effect of C/L ratio on compressive strength of LR samples LR11 & LR18. (pH = 9.6, see Table 2 for dilution values).

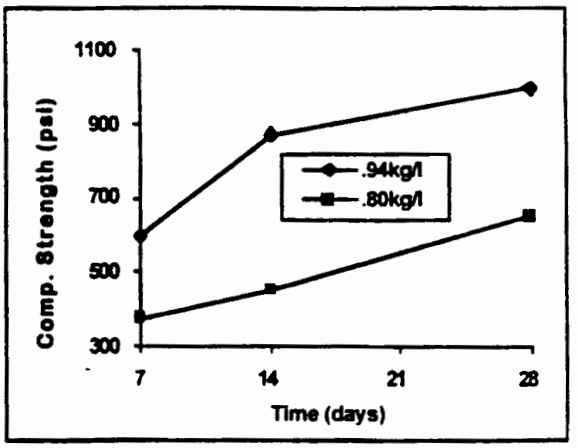

Fig. 5b. Effect of C/L ratio on compressive strength of OX samples OX16 & OX19. (pH = 9.5, water dilution = 10%).

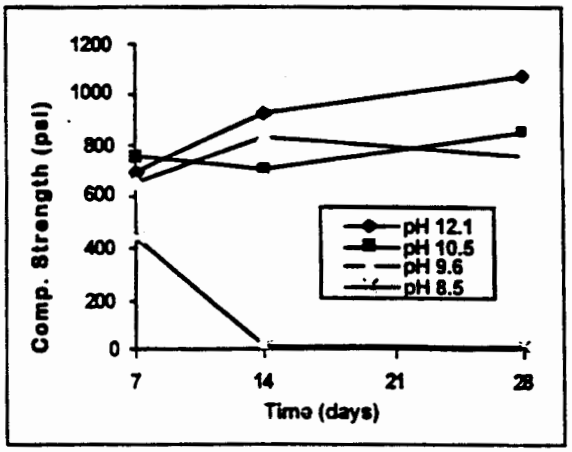

Fig. 6a. Compressive strength of LR samples LR14, LR15, LR16, & LR18. (C/L ratio = 0.80 kg/l, see Table 2 for dilution values).

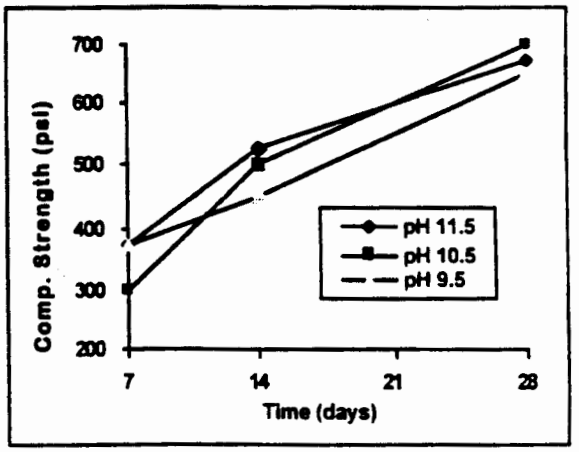

Fig. 6b. Compressive strength of OX samples OX14, OX15, & OX16. (C/L ratio = 0.80 kg/l, water dilution = 10%).

TCLP Results. TCLP testing was done on selected OX samples. The OX samples were analyzed for the RCRA metals Cr, Cd, Pb, Ba, and Ni. The RCRA metals not included in the TCLP testing were present in concentrations that would be well below the TCLP limit even if 100% of each metal leached out in the TCLP. The samples for TCLP analysis were taken from the pH categories of

9.5, 10.5, and 11.5 and included the sample with the lowest compressive strength in each category. The sample with the lowest compressive strength was considered the worst-case condition for leaching resistance because of the connection of compressive strength to leaching resistance through a common dependency on permeability.[21] The analytical results in Table 3 show that all samples passed the TCLP standards for nonhazardous waste.

Table 3: TCLP Results for Oxalate Surrogate Samples
(all concentrations in ppm)

Sample ID	Cr	Cd	Pb	Ba	Ni
OX7	0.11	<0.003	<0.011	2.6	0.01
OX8	0.12	<0.003	<0.011	2.4	0.01
OX9	0.12	<0.003	<0.011	3.0	<0.01
OX14	0.10	<0.003	<0.011	2.7	0.01
OX15	0.13	<0.003	<0.011	2.6	<0.01
OX16	0.11	<0.003	<0.011	2.7	<0.01
OX17	0.14	<0.003	<0.011	2.7	0.01
OX18	0.11	<0.003	<0.011	2.9	0.01
OX19	0.17	<0.003	<0.011	2.8	0.05
Leach Resistance**	99.9%	>97.9%	>99.6%	69.0%	99.9%

**These values represent the lowest leach resistance for each metal calculated from the TCLP results in Table 3. The definition and the calculation of leach resistance is presented in Figure 1.

Radiolytic Free-Liquid Generation. The testing for susceptibility to radiolytic free-liquid generation was conducted with OX samples. Gamma irradiation was used to simulate the radiolytic degradation caused by alpha irradiation in the actual wastes.* To achieve 10^8 rads quickly, the test was conducted using a 380 rad/sec ^{60}Co gamma source at Sandia National Laboratories. The samples were taken from the pH categories of 9.5, 10.5, and 11.5 and included the sample with the lowest compressive strength in each category. Compressive strength was again used as the worst-case indicator because of its common dependency with free-liquid generation on permeability. The test results, presented in Table 4, show that no Portland cement sample produced free liquid during the irradiation test.

Two Envirostone-cemented samples were also included in this test, a short-mixed (OX20) and a long-mixed (OX21) sample. These were included to investigate a correlation found in previous work between longer mixes and suppression of radiolytic free-liquid generation in Envirostone cemented waste forms.[9,10,22] During the irradiation test, free liquid was found in both samples, the short-mix sample at 1.7×10^7 rads and the long-mix sample at 4.4×10^7 rads. The liquid generated by the long-mix sample was actually greater in volume than that of the short-mix sample (see Table 4).

* Gamma irradiation is less efficient than alpha irradiation in the production of radiolytic H_2. The G value (H_2 molecules produced per 100 eV of energy absorbed) from gamma radiolysis has been reported to be approximately 33% lower than that from alpha radiolysis.[14]

Based on these results, it can be concluded that the long-mix technique cannot be consistently relied upon to suppress radiolytic free-liquid generation in Envirostone cemented waste forms.

Table 4: Gamma Irradiation

Cumulative Dose: (rad)	8.3×10^6	1.7×10^7	4.4×10^7	5.3×10^7	7.2×10^7	9.8×10^7
Sample ID	Liquid?	Liquid?	Liquid?	Liquid?	Liquid?	Liquid?
OX14	no	no	no	no	no	no
OX15	no	no	no	no	no	no
OX16	no	no	no	no	no	no
OX17	no	no	no	no	no	no
OX18	no	no	no	no	no	no
OX19	no	no	no	no	no	no
OX20 short-mix Envirostone	no	Yes (damp[++])	Yes (1.55g)	Yes (3.35g)	no	no
OX21 long-mix Envirostone	no	no	Yes (3.59g)	Yes (4.91g)	no	no

[++]Weights in parentheses are totals from the start of free-liquid generation.

Full-Scale Testing: The full-scale test was conducted with LR-based surrogate and was designed to simulate the bench-scale sample LR16. The full-scale sample had a pre-cement pH of 10.3 and a C/L ratio of 0.80 kg/l, which corresponded to 220 lb of Portland cement. The EV, NaOH, and water volume percentages in the full-scale sample were 41%, 28%, and 31%, respectively. All performance standards were monitored in the full-scale test except RCRA-metal leaching resistance and radiolytic generation of free liquid. Compressive strength was used as an indicator of performance in these nonmonitored areas because of its common dependence with them on permeability. The results relative to each performance standard are reported below.

Mixability. The mixing equipment had no difficulty in providing center-to-edge mixing of the cement paste.

Free Liquid. No bleed water was present at any time after cementation.

Initial Set Time. Initial set occurred at 20.6 hours after cementation, approximating the 20-hour initial set time of LR16. The profile of the penetration resistance is shown in Figure 7.

Temperature. The full-scale test was useful in illuminating several areas of uncertainty concerning temperature behavior. The temperature was monitored approximately halfway down at the center and edge (1 inch in) of the cement monolith and between the rigid liner and bag. The temperature reached a maximum of 163°F at the center, 150°F at the edge, and 103°F at the bag. The full-scale temperature profile at the center of the monolith is shown in Figure 8.

The maximum center temperature of the full-scale sample was 66°F higher than that of LR16. This corresponds to an increase of 30°F per 100 lb of Portland cement ([66°F / 220 lb] x 100). This is twice as high as the industry standard of 15°F per 100 lb used to establish the temperature ceiling for the bench-scale tests. This difference is likely a result of the industry standard being based on concrete, in which the aggregate would act as a heat sink. Another meaningful finding was that the temperature at the bag location was 60°F lower than at the center of the monolith. This resulted in a temperature significantly below the bag-failure temperature of 145°F even though the center temperature was higher than expected. These data can now be used to back-calculate a revised temperature ceiling of 139°F for the bench-scale tests (145°F + 60°F - 66°F). At this higher ceiling, the pH 12.1 LR sample, previously rejected with a maximum temperature of 107°F, falls within in the acceptable temperature range.

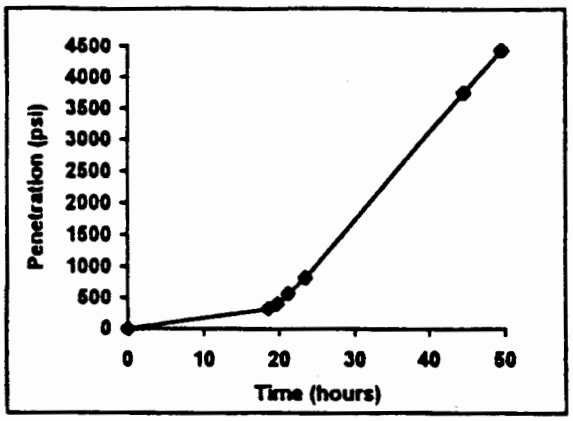

Fig. 7. Penetration resistance of full-scale test.

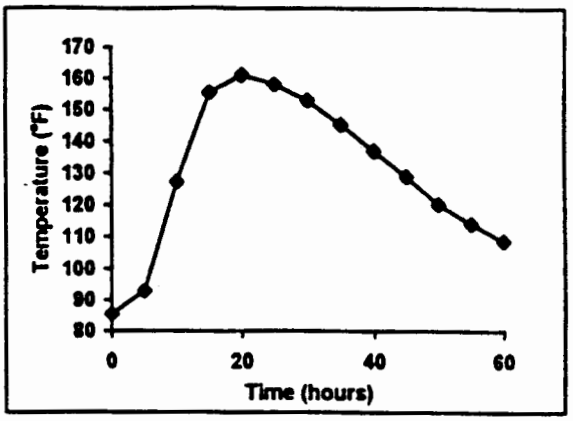

Fig. 8. Temperature profile of full-scale test (center).

Compressive strength. The compressive strength values of the full-scale sample exceeded those of LR16 after the first week of the 28-day monitoring period. This indicates no damage to the monolith occurred as a result of increased hydration temperature from scale-up. The compressive strength profiles of the full-scale sample and LR16 are shown in Figure 9.

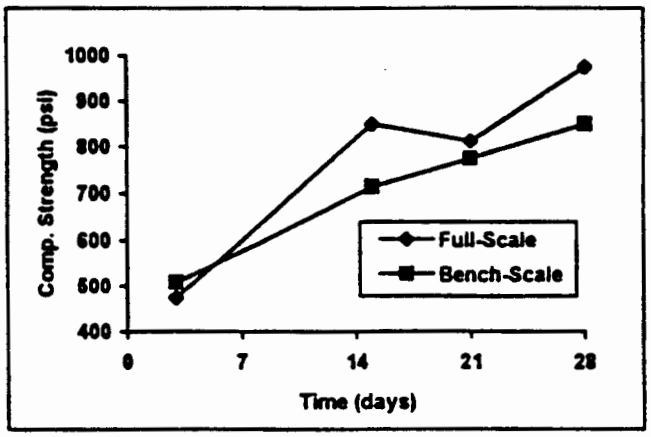

Fig. 9. Compressive strength comparison of full- and-bench-scale (LR16) samples. (Full-scale data taken at 3, 15, 21, and 28 days. LR16 data at 3, 15, and 21 days were extrapolated from Table 2 values).

Quality Assurance: Some care must be taken in procuring the Type I/II cement for the TA-55 cementation operation. Type I/II Portland cement is defined by a range for each of its chemical, mineralogical, and physical properties. The ranges are broad enough that variations in setting characteristics and sensitivity to chemical interferences are sometimes observed between Type I/II cements from different sources. It is recommended, therefore, that the QA program for this operation assure that procurement be from a source offering a Type I/II cement with similar composition to that used in this development project. The particular cement used in this project is a low-alkali Type I/II cement. The source of the cement was Rio Grande Portland Cement Corporation in Tijeras, New Mexico. The chemical and mineralogical composition and other pertinent physical characteristics for this cement are listed in Appendix 3.

Costs: For a production rate of 3 cement drums per week, the Portland cement required for operating the TA-55 cement process will be approximately 20.6 tons per year.* The current delivered cost from Rio Grande Portland Cement Corporation is $77.20 per bulk ton, resulting in a yearly cost of $1,592. This compares to $1,300 per ton and $33,627 per year for Envirostone cement.# The resulting savings in cement cost with the use of Portland cement will be approximately $32,035 per year. The increased volume of NaOH solution required by the Portland operation should result in an increased cost for NaOH of no more than $5,000 per year. This results in a net savings in raw materials of approximately $27,000 per year. The cost for Portland cement from Rio Grande Portland Cement Corporation is for bulk powder cement. Bulk delivery is less costly and has the advantage of allowing pneumatic delivery into the TA-55 cement silo. Pneumatic delivery will also reduce the number of person-hours required to load the silo from approximately 20 hours for the current manual technique to 1 hour per year.

Other Wastes: The recommendations of this report should be compatible with the addition of dry, non-reactive particulate wastes. In the present TA-55 operation, this type of waste is added to a pre-established cement paste to prevent the solids from settling during mixing. The primary effect of using this technique will be an increase in paste viscosity, which can be addressed by additional dilution with water if necessary. Slurries (wet particulates) with a pH outside the range recommended in this report should be pretreated to properly adjust the pH before addition to the cement paste. Low-density slurries that do not require addition to a pre-established paste can be added to the EV waste and their pHs adjusted with the EV waste before cement is added. Ion-exchange resins present an additional concern in that they have been reported to cause setting and free-liquid problems under certain conditions.[35,36] Non-EV aqueous wastes may also cause chemical interferences with the Portland setting reaction. Thus, it is recommended further studies be conducted before ion-exchange resins or non-EV aqueous wastes are incorporated into the cementation operation.

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* Calculated at 150 drums per year at 275 lb of Portland cement per drum.

Calculated at 150 drums per year at 340 lb of Envirostone cement per drum.

No testing was done with organic wastes in this development project. Portland cement is not as efficient as Envirostone in treating nonpolar organics, but methods exist that permit satisfactory treatment of some organics with Portland cement.[37] It generally is adequate for limited volumes of nonpolar organics if they are prepared in a well-dispersed, oil-in-water emulsion. It does not perform well with inverse emulsions (water-in-oil) or pure solvents.[34] Organics with high solubility (polar) generally are not immobilized well by Portland cement.[34] It is recommended additional development be conducted before organic wastes are incorporated into the cementation operation.

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- Earl McDaniel of Oak Ridge National Laboratory for technical support.
- NMT-7 cement process personnel for assistance in conducting the full-scale test.

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Appendix 1

Analysis of Evaporator Bottoms[a]
(in g/l except as noted)

	Lean Residue	Oxalate Filtrate	Hot Distillate
Fe	17.0	7.9	16.9
Ca	61	10.5	39.1
K	17.6	4.8	14.6
Mg	58.7	13.3	41.9
Na	7.4	23.9	9.4
Al	4.6	2.3	4.7
NH_4	0.025	0.090	0.035
Cl	1.1	0.265	1.35
NO_3	457	398	419
SO_4	1.6	<1	1.57
C_2O_4	3.3	33.8	11.3
F	5.4	1.7	5.1
H^+ (molar)	1.0	4.6	1.75
TDS[b]	629	330	600
Ag	<0.005	<0.001	<0.002
As	<0.015	<0.005	0.010
Ba	0.035	0.018	0.029
Cd	0.014	0.003	0.014
Cr	3.0	1.94	2.35
Hg	<0.025	<0.005	0.010
Ni	1.8	1.205	1.60
Pb	0.19	0.056	.125
Se	<0.008	<0.008	0.009
Tl	<0.060	<0.020	<0.060

[a]Median values of all analyzed EV samples; [b]Total dissolved solids

Appendix 2

Equipment for Sample Preparation & Data Acquisition

The bench-scale equipment was assembled to match the design and proportions of the TA-55 full-scale process as much as possible. A programmable laboratory mixer (Lightnin Labmaster TSM2510) was used as the mixing device. The mixing profile was matched to the TA-55 process with the rpm being ramped up from 250 rpm to 400 rpm to maintain adequate mixing during cement addition. A program was written for the laboratory mixer that automatically and continuously recorded the mixer power, mixer rpm, and mixing time for each bench-scale test. For the full-scale test, the mixing container consisted of a 1/4-inch thick polyvinyl chloride rigid liner, inside a 12-mil vinyl bag, inside a 55-gallon drum (DOT 17-C). This configuration was identical to that used in the TA-55 operations minus the lead between the bag and drum. The mixer (Lightnin XJ350), mixer shaft, and propellers (12.4-inch diameter Lightnin A100) were identical to those used in the TA-55 process. The two propellers were separated on the shaft by one propeller diameter with the lower propeller being one-half diameter above the bottom of the liner. The full-scale test had a mixing rpm of 400 during the entire test.

The following equipment was used for data acquisition in the bench- and full-scale tests. The bench-scale mixing profiles were recorded by a Compac Model M84 personal computer through an RS-232 interface with the mixer. Temperature was monitored using a Yokogawa Model LR4120 strip chart recorder and a type-K, Omega thermocouple (5TC-TT-K-24-36). The thermocouple was Teflon-coated to ease its removal from the hardened cement monoliths. The compressive strength of all waste forms was determined using a model C, 12-ton hydraulic press from the Carver Laboratory Press Company. The compressive strength data were taken on 2-inch cubes cast in Humboldt H-2810 cube molds. Penetration resistance was determined using a Humboldt model H-3143 hydraulic penetrometer. The gamma irradiation data for the bench-scale samples were collected with the use of a 380 rad/sec ^{60}Co gamma source located at Sandia National Laboratories.

Appendix 3

Composition of Type I/II Portland Cement Used in Tests
(in %, except where noted)

	#1 1/1/93 - 12/12/93[a] Average	(Std. Dev.)	#2 5/13/94 Bin 9 - Test 114	#3 7/1/94 - 10/31/94 Average	#4 9/1/94 - 1/18/95 Average
SiO_2	20.9	(0.22)	20.9	21.1	21.1
Al_2O_3	4.3	(0.087)	4.3	4.3	4.3
Fe_2O_3	3.1	(0.082)	3.1	3.0	3.1
CaO	63.3	(0.55)	63.3	63.6	63.6
MgO	2.8	(0.51)	2.4	2.5	2.6
SO_3	2.9	(0.08)	3.0	3.0	2.0
Na_2O	0.18	(0.023)	0.21	0.19	0.21
K_2O	0.52	(0.029)	0.52	0.55	0.52
Loss On Ignition	1.5	(0.12)	1.2	1.4	1.40
Insoluble residue	0.24	(0.022)	0.22	0.23	0.24
C_3S[b]	57.8	(2.48)	57	57	57
C_2S[c]	16.3	(2.1)	17	17	18
C_3A[d]	6.1	(0.24)	6.3	6.3	6.0
C_4AF[e]	9.5	(0.24)	9	8	10
Alkalies (Na_2O equiv.)	0.52	(0.033)	0.57	0.55	0.55
Blaine, (sq. M / kg.)	353	(8.0)	349	359	361
-325 mesh fineness	93.3	(1.19)	92.7	93.3	95.2
Autoclave expansion	0.05		0.03	0.05	0.05
Air content	8.5		8.9	8.2	8.1

[a] Dates are date of manufacture; [b] C = CaO; [c] S = SiO_2; [d] A = Al_2O_3; [e] F = Fe_2O_3

APPENDIX D

FINAL LETTER REPORT ON PYROLIZATION TREATMENT AT TECHNICAL AREA 55

Hard copies of this document were provided to
the New Mexico Environment Department.

Department of Energy
Field Office, Albuquerque
Los Alamos Area Office
Los Alamos, New Mexico 87544

OCT 28 1994

CERTIFIED MAIL - RETURN RECEIPT REQUESTED

Barbara Hoditschek, Program Manager
RCRA Permits Program
Hazardous and Radioactive Materials
Bureau
New Mexico Environment Department
525 Camino de los Marquez
P. O. Box 26110
Santa Fe, NM 87502

Dear Ms. Hoditschek:

The Department of Energy (DOE) and the Management and Operating contractor for the Los Alamos National Laboratory (LANL), the University of California, are providing this final report on the treatment of plutonium-impregnated nitrated cheesecloth at LANL by the Inert Atmosphere Decomposition Unit (IADU) located at Technical Area 55, in Building PF-4, Room 420, pursuant to Section II.K.1 of the 90-day emergency permit number NM0890010515-EP1. This letter also constitutes the certification required by Section II.K.2 of the permit.

On July 7, 1994, LANL began treating plutonium-impregnated nitrated cheesecloth managed as a mixed waste for ignitability and reactivity. A total of 3,841 grams of this cheesecloth was treated from July 7, 1994 through August 31, 1994. In addition to the nitrated cheesecloth, 27,628 grams of plutonium-impregnated, non-nitrated (and non-hazardous) cheesecloth was also treated at the same time. A total of 90 runs were required to work off these wastes. As a result of treatment, 6,630 grams of non-hazardous treatment residue was generated. Enclosed is a table showing each process run, including amount of nitrated and non-nitrated cheesecloth processed, date, start and end times of each process, and beginning and maximum temperatures.

As shown in the enclosed table, the IADU was operated at a process temperature below 800°C during the first 55 process runs, which was below the process temperature prescribed in the permit. The cause of the lower operating temperature was insufficient sizing of the heating coils and excessive cooling of the unit by the inert argon atmosphere. This was further exacerbated by the placement of the thermocouple used to measure process temperature too distant from the waste being processed and inadequate insulation of the process unit. Therefore, IADU

process temperatures did not reach 800°C during runs 1 through 55. On August 12, after relocating the thermocouple, installing high temperature heating coils and additional insulation, the IADU began operating at 800°C. On August 18, during run 66, the IADU failed to register 800°C. To assure proper deactivation of the hazardous components, all treatment residues generated below 800°C were reprocessed. The final 34 runs, except for run 66, including one run for reprocessing all residues generated below 800°C (i.e., runs 1-55) and a rerun on residue from run 66, were performed at or above 800°C. Thus, all the waste including all nitrated cheesecloth stored in Building 4, was ultimately subjected to temperatures at or above 800°C and achieved complete pyrolysis and thereby rendered non-hazardous.

LANL performed a review of the potential non-radioactive off-gassing that may have occurred during the processing of plutonium-impregnated nitrated and non-nitrated cheesecloth below 800°C. The decomposition of cheesecloth in the IADU may result in off-gassing of aldehydes, ketones, and alcohols into the caustic scrubber. At 131°C, or the minimum temperature measured during run number 10, the maximum estimated hourly emission for each of these compounds was found to be 0.57 lb/hr (1.71 lb/hr total). The emission limit, in accordance with Section 702 of the New Mexico Air Quality Regulations (AQCR) is 10 lb/hr. Therefore, the AQCR standard was never exceeded as a result of lower treatment temperatures.

Two discrepancies were found in the operating parameters provided by LANL in the permit application: (1) Attachment A, page 4, described feed rate at 10 percent by weight when the operating procedures for the IADU recommended 10 percent *by volume*; and (2) Attachment A, page 6, required replacement of scrubber water upon discoloration when the actual condition should have been pH. On August 17, Mr. Juan Corpion spoke with Ms. Stephanie Kruse of your staff regarding the discrepancies in the operating permit. Subsequently, on August 18, Ms. Kruse called Mr. Corpion and recommended that DOE and LANL send a letter clarifying the language in the permit to coincide with the operating parameters of the IADU. Ms. Kruse also advised that this action was not considered by the New Mexico Environment Department (NMED) as a permit modification. This clarification was sent to NMED in a letter from DOE on September 2, 1994. A response approving the parameters described in the September 2 letter was provided to DOE in a letter from NMED dated September 12, 1994.

OCT 28 1994

If you have any questions regarding this matter, please contact Joseph Vozella at (505) 665-5027, or Jon Mack at (505) 665-5026.

Sincerely,

Joseph C. Vozella 10/20/94

Joseph C. Vozella Date
Acting Asst. Area Manager
Office of Environment and Projects
U. S. Department of Energy

LAAMEP:2JM-014

10/28/94

Dennis Erickson, Director Date
Environment, Safety, and Health
Division
Los Alamos National Laboratory

Enclosure

bcc w/enclosure:
J. Mack, AAMEP, LAAO
L. Cummings, Counsel, LAAO
A. McMillan, ESH-DO, LANL,
MS-K491
J. Corpion, ESH-19, LANL,
MS-K498
A. Barr, ESH-19, LANL,
MS-K498
D. Christensen, NMT-DO, LANL,
MS-E500
J. Balkey, NMT-7, LANL,
MS-E501
Q. Appert, NMT-2, LANL,
MS-E511
J. Rochelle, LC/GL, LANL,
MS-A187

Table 1. Record of Nitrated and Non-Nitrated Cheesecloth Process Runs

Run	Date	Start Time	End Time	Nitrated Rag Weight (g)	Non-Nitrated Rag Weight (g)	Beginning Temp. (°C.)	Maximum Process Temp. (°C.)
1[a]	7/6/94	13:00	14:10	0.0	128.8	25.0	328.0
2	7/7/94	10:20	12:25	22.0	216.4	25.0	500.0
3	7/8/94	10:11	11:45	22.5	225.8	61.0	328.0
4	7/11/94	10:00	11:08	21.7	218.0	21.0	438.0
5	7/12/94	09:35	11:20	16.3	212.5	25.0	380.0
6	7/13/94	09:30	11:07	26.4	251.3	23.0	362.0
7	7/14/94	07:55	08:36	24.8	240.9	25.0	400.0
8	7/14/94	14:21	15:42	21.2	212.4	25.0	160.0
9	7/15/94	09:30	11:20	24.8	257.3	25.0	140.0
10	7/18/94	07:40	08:54	22.6	226.5	25.0	131.0
11	7/18/94	10:18	11:40	30.0	300.0	50.0	151.0
12	7/18/94	13:07	14:35	28.3	283.6	75.0	141.0
13	7/19/94	07:45	09:52	22.3	228.0	25.0	146.0
14	7/19/94	11:14	14:05	27.2	277.6	30.0	150.0
15	7/19/94	15:25	17:00	52.3	523.3	50.0	166.0
16	7/20/94	07:52	10:02	58.9	589.0	25.0	530.5
17	7/20/94	13:03	14:36	42.2	422.6	55.0	451.0
18	7/20/94	15:01	17:00	33.1	331.5	50.0	530.0
19	7/21/94	07:46	09:00	27.9	279.1	24.0	579.0
20	7/21/94	10:35	12:30	26.7	266.9	50.0	390.0
21	7/21/94	14:30	17:00	40.2	400.9	50.0	380.0
22	7/22/94	07:42	10:35	43.2	432.5	25.0	442.8
23	7/22/94	11:15	13:20	52.4	524.6	45.0	445.0
24	7/25/94	07:54	10:53	49.0	490.2	25.0	460.0
25	7/25/94	13:03	14:25	27.7	273.5	25.0	410.0
26	7/25/94	15:27	17:19	21.8	218.7	25.0	143.0
27	7/26/94	07:40	09:15	35.4	353.2	25.0	292.0
28	7/26/94	10:05	11:44	35.5	353.9	25.0	373. 6
29	7/26/94	12:35	13:57	21.7	217.2	50.0	460.0
30	7/26/94	14:46	16:36	40.3	403.8	25.0	536.0
31	7/27/94	07:42	09:17	27.0	270.5	25.0	55 0.0
32	7/27/94	09:57	12:00	35.0	350.4	35.0	458.0
33	7/27/94	13:00	13:45	21.3	217.1	25.0	654.0
34	7/27/94	14:15	15:00	27.9	279.6	100.0	520.0
35	7/27/94	15:15	16:00	20.0	196.5	50.0	363.0
36	7/29/94	07:46	11:15	23.9	239.8	25.0	299.0
37	8/1/94	07:52	11:30	34.6	346.6	25.0	394.0
38	8/1/94	12:15	14:17	23.2	232.0	25.0	300.5
39	8/1/94	14:45	16:58	29.8	298.2	25.0	279.0
40	8/2/94	07:30	09:00	32.0	160.5	25.0	270.0
41	8/2/94	09:56	11:22	57.0	285.6	100.0	230.0
42	8/2/94	12:00	14:30	41.6	208.3	50.0	300.0
43	8/3/94	07:30	10:30	55.5	275.2	25.0	352.0
44	8/3/94	11:03	13:44	90.4	452.0	25.0	360.0
45	8/3/94	14:40	17:05	84.4	422.0	25.0	235.0
46	8/4/94	07:45	10:30	80.9	404.7	25.0	282.0
47	8/4/94	11:00	11:45	43.6	218.0	25.0	220.0
48	8/4/94	13:00	16:06	55.4	277.0	25.0	293.0
49	8/8/94	08:15	10:45	40.4	204.4	25.0	412.0
50	8/8/94	11:15	12:30	42.4	212.4	50.0	415.0
51	8/8/94	13:00	15:20	61.6	308.3	50.0	591.0
52	8/9/94	07:45	10:30	83.8	419.3	25.0	596.0
53	8/9/94	11:45	15:43	95.0	475.5	25.0	616.0
54	8/10/94	11:35	13:00	67.8	339.9	25.0	465.0

Table 1. Record of Nitrated and Non-Nitrated Cheesecloth Process Runs

Run	Date	Start Time	End Time	Nitrated Rag Weight (g)	Non-Nitrated Rag Weight (g)	Beginning Temp. (°C.)	Maximum Process Temp. (°C.)
55	8/11/94	10:30	16:58	17.9	179.6	25.0	760.0
56	8/12/94	07:35	08:45	18.7	187.5	25.0	802.0
57	8/12/94	09:50	10:55	20.8	208.7	25.0	814.0
58	8/12/94	12:08	12:50	34.0	340.9	50.0	817.0
59	8/15/94	07:45	08:50	26.2	262.1	25.0	820.0
60	8/15/94	11:00	12:11	38.7	387.5	25.0	800.0
61	8/15/94	14:30	15:38	36.9	369.0	50.0	800.0
62	8/16/94	07:45	09:35	41.0	410.7	25.0	800.0
63	8/16/94	11:15	12:21	44.8	448.2	50.0	833.0
64[b]	8/16/94	14:49	17:10	N.A.	N.A.	25.0	805.0
65	8/18/94	07:35	10:05	33.8	338.2	25.0	836.0
66	8/18/94	12:40	14:00	34.0	340.5	25.0	655.0
67	8/19/94	07:35	08:30	60.0	300.0	25.0	805.0
68[c]	8/22/94	07:55	10:20	39.4	196.7	25.0	800.0
69	8/22/94	14:50	15 :53	40.2	400.7	100.0	800.0
70	8/23/94	07:55	08:50	42.6	426.5	25.0	809.0
71	8/23/94	13:55	15:08	40.5	400.7	100.0	802.0
72	8/24/94	07:45	08:45	25.6	256.6	25.0	800.0
73	8/24/94	13:00	14:00	24.0	240.8	25.0	804.0
74	8/25/94	07:30	08:36	38.9	389.5	25.0	800.0
75	8/25/94	10:10	11:35	30.2	302.4	100.0	809.0
76	8/25/94	12:45	13:50	50.0	378.4	100.0	906.0
77	8/25/94	15:15	16:15	49.0	298.8	100.0	800.0
78	8/26/94	07:40	08:50	49.0	200.7	25.0	800.0
79	8/26/94	10:10	10:55	49.0	401.1	50.0	800.0
80	8/26/94	12:25	13:26	49.0	411.3	100.0	800.0
81	8/26/94	14:55	15:31	96.0	377.7	75.0	800.0
82	8/29/94	07:40	08:55	96.0	407.5	25.0	800.0
83	8/29/94	12:30	13:30	99.5	192.4	75.0	800.0
84	8/29/94	14:30	15:40	99.0	355.4	100.0	800.0
85	8/30/94	07:30	08:50	30.9	309.1	25.0	800.0
86	8/30/94	11:05	11:50	90.0	246.8	50.0	800.0
87	8/30/94	13:05	14:00	90.0	310.4	25.0	800.0
88	8/31/94	07:37	08:35	65.9	304.5	25.0	800.0
89	8/31/94	10:13	11:00	48.4	268.2	50.0	800.0
90	8/31/94	12:30	13:28	98.5	335.4	50.0	800.0
Total Amount Processed				**3,841**	**27,638**		

Notes: a) Dry run, no nitrated cheesecloth introduced into the IADU.
b) Reprocessing of treatment residue generated during runs 1-55 to 800° C.
c) Processing of 39.4 g. of nitrated cheesecloth and reprocessing of treatment residue generated during run 66 to 800° C.

CERTIFICATION

I certify under penalty of law that this document and all attachments were prepared under my direction or supervision in accordance with a system designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who manage the system, or those persons directly responsible for gathering the information, the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations.

Dennis J. Erickson
Division Director for Environment, Safety, and Health Division
Los Alamos National Laboratory
Operator

06/18/01
Date Signed

Joseph C. Vozella
Assistant Area Manager, Los Alamos Area Office
U.S. Department of Energy
Albuquerque Operations
Owner/Operator

6/18/01
Date Signed