DEPARTMENT OF ENERGY • UNITED STATES OF AMERICA

PLUTONIUM FOCUS AREA

Technology Summary

August 1996

The information in this book represents information available and current through February 1996.

PLUTONIUM FOCUS AREA

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INTRODUCTION

To ensure research and development programs focus on the most pressing environmental restoration and waste management problems at the U.S. Department of Energy (DOE), the Assistant Secretary for the Office of Environmental Management (EM) established a working group in August 1993 to implement a new approach to research and technology development. As part of this new approach, EM developed a management structure and principles that led to the creation of specific Focus Areas. These organizations were designed to focus the scientific and technical talent throughout DOE and the national scientific community on the major environmental restoration and waste management problems facing DOE. The Focus Area approach provides the framework for inter-site cooperation and leveraging of resources on common problems.

After the original establishment of five major Focus Areas within the Office of Technology Development (EM-50, now called the Office of Science and Technology), the Nuclear Materials Stabilization Task Group (EM-66) followed the structure already in place in EM-50 and chartered the Plutonium Focus Area (PFA). The following information outlines the scope and mission of the EM, EM-60, and EM-66 organizations as related to the PFA organizational structure.

EM MISSION

EM is responsible for managing the cleanup of DOE wastes and nuclear materials from past nuclear weapons production and current operations. The EM mission is to bring DOE sites into compliance with environmental regulations while minimizing risks to the environment, human health and safety posed by the generation, handling, treatment, interim storage, transportation, and disposal of DOE waste. Also, the EM mission is to stabilize plutonium and other nuclear materials and to deactivate nuclear facilities.

THE OFFICE OF NUCLEAR MATERIAL AND FACILITY STABILIZATION (EM-60)

The mission of the Nuclear Material and Facility Stabilization program is to stabilize nuclear materials for the protection of workers, the public, and the environment; deactivate surplus facilities in a manner that provides savings to the government; and maintain an infrastructure to facilitate interim storage, inspection/monitoring, and final disposition of excess nuclear materials. As such, the Office provides the leadership necessary to accomplish the mission and carries out those program planning and budgeting, evaluation and intervention, and representation functions associated with the stabilization of nuclear materials and the deactivation of surplus facilities.

THE NUCLEAR MATERIALS STABILIZATION OFFICE (EM-66)

The mission of the Nuclear Materials Stabilization Office (also known as the Nuclear Materials Stabilization Task Group [NMSTG]) is to integrate the Department's programs for stabilizing excess nuclear materials to achieve safe, stable interim states for interim and long-term storage pending disposition. NMSTG functions include:

- Provide, through the Deputy Assistant Secretary, program direction and policy for the integrated management of the stabilization of excess nuclear materials.
- Designate materials within the scope of the program.
- Form and direct an Integration Working Group (IWG) to identify and evaluate stabilization requirements, capabilities, operational barriers, and integration opportunities.
- Direct the research and technology development needed to support the projects.
- *Form and direct a Plutonium Focus Area to identify research and technology requirements, evaluate proposals for addressing requirements, and prepare appropriate task directions for laboratory work.*
- Develop and supplement guidelines for Site Management Plans, including reporting vehicles necessary to monitor progress. Control changes to the Implementation Plan.
- Recommend, oversee, and/or direct trade studies necessary for determining preferred alternatives for treating and storing materials included in the program.
- Advise senior line managers of schedule variances and their impacts on commitments and progress to desired end states, and recommend appropriate management action.
- Initiate the development of standards and procedures needed for the program.
- Initiate reports to the Defense Nuclear Facilities Safety Board (DNFSB) on changes to milestones in the Implementation Plan for the Board's Recommendation 94-1. Forward an Annual Report to the Board on the progress toward meeting the commitments in the Implementation Plan.
- Initiate a quarterly report to the Under Secretary on the progress of the Department in implementing the Implementation Plan, recommending appropriate actions to address funding or progress shortfalls.

PLUTONIUM FOCUS AREA OVERVIEW

THE CHALLENGE

When the nuclear weapons program constituted the main thrust of the Department's efforts during the Cold War, the majority of fissile materials, scrap, and materials from retired weapons was recycled. During this period, it was cost-effective and timely to recover fissile material from high-assay scrap and retired weapons. As a result, little fissile material scrap was considered waste, and thus, these materials were handled, packaged, and stored with the intent of being recovered within a short period of time. When weapons production was halted in the early 1990's, large quantities of unstable materials were left in the production line or stored in a condition unsuitable for long-term stability. Consequently, residues and other processing intermediates are presently in various states at several sites under conditions that cannot assure long-term safety. With increasing frequency, the DOE complex has experienced unexpected and unsafe behavior from various materials such as excessive generation of hydrogen gas, container pressurization, generation of pyrophoric materials that threaten ignition and spread of radioactive contamination, and leakage from containers of radioactive, acidic solutions. Corrective actions are clearly needed in the short term; however, development of an adequate knowledge and technology base is required to resolve safety issues while transitioning into interim storage and final dispositioning programs.

To address these critical issues, the Defense Nuclear Facilities Safety Board (DNFSB) was established by Congress (Public Law 100-456) in September 1988. DNFSB is responsible for the independent, external oversight of all activities in the DOE nuclear weapons complex affecting public health and safety. DNFSB reviews operations, practices, and occurrences at DOE's defense nuclear facilities and makes recommendations to the Secretary of Energy regarding the protection of public health and safety.

On May 26, 1994, the DNFSB issued Recommendation 94-1, which expressed the Board's concern about nuclear materials left in the manufacturing "pipeline" after the United States halted its nuclear weapons production activities. The DNFSB emphasized the need for an integrated remediation plan for these materials. DOE accepted DNFSB Recommendation 94-1 on August 31, 1994. DOE issued an implementation plan to address these concerns ("Defense Nuclear Facilities Safety Board Recommendation 94-1 Implementation Plan," February 28, 1995) and established the Nuclear Materials Stabilization Task Group (NMSTG).

Recommendation 94-1, sub-recommendation 2 stated, "... a research program [should] be established to fill any gaps in the information base needed for choosing among the alternate processes to be used in safe interim conversion of various types of fissile materials to optimal forms for safe interim storage and the longer term disposition. Development of this research program should be addressed in the program plan called for by [the Board]."

Consequently, in March 1995 the NMSTG chartered the Research Committee (RC) to accomplish the following:

- Assess the nuclear materials stabilization program outlined in the Implementation Plan
- Formulate the 94-1 R&D (Research & Development) Plan to address the technology and core program needs of the stabilization program
- Prepare task statements defining 94-1 R&D activities required to accomplish program objectives.

The RC was disbanded in September 1995 at the issuance of Revision 0 of the 94-1 R&D Plan. Responsibility for implementing the 94-1 R&D Plan, tracking the information, and preparing updates became the charter of the Plutonium Focus Area (PFA).

PFA PRODUCT LINES

Plutonium exists within the DOE complex in various forms and concentrations, in both pure and impure states. Plutonium metals have been safely stored in "pits" (a "pit" is the core of a nuclear weapon) for decades. Other forms, including oxides and residues, were staged and recycled rather than stored. Plutonium residues such as solutions, high-hazard combustibles, and organic materials must be stabilized in the near term (i.e., three to eight years). In recent years, with significant inventories of plutonium residues in storage, several instances of packaging and container deterioration and weakening have been noted.

PFA technology baseline requirements, defined as those sets of requirements that must be addressed by existing or proposed programs, have been categorized and further defined in these six areas:

1. standards and specifications development
2. stabilization process development
3. packaging
4. surveillance and monitoring
5. transportation and remediation infrastructure
6. core technology support

1. Standards and Specifications Development

Continuing R&D work on standards will provide a better understanding of requirements for the safe stabilization and storage of plutonium. As an example, a standard has been issued for storage of plutonium metals and oxides (DOE-STD-3013). Further R&D is needed to improve the Department's understanding of how various factors influence the long-term stability of oxides. Additionally, standards are required for storage and stabilization of other 94-1 nuclear materials. Since each isotope will be stored at only one or two locations, site-specific standards provide a practical resolution to this need. Another area of standardization is development of specifications for central procurement of stabilization and packaging systems.

2. Stabilization Process Development

Stabilization process development applies to plutonium and other 94-1 materials that require stabilization before being placed in interim storage awaiting ultimate disposition. The complexity of the at-risk 94-1 plutonium inventory and the variety of physical forms (e.g., oxides, salts, ash, solutions, etc.) creates the greatest need for additional R&D.

3. Packaging

Plutonium and other actinide materials are currently stored in a variety of packages, including tanks, piping, plastic containers, and metal containers. When production operations were terminated in the early 1990s, these materials were being held for staging into production process systems. DOE-STD-3013 requires that plutonium metals and oxides be stored in dry atmospheres in hermetically sealed containers that do not contain organic materials, and promotes standard package interfaces.

4. Surveillance and Monitoring

Surveillance of packages is required to ensure that container integrity is maintained and that all materials comply with environmental, safety, and health requirements. Surveillance and monitoring processes and equipment should be standardized complex-wide. Nonintrusive surveillance offers distinct advantages over conventional methods (e.g., inspection by opening packages).

5. Transportation and Remediation Infrastructure

The technology baseline for intra-site transport requires that containers be developed and certified for the transport of large volumes of dilute plutonium solutions to facilitate stabilization. Utilization of modular treatment systems will reduce facility re-start requirements and provide stabilization capability on a more predictable schedule.

6. Core Technology Support

The objective of core technology support is to augment the knowledge base about general physical and chemical processing and about storage behavior of actinide-containing materials and to assure safe interim nuclear material storage until disposition policies are formulated. Core technology support is required from R&D organizations throughout the life of the stabilization program.

CONTACTS

William L. Scott
U.S. Department of Energy
Idaho Operations Office
785 DOE Place
Idaho Falls, ID 83401-1562
Phone: (208) 526-8189
e-mail: scottwl@inel.gov

Dr. Finis H. Southworth
Advanced Engineering
Development Laboratory
Lockheed Martin Idaho Technologies
Company
2525 Fremont Avenue
P.O. Box 1625
Idaho Falls, ID 83415-3750
Phone: (208) 526-8150
e-mail: fin@inel.gov

1.0 STANDARDS DEVELOPMENT

Continuing R&D (research & development) work on standards will provide a better understanding of requirements for the remediation and safe storage of plutonium. Although a standard has been issued for storage of plutonium metals and oxides (DOE-STD-3013), further R&D is needed to improve the Department's understanding of how various factors influence the long-term stability of oxides.

Additionally, standards are required for remediaton and storage of the remaining 94-1 materials. Even though each isotope may be stored at only one or two locations, standardized practices and containers assure smooth transfers and interfaces.

Technology baseline requirements for standards are as follows:

a. *Storage Standard for Plutonium Oxides*: The behavior of plutonium oxides must be studied in a variety of conditions and environments to ensure its long-term stability.

b. *Standards for other 94-1 Materials*: Standards must be developed for the storage of special isotopes, including americium/curium, neptunium, Pu-238, Pu-242, and other 94-1 nuclear materials.

1.1 PLUTONIUM STABILIZATION AND PACKAGING SYSTEM SPECIFICATION DEVELOPMENT

TECHNOLOGY NEED

The Plutonium Stabilization and Packaging System (PuSPS) project was driven by the need for a complex-wide plutonium (Pu) technology for inspecting, disassembling, stabilizing, repacking, and labeling failed Pu product containers. The resulting system will be obtained from one design effort and one procurement package, and yet will meet the needs of each site. This will provide necessary standardization of stabilized materials and reduce the duplication of efforts and costs that would be incurred if individual sites continued development. The standardized stabilizing, packaging, labeling and transfer system will simplify handling, accountability, inspection, identification, and transportation. Ease of inspection and some automated support features will increase safety and reduce personnel exposure. The equipment must be operable under glovebox conditions, must be automated to reduce exposure, and must stabilize and package material to acceptable standards.

TECHNOLOGY DESCRIPTION

In response to the DNFSB's Recommendation 94-1, the DOE committed to stabilize and package plutonium metal and oxide in accordance with Department standards. However, none of the affected facilities developed an integrated container for use throughout the complex or had all of the equipment necessary for completing this task in-place. The PuSPS was initiated by the NMSTG for the purpose of scoping, specifying, and conducting a procurement

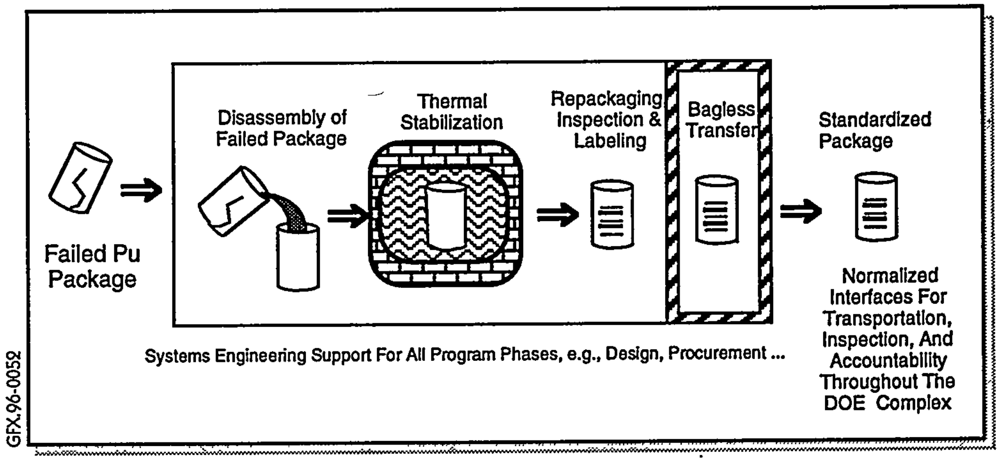

Figure 1.1-1. Functional Flow Diagram for Plutonium Stabilization and Packaging

of a standardized set of packaging and stabilization equipment to be installed at each of DOE's plutonium sites. Although this effort was initiated and completed by the NMSTG, the Plutonium Focus Area (PFA) contributed necessary technical and systems engineering support to support the standardized design.

BENEFITS

Utilization of a standardized specification for complex-wide procurement will dramatically reduce total life-cycle costs. Utilization of a standardized package size will also assure smooth transfers between sites and improved accountability. These savings will be available for investment in expanded R&D scope.

COLLABORATION/TECHNOLOGY TRANSFER

A complex-wide task team with membership from Savannah River Site (SRS), Rocky Flats Environmental Technology Site (RFETS), Los Alamos National Laboratory (LANL), Hanford, and Idaho National Engineering Laboratory (INEL) and DOE field offices was assembled to review existing designs, develop specifications, and develop the procurement package for a complex-wide piece of equipment to provide for stabilization and transfer of material. This piece of equipment will be used at various sites. The package will be procured from industry.

ACCOMPLISHMENTS

The first stage of the project consisted of scoping the procurement to include that equipment considered necessary for conducting the stabilization and packaging operations. The scope was determined by the PuSPS working group. Another part of the scoping process included an initial evaluation (i.e., trade study) of the available commercial options, which determined that vendor-supplied technologies were sufficiently mature to warrant a commercial procurement.

Another important part of the PuSPS effort was preparing documents for commercial bids. The PFA contributed mechanical engineering support by assimilating the requirements for the container into a container specification. Systems engineering support was provided to participate in development of container marking requirements and to incorporate those requirements into a container marking specification. Both of these sub-specifications were made part of the larger procurement specification.

Systems engineering support was provided to develop data item descriptions for a systems specification and a systems design document. These key documents were reviewed at the prime contractor's design review.

CONTACT

Robert Price
DOE-NMSTG
Forrestal Building
1000 Independence Avenue
Washington, D.C. 20585
Phone: (202) 586-1687
e-mail: robert.price@em.doe.gov

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None at this time.

2.0 STABILIZATION PROCESS DEVELOPMENT

Stabilization process development applies to plutonium and special isotope materials that require stabilization before being placed in interim storage. The complexity of the at-risk 94-1 plutonium inventory and the variety of physical forms (e.g. solutions, residues, etc.) creates the greatest need for additional research and development (R&D).

For ease of addressing R&D needs, plutonium is subdivided into metals, oxides, and residues, with residues being further subdivided into plutonium metal alloys and intermetallic compounds, oxide-like materials and compounds, salts, combustibles, and inorganic solids. Special isotopes include americium/curium, neptunium, Pu-238, and Pu-242.

PLUTONIUM SOLUTIONS

Plutonium solutions are acidic, inherently unstable, and corrosive; can leak from containment; and undergo continuous degradation from radiolysis. Radiolysis results in loss of acidity, plus the potential for precipitation from reaction with degradation products and impurities. Radiolysis will also generate hydrogen, which can accumulate in explosive concentrations if not vented or removed. Because of the unstable nature of these solutions, the plutonium must be separated and stabilized in the near term. Plutonium concentrations vary from less than 1 gram per liter to more than 300 grams per liter. Acid solutions exist in both nitrate and chloride forms.

Shipment of plutonium in solution to a common processing location could provide a general solution to the disposition of these materials; however, shipment of kilogram quantities of plutonium in solution via commercial transport has been expressly prohibited by the U.S. Nuclear Regulatory Commission for over 20 years. Unless an exemption is obtained from existing regulations, the processing of plutonium solutions must be performed at the sites where they are stored. Each site must assess the facilities, equipment, and technologies necessary to facilitate stabilization. Development of selected processes must be based on the safe and economic stabilization of plutonium and must be accomplished within the 94-1 schedule.

Technology baseline requirements for the stabilization of plutonium solutions are as follows:

a. *Stabilization of Plutonium Solutions*: Site-specific processes must be developed to extract plutonium from nitrate and chloride solutions for conversion into solid form and for subsequent stabilization.

b. *Equipment for use with Plutonium Solutions*: Site-specific equipment development will be continued to separate plutonium from solutions and to stabilize the plutonium into solid form.

c. *Pretreatment of Plutonium Solutions for the Vertical Calciner*: A process must be developed to remove reactive elements from plutonium solutions to facilitate more efficient operation of the vertical calciner.

d. *Post-Treatment of Plutonium Stabilization Effluents*: Processes must be developed to remove residual actinides from process solution effluents to meet site-specific liquid discharge requirements.

PLUTONIUM RESIDUES

Plutonium residues are further subdivided into five categories (as proposed in the DOE-STD-Draft-SAFT-0045). Plutonium residues are high-bulk items (>90,000 kilograms), usually containing less than 10 percent plutonium.

a. *Plutonium Metal Alloys and Intermetallic Compounds* - Materials in this category include anode heels from electrorefining, scrub alloys, and a variety of alloys in which plutonium is a minor constituent (<50% plutonium).

b. *Oxide-Like Materials* - Potential hazards associated with these materials includes gas generation, dispersion of respirable fines, flammability, and chemical reactivity. Oxide-like materials include incinerator ash, filter residues, miscellaneous plutonium compounds, dissolver heels (insolubles), sludge, and mixed oxides.

c. *Pyrochemical Salts* - Potential hazards associated with pyrochemical salts include corrosion of containers, gas generation from radiolysis of salts, and the presence of reactive metals. These salts were generated from electrorefining, molten salt extraction, direct oxide reduction, and other pyrochemical operations, including salt scrub, molten salt extraction from pyrochemical operations and large inventories of sand, slag, and crucible (SS&C) residues from metal reduction operations.

d. *Plutonium-Contaminated Combustibles* - Potential hazards associated with plutonium-contaminated combustibles include spontaneous combustion from nitrated combustibles and/or pressure buildup from gas generation due to radiolysis. Such combustibles include cartridge filter elements, graphite, rubber (leaded and nonleaded gloves), ion exchange resins, plastics, and cellulose materials (e.g., paper, wipes).

e. *Inorganic Solids* - Potential hazards associated with inorganic solids include gas generation and a release involving respirable fines containing plutonium. Inorganic solids include insulation, ceramics, contaminated scrap metals, fire brick, and LECO crucibles. Although combustible constituents are usually not present, their solids may be stored in plastic.

Technology baseline requirements for stabilization of plutonium residues are outlined in the 94-1 R&D plan.

SPECIAL ISOTOPES

Special isotopes (Pu-238, Pu-242, and isotopes of neptunium and americium/curium) in solution will be stabilized. The americium/curium mixtures contain other actinides, including plutonium.

2.1 RESEARCH AND DEVELOPMENT REQUIREMENTS "GAP" ANALYSIS

TECHNOLOGY NEEDS

The gap analysis supports both near-term (3 to 8 years) and long-term stabilization technologies. By identifying preferred end states and by developing preferred processes which focus on specific requirements, dilution of research efforts and funding will be minimized.

TECHNOLOGY DESCRIPTION

As part of the Plutonium Focus Area's (PFA) responsibilities to implement the R&D plan and use a systems engineering approach, the PFA has begun an analysis of requirements for research. These requirements will be used to assess the need for proposed research and to evaluate progress of on-going research. The definition of requirements is focused on collection and correlation of sets of research requirements and activities from three sources: the NMSTG 94-1 Implementation Plan, the 94-1 R&D Plan, and the on-going research currently planned or in-place at DOE sites.

Correlation of these requirements results in mismatches or gaps between what is being done and what needs to be done, called "gaps". These can be from research called for by the 94-1 Implementation Plan but not identified in the R&D Plan; research called for by the 94-1 R&D Plan, but not by the Implementation Plan; research being performed or being planned at DOE sites, but not identified in the R&D plan; and new research suggested by sites, universities, and commercial organizations.

The gap analysis will result, as shown in Figure 2.1-1, in a single list of research requirements and activities called the Baseline Research and Development Technical Requirements Document.

BENEFITS

The value of the gap analysis is to focus effort and funding on the research areas that are needed most. It also provides a means to identify peripheral, related research which should be followed to support NMSTG needs. It is efficient to have such a straightforward checklist to enable researchers to go directly to a source for requirements without having to spend time analyzing documents to be sure none are missed.

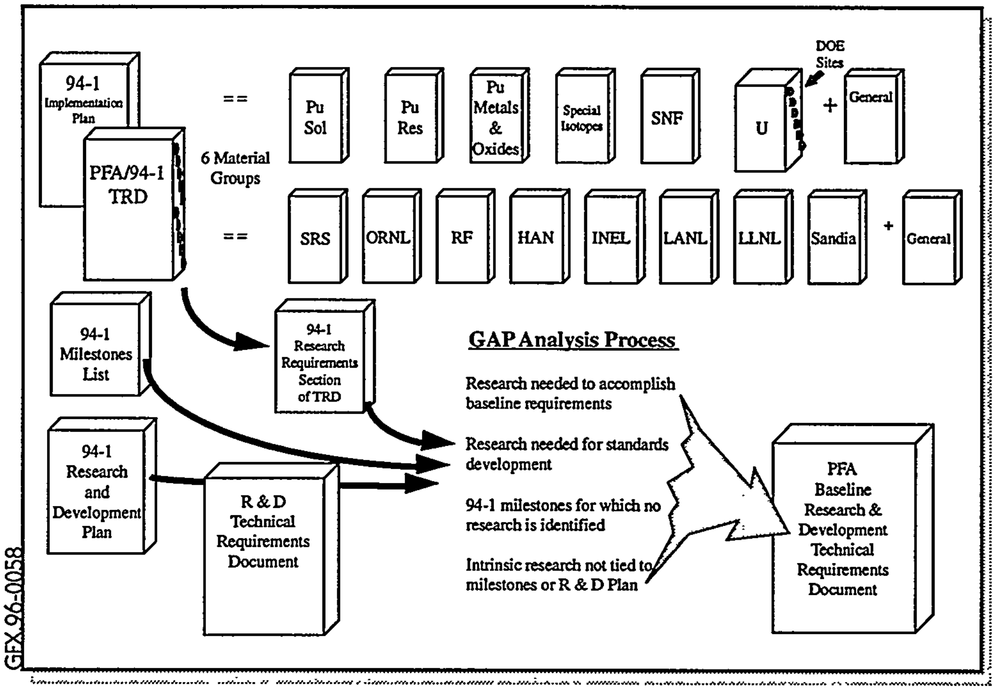

Figure 2.1-1. The Gap Analysis Process

COLLABORATION/TECHNOLOGY TRANSFER

The gap analysis will provide a baseline for PFA's TAP (Technical Advisory Panel) in their solicitations for collaborative research with industry and universities. Resolution of requirement conflicts will reduce the technology transfer cycle time and overall cost.

ACCOMPLISHMENTS

To help define the present gaps, complete, succinct statements of requirements are developed from source documents in the form of "requirements analyses." These are formed into lists of requirements statements and are published as Baseline Research and Development Technical Requirements Documents (TRDs).

CONTACTS

Don Schilling
Advanced Engineering
Development Laboratory
Lockheed Martin Idaho
Technologies Company
2525 Fremont Avenue
P.O. Box 1625
Idaho Falls, ID 83415-3750
Phone: (208) 526-0248
e-mail: dons@inel.gov

Dr. C. Robert Kenley
Advanced Engineering
Development Laboratory
Lockheed Martin Idaho
Technologies Company
Forrestal Building
Washington, D.C. 20585
Phone: (202) 586-6183
e-mail: kenlcr@inelmail.inel.gov

Dr. Finis H. Southworth
Advanced Engineering
Development Laboratory
Lockheed Martin Idaho Technologies Company
2525 Fremont Avenue
P.O. Box 1625
Idaho Falls, ID 83415-3750
Phone: (208) 526-8150
e-mail: fin@inel.gov

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2.2

CERAMIFICATION FOR STABILIZATION AND IMMOBILIZATION OF PLUTONIUM-CONTAINING COMPOUNDS

TECHNOLOGY NEEDS

DOE has over 5 metric tons (MT) of plutonium in the form of plutonium oxide across the complex, of which over 3 MT are maintained at RFETS. Although the amount of plutonium oxide is relatively small, plutonium oxide poses the largest risk among plutonium compounds of interest.

Ceramification is appropriate for stabilization and immobilization of oxide because it has the potential to eliminate or reduce the respirable fraction of oxides by a factor of 60 or better for the same cost and time as required for calcination at 950°C. Calcination reportedly reduces the respirable fraction of oxides by a factor of three to ten (Conrad, et al., 1995, Rickets 1995). Ceramification offers reduction in risk equivalent to calcination with no additional cost or schedule delay.

TECHNOLOGY DESCRIPTION

Ceramification is a new plutonium stabilization/immobilization process under development at RFETS. Ceramification, which is based on a coating process proven in weapons applications, is appropriate for the stabilization and immobilization of plutonium solutions, plutonium oxides, and some other respirable and dispersible plutonium compounds (i.e., residues). In the process, these plutonium compounds are converted into solid plutonium oxide articles appropriate for low-risk, interim storage requiring minimal surveillance. Portions of this process are proprietary and are not presented in detail.

During ceramification, a bonding precursor is added to the dispersible plutonium matrix and thermally decomposed at 300°C to 600°C. The thermal decomposition products of the precursor bond the matrix into a solid, nondispersible porous ceramic. To improve strength, the precursor is reapplied to the solid article, which is then thermally cured a second time. This treatment scheme can be repeated as many times as necessary to achieve a desired strength. Additionally, strength can be improved by performing the ceramification process directly in a reinforced stainless steel vessel to which the final solid oxide article will be intimately bonded. The ceramic-filled stainless steel vessel can be considered a single composite article, with properties being a summation of all materials within the bounds of the vessel. Figure 2.2-1 provides a simple process flow diagram for ceramification.

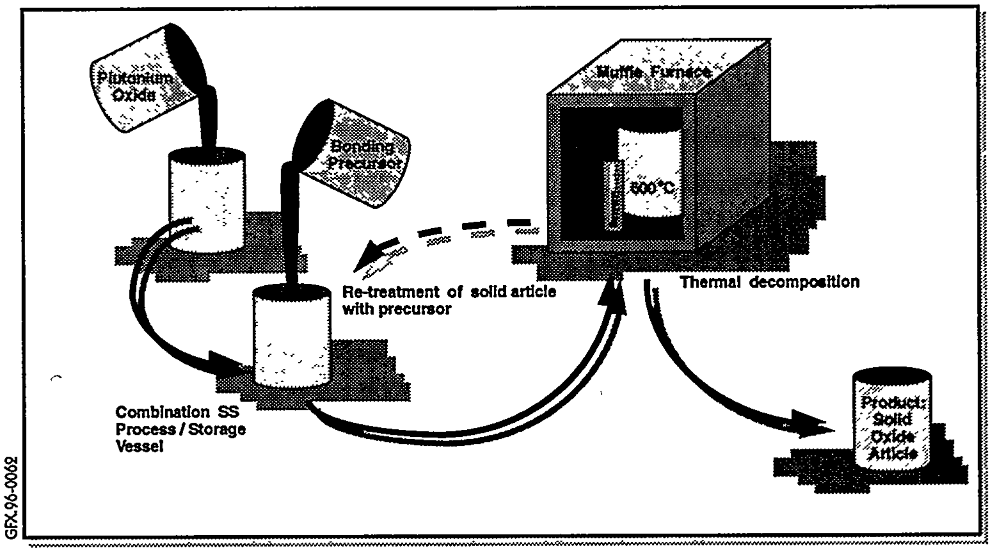

Figure 2.2-1. The Ceramification Process

The final products of ceramification can be tailored to meet DOE's stabilization and reuse objectives. Final homogeneous products can consist of essentially pure plutonium oxide or plutonium oxide blended with additives to increase strength, increase proliferation resistance, and/or incorporate neutron absorption.

BENEFITS

Ceramification eliminates or reduces the dispersibility of plutonium compounds and significantly reduces safety requirements and risks of interim storage and transport. A nondispersible product reduces potential contamination of facilities, equipment, and personnel during material handling. Ceramification produces articles with high waste loading and little or no volume increase. Achievable plutonium loadings in the solid article are estimated at more than 80% plutonium oxide. Final articles are homogeneous. Ceramification is carried out at low process temperatures (300 to 600°C). Full-scale processing times have ranged from one to three hours in batch tests.

COLLABORATION/TECHNOLOGY TRANSFER

RFETS advertised for an industrial partner to commercialize this technology in the Commerce Business Daily in FY95. Several interested partners have been identified and are being considered.

ACCOMPLISHMENTS

In FY95, ceramification was successfully demonstrated on surrogates including cerium oxide, zirconium oxide, aluminum oxide, and cerium nitrate. Full-scale (equivalent to 1 kg plutonium), nondispersible articles were produced using prescribed precursors at 300°C to 600°C in under two hours of total treatment time, with three re-treatments to provide strength to the article.

Funding to develop ceramification at RFETS for immobilization of plutonium compounds was allocated in the second-quarter of FY96. This funding will be used to verify the performance of ceramification on plutonium oxide, plutonium nitrate, and plutonium contaminated ash in FY96.

CONTACTS

W. C. Rask
Department of Energy
RFETS Field Office
Mailstop Bldg. 460
P. O. Box 928
Golden, CO 80402-0928
Phone: (303) 966-2648
no e-mail capability

A. G. Phillips
Safe Sites of Colorado
Rocky Flats Environmental Technology Site
Mailstop Bldg. 881
P. O. Box 464
Golden, Colorado 80402-0464
Phone: (303) 966-4346
no e-mail capability

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Rickets, T. "Mass Fraction of Respirable and Dispersible Particles as a Function of Processing History and Thermal Treatment," *Letter Report to Jerry Stakebake*, RFETS Environmental Technology Site (October 19, 1995).

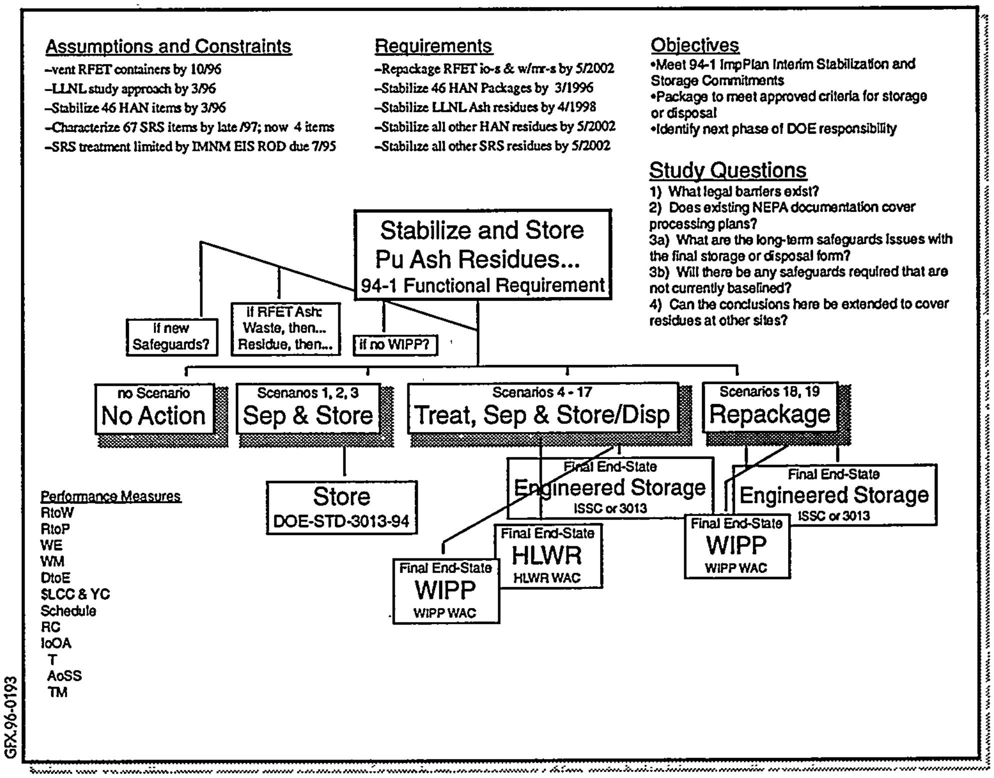
2.3 TRADE STUDY ON END STATE OF ASH RESIDUES (ESAR)

TECHNOLOGY NEEDS

The Trade Study is intended to identify the best possible technologies for the ash residues issue based on technical considerations. The recommended technologies are presented to the NMSTG, which is responsible for incorporation of stakeholder concerns, policy issues, and other non-technical concerns.

TECHNOLOGY DESCRIPTION

The PFA has begun an analysis to determine the preferred method for dealing with plutonium-bearing ash residues located at RFETS, LANL (Los Alamos National Laboratory), Hanford, SRS (Savannah River Site), and other sites. This effort seeks the most desirable pathway(s) to an acceptable end-state, using efficiency, cost, waste, facility capabilities, worker safety and public safety as elements of the preference ranking criteria.



Assumptions and Constraints
-vent RFET containers by 10/96
-LLNL study approach by 3/96
-Stabilize 46 HAN items by 3/96
-Characterize 67 SRS items by late /97; now 4 items
-SRS treatment limited by IMNM EIS ROD due 7/95

Requirements
-Repackage RFET io-s & w/mr-s by 5/2002
-Stabilize 46 HAN Packages by 3/1996
-Stabilize LLNL Ash residues by 4/1998
-Stabilize all other HAN residues by 5/2002
-Stabilize all other SRS residues by 5/2002

Objectives
•Meet 94-1 ImpPlan Interim Stabilization and Storage Commitments
•Package to meet approved criteria for storage or disposal
•Identify next phase of DOE responsibility

Study Questions
1) What legal barriers exist?
2) Does existing NEPA documentation cover processing plans?
3a) What are the long-term safeguards issues with the final storage or disposal form?
3b) Will there be any safeguards required that are not currently baselined?
4) Can the conclusions here be extended to cover residues at other sites?

Performance Measures
RtoW
RtoP
WE
WM
DtoE
$LCC & YC
Schedule
RC
IoOA
T
AoSS
TM

Figure 2.3-1. Requirements and Alternatives Considered by the ESAR Trade Study

The task is conducted by the Trade Study Group, comprised of members from sites impacted by the study and those persons having interest in its outcome.

The study requires consideration of the 94-1 milestones, the initial conditions, and the assumptions and questions shown in Figure 2.3-1. The categories of alternatives that must be considered to meet the requirements are also shown in the boxes at the bottom of the figure.

The task requires, for each alternative assessed, identification of a process description, facility requirements, equipment requirements, throughput capability, storage location and capacity, surveillance and monitoring activities, transportation requirements, and National Environmental Policy Act (NEPA) requirements.

Each alternative will be scored in terms of nine Performance Measures (PM): Risk to Worker (RtoW); Risk to Public (RtoP); Worker Exposure (WE); Secondary Waste Generation (WM); Discharge to Environment (DtoE); Cost (LCC); Schedule; Regulatory Compliance (RC) and Impact on Other Activities (IoOA).

BENEFITS

The study is the first of a three-part effort to contribute to a programmatic decision by NMSTG. Part III will involve a decision based on both the technical basis provided by the trade study and other, non-technical factors identified in Part II.

COLLABORATION/TECHNOLOGY TRANSFER

The Ash Trade Study is in progress as of this writing, and as such collaboration/technology transfer is premature.

ACCOMPLISHMENTS

The study team identified and investigated 28 specific process-scenario combinations, 19 of which are currently being evaluated and ranked according to Performance Measure (PM) scores. Performance Measure algorithms are also being developed by the Trade Study Team.

Issues and candidate options for resolution have been defined. Issues center on the best dispositioning approach, given the balance of inventory among sites and the plutonium concentration, which are indicated in Figure 2.3-2. Other issues include:

- Impact of certain inventories' classification outside the "residues" category
- Transportation regulations
- Safeguards impacts
- Concerns about the likelihood of achievement of end states

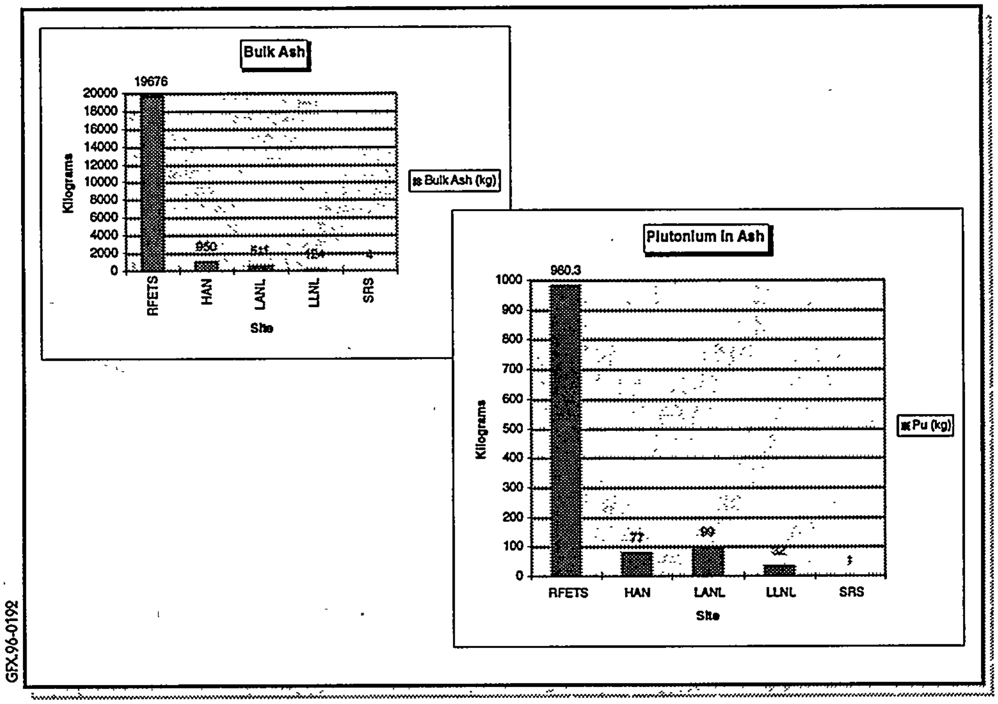


Figure 2.3-2. Ash and Plutonium Inventory Distribution

CONTACTS

Charles Gilmore
Department of Energy,
Idaho Operations Office
850 Energy Drive
Idaho Falls, ID 83401-1563
Phone: (208) 526-1808
e-mail: gilmorce@inel.gov

Don Schilling
Advanced Engineering
Development Laboratory
Lockheed Martin Idaho
Technologies Company
2525 Fremont Avenue
P.O. Box 1625
Idaho Falls, ID 83415-3750
Phone: (208) 526-0248
e-mail: dons@inel.gov

Dr. Finis H. Southworth
Advanced Engineering
Development Laboratory
Lockheed Martin Idaho Technologies
Company
2525 Fremont Avenue
P.O. Box 1625
Idaho Falls, ID 83415-3750
Phone: (208) 526-8150
e-mail: fin@inel.gov

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2.4 HIGH-GRADIENT MAGNETIC SEPARATION

TECHNOLOGY NEED

Caustic or neutral liquid waste generated by chloride processing operations in the Los Alamos National Laboratory Plutonium Facility (TA-55) can produce large volumes of liquid with an average alpha activity of 4500 mCi/L. The liquid effluents are transferred to the Los Alamos Waste Management Facility (TA-50) for treatment and disposal, ultimately producing several tons of TRU (transuranic) solid waste per year. In order to avoid the TA-50 treatment, the goal at TA-55 is to reduce TRU activity in the process waste streams to the industrial waste disposal criteria (<0.52 mCi/L).

TECHNOLOGY DESCRIPTION

High-Gradient Magnetic Separation (HGMS) is a technology that can be used selectively to extract plutonium contaminants from liquid waste streams. HGMS is capable of concentrating the actinides in process waste streams to form a low-volume, actinide-rich stream for subsequent processing and a high-volume, actinide-lean stream for direct discard to the industrial waste stream. HGMS applies intense magnetic fields to separate actinide particles from other materials. In HGMS, a particulate slurry is passed through a magnetized matrix volume. Magnetic particles (i.e. actinide oxides and hydroxides) are extracted from the slurry by the matrix while the nonmagnetic fraction passes through the magnetized volume. The magnetic fraction later is flushed from the matrix to produce a low-volume residue that can be recycled, stored, or treated for disposal. HGMS effectively addresses ~0.3 to 90+ sized particles. The majority of radioactive components in contaminated slurries have been found to concentrate in the fine particle size range (<35 +), which is effectively treated by HGMS.

On a laboratory scale at TA-55, the proposed technology has been successfully demonstrated with a specific chloride waste stream. Results from screening experiments on TA-55 caustic liquid waste water effluent indicate that >99.6% extraction of plutonium activity can be achieved using HGMS. These results represent decontamination levels of three orders of magnitude to 0.2 mCi/L, which meets the industrial waste water discharge criteria.

Initial work requires the continued testing of the varied TA-55-produced radioactive waste streams. Neutralized samples from both chloride and nitric acid processes will be tested for HGMS feasibility. Pretreatment protocols and processing protocols will be established. A decision to proceed will be made at the completion of these laboratory-scale tests. Once the HGMS feasibility is established, the engineering design for TA-55 implementation will be conducted in the second year. The final phase of work is the purchase, installation, and testing of a magnet unit for implementation in the following year at TA-55.

BENEFITS

Implementation of HGMS will offer a reduction in TRU waste produced by TA-50. Up to 15 TRU drums at TA-50 originally containing 3,000 L of radioactive caustic liquid waste will be eliminated. In the new process, 0.24 kg of plutonium and approximately 0.02 kg of americium can be concentrated and collected at TA-55. This material can then be recycled or disposed of in approximately 2 drums of TRU cemented waste.

A cost savings benefit is also realized by the reduction of the generated TRU waste, discussed above. In addition, a significant cost savings will occur in one of two other potential sources: either (1) reduced need for the current Pretreatment Room at TA-50 (approximately $500,000 operational expense), or (2) elimination of a full-sized TRU pretreatment facility and separate collection system in the planned Radioactive Liquid Waste Treatment Facility (approximately $6,800,000 capital expense). With TRU liquid waste treatment contained at TA-55, the need for a TRU pretreatment facility and separate collection system to comply with Hazard Class II standards is also eliminated.

Physical separation processes, such as HGMS, are particularly attractive because no additional waste is generated during processing. HGMS is capable of concentrating the actinides in process waste streams to form a low-volume, actinide-rich stream for subsequent processing and a high-volume, actinide-lean stream for industrial waste discard. In addition, treatment of TA-55 effluent prior to off-site treatment would eliminate the hazard of pumping radioactive liquid waste to off-site facilities. This would also eliminate worker radiation exposure at the off-site facility.

COLLABORATION/TECHNOLOGY TRANSFER

This is a new initiative. Opportunities for collaboration and technology transfer are currently being solicited.

ACCOMPLISHMENTS

A task plan has been developed to outline the technology development requirements. This activity is currently unfunded.

CONTACT

Laura A. Worl
Principal Investigator
MS E510
Los Alamos National Laboratory
Los Alamos, NM 87545
Phone: (505) 665-7149
e-mail: lworl@lanl.gov

BIBLIOGRAPHY OF KEY PUBLICATIONS

None at this time.

2.5 SALT DISTILLATION

TECHNOLOGY NEEDS

The 16 metric tons (MT) of pyrochemical salts at RFETS contain about 1 MT of plutonium. Eleven MT of these salts are composed of a sodium chloride-potassium chloride matrix. The remainder consists of a calcium chloride matrix. DOE has committed to mitigate the problems associated with 6 MT of high-hazard pyrochemical salts at RFETS by May 1997, and an additional 4 MT by December 1997. These residues may pose a safety risk if the reactive metals in the residues come into contact with water, creating hydrogen gas that could cause container pressurization. Even if no hazard from reactive metals is present, these salts can adsorb moisture from the atmosphere, resulting in corrosion and breach of containment. The full 16 MT inventory is to be made safe by May 2002. In addition to mitigation of hazards, these residues must also be made acceptable for eventual disposal. An additional 2 tons of these salt residues exist at Los Alamos and require stabilization by May 2002.

TECHNOLOGY DESCRIPTION

Equipment designed to separate pyrochemical salts into a very lean salt fraction (<100 ppm plutonium) and plutonium oxide meeting the requirements of DOE-STD-3013 will be obtained, assembled, tested and demonstrated.

Distillation separation is based on the large difference in vapor pressures at high temperature between chloride salts, which constitute most pyrochemical residues, and the actinide oxides. However, the plutonium content in these salts can be in the form of plutonium trichloride. Vapor pressure differences between alkali and alkaline earth chlorides and plutonium trichloride are too small to effect a good separation, therefore $PuCl_3$ must be converted to an oxide through an oxidation process. The oxidation process developed at Los Alamos uses carbonate salts to act as an oxidant. This has proven very

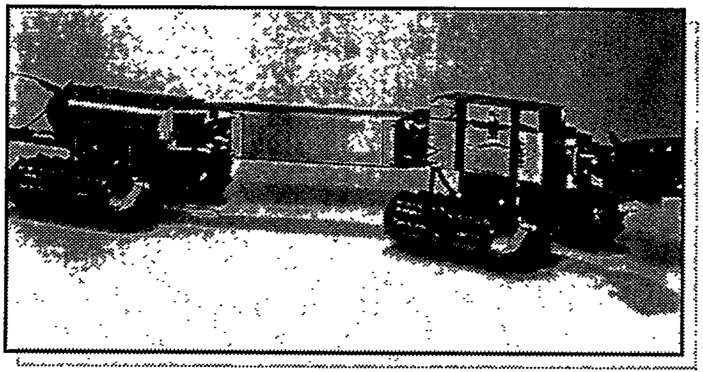

Figure 2.5-1. Results of Modeling of Plutonium Concentration in Distilled Salts

effective in converting all plutonium species into plutonium dioxide and can be ultimately combined with a distillation separation process.

The vapor pressure of sodium chloride and potassium chloride at 850°C is about 1 torr. The vapor pressure of plutonium dioxide at this same temperature is 10^{-16} torr. This tremendous difference in physical properties forms the basis for a very efficient physical separation. Simple modeling results, shown in Figure 2.5-1, indicate that the plutonium concentration in sodium chloride-potassium chloride salts can be reduced to 10^{-10} ppm. While it is theoretically possible to produce a salt that would meet the criterion for low-level waste (LLW) of 100 nCi/g (~1 ppm), efforts are focused on obtaining a product salt that contains <100 ppm plutonium. At this level of contamination the salts are still TRU waste, but 55-gallon drums can be filled on a volume limitation of the waste rather than a radionuclide loading. This can result in a 10-100 fold decrease in the amount of drums sent to the Waste Isolation Pilot Plant (WIPP).

BENEFITS

Separation of the plutonium from the waste salts will lead to a large reduction in the cost of disposal, even if the salts do not meet LLW disposal criteria. Current NRC (Nuclear Regulatory Commission) Certificate of Compliance (CofC) would result in an average maximum of 23 fissile gram equivalents (FGE) per 550 gallon drum. In the best possible circumstance this would lead to over 50,000 drums for disposal at WIPP. A request has been submitted to the NRC to amend the CofC to allow up to 200 FGE per drum within a 14-drum TRUPACT (shipping container for transuranic waste). This would still result in 8,000 drums. If the plutonium in the salts can be reduced to below 100 ppm, a drum could be filled with salt without impacting even the present 25-g plutonium limit. In this case, about 200 drums would be generated for WIPP disposal. At a cost of $10K per drum, the cost savings realized could total tens of millions of dollars. The separated plutonium, consisting of 1 MT of plutonium dioxide, could be packaged per DOE-STD-3013. Costs incurred by storage of the plutonium oxide would be greatly offset by savings realized from WIPP disposal. Recent estimates of the total cost to process the salt inventory at RFETS by distillation are $71 million, compared to $103 million for disposal at WIPP with modified NRC CofC, and $534 million with present NRC CofC.

COLLABORATION/TECHNOLOGY TRANSFER

As this is a new initiative, opportunities for collaboration and technology transfer are currently being solicited.

All electrical, thermocouple, and vacuum feedthroughs and fittings needed for glovebox installation were installed. A new cover incorporating the appropriate vacuum feedthrough to replace the existing furnace well cover was designed and fabricated. This new cover, along with required fittings and feedthroughs, is now in place in the glovebox.

The vacuum seals, where the distillation and receiver chambers are loaded and unloaded, are water cooled o-rings. However, the spool connecting the two chambers must be at an elevated temperature to permit smooth flow of salt vapors. Tests were conducted on the high-temperature metal c-ring seals used in this spool. An acceptable performance criterion was established for the vacuum seal at <0.01 torr. The results of the test runs are marginal, and efforts are being made to improve seal characteristics.

Uncontaminated salts were used to verify the feasibility of the salt distillation process. These tests employed existing equipment and have been used to determine distillation rates as a function of temperature. A target rate of 3 kg per unit per day had been established. Results for sodium chloride-potassium chloride indicated that such a rate was easily achievable. Distillation rates for calcium chloride were found to be too slow below 1100°C. Calculated distillation rates are shown in Figure 2.5-2. Further study is required to determine the applicability of distillation for the calcium salt mixtures. These tests with uncontaminated salts have also been used to provide input for equipment designed expressly for the salt distillation process. The process was demonstrated on a small scale with actual contaminated salts, and these runs showed decontamination to less than glovebox interior level.

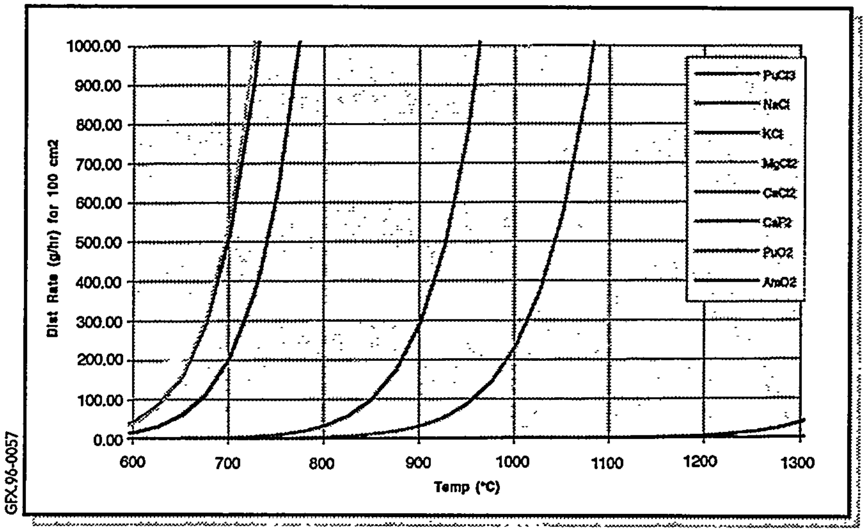

Figure 2.5-2. Calculated Distillation Rates for Various Chloride Salts

Individual components will be assembled and tested prior to introduction into a glovebox environment. The equipment will then be introduced into an existing plutonium-contaminated glovebox. After assembly in the glovebox, testing will be scaled up to 3 kg per day runs. Once testing is complete, a full-scale processing run will be performed to establish reliability and robustness of the process and equipment.

Fabrication of the Inconel parts is progressing. Problems encountered by the manufacturer in machining to the design specifications have now been resolved. All other components and sub-assemblies have been fabricated and received.

CONTACT

Dr. Eduardo Garcia
Principal Investigator
MS E511
Los Alamos National Laboratory
Los Alamos, NM 87545
Phone: (505) 667-0794
e-mail: egarcia@lanl.gov

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2.6 ELECTROLYTIC DECONTAMINATION

TECHNOLOGY NEEDS

Repackaged containers must be contamination-free for out-of-line interim storage, and container weld ends must remain hermetically sealed after cleaning.

TECHNOLOGY DESCRIPTION

The task is designed to decontaminate material containers so that these containers can be released into the laboratory room for handling outside the glovebox environment.

Electrolytic decontamination is a technology under development for several applications. The original purpose of the technology was to decontaminate equipment and uranium weapons parts for disposal at RFETS, and it has been successfully demonstrated for these applications. Its potential for providing contamination-free containers as part of an automated system led to the integration of electrolytic decontamination with the Sandia can-out system, a robotic-assisted method for removing filled containers from the glovebox. It is also being applied as the means of container decontamination in the ARIES pit conversion project. The 94-1 R&D project supports electrolytic decontamination to demonstrate its utility in removing contamination from the exterior of containers.

BENEFITS

Once the material can is set up in the fixture, no more handling by operators is required during the decontamination process. This minimizes radiation exposure to personnel. The electrolyte solution is recycled, which minimizes waste generation.

COLLABORATION/TECHNOLOGY TRANSFER

This project has collaborative efforts with Sandia for the automatic bagless can-out, and with Lawrence Livermore National Laboratory, who is a partner with Los Alamos in the ARIES project.

ACCOMPLISHMENTS

Preliminary hot demonstration tests of electrolytic decontamination of material containers are being conducted.

Three empty externally contaminated 304 stainless steel material containers, which had been contaminated in the glovebox line of the repackaging project, were decontaminated below the specifications for release into the laboratory room. The swipeable limit for room release is 20 disintegrations per minute (dpm)/100 cm^2. For the sides of the containers, contamination was reduced from an average direct alpha contamination reading of 25,000 dpm/100 cm^2 and an average swipeable alpha contamination reading of 5,000 dpm/100 cm^2, to no detectable and no swipeable contamination. For the weld ends of the containers that make contact with the glovebox floor, these hermetically sealed containers were cleaned from an average direct alpha contamination reading of 200,000 dpm/100 cm^2 and an average swipeable alpha contamination reading of 10,000 dpm/100 cm^2, to less than the required 500 dpm/100 cm^2 direct contamination and no swipeable contamination.

After cleaning, these containers were helium leak-tested to 1×10^{-7} std cm^3/s as required by DOE-STD-3013, as they had been prior to cleaning, and no detectable leaks were found. The recycled electrolyte was analyzed by liquid scintillation and gas proportional alpha counting, and no detectable plutonium could be found. Precipitate analysis verified plutonium contamination in the precipitate. An ultrafiltration testing report was issued. Ultrafiltration would be an upgrade to the Buchner funnel method currently used.

CONTACT

Tim O. Nelson
Principal Investigator
MS E510
Los Alamos National Laboratory
Los Alamos, NM 87545
Phone: (505) 667-2326
e-mail: TON@lanl.gov

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2.7 EXTRACTION CHROMATOGRAPHY

TECHNOLOGY NEED

Plutonium purification and processing operations produce aqueous effluent streams containing americium-241 and a small fraction of plutonium.

TECHNOLOGY DESCRIPTION

The processes developed in this work will treat aqueous effluent streams at the source, in order to selectively extract actinide elements. Chromatographic treatment of these effluents offers actinide extraction advantages, including chemical specificity, moderately high loading capacity, multistage character, and rapid kinetics. Successful implementation will effect a separation of the effluent stream into a small volume of high-actinide concentration and the original aqueous effluent stream, now decontaminated of most alpha activity. The concentrated actinides will be recovered in relatively pure form suitable for vault storage, additional recovery operations, or disposition. Downstream treatment operations on the aqueous effluents will be simplified and made less hazardous due to the reduction of radioactivity.

Extraction chromatography with organic extractants has been applied full scale at Los Alamos National Laboratory (LANL) to high-concentration (5 to 8M) hydrochloric acid effluent streams from solvent extraction and anion exchange processing using commercially available materials. More than a dozen full-scale tests have been performed, removing tens of grams of plutonium and americium from hundreds of liters of process effluent. This work has suggested several engineering and process chemistry improvements. Extractive resins will be selectively developed to remove actinides from high-concentration nitric acid ion-exchange effluents and from lower-acid-concentration effluents typical of oxalate or hydroxide filtrates from hydrochloric acid and nitric acid operations.

A carbamoylmethylphosphine oxide (CMPO) derivative has proven to be more efficient for extraction of trivalent actinides from hydrocholoric acid effluent streams. A large amount of this ligand will be synthesized to continue full-scale implementation of extraction chromatography for this process. A series of CMPO derivatives, which are unknown in the literature and systematically varied in structure, have been synthesized. These will be characterized fully and loaded onto inert support materials for testing. A select group of organic and inorganic actinide absorber materials will be tested at small scale with actinide solutions and competing elements under conditions similar to those observed in the nitric acid and hydrochloric acid process effluent solutions. The promising candidate absorber materials will be obtained in larger quanties and tested at a pilot scale with actual effluents from plutonium processes.

BENEFITS

Application of actinide extraction technologies to effluent streams at the source minimizes the volume of effluent that must be treated, and simplifies the separations chemistry requirements. The improved separations result in the following: (1) production of relatively pure actinide oxides for storage or disposition; (2) avoidance of dilute actinide residues of high activity that require vault storage or additional processing; and (3) reduction in activity levels, hazards, and exposures associated with storage and handling of the final solid waste types generated.

COLLABORATION/TECHNOLOGY TRANSFER

This is a new initiative. Opportunities for collaboration and technology transfer are currently being solicited.

ACCOMPLISHMENT

A task plan has been developed to outline the technology development requirements. This activity is currently unfunded.

CONTACT

Gordon D. Jarvinen
Principal Investigator
MS E510
Los Alamos National Laboratory
Los Alamos, NM 87545
Phone: (505) 665-0822
e-mail: gjarvinen@lanl.gov

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None at this time.

2.8 PYROCHEMICAL SALT OXIDATION

TECHNOLOGY NEED

Treatment methods are needed for oxidizing wet or reactive pyrochemical salts to satisfy the near-term Defense Nuclear Facilities Safety Board (DNFSB) 94-1 schedule for reducing, within three years, the risk of potentially unstable residues.

TECHNOLOGY DESCRIPTION

This project will continue development of chemical oxidation approaches to treating spent pyrochemical salts in order to mitigate all safety concerns regarding interim on-site storage. The effort will focus on optimization of the oxidation process that prepares feed for the salt distillation process that requires oxidized salt as feed. Parametric studies will be performed to minimize the amount of oxidant needed to ensure that the oxide heel from the distillation process has the maximum special nuclear material (SNM) concentration and that the distilled salt has the minimum amount of actinide. This is a continuation of a previous effort in which the chemical oxidation parameters for electrorefining, molten salt extraction, and direct oxide reduction salts were developed to stabilize pyrochemical salts in standard pyrochemical furnaces with chemical oxidants. Effectiveness of treatment will be verified by determination of the hydrogen gas evolution of treated salts in water and by the performance of the oxidized salt in the distillation process. Manpower, equipment, and facility needs will be identified for each oxidation technique, and recommendations for treatment processes will be provided. Upon completion of the near-term process development efforts, personnel from RFETS will come to Los Alamos National Laboratory to participate in the development effort for transfer of the process to RFETS. Crucibles from pyrochemical processes contain salt residues and therefore have the same potentially reactive species and stability concerns as pyrochemical salts. Another goal of this project will be to develop a process to chemically oxidize and to stabilize crucibles for safe, interim storage.

BENEFITS

Successful completion of this phase of the project will fully integrate the oxidation process into a feed treatment process for the salt distillation process. The coupled process will result in a stabilized oxide product and a salt residue that is extremely low in actinide concentration and that can be shipped to the Waste Isolation Pilot Plant (WIPP) in the least number of containers. The processing techniques will be directly applicable to meeting 94-1 concerns at LANL and at RFETS, and to meeting these concerns in the most cost-effective manner.

COLLABORATION/TECHNOLOGY TRANSFER

This is a new initiative. Opportunities for collaboration and technology transfer are currently being solicited.

ACCOMPLISHMENTS

- Completed production demonstration on molten salt extraction, electrorefining, and direct oxide reduction salts
- Completed Sensitivity Matrix for molten salt extraction salts
- Completed Sensitivity Matrix for direct oxide reduction salts
- Issued final report for RFETS on Salt Oxidation Project for October 1995 through March 1996
- Initiated contract with Kaiser-Hill for joint development project on crucible oxidation process.

CONTACT

James A. McNeese
Principal Investigator
MS E511
Los Alamos National Laboratory
Los Alamos, NM 87545
Phone: (505) 665-0152
e-mail: jmcneese@lanl.gov

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None at this time.

2.9 POLYCUBES

TECHNOLOGY NEED

Approximately 1600 polycubes are being stored at Hanford. The cubes range in size from 1/2 in. X 2 in. X 2 in. up to 2 in. X 2 in. X 2 in. and are sealed with a coating of aluminum paint and/or tape. The cubes are packaged in vented food pack cans with approximately 5-8 cubes per can. The objective of this work is to design, test, and implement a pyrolysis system that will effectively destroy and remove the polystyrene matrix from the plutonium and uranium oxides, leaving the oxides as free-flowing powders that are suitable for interim storage. The pyrolysis system will be designed with a series of downstream technologies to treat the pyrolysis off-gas, allowing it to operate in compliance with environmental regulations.

TECHNOLOGY DESCRIPTION

Pyrolysis is a pyrochemical technique using a high-temperature inert environment to break down and volatilize polymeric materials. Los Alamos National Laboratory has recently designed a gas-tight pyrolysis reactor that is suitable for glovebox applications. Preliminary tests have shown that this design allows for nearly 100% removal of the polystyrene without the formation of tars or other constituents that could adversely affect the reactor's operation. At this point, a series of downstream technologies need to be introduced to break down the components in the reactor effluent to an environmentally benign form. This is a necessary step in allowing pyrolysis to operate in a manner that is both safe and consistent with environmental concerns. Three approaches to off-gas treatment are to be considered in this program. Each is designed to oxidize the organic constituents to carbon dioxide and water.

<u>Catalytic Conversion</u>. In this approach, the off-gas is fed with air to a catalytic converter maintained at an elevated temperature. The catalyst allows the oxygen to oxidize the organic constituents more effectively.

<u>Secondary Combustion</u>. This approach uses a resistance heating element, the pyrolysis off-gas, and oxygen to oxidize the products of pyrolysis. Typical commercial operating parameters necessary to meet environmental requirements are a residence time of 2 s at a temperature of 1000°C.

<u>Silent Discharge Plasma</u>. In this approach, a plasma field is formed in cells consisting of two glass plates charged to a potential difference of 15—50 kV. Microdischarges are formed between the plates that react with water and oxygen to give free radicals that readily oxidize the organic constituents.

Decision points to evaluate the progress of the development and demonstrations of these technologies have been established in the schedule. Down selections will be made as the work progresses and confidence levels with each technology will be established. This work will provide the "hands-on" experience required to determine which technology is most suitable for glovebox application.

The secondary combustion unit will be designed by an engineering firm and procured from industry. Engineering demonstration of the unit will be done at LANL. A full-scale pyrolysis system will be built, fully tested, and transferred to the Plutonium Finishing Plant for the processing of polycubes. The processing of actual polycubes at LANL on a demonstration scale has been discussed and is possible, but such processing is not included in the present scope of the work. The relative merit of this type of demonstration on radioactive materials will be evaluated as this development and demonstration project proceeds.

BENEFITS

Polycubes are not suitable for long-term storage. Pyrolysis was once used at Hanford to process polycubes. The primary problem was frequent line plugging in the solvent-scrubbing system used to treat off-gas. Leakage of the styrene monomer was also problematic. In addition, the only mode of off-gas treatment was to pass the reactor effluent through a carbon tetrachloride scrubber. A new process must be implemented that minimizes polycube handling and that more effectively processes the off-gas. The full-scale pyrolysis system designed and tested in this effort is expected to accomplish that end and to allow the remaining 1600 polycubes to be processed.

COLLABORATION/TECHNOLOGY TRANSFER

The objective of this task is to develop and deliver a pyrolysis unit to Hanford. Hanford personnel are involved in the research efforts to facilitate the technology transfer.

ACCOMPLISHMENTS

- In order for pyrolysis to run in compliance with both safety and environmental regulations, an off-gas treatment technology needs to be introduced to break down the components in the reactor off-gas to an environmentally benign form. Three off-gas treatment technologies were identified as potential candidates for this application: catalytic conversion, silent discharge plasma, and the use of an afterburner.
- Properly designing each of the off-gas treatment technologies depends on knowing the rate at which the off-gas leaves the pyrolysis reactor, the composition of the off-gas, and how the rate and composition change with

time. To determine these parameters, an experimental setup was constructed and used to capture, sample, and measure the rate at which the off-gas leaves the reactor during an actual pyrolysis run. Tests were conducted on a number of cubes in different forms (whole or crushed cube) and with different surface coatings (aluminum paint, PVC tape, and Shurtape). Multiple cube tests were also conducted to determine how the off-gas generation rate was affected by loading more than one cube in the reactor.

- Gas chromatography/mass spectroscopy analyses were conducted to analyze the composition of the off-gas. Tests were conducted to determine how the composition was affected by the different forms of cubes and by the presence of surface coatings.
- The rate and composition data were used to conduct a series of mass and energy balances, the results of which were used to design a catalytic converter that would be suitable for glovebox operations.
- A number of catalyst companies were identified as having catalysts suitable for this application. These companies are now sending catalysts to be evaluated for this application.
- The results of the rate tests were used to design and build a coaxial silent discharge plasma prototype unit. An experimental setup was also constructed to evaluate the performance of this unit using styrene and other organic liquids as surrogates for the reactor off-gas. Preliminary tests are now being conducted. Once the unit is optimized, it is to be configured with the pyrolysis reactor setup for actual pilot-scale testing and evaluation.

CONTACTS

Daniel J. Kathios
MS E511
Los Alamos National Laboratory
Los Alamos, NM 87545
Phone: (505) 665-9026
e-mail: dkathios@lanl.gov

John Coogan
MS E525
Los Alamos National Laboratory
Los Alamos, NM 87545
Phone: (505) 665-0186
e-mail: jcoogan@lanl.gov

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None at this time.

2.10

THE GLASS MATERIAL OXIDATION AND DISSOLUTION SYSTEM

TECHNOLOGY NEED

The Glass Material Oxidation and Dissolution System (GMODS) addresses the need for a process to convert complex waste mixtures - containing ceramics, metals, organics, halogens, and amorphous solids with ill-defined chemical compositions - into a homogeneous, high-quality waste form: borosilicate glass. Several processes (plasma torch, etc.) can convert complex mixtures into semihomogeneous waste forms. Moreover, many processes (conventional vitrification, synrock synthesis, etc.) can convert homogenous wastes into high-quality waste forms. However, no current processes accept almost all wastes and produce a high-quality waste product. If complex mixtures are to be converted to high-quality waste forms using existing technologies, complex, expensive, multistep processes are required.

Because plutonium scrap and residues are both complex and hazardous, the need exists for general purpose processes to convert these materials into high-quality glass for safe, interim storage or disposal.

TECHNOLOGY DESCRIPTION

GMODS is a general-purpose, waste-treatment process designed to convert (1) ceramics, metals, organics, and amorphous solids to borosilicate glass; (2) oxidize organics with the residue converted to borosilicate glass; and (3) convert halogens [such as chlorides] to low-halogen-containing borosilicate

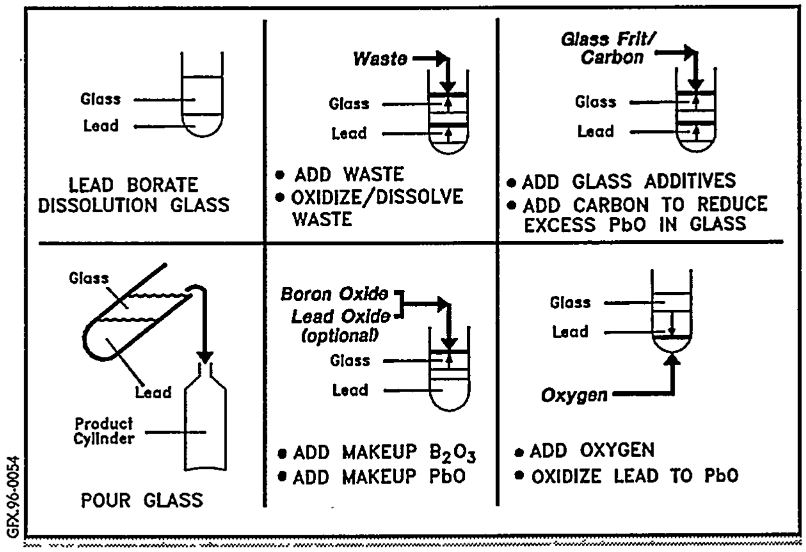

Figure 2.10-1. GMODS Batch Processing of Waste to Borosilicate Glass

glass and a secondary, clean, sodium-halogen stream. It is being developed to process those plutonium-containing wastes that cannot be easily and economically processed by other technologies.

GMODS converts plutonium-containing materials into glass inside a melter. The process can operate as a batch (Fig. 2.10-1) or continuous process. For batch operation, the starting conditions are a glass melter filled with molten, lead-borate dissolution glass. Oxides dissolve in glass, but metals and organics do not. GMODS uses lead oxide (PbO) in the molten glass to oxidize (a) metals to metal oxides and (b) organics to carbon oxides. The resultant metal oxides dissolve into the glass, and the carbon oxides exit the melter as gases. The lead metal reaction product separates from the glass and forms a separate layer at the bottom of the melter.

After dissolution of the wastes, silicon oxide and other additives are added to the glass to produce a high-quality product glass. Excess PbO is removed from the glass by adding carbon, which converts the PbO to lead metal and carbon dioxide (CO_2). The final glass may have some or no PbO depending upon the desired product glass. The product glass is poured from the melter into the waste packages. To generate the next batch of dissolution glass, boron oxide is added to the melter, and the lead metal is oxidized to lead oxide.

GMODS also converts halogen-containing materials to glass, a process which creates a separate nonradioactive sodium halogen waste stream. Halogens, such as chlorides, make poor-quality glasses; hence, they must be separated from other components. In the dissolution glass, chlorides in the feed form lead chloride ($PbCl_2$), which is volatile at glass melter temperatures and which exits to the aqueous sodium hydroxide (NaOH) scrubber. In the scrubber, the $PbCl_2$ reacts with the NaOH to yield insoluble lead hydroxide [$Pb(OH)_2$] and soluble NaCl. The insoluble $Pb(OH)_2$ is recycled back to the melter, in which it decomposes to PbO and steam, while the aqueous NaCl stream is cleaned and discharged as a chemical waste.

BENEFITS

Glass is recognized worldwide as a preferred waste form for radioactive and chemically hazardous wastes. There is, however, a major limitation: all existing glass processes require that the waste be in the form of oxides or oxide-like materials before vitrification. Oxide-like materials are compounds such as nitrates and carbonates that decompose to oxides at high temperatures. Conversion of wastes to oxide-like forms before vitrification is a complex and expensive task.

GMODS allows the direct conversion of oxides, metals, ceramics, organics, halogens, and amorphous solids to glass. This allows complex waste mixtures (filters, process wastes, laboratory wastes, etc.) to be directly processed to glass without preprocessing. The alternatives are to separate the wastes into specific categories and treat each category of waste separately or process (oxidize, dechlorinate, etc.) each waste before vitrification.

COLLABORATION/TECHNOLOGY TRANSFER

Discussions are underway for industrial cooperation to develop the process. Initial patents were granted October 24, 1995.

ACCOMPLISHMENTS

- A U.S. patent (5,461,185) on the GMODS process was granted to Martin Marietta Energy Systems, Inc. on October 24, 1995. Other domestic and foreign patents are pending.
- An initial thermodynamic analysis of the process has been completed and conversion to glass has been demonstrated on a laboratory scale (100-200g) with a variety of feed materials, including aluminum, zircaloy, stainless steel, carbon, uranium, cerium, aluminum oxide, uranium oxide, and cerium oxide. Key physical properties (viscosity, density, etc.) of the molten dissolution glass have been measured.
- Flowsheets have been developed, and equipment requirements for different scales of operation have been identified. Equipment options potentially exist to process wastes from 1 to 100,000 t/year. Significant development work remains.

CONTACTS

Dr. Charles W. Forsberg
Principal Investigator
Oak Ridge National Laboratory
P.O. Box 2008
Oak Ridge, TN 37831-6495
Phone: (423) 574-6783
e-mail: forsbergcw@ornl.gov

E. C. Beahm
Principal Investigator
Oak Ridge National Laboratory
P.O. Box 2008
Oak Ridge, TN 37831-6495
Phone: (423) 574-6851
e-mail: beahmec@ornl.gov

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2.11 PYROLYSIS

TECHNOLOGY NEEDS

Plutonium-contaminated combustible residues are currently located around the DOE complex. Combustibles consist of paper, plastics, rags, gloves, ion-exchange resins, filters, and oil- and grease-contaminated residues. All combustibles generate hydrogen from the radiolysis of the matrix. However, the risk resulting from the pressure build-up has been mitigated at RFETS in the short term by venting the storage drums. Ion exchange resins in nitrate form constitute a fuel and oxidizer in intimate contact. Some of the combustibles contain cellulosic materials, which are of concern due to the presence of nitric acid and nitrates or oil. The best long-term technical approach to address all of the above combustible safety issues is to destroy the matrix, which involves incineration. However, obtaining an incinerator permit can be a difficult and lengthy process. Consequently, DOE is evaluating alternatives to incineration in a trade study. The objective of the research is to provide data for the trade study and develop and transfer technologies selected through the trade study process.

TECHNOLOGY DESCRIPTION

Pyrolysis is a pyrochemical technique that uses a high-temperature, chemically inert environment to break down and volatilize polymeric materials. The purpose of this work is to design, develop, and implement a pyrolysis system to process polymeric materials commonly generated during nuclear materials applications. The pyrolysis system designed in this effort will destroy the matrix of the polymer, allowing its decomposition products to be removed as a gas. The remaining material containing the radionuclides will be reduced significantly in both mass and volume, and will be in a form suitable for long-term storage, cementation, or subsequent actinide recovery. The pyrolysis system designed in this effort will be tailored specifically for the glovebox environment and will be designed to minimize the formation of organic liquids. The design will also incorporate a series of downstream technologies to treat the off-gas decomposition products, allowing the process to operate in compliance with environmental regulations.

LANL has recently designed a gas-tight pyrolysis reactor that is suitable for glovebox applications. Preliminary tests of nonradioactive material have shown that this design allows for 80-100% reduction in mass of selected polymeric materials commonly generated in glovebox applications. Only a small amount of a dry, brittle, black material (presumed to be a form of carbon) is left in the reactor. The design also avoids the formation of tars and other constituents that could adversely affect its operation. In this work, a pyrolysis system is to be designed and optimized in order to (1) accommodate a more comprehensive array of polymeric materials, (2) minimize the formation of organic liquids, and (3) oxidize components of the off-gas, making the process consistent with air quality concerns. Once optimized, the pyrolysis system is to be deployed at the Los Alamos Plutonium Facility (TA-55) for the processing of combustible wastes.

Reactor Design. The original reactor was designed to be housed in a 2500-watt Lindberg vertical clamshell furnace. A larger reactor suitable for a 5000-watt furnace is also to be designed and built. This reactor will allow for more material to be processed per pyrolysis run and will be compatible with 5000-watt furnaces already available at LANL and at other facilities in the nuclear complex. This more powerful furnace will also provide for greater control over the reactor's temperature/time profile, allowing the system to be optimized for more time efficient processing of the materials. The new reactor also will be designed to accommodate the steam reformation reaction. This option could provide a further reduction in the mass of the material remaining in the reactor by removing residual carbon as either carbon monoxide or carbon dioxide. The introduction of water and dilute solutions of hydrogen peroxide are to be considered for this purpose.

Condensation. Condensation is used to remove hydrocarbons that are liquids at ambient conditions from the reactor off-gas. These long-chain hydrocarbons leave the reactor before they are broken down to smaller constituents that would be more suitable for subsequent processes, like catalytic conversion. Preliminary tests have shown that pyrolyzing condensate (collected from the pyrolysis of a variety of polymeric materials) reduced the mass of that condensate by 39%. Thus, recycling the condensate to the reactor for further pyrolysis is a viable approach to minimizing the formation of organic liquids. In this work, a reflux coil is to be designed and situated on the top of the reactor in order to allow for the immediate condensation and return of these long-chain hydrocarbons to the pyrolysis environment. The reflux coil will be tested by pyrolyzing a variety of polymeric materials and progressively will be modified to effectively condense long-chain hydrocarbons emanating from any polymeric material considered in this analysis.

Caustic Scrubbing. Caustic scrubbing is used to remove acidic gases that are formed during the breakdown of the polymeric materials. These include gases such as hydrochloric acid from polyvinyl chloride, hydrogen fluoride from Teflon, and carbonic acid from cellulose. Effective removal of these gases is required to maximize the longevity of subsequent downstream technologies (e.g., the catalytic converter) and to ensure that the process operates in compliance with regulations. Preliminary tests with a 1 M sodium hydroxide scrubber yielded positive results. At this point, the scrubber solution volume and sodium hydroxide concentration must be optimized to accommodate the comprehensive array of polymeric materials in this analysis.

Catalytic Oxidation. Catalytic conversion is used to oxidize small-chain hydrocarbons and carbon monoxide in the gas effluent to carbon dioxide, thus allowing the process to operate in compliance with regulations. A number of commercially available catalysts will be considered in this analysis. These include various platinum/palladium/rhodium catalysts supported on alumina and other inorganic substrates. These catalysts are to be evaluated for their effectiveness in oxidizing the pyrolysis off-gas hydrocarbons to carbon dioxide, for their longevity, and for their ability to be cemented easily for subsequent disposal.

BENEFITS

A considerable amount of plutonium-contaminated combustible waste exists at virtually all sites in the DOE complex. There has been and will continue to be difficulty in permitting the incineration of these wastes. At TA-55, pyrolysis is already being used for the processing of plutonium-contaminated cellulose. The development work conducted in this effort will provide an avenue through which a more comprehensive array of combustible wastes can be processed. Once designed and optimized, this process can be readily applied at other facilities in the complex for the processing of their combustible wastes.

COLLABORATION/TECHNOLOGY TRANSFER

LANL and Hanford Site collaborate on this technology. As this is a new effort, other opportunities for collaboration and technology transfer are currently being solicited.

ACCOMPLISHMENTS

Completed testing of gas-tight pyrolysis reactor. Preliminary tests have shown that this design allows for 80-100% reduction in mass of selected polymeric materials commonly generated in glovebox applications. Modifications to test reactor are underway to accommodate use in a glovebox.

Gas chromatography/mass spectroscopy analysis capabilities have been established to determine the composition of the off-gas stream.

Initiated Process Hazards Analysis for pyrolysis of combustible wastes. Processing authorization is anticipated on October 30, 1996.

CONTACT

Quentin D. Appert
Principal Investigator
MS E511
Los Alamos National Laboratory
Los Alamos, NM 87545
Phone: (505) 667-9241
e-mail: qappert@lanl.gov

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None at this time.

2.12 CATALYZED CHEMICAL OXIDATION

TECHNOLOGY NEED

Plutonium-contaminated combustible residues are currently located around the DOE complex.

Combustibles consist of paper, plastics, rags, gloves, ion-exchange resins, filters, and oil- and grease-contaminated residues. All combustibles generate hydrogen from the radiolysis of the matrix. However, the risk resulting from the pressure build-up has been mitigated at RFETS in the short term by venting the drums. Ion exchange resins in nitrate form constitute a fuel and oxidizer in intimate contact. Some of the combustibles contain cellulosic materials, which are of concern due to the presence of nitric acid and nitrates or oil. The best long-term technical approach to address all of the above combustible safety issues is to destroy the matrix by incineration. However, permitting an incinerator can be a difficult and lengthy process. Consequently, DOE is evaluating alternatives to incineration in a trade study. The objective of the research is to provide data for the trade study and develop and transfer technologies selected through the trade study process.

TECHNOLOGY DESCRIPTION

Combustible stabilization by chemical oxidation is achieved by the destruction (oxidation) of all solid and liquid organic material within the residue matrix and by solution of all radionuclides and active metals. The result of the process is that no combustible material or hydrogen-generating constituents remain. The actinides can be separated and stored as a salt or oxide, or the actinides can be disposed of with the other metal salts and insoluble materials. Some chemical processes are sufficiently aggressive that they oxidize even hard-to-destroy materials, such as polyethylene and polyvinyl chloride. Space constraints within gloveboxes, as well as criticality requirements, can be met because of the relative small size of the reaction vessels required. The chemistry of the process will deal with the multiple hazardous waste codes associated with residues at RFETS that fall within Resource Conservation and Recovery Act (RCRA) regulation.

Development to date has focused on the Delphi Research DETOX^{SM} process and has been highly successful. Delphi Research, LANL, and RFETS now have two years experience operating this process with surrogate materials at the four-liter reactor vessel size, which is full scale for residue processing. Full-scale demonstration using actual plutonium residues is now needed.

Reconfiguration and upgrading of the current development system for glovebox operation will be required, followed by testing with radioactive materials.

The process uses iron with a combination of homogeneous metal catalysts, such as platinum and palladium, in a chloride solution. This is a variation of wet chemical oxidation. In the DETOX^{SM} process the metal in solution plays the major role in the destruction of the organic material. The process operates at 200°C and 0.7 megapascals (MPa) or 100 pounds per square inch (psi). The majority of required development on this process has been completed. Bench-scale testing of surrogate materials has demonstrated that the DETOX^{SM} process can process a wide variety of residues, including those contaminated with mercury and cyanide. The average destruction rate for combustible material is 0.2 kg/hr in 2 L of DETOX^{SM} solution in a 4-L stirred reaction vessel. An independent study conducted by the Materials Department at the Colorado School of Mines has identified the materials of construction that offer the greatest corrosion resistance for this application. A process design and a safety review have been completed for a low-level mixed waste pilot demonstration system that currently is being fabricated.

BENEFITS

Although the relative hazard of combustible residues varies with the type of residue, the potential safety problems with combustible residues include the following:

1. Radiolytic generation of hydrogen and other flammable and corrosive gases from hydrogenous material, plastic packaging material, and reactive metals
2. Reactive/pyrophoric metals
3. Nitrated/fuel mixtures
4. Plutonium metal/organohalide mixtures
5. Degradation of plastic packaging material

This research will aid in the stabilization and safe, interim storage of these high-risk combustibles.

COLLABORATION/TECHNOLOGY TRANSFER

This is a collaborative effort with Delphi Research and RFETS. RFETS personnel are involved to facilitate technology transfer.

ACCOMPLISHMENTS

Development to date has been highly successful. Bench-scale testing of 16 different surrogate materials has demonstrated that the DETOX^{SM} process can process all RFETS liquid and solid combustible waste types. Process information of surrogate destruction, process information on the fate of hazardous organic and inorganic compounds in the DETOX^{SM} solution, and component testing on stirrers, solid feed, condenser design, and secondary reactors have been incorporated into the design of proposed pilot-scale systems for low-level mixed waste and plutonium residues. Materials testing at the Colorado School of Mines has successfully found and provided independent data on materials of construction with little corrosion in this application.

An initial engineering evaluation of a four- to eight-liter combustible residue system has been completed. A contract is being placed for support from Delphi Research in configuring a residue system. In conjunction with LANL and RFETS personnel, Delphi Research will design, procure, and assemble a catalytic chemical oxidation system for the processing of combustible plutonium residue material. LANL and Delphi personnel will jointly complete operational testing of the system in a glovebox mockup. The intent of the procurement is to provide a system ready for glovebox installation at LANL TA-55 to be used to treat residues.

The chemistry of plutonium in the DETOX^{SM} solution and the optimum method of treating the spent solution is being investigated. Disposal of spent solution by precipitation of metals as hydroxides is possible, but filtration and washing of the gelatinous precipitate is difficult. The feasibility of hydrolysis of the solution at elevated temperature was tested. It produces a coarse ferric oxide and a scrubber solution of hydrochloric acid. Work has begun to optimize hydrolysis conditions, define the fate of metals and their disposal, and test the final methodology on actual plutonium residues.

CONTACT

Charles Brown
Principal Investigator
Kaiser Hill Company
RFETS Environmental Technology Site
P. O. Box 464
Golden, CO 80402-0464
Phone: (303) 966-5277
no e-mail capability

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2.13 HIGH-RISK COMBUSTIBLE WASHING

TECHNOLOGY NEED

Plutonium-contaminated combustible residues are currently located around the complex.

Combustibles consist of paper, plastics, rags, gloves, ion-exchange resins, filters, and oil- and grease-contaminated residues. All combustibles generate hydrogen from the radiolysis of the matrix. However, the risk resulting from the pressure build-up has been mitigated at RFETS in the short term by venting the drums. Ion exchange resins in nitrate form constitute a fuel and oxidizer in intimate contact. Some of the combustibles contain cellulosic materials, which are of concern due to the presence of nitric acid and nitrates or oil. The best long-term technical approach to address all of the above combustible safety issues is to destroy the matrix, which involves incineration. However, permitting an incinerator can be a difficult and lengthy process. Consequently, DOE is evaluating alternatives to incineration in a trade study. The objective of the research is to provide data for the trade study and develop and transfer technologies selected through the trade study process.

TECHNOLOGY DESCRIPTION

Washing can be as simple as a water wash of the matrix material. Additives could promote plutonium removal, thereby ameliorating the hydrogen generation problem. Water alone may be adequate for removing nitrates from most combustibles, with the exception of ion-exchange resins in which the addition of salicylate has been demonstrated to promote the removal of nitrates, and to help render these materials safe for interim storage. Corrosion primarily results from nitric acid or from halocarbons present in the wastes. Both are addressed adequately by an aqueous wash or an aqueous wash with the addition of a detergent. The one possible exception might be the RFETS Ful-Flo™ filters used in the purification process for carbon tetrachloride. For these, bulk solvent removal (low-temperature thermal desorption) before washing may be advantageous, because it would substantially reduce waste water treatment.

Aqueous washing is straightforward and has been used at most DOE sites in one form or another on residue materials, particularly for nitrate removal. Additives and sonication were briefly evaluated for plutonium/americium removal from combustible matrices in a research and development effort at LANL (February 1-April 30, 1995). The initial results were encouraging, and plutonium removal efficiencies as high as 90% were observed when plutonium-selective chelates on water-soluble polymers were used. Washing with aqueous solutions containing salicylates appears to have been successful in nitrate replacement on ion-exchange resins and is in the final evaluation phase at LANL. Detergents that promote solvent removal from residue matrices are readily available from the commercial laundry industry, but these detergents require evaluation and testing for specific treatment applications.

BENEFITS

The major benefits of combustible washing are:

- It is a simple technology and is easily implemented with little development required.
- It can ameliorate immediate safety concerns and render combustibles and other residues safe for interim storage.
- It can be implemented faster than most baseline technologies.
- It is compatible with final treatment, if required.
- In some cases, further treatment is not necessary unless it proves cost effective to do so.

COLLABORATION/TECHNOLOGY TRANSFER

This is a new initiative. Opportunities for collaboration and technology transfer are currently being solicited.

ACCOMPLISHMENTS

Testing the batch-contact extraction (washing), filtration/decantation, thermal desorption, and stabilization steps of the baseline flow sheet began in March 1996. The equipment and chemicals for the project have been obtained. The Texaco Transultex A and H cutting oils were obtained. Surrogate was obtained from Safe Sites of Colorado (SSOC). The experimental plan and project work scope were written, negotiated, and completed.

Completed solid residue washing experimental plan, which includes a detailed experimental plan using a surrogate containing cerium metal for the initial scoping studies followed by tests with plutonium loaded surrogate.

CONTACT

Norman C. Schroeder
Principal Investigator
MS J514
Los Alamos National Laboratory
Los Alamos, NM 87545
Phone: (505) 667-0965
e-mail: nschroeder@lanl.gov

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None at this time.

2.14 MEDIATED ELECTROCHEMICAL OXIDATION

TECHNOLOGY NEEDS

Plutonium-contaminated combustible residues are currently located around the complex.

Combustibles consist of paper, plastics, rags, gloves, ion-exchange resins, filters, and oil- and grease-contaminated residues. All combustibles generate hydrogen from the radiolysis of the matrix. However, the risk resulting from the pressure build-up has been mitigated at RFETS in the short term by venting the drums. Ion exchange resins in nitrate form constitute a fuel and oxidizer in intimate contact. Some of the combustibles contain cellulosic materials, which are of concern due to the presence of nitric acid and nitrates or oil. The best long-term technical approach to address all of the above combustible safety issues is to destroy the matrix, which involves incineration. However, permitting an incinerator can be a difficult and lengthy process. Consequently, DOE is evaluating alternatives to incineration in a trade study. The objective of the research is to provide data for the trade study and develop and transfer technologies selected through the trade study process.

TECHNOLOGY DESCRIPTION

The purpose of this work is to continue development of the technique of mediated electrochemical oxidation (MEO) as a viable technology for the treatment of combustible waste and for the removal of surface contamination from selected matrices. The final goal is to demonstrate the feasibility of this process on the full production scale using real combustible waste as feed. Results from these studies will be used to compare the applicability and utility of this treatment technology against other treatment technologies currently under development.

This technology has the capability of destroying the organic component and recovering the radioactive component of selected mixed wastes. For example, it has been shown that chlorinated hydrocarbons, such as tetrachloroethylene, carbon tetrachloride, and chloroform, can be converted very efficiently to carbon dioxide at room temperature. However, other organic materials like polypropylene and polyethylene are fairly inert. MEO, as a room-temperature process, would be an ideal technology for the treatment of a waste such as the full-flow filters that are contaminated with carbon tetrachloride and plutonium. Treatment with the MEO process would convert the carbon tetrachloride to carbon dioxide, dissolve the plutonium for subsequent recovery by standard recovery procedures, and leave the filters free of both organic and radioactive contamination – in other words, suitable for compaction and disposal.

2.15 STABILIZATION OFF-GAS ANALYSIS SYSTEM

TECHNOLOGY NEEDS

DOE needs to analyze the off-gas produced during calcination of plutonium oxides. DOE-STD-3013 requires thermal stabilization at 1000 +/- 50 C for one hour in an oxidizing atmosphere for greater than 50 weight percent plutonium. However, there are questions whether this is acceptable for the variety of materials which are less than 50 weight percent plutonium. Analysis of the off-gas during thermal stabilization would provide information which may be useful in determining acceptability for interim storage.

TECHNOLOGY DESCRIPTION

The proposed system would analyze the gas generated during thermal stabilization of plutonium. Analysis would include organic decomposition products, volatiles, and others.

The system would be composed of the furnace, piping, and a residual gas analyzer with computer control.

BENEFITS

This technology would develop a system which supports the determination of interim storage acceptability.

COLLABORATION/TECHNOLOGY TRANSFER

This is a new initiative. Opportunities for collaboration and technology transfer are currently being solicited.

ACCOMPLISHMENTS

This is a new initiative. No accomplishments have been made for this effort.

CONTACT

Lee A. ReFalo
Principle Investigator
Savannah River Technology Center
Building 305-A
Aiken, SC 29808
Phone: (803) 725-4473
e-mail: lee.refalo@srs.gov

BIBLIOGRAPHY OF KEY PUBLICATIONS

None at this time.

2.16 PLASMA HEARTH PROCESS DEMONSTRATION OF THE TREATMENT OF PLUTONIUM-BEARING MATERIALS

TECHNOLOGY NEEDS

The PFA is chartered with developing technologies to treat, recover, stabilize, or otherwise manage plutonium residues in the DOE system. The stabilization of appropriate residue matrices is required to meet a variety of regulatory and safety requirements that specify compliant interim storage or disposal of these materials. The residue streams targeted for this stabilization demonstration are the variety of plutonium-bearing ash residues, the heterogeneous mixtures of solid materials with plutonium (combustible and non-combustible) and any other plutonium residue streams found to be treatable in this process.

TECHNOLOGY DESCRIPTION

The Plasma Hearth Process (PHP) is a high-temperature thermal treatment process using a plasma arc torch in a stationary, refractory-lined chamber that destroys organics and stabilizes the residuals in a nonleaching, vitrified waste form, greatly improving the disposability of the waste.

The term "plasma" refers to a highly-ionized, electrically conductive gas. The type of plasma produced in the PHP application is a DC arc-generated thermal plasma created by a device known as a "plasma torch." The plasma torch used in the PHP technology operates in the transferred arc mode. Basically, the transferred arc torch uses a flow of gas to stabilize an electrical discharge (arc) between a high voltage electrode (inside the torch) and a molten pool of waste (maintained at ground potential). Because of the very high resistance to electrical current flow through a gas, electrical energy is converted to heat which in turn destroys or stabilizes treatment matrices.

The PHP technology is chiefly applicable to solid or sludge wastes where a stabilized byproduct is required for disposal. The technology is ideally suited for heterogeneous wastes of nearly any type that are difficult to treat by conventional thermal technologies. It is currently being developed for DOE alpha-contaminated solid mixed wastes, both low-level and transuranic (TRU). One of the key advantages of the PHP technology is the ability to process a wide variety of waste materials, often without regard to physical or chemical nature. The process oxidizes the matrix constituents and stabilizes them in a single step, eliminating the need to convert many non-oxide compounds, such as nitrates and carbonates, to oxides prior to stabilization. Whole containers of waste, including heterogeneous matrices, may be fed into the process chamber, which minimizes pretreatment characterization, sorting, sizing, and other handling and potential exposure to toxic and radioactive contaminants.

The primary functional units of the PHP system are the feed system, plasma chamber, slag/metal removal system, secondary combustion chamber, and air pollution control system. Processing begins as unopened containers of waste are fed to the fixed hearth plasma chamber, where heat from the plasma torch initiates a variety of chemical and physical changes. Complex organic compounds break down into non-complex gases that are drawn from the chamber, while the remaining inorganic material materials are "drip melted" slowly into a crucible. The molten material separates into two phases: a vitrified product and metal. The molten materials can be poured into waste containers by a simple hearth tilting mechanism to avoid tap hole plugging problems. A sketch of the bench-scale system is shown in Figure 2.16-1.

Induced draft fans pull gases from the plasma chamber to a secondary combustion chamber. Once there, excess oxygen and the product gases are mixed in a very hot and turbulent environment for a sufficient residence time to complete the conversion of the gaseous organic compounds to CO_2 and H_2O. This secondary chamber guarantees the hazardous organics are treated completely to achieve the high-destruction-efficiency required by the EPA (Environmental Protection Agency). The off-gas is then scrubbed by state-of-the-art air pollution control technologies capable of removing a high degree of pollutants, producing very clean process emissions.

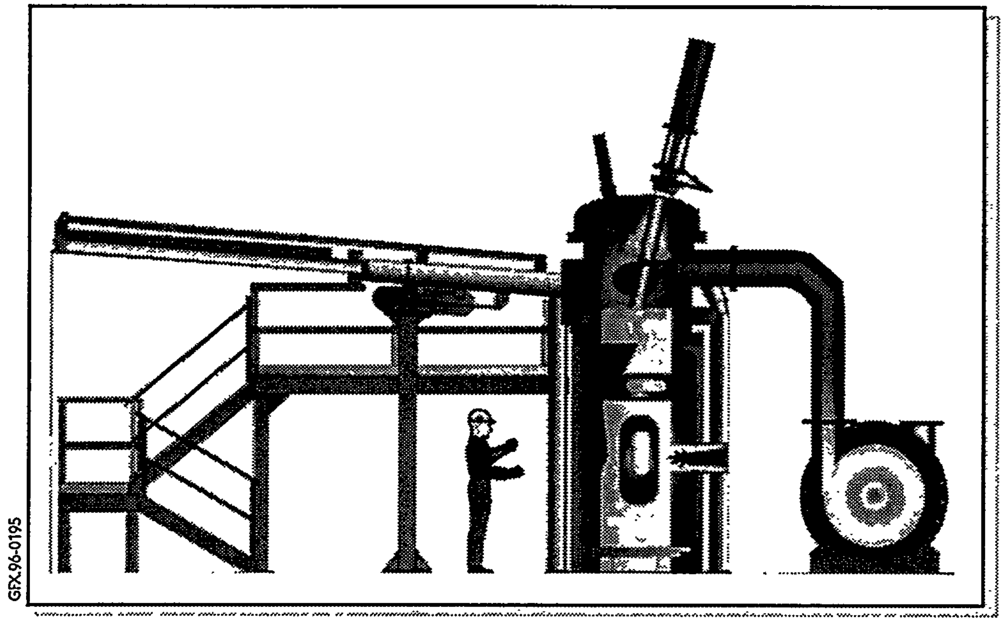

Figure 2.16-1. Bench-Scale Plasma Hearth Process System

BENEFITS

The molten product resulting from PHP processing can form two phases: 1) a high-integrity, leach-resistant vitrified product is produced in any operating mode; and 2) a separate reduced metal phase is produced when the process is operated in a reducing (oxygen deficient) mode. The two phases segregate naturally due to density differences and can be recovered separately. In an oxidizing mode, the metals are also oxidized, resulting in a single vitrified phase. The chemistry in the melt can be controlled through additives, if necessary, to optimize the properties of the product, including criticality control.

Although the high-integrity, vitrified final waste form has a primarily crystalline structure and is not a glass, vitrified PHP products from a variety of waste matrices have repeatedly shown the ability to meet or exceed disposal requirements instituted by the EPA. The properties of these semi-homogeneous waste forms also compare favorably to the Savannah River high-level waste glass (SRL 200) and the benchmark EA (environmental assessment) glass, using the PCT (Product Consistency Test) developed by the High-Level Waste Program as an indicator.

Waste streams generated by this process include spent filters, a scrubber blowdown, and refractory. In the full-scale process, spent filters can be processed in the PHP. Because off-gas scrubbing for acid gases occurs downstream of system HEPA (High-Efficiency Particulate Air) filters, the scrubber liquor and resulting blowdown will be non-radioactive or low-level at worst. Refractory materials spent in the process can also be processed through the PHP.

COLLABORATION/TECHNOLOGY TRANSFER

The PHP, in its sixth year of DOE-funded development, is very mature. Plasma arc technology is an existing, well-understood, commercially available technology. Industry has been using plasma arc torches in various applications for many years. Fixed hearth plasma technology has been used for many years in the processing of speciality metals, and the PHP is currently being demonstrated by EM-50 for the treatment of TRU and alpha low-level mixed wastes, leveraging investments from other Focus Areas. Therefore, the PHP Project involves adapting commercial plasma torch metal refining technology to the vitrification of waste materials. Because of the maturity of plasma torch technology and the similarity of the applications, the development has been rapid.

The PHP has been demonstrated at engineering scale and near-full scale on simulated DOE wastes. It has been demonstrated, at engineering-scale, to be highly applicable to the treatment of incinerator and other thermal-treatment ash. Science Applications International Corporation (SAIC) has processed thermal-treatment system ash representing baghouse flyash, dry scrubber flyash, and heavily-chlorinated flyash into stable waste forms in their STAR Center PHP system. Cerium was used as a plutonium surrogate for these tests. In addition to plutonium (Pu) ash, the PHP process has been demonstrated (using surrogates) to treat Pu-bearing wastes in a variety of waste matrices, including complex physical and chemical mixtures, into stable waste forms.

The Project is currently completing installation of two advanced PHP demonstration systems, one a full-size (1.2 MW - 55 gal drums), continuous-feed pilot demonstration system utilizing surrogate waste feeds, the other a radioactive engineering-scale (200 kW - 1 gal cans) system for evaluation of actinide behavior and treatment of plutonium-bearing wastes. The smaller unit, designated the Radioactive Bench-Scale System, is a batch system installed at the TREAT facility at ANL-W (Argonne National Laboratory-West) that is scheduled to commence radioactive operations in July 1996.

Primary R&D requirements for the PHP for these types of wastes would be in the areas of additives for criticality control and assessment of actinide partitioning in the system at the increased Pu levels present. The Bench-Scale System, as currently installed, was designed for processing Pu wastes. However, some additional analyses would be required to evaluate whether the system requires modification in order to meet the more stringent requirements for higher fissile material content. The facility Hazard Category would need to be reevaluated.

It is believed that processing of the PFA's Pu ash could be demonstrated in the Bench-Scale System in FY97 with no change in the system hardware. The system, rated at a throughput of two one-gallon cans per hour, could easily process materials with up to 10% Pu (subject to SAR and NESHAP reviews) by employing silica (sand) as an additive to control criticality. The ash would be fed to the process in one-gallon cans, up to eight cans per batch. The processing of other PFA waste streams can also be demonstrated, possibly in FY97 and FY98.

CONTACT

Robert L. Gillins
Science Applications International Corporation
545 Shoup Ave., Suite 200
Idaho Falls, ID 83402-3575
Phone: (208) 528-2114
e-mail: Bob_Gillins@cpqm.saic.com

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None at this time.

2.17 MICROWAVE VITRIFICATION OF MIXED WASTES AND RESIDUES

TECHNOLOGY NEED

Currently, many metric tons of high activity residue materials are being stored at RFETS Environmental Technology Site in sub-standard forms. The list of stored residues include sludges, sand/slag/crucible from foundry operations, incinerator ash, oxides, pyrochemical salts, and a variety of other inorganic materials. These materials are found throughout the DOE complex, making the problem common to numerous sites and programs. The DNFSB identified these materials as posing a significant safety risk to the DOE complex (refer to DNFSB 94-1 and 94-3). In DNFSB 94-1, the Board stated that high-risk residues must be completely processed. In response to DNFSB Recommendation 94-3, microwave vitrification is being considered as an option for treating plutonium oxide stored at RFETS.

TECHNOLOGY DESCRIPTION

Microwave Vitrification has been developed as a treatment process that would be appropriate for many residue waste forms. Preliminary scoping studies indicate that this technology could successfully treat 80% of the residue inventory at RFETS. The process uses microwave energy to heat and vitrify waste materials. Microwave vitrification is an innovative technology for the vitrification of inorganic mixed and high activity wastes. It produces a durable, leach-resistant final form that meets Department of Transportation requirements and is ideal for interim storage. The microwave vitrification system is a flexible heat source that is readily adaptable to other applications such as drying and calcination.

The final waste form produced by microwave vitrification is a glass or mineral-like monolith that does not contain free-liquid or releasable particulates, and is highly leach resistant. It is extremely robust and is especially suited for hazardous and mixed wastes. The microwave vitrified waste form meets all criteria for shipment and disposal at either the Nevada Test Site (NTS) or WIPP.

Vitrification is also attractive from a life-cycle cost perspective, due to the significant waste volume reductions that are possible - up to 97%.

Significant work with solidification of sludges has been performed at RFETS since 1986. Currently, a surrogate hydroxide coprecipitation sludge is successfully being processed using a full-scale demonstration system. Other wastes that passed preliminary screening tests using a bench-scale system include ash, oxides, sand, slags, crucibles, pyrochemical salts, fire brick, fluidized bed incinerator ashes, nitrate salts, sludge, soils, pondcrete, saltcrete, pond sludge, raschig rings, miscellaneous glass, and asbestos.

The microwave vitrification technology is mature and ready for implementation. Basic research and development efforts for the technology have been completed and current activities are focused on confirming the waste formulations and system design for implementation. The feasibility of production implementation of the microwave vitrification process has been proven using a full-scale demonstration system which has been in operation since 1989. Production implementation efforts have been initiated. A conceptual design report has been submitted to DOE and preparation of the design criteria was initiated. A small-scale system for the treatment of high activity transuranic wastes is being fabricated for deployment in a production facility at RFETS.

BENEFITS

Microwave vitrification produces a robust final waste form that meets the waste acceptance criteria for shipping and disposal. The process developed at RFETS is an in-drum melting system designed to isolate the waste material in a shipping container during the process. Several advantages to the microwave solidification process have been identified through the development program.

- The reduction of fines eliminates respirable dust concerns.
- A material loses attractiveness when locked into a glass matrix. The borosilicate glass contains boron, which acts as a neutron poison, thus providing a degree of safety for criticality concerns.
- Direct heating of materials eliminates the need for electrodes and other consumables.
- Radioactive exposures to workers are lowered due to remote treatment processing.
- Material throughput is increased due to shorter cycling times.

COLLABORATION/TECHNOLOGY TRANSFER

The microwave process is applicable to a variety of inorganic waste materials that are found throughout the DOE complex. The process is very flexible and can be applied to nearly 80% of the legacy wastes at RFETS. Further, these waste streams can be combined in the process to increase waste loading up to 100%. This opportunity reduces waste disposal costs and improves the cost/benefit perspective of the process.

Results of a recent study, "Technical Assessment of Microwave Solidification," were published by the Global Environment and Technology Foundation. The study evaluated technical aspects of the microwave vitrification for the purpose of commercialization and marketing of the technology to the private sector. The evaluation compared the microwave process to competing technologies on the basis of advantages, maturity, regulatory acceptance, implementability, health and safety, and applications. The report ranked microwave vitrification higher than competing technologies and recommended it for further commercialization efforts.

ACCOMPLISHMENTS

Basic research and development efforts for the technology have been completed and current activities are focused on confirming the waste formulations and system design for implementation. The feasibility of production implementation of the microwave vitrification process has been proven using a full-scale demonstration system which has been in operation since 1989. Production implementation efforts have been initiated and a conceptual design report has been submitted.

Numerous other scoping studies using the bench-scale systems were subsequently conducted to determine the full application potential of the technology on a wide variety of newly assigned waste streams. Preliminary scoping studies have been completed on surrogate backlogged sludge, incinerator ash, soils, activated carbon, raschig rings, miscellaneous glass, cerium oxide, nitrate salt, molten salt extraction materials, and electro-refining salt. These studies have also examined the possibility of combining waste materials to achieve higher waste loads which provides further improvements in the process economics. One hundred percent (100%) waste loading has been achieved by combining raschig rings or miscellaneous glass as the silica source in the process waste matrix.

A series of studies was conducted on actual hydroxide sludge from the liquid waste treatment facility. The tests achieved a 72% waste reduction, 2.17 g/cc density, and 60% waste loading. These results were similar to those obtained on the non-radioactive surrogates, thus validating the applicability of the technology on the actual waste material. Further studies on the actual hydroxide sludge were completed to confirm leach performance of the product. Additionally, a test program was completed on actual soil material from Operable Unit #2 at RFETS. These remediation materials were tested to determine parameters such as waste loading, processing rate, and a product's physical and leach characteristics. The results were very encouraging and indicated that the process is applicable to soil materials.

CONTACT

Greg Sprenger, Principal Investigator
Kaiser-Hill RFETS
Rocky Flats Environmental Technology Site
Bldg. 750
P.O. Box 464
Golden, CO 80402-0464
Phone: (303)966-3159
No e-mail capability

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3.0 PACKAGING

Plutonium and other actinide materials are currently stored in a variety of packages, including tanks, piping, plastic containers, and metal containers. When production operations were terminated in the early 1990s, these materials were being held for staging into production process systems. They are now being repackaged to meet criteria for interim storage per DOE-STD-3013. This standard requires plutonium metals and oxides be stored in dry atmospheres in hermetically sealed containers that do not contain organic materials.

Packaging of residues containing small quantities of plutonium has historically been in large containers (e.g., 30- and 55-gallon drums, 5-gallon pails). Smaller packages containing plutonium-bearing residues are stored in drums and pails. Many of these packages are larger than those required by the standard. Packaging of pyrochemical chloride salts may require use of special containers with corrosion-resistant properties.

Technology baseline requirements for packaging are as follows:

a. *Load-Out Transfer System* - A transfer system must be developed to eliminate organics and plastics from storage packages.

b. *Large Package Storage for Plutonium Residues* - Containers must be developed to store low-assay plutonium residues in larger quantities than prescribed by DOE-STD-3013.

c. *Corrosion-Resistant Containers* - The use of corrosion-resistant containers for storing pyrochemical salts and other corrosive residues must be evaluated. Containers must be developed and tested, as required.

d. *Storage Container for Neptunium Oxides* - Shielded containers must be developed for the long-term storage of neptunium. The protactinium daughter of this element emits a strong gamma ray (0.31 Mev), producing high radiation levels in less than one month.

e. *Storage Container for Pu-238* - The high heat load of Pu-238 requires special cooling in long-term storage. The buildup of helium during storage requires periodic venting of the container.

f. *Storage Container for Pu-239* - A long-term storage container for Pu-239 must be developed, tested, and certified to meet the requirements of DOE-STD-3013.

3.1 ROBOTICS TECHNOLOGY DEVELOPMENT IN SUPPORT OF PLUTONIUM PROCESSES

TECHNOLOGY NEEDS

The Plutonium Focus Area (PFA) is currently concentrating on the development of processes to stabilize and assay various plutonium forms. These materials and the processes associated with them have special handling requirements due to security, radiation exposure, and contamination concerns. Existing processes and technology have relied heavily upon workers conducting manual tasks. Advancements in automation and robotics technology, and a greater sensitivity to worker safety, are creating a demand for technology that will place the operator in more of a supervisory capacity. The Robotics Technology Development Program has been providing support to the DOE, Office of Environmental Management for more than six years. Along with partners in industry and academia, the Robotics Technology Development Program will bring years of nuclear material handling experience to support the efforts of the PFA and their customers.

While robotics have become commonplace in manufacturing production lines around the world, adaptation of this technology to the special needs associated with plutonium processes is still a major challenge. Integration of cutting edge control and sensing systems with highly reliable hardware is essential to success. Size and performance requirements associated with tasks in glove box processing lines create unique challenges, and make it crucial for operators to be comfortable with and confident in the systems.

FY97 marks the beginning of the Plutonium Processes technical area within the Robotics Technology Development Program. The development of future programs, with an emphasis on systems engineering and life-cycle benefits, will be a major task in the coming year. However, two projects have already been deemed beneficial to existing and planned processes.

An Automated Plutonium Container Unpacking System will be capable of transporting, opening and dispensing the contents of inner vessel plutonium packages. The system will be capable of automatically processing the majority of containers in the complex, while providing simple operator intervention for off normal packages.

A Flexible Automation System for Glovebox Processing will provide a cost-effective technology for applying automation to multiple tasks in multiple stages of a process. The system will be capable of supporting significant process changes, and be easily installed and removed from glovebox lines.

Automated Plutonium Container Unpacking System: The plutonium container will be introduced through an isolation chamber. Once the outer door is sealed, the inner portal will open and the container will be delivered to the container staging area automatically. In the staging area, a clamping mechanism will hold the container firmly in place, applying the appropriate force with the use of compliant clamps. For containers that are eligible for automated opening, a mechanism for cutting and lid removal will lower and clamp over the container. This device will roll cut the container, avoiding problems associated with thermal or residue generating operations. Plutonium residue will automatically be removed from the lid, and the lid will then be placed in the scrap area for bag out. The open container will then be delivered to the material out port, or be placed in the plutonium removal station. At the plutonium removal station, a system of mechanical and electrostatic technology will ensure contained extraction of plutonium oxides from the containers. Material needing further segregation will require manual operator intervention. The extracted material will be transferred to the material out port for introduction into the process. Empty, residue-free containers will be delivered to the scrap area for bag out with the lid.

Flexible Automation System for Glovebox Processing (see Figure 3.1-1): There is currently no automation system that is capable of adapting to multiple tasks in multiple glovebox lines with the appropriate level of dexterity and reliability. As a result, automated systems are usually expensive, custom designs, with no function beyond the original glovebox task for which they were designed. A flexible automation system will provide autonomous control for repetitive tasks, and operator assisted tele-operation for off-normal or highly unstructured tasks such as maintenance. The system will be capable in installation and removal from hot glove boxes, and exchangeable between gloveboxes. It will have a high degree of dexterity allowing a single configuration to be applied

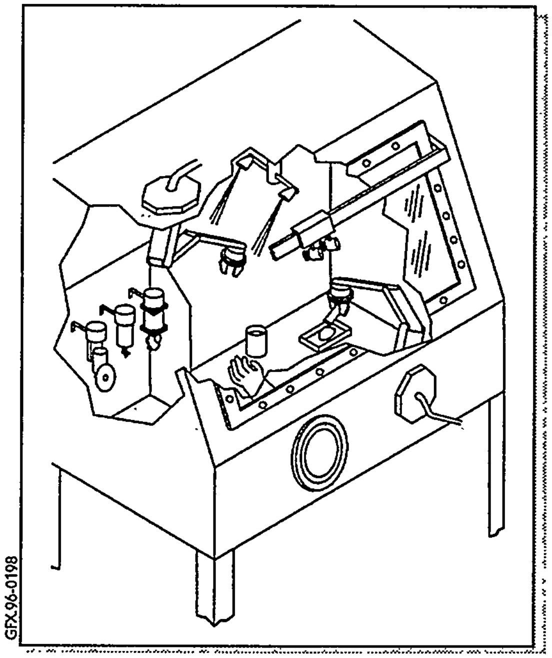

Figure 3.1-1 Artist's Concept: "Flexible Automation for Glovebox Processing"

to a varied array of tasks. The proposed technology has six major sub-systems which include: a robotic manipulator with a quick disconnect that provides remote and automated access to the majority of the glove box work space; a set of quick disconnect tools for the manipulator that will allow a broad range of applications; fixtures and glovebox interface technology to provide easy installation and removal of the system; interfaces that will provide operators with intuitive control at the glove box or from a remote location; work cell modeling with near real time updating to provide path planning, collision avoidance, and object determination; control system hardware and software.

BENEFITS

The benefits of these two systems are similar. The major benefit is a significant reduction in worker exposure to radiation and potentially hazardous working environments. The systems are each cost effective relative to their life cycle. The unpacking system will be designed to reliably accommodate the majority of package types within the DOE complex. Because the system is dedicated to this process, and because it will rely on operators for off-normal packages, the cost will be low. The flexible automation system will be significantly more expensive, however the ability to reuse the system and apply it to new tasks when necessary, will make its life-cycle cost attractive. Improved productivity is also a potential benefit for some activities.

COLLABORATION/TECHNOLOGY TRANSFER

Technologists from four laboratories (Savannah River Technology Center, LANL, Idaho National Engineering Laboratory, and Sandia National Laboratory) with plutonium process automation experience will be involved in the development of these systems. It is expected that commercial partners will be able to provide these systems to the DOE upon completion of successful demonstrations and appropriate transfer of the technology.

ACCOMPLISHMENTS

An initial review of these systems has been completed with potential customers within the complex. A need has been determined, and preliminary concepts agreed upon.

CONTACT

Brad Jenkins
Plutonium Processes Coordinator
Robotics Technology Development Program
Savannah River Technology Center, 773-A
Aiken, SC 29808
Phone: (803) 725-8764
e-mail: brad.jenkins@srs.gov

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None at this time.

4.0

SURVEILLANCE AND MONITORING

Surveillance of packages is required to ensure that container integrity is maintained and that all materials comply with environmental, safety, and health requirements. Surveillance measures should be standardized for stored materials. Nonintrusive surveillance offers distinct advantages over conventional methods (e.g., inspection by opening packages).

Technology baseline requirements are as follows:

a. *Plutonium Metals and Oxides* - Most of the materials requiring interim storage are categorized as plutonium metals and oxides. Surveillance requirements for these materials include monitoring for pressure buildup and distortion of containers from pressurization, corrosion, and change of form within the container.

b. *Plutonium Residues* - Plutonium residues that have not been conditioned for long-term storage require constant surveillance. The large volume of residues currently on hand will necessitate interim storage for several years. These residues may be recategorized as waste and held for disposition to WIPP (Waste Isolation Pilot Plant).

c. *Special Isotopes* - The special isotope category contains mostly one-of-a-kind types of materials. Surveillance requirements will likely be tailored for each special isotope.

4.1 TOMOGRAPHY

TECHNOLOGY NEED

This project will develop a noninvasive, nondestructive method for determining the contents and the condition of the transuranic (TRU) materials in containers currently being used. The DOE complex has many containers of plutonium with poorly characterized contents and packaging. Processing or safe repackaging of plutonium-containing materials often requires opening packages in gloveboxes with non-inerted atmospheres. Unexpected consequences can be produced. Development of more sophisticated and precise noninvasive analytical tools is needed.

TECHNOLOGY DESCRIPTION

Researchers will investigate the feasibility of using x-ray tomography to determine discrete chemical and elemental characteristics within currently stored inventories of plutonium. Using surrogate materials, DOE will evaluate commercially available x-ray tomography equipment for its ability to distinguish between plutonium compounds in sealed metal containers. Identification of plutonium corrosion products, such as plutonium hydride and plutonium oxide, will be investigated. Being able to determine the existence of pyrophoric material before containment is lost will be invaluable. The specific location and amount of hydride formation within a packaged plutonium matrix will be investigated. Los Alamos National Laboratory (LANL) and Savannah River Site (SRS) will begin to investigate measurement techniques that are complementary to computed tomography (CT), such as gamma-ray backscattering and alpha/n neutron emission, including algorithms for integration of results with CT data. Los Alamos will install and evaluate an existing prototype instrument, the tomographic gamma scanner (TGS), that utilizes related technology. This instrument is a large-scale tomographic system used for nondestructive assay (NDA) of nuclear materials in containers up to 55-gallon-drum size.

BENEFITS

For safety and processing efficiency, DOE needs to be able to recognize specific identifiable characteristics within sealed containers. Unprocessed materials currently located on several DOE sites often require extensive handling to determine their condition. Such handling increases worker exposures, contamination incidents, and inhalation risks. Development of noninvasive analytical tomography equipment for semi-remote handling would vastly improve current knowledge of stored inventories. X-ray tomography may reveal potential hazards that other analytical tools miss, improving worker safety and reducing environmental contamination.

COLLABORATION/TECHNOLOGY TRANSFER

This is a new initiative. Opportunities for collaboration and technology transfer are currently being solicited.

ACCOMPLISHMENTS

- Installed TGS in the LANL Plutonium Facility. Milestone completed January 1996.
- Completed a review of stored nuclear materials and failure mechanisms from recent incidents at the Los Alamos Plutonium Facility storage vault. Milestone completed February 1996.
- Initiated computer modeling studies to evaluate the use of CT to detect and quantify changes in interim-stored nuclear materials, and to optimize CT equipment for this application.
- Designed test objectives, using high-density non-nuclear materials, to test and evaluate CT systems. Initiated fabrication of test objects. Test objects will be imaged by both DOE facility DR (Digital Radiography)/CT systems and commercial vendors of CT equipment in a round-robin test plan.
- Completed instrument setup and calibration of a TGS system in the Plutonium Facility.
- Began using the TGS to assay nuclear materials at the LANL Plutonium Facility that have been historically difficult to measure, such as salts from plutonium processing operations. These measurements will be used to evaluate the TGS technique against current NDA technologies.
- SRTC has produced two CT test objects that contain a set of chemical compounds with varying density and effective atomic number. These test objects will be used by SRTC and LANL to study the efficacy of dual-energy CT analysis.

CONTACT

Lynn A. Foster
Principal Investigator
MS E513
Los Alamos National Laboratory
Los Alamos, NM 87545
Phone: (505) 665-8261
e-mail: laf@lanl.gov

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None at this time.

4.2 PRESSURE-SENSITIVE DEVICES

TECHNOLOGY NEED

The possible generation of hydrogen gas in plutonium storage containers could jeopardize the integrity of the containment vessel. Accurate determination of hydrogen gas generated in the container is important for understanding the chemical processes that occur during long-term storage.

TECHNOLOGY DESCRIPTION

This project will result in the development of a non-invasive, non-intrusive hydrogen sensor and a data telemetry system to monitor for the presence of hydrogen in nuclear material vessels. The system will be capable of measuring hydrogen concentrations ranging levels from parts-per-million (ppm) to levels approaching 100% hydrogen. The acquired data will be transmitted from the container using a telemetry system based on magnetic coupling. This data transmission technique will preserve the integrity of the container by avoiding breaching the container with wires or cables.

This multiyear project is aimed at providing appropriate sensing elements for plutonium storage containers to characterize fully the chemical and physical changes that occur during storage. The requirements include development of (1) a sensor that measures hydrogen concentration within the inner container of the plutonium storage vessel, and (2) a data telemetry system that transmits the data out of the container. The sensor and the telemetry system must not breach the container or introduce any organic materials into the container. These requirements will be met by using the Robust Hydrogen Sensor developed by Sandia National Laboratories/New Mexico (SNL/NM). The sensor will be modified to meet environmental constraints imposed by the container application. A magnetic field-based telemetry system will be developed to transmit the data. The complete system will be tested first on an empty container to verify performance of the telemetry technique. The system will then be installed on a filled container so that hydrogen concentration levels can be monitored.

BENEFITS

The immediate benefit of the monitoring system will be to contribute to the understanding of the chemical reactions that occur in stored plutonium-bearing materials. This knowledge will assist in designing a long-term monitoring system that will include a wider range of sensing elements. Development of a sensor that can be used for long-term monitoring of hydrogen in containers will alleviate concern over pressure buildup and possible leaking of material. The use of the sensor will also minimize stay times of personnel to periodically unpackage and inspect containers.

COLLABORATION/TECHNOLOGY TRANSFER

This project is a collaborative effort between LANL and Sandia.

ACCOMPLISHMENTS

- The specific environment to be monitored was determined. The initial monitoring device will monitor only hydrogen and temperature.
- The sensor modification and packaging milestone is 50% complete as of April 1996. Sandia has developed a hydrogen sensor that will operate in the correct pressure range. Packaging the sensor may require shielding. Radiation effects and dose rates will be determined empirically and by modeling.

CONTACT

David R. Horrell
Principal Investigator
MS E513
Los Alamos National Laboratory
Los Alamos, NM 87545
Phone: (505) 665-7630
e-mail: dhorrell@lanl.gov

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None at this time.

4.3 CALORIMETER TO INTERFACE WITH THE PLUTONIUM STABILIZATION AND PACKAGING SYSTEM CONTAINER

TECHNOLOGY NEEDS

The Putonium Focus Area (PFA) has identified the need to design, fabricate, model, and optimize a calorimeter and calorimeter control system specifically to interface with the Plutonium Stabilization and Packaging System (PuSPS) material container (the PuSPS is described in section 1.1).

TECHNOLOGY DESCRIPTION

Several calorimeters will be required for the Savannah River Site's Actinide Packaging and Storage Facility and elsewhere in the DOE complex for measurement of the PuSPS container. While multiple calorimeters exist which could measure this container, none are optimized. Benefits of this effort would include optimized measurement time while maintaining necessary system accuracy.

This proposed effort would design and fabricate the calorimeter to interface with the PuSPS container. Computer modeling of the calorimeter's thermodynamics would be completed to support advanced control system development. Control system development would include testing of PID, Fuzzy Logic, and State Space techniques.

BENEFITS

This technology would develop an optimized calorimeter system to interface with the PuSPS container. The most significant benefit would be an optimal measurement time (throughput) system which meets accountability requirements. This leads to an increased sample throughput and/or reduced floor space requirements.

COLLABORATION/TECHNOLOGY TRANSFER

This is a new initiative. Opportunities for collaboration and technology transfer are currently being solicited.

ACCOMPLISHMENTS

Calorimetry design is well established at the Savannah River Site. Multiple calorimeters are in production use and independent testing of the design has been completed.

State Space control techniques have been developed for another SRS calorimeter. Testing of this technique indicated significant time reduction for a cold power startup and potentially significant time reductions for sample measurements. This effort was stopped prior to completion so the calorimeter could be put into production use.

Currently, an effort is underway for a Fuzzy Logic control system development with the APSF 2R & PCV calorimeter. The results of this effort will indicate the benefits of development for the calorimeter to interface with the PuSPS container.

As this is a new initiative, no efforts have been made for the development of an optimized calorimeter to interface with the PuSPS container.

CONTACT

Lee A. ReFalo
Principle Investigator
Savannah River Technology Center
Building 305-A
Aiken, SC 29808
Phone: (803) 725-4473
e-mail: lee.refalo@srs.gov

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None at this time.

4.4 ACOUSTIC RESONANCE SPECTROSCOPY

TECHNOLOGY NEED

This project will continue to develop a noninvasive, nondestructive method for determining the gas composition and pressure build-up in metal, oxide, and residue containers.

TECHNOLOGY DESCRIPTION

Researchers are investigating the feasibility of using acoustic resonance spectroscopy (ARS) to detect pressure increase and gaseous components. Surveillance must be performed on containers of plutonium-containing materials. Development of more sophisticated and precise, noninvasive analytical tools is needed. ARS has been sufficiently well developed at this decision point to determine its viability for this application.

Studies will continue, using surrogate materials in cans to represent plutonium metal, oxide, and residues. Measurement of partial pressures for hydrogen, oxygen, and helium components will be researched. Experiments are being designed to elucidate the effects of such factors as temperature, data acquisition rate, frequency range, and step size on the ability to detect changes within a can. Further areas of needed investigation are changes in pressure and composition of the gas, changes in the can itself, and the ability to observe changes in a can within a second can. DOE is monitoring changes and trying to determine whether the technique will detect changes. Causes of changes are subjects of other studies.

BENEFITS

For safety, the recognition of specific identifiable characteristics within sealed containers is significant. Development of ARS for application to containers would vastly improve current capabilities to detect changes in the containers. Using ARS, the changes in pressure and composition of the gas within the container can be detected. Theoretically, a defective weld in a container also should be detectable.

COLLABORATION/TECHNOLOGY TRANSFER

This is a new initiative. Opportunities for collaboration and technology transfer are currently being solicited.

ACCOMPLISHMENTS

A technical report was prepared detailing findings from previous experiments. The report summarizes a set of experiments designed to evaluate the feasibility of measuring gas pressure and gas composition inside the proposed container with acoustic resonance spectroscopy. Gas resonances are easily detected inside an otherwise empty container. The amplitude of the gas resonances varies with gas pressure and the frequency varies with gas composition, as predicted by theory. In addition, it is observed that the frequencies of the shell resonances vary with gas pressure as well, providing a potential second measure of pressure. Unfortunately, when even small amounts of simulated plutonium oxide (sand) are added to the cylinder, the sand absorbed enough energy so that the gas resonances become undetectable. A simple modification to the container created an analysis chamber in which the gas resonance can be excited and monitored, even though the rest of the container is full of sand. Thus, the feasibility of monitoring gas composition and pressure inside a modified inner residue cylinder with acoustic resonance spectroscopy has therefore been demonstrated. The sensitivity of ARS to detect changes in gas composition based on shifts in the frequency of the gas peak by 1 Hz have been calculated for mixtures of helium, hydrogen, and argon. Although helium and hydrogen are similar in mass, the frequency shift when hydrogen is mixed with helium is nevertheless easily detectable. The calculations predict that an increase of less than 0.5 torr hydrogen in a local atmosphere of helium (590 torr) would be detectable.

Future work will address two issues: demonstration of a capability to observe gas modes of an inner container with transducers mounted on an outer container, and demonstration of the ability to deconvolute acoustic resonances due to the container and those due to gas modes. A set of experimental chambers has been designed to test the ability to couple acoustic responses between an inner and outer container using a variety of coupling geometrics. These chambers will be used to test for the optimum geometry of a container-to-container coupler, for the optimum thickness of lids for containers, and for other container parameters that will affect the successful application of ARS to container surveillance. Theoretical modeling of the acoustical response of a gas mode interacting with a container mode has been started with very encouraging results; i.e., the theoretical response curve reproduces many of the characteristics of experimentally observed response curves. Quantitative comparison of experiment and theory will be started. Mathematical approaches to deconvolute experimental data to obtain quantitative information on the gas modes will be started.

CONTACT

D. Kirk Veirs
Principal Investigator
MS E510
Los Alamos National Laboratory
Los Alamos, NM 87545
Phone: (505) 667-9291
e-mail: veirs@lanl.gov

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4.5

Systems Engineering Development of An Integrated Surveillance System for Stabilized Plutonium-Bearing Materials

Technology Need

This task addresses a summary listing of requirements from the draft Baseline R&D Technical Requirements Document.

Table 4.5-1. Requirements Addressed

Legend for Table 1:
PR = Program Requirement
PrD = Process Development Requirement
SR = Undefined requirement

Req. #	Req. Type	Page #	Requirement Text
1	PR	3	A Systems Engineering approach is required to provide an integrated surveillance system for stabilized plutonium-bearing materials. This research involves: defining surveillance requirements, defining system functions, developing approaches to meet the requirements, performing trade studies to select the best approaches, and evaluating and validating how well the selected approach meets the requirements.
2	PrD	4	A technology-specific program is required that is focused on treating and storing materials safely, with concurrent development of storage criteria and surveillance requirements, centered around the 3- and 8- year targets.
3	PrD	6	Develop sampling, analytical procedures, more effective processing and a storage package surveillance/demonstration program.
4	SR	9	Develop standard requirements for the surveillance and maintenance activities associated with the inventory.
5	PR	32	Develop an integrated approach for the interim storage of plutonium packages, using nonintrusive technologies that minimize personnel exposures and maximize safeguards and security.
6	PrD	43	To meet DOE-STD-3013 requirements for interim storage of plutonium metals and oxides, a permanent marking system must be developed and successfully demonstrated.

TECHNOLOGY DESCRIPTION

This task will identify consistent surveillance requirement sets and process functions using a proven system engineering process. The systems engineering process will begin with definition of customer needs for an integrated system for surveillance and monitoring of stabilized plutonium packages. This will be incorporated with detailed definition of the major functions of the system including characterization of stabilized plutonium-bearing material packages; surveillance and monitoring of the containers and contents; and tracking containers and contents throughout the DOE complex until final disposition. An integrated data acquisition, storage, and retrieval system must be developed for use throughout the life of these materials to ensure compliance with the safeguard and accountability requirements. Figure 4.5-2, An Integrated System for Monitoring Stabilized Plutonium-Bearing Material Packages, is a depiction of the proposed surveillance system functions

BENEFITS

The benefits derived from an integrated DOE wide approach to surveillance will include:

- Identify and preclude duplication of efforts
- Cut cost by eliminating redundant work
- Raise public confidence by implementing a consistent, standardized, and verifiable method of surveilling, monitoring, and tracking packaged, stabilized plutonium across the complex
- Provide systematic standardized data acquisition, storage, and retrieval throughout the DOE complex for well-focused analysis

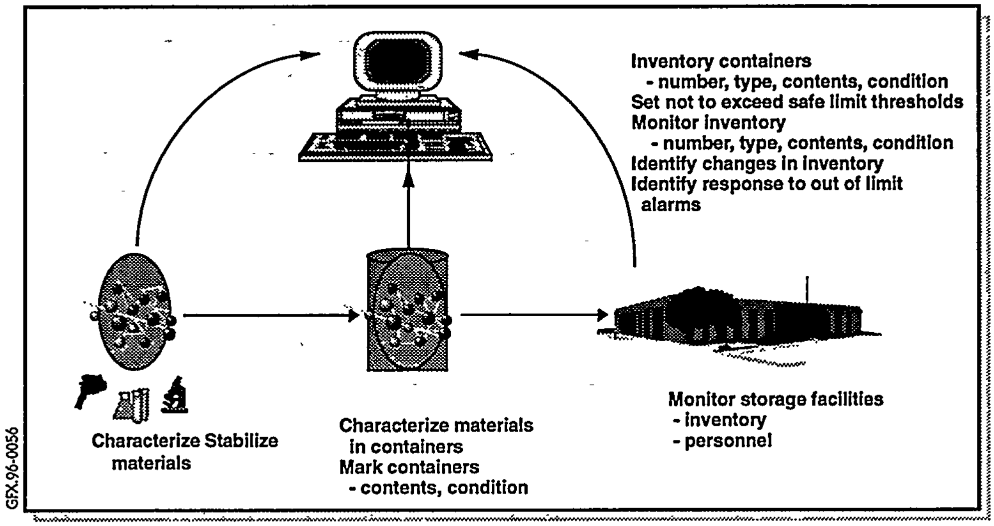

Figure 4.5-2. An Integrated System for Monitoring Stabilized Plutonium-Bearing Material Packages.

- Provide a means to establish a records system that meets regulatory and quality control requirements for tracking this material throughout its lifetime
- Provide proliferation control and tracking of material across the complex
- Provide a growth path for infusing technology to continuously improve system effectiveness

COLLABORATION/TECHNOLOGY TRANSFER

The main goal of this task is to provide an integrated set of requirements for monitoring and surveillance of stabilized plutonium-bearing material packages across the DOE complex. Collaborative efforts will be identified and pursued after evaluating technologies that meet the requirements.

ACCOMPLISHMENTS

A white paper has been submitted to the PFA Technical Advisory Panel identifying the requirements for an Integrated Surveillance System for Monitoring Stabilized Plutonium-Bearing Material Packages. The tasks include:

- Definition of Characterization Functions and Requirements
- Definition of Surveillance Functions and Requirements
- Definition of Tracking and Inventory Functions and Requirements
- Definition of Data Acquisition and Analysis Functions and Requirements
- Production of a System Level Specification

CONTACTS

Gloria Power
Principle Investigator
Lockheed Martin Idaho
Technologies Company
P.O. Box 1625
Idaho Falls, ID 83415-3750
Telephone: (208)526-4423
Fax: (208)526-4313
e-mail: poweg@inel.gov

Bill Motes
Department Manager of Radiation
and Environmental Measurement
Lockheed Martin Idaho
Technologies Company
P.O. Box 1625
Idaho Falls, ID 83415-2202
Telephone: (208)526-3577
Fax: (208)526-3360
e-mail: bmotes@inel.gov

BIBLIOGRAPHY OF KEY PUBLICATIONS

None at this time.

4.6 PORTABLE GLOVEBOX INSTRUMENTATION

TECHNOLOGY NEEDS

The PFA needs to develop a system for performing material characterization and/or accountability in a glovebox line without sample removal or unnecessary utilization of valuable glovebox space. There is a need for the ability to achieve measurements without the spread of contamination, while offering significant flexibility in measurement location.

TECHNOLOGY DESCRIPTION

The proposed measurement system would be configured for glove port mounting with the use of a modular connection device.

Measuring systems such as calorimeters and neutron coincidence counters are fabricated with measurement wells. The measurement well could be made with a modular connection device permanently mounted (and intrinsically sealed). The modular connector would allow for attachment and detachment of the measurement device without breaking glovebox confinement.

Calorimetry, neutron coincidence counting, and modular connectors all exist. Combining the technologies will create a highly useful instrument with very little associated risk.

BENEFITS

This technology would eliminate the need for dedicated glove box space for measurement systems. Additionally, the need for sample removal would be significantly reduced or eliminated.

This technology could be extended from calorimetry or neutron counting to other types of measuring systems.

COLLABORATION/TECHNOLOGY TRANSFER

This is a new initiative. Opportunities for collaboration and technology transfer are currently being solicited.

ACCOMPLISHMENTS

Calorimetry design is well established at the Savannah River Site. Multiple calorimeters are in production use and independent testing of the design has been completed. Modular connection devices were developed for similar purposes as proposed here. As this is a new initiative, no accomplishments have been made towards combining the two technologies.

CONTACT

Lee A. ReFalo
Principle Investigator
Savannah River Technology Center
Building 305-A
Aiken, SC 29808
Phone: (803) 725-4473
e-mail: lee.refalo@srs.gov

BIBLIOGRAPHY OF KEY PUBLICATIONS

None at this time.

4.7 DIGITAL RADIOGRAPHY INSPECTION

TECHNOLOGY NEED

Historically, conventional radiographic techniques provided the necessary qualitative and quantitative information on special nuclear materials, but with several drawbacks. Radiographic films are inefficient capturers of X-ray photons, thus million-volt accelerators were required, which meant thick-walled vaults had to be built. Film processing generates hazardous waste. Each radiographic film is considered classified and potentially contaminated, so a secure, climatically and radiologically controlled repository was required. Personnel exposure to radiation was inevitable since the material manipulation and radiographic film exchange were done manually.

TECHNOLOGY DESCRIPTION

A digital radiography inspection (DRI) station will screen thousands of high-assay containers while primarily looking for oxides, metals, foreign objects, and liquids. The intent is to provide a cost-effective way of placing the fissile plutonium solids and fissile mixed solids containers into interim storage. DRI will be tasked to sort containers into suspect containers that need to be opened and those that do not need to be opened, inspected, and either sent to processing in a suitable form or repackaged using current packaging methods for later treatment.

BENEFITS

An automated, computer-controlled digital radiographic surveillance station would eliminate the hazardous waste associated with the developing of films; eliminate the need for a large, climatically and radiologically controlled vault; and reduce costs since fewer installations would be required.

COLLABORATION/TECHNOLOGY TRANSFER

The Savannah River Technology Center (SRTC) led a complex-wide round-robin exercise to address the current capability of DRI. The exercise involved the participation of six different DOE sites and commercial vendors including Allied Signal Aerospace Company, SNL, LANL, LLNL, Synergistic Detector Designs, and Lockheed's NDT Development Laboratory.

ACCOMPLISHMENTS

SRTC has developed phantoms and mockup containers that were used to produce both digital radiographs and computed tomograms of plutonium pits encased in the mockup containers. This was the first time that digital radiography was substituted for conventional radiography to obtain qualitative and quantitative information on a pit. ADRIS, SRTC's new automated digital radiography inspection system, is a mobile station that has recently been used to inspect the welds on over 1500 heavy-water drums.

CONTACTS

Rick Poland
Principal Investigator
Savannah River Technology Center
723A
Aiken, SC 29808
Phone: (803) 725-3484
e-mail: rick.poland@srs.gov

Boyd Howard
Principle Investigator
Savannah River Technology Center
730A
Aiken, SC 29808
Phone: (803) 725-3765
e-mail: boyd.howard@srs.gov

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4.8 CONVECTION CALORIMETER

TECHNOLOGY NEEDS

Technology needs are to develop a system for performing material accountability with a very fast (< 2 hours per measurement) calorimeter while maintaining necessary accuracy.

TECHNOLOGY DESCRIPTION

The proposed measurement system would be configured for measurement of a 5" diameter, 10" tall container. The concept of operation utilizes an air flow technique developed at the Savannah River Site which has been demonstrated to perform measurements in less than 2 hours.

Two instruments have been put into production use at the Savannah River Site which have used this technique. The technique is based on a repeatable, temperature differential in air flow. Previously, minimal effort was placed on achieving optimal accuracy. Results have been on the order of 1% relative standard deviation. The proposed effort would be based on the current technique, lessons learned, and a focus of achieving 0.5% or less relative standard deviation.

BENEFITS

This technology would significantly reduce the number of calorimeters required for accountability measurements. Benefits could be realized in fewer calorimeters, less expensive calorimeters, reduced floor space requirements, reduced radiation exposure, and simpler systems.

COLLABORATION/TECHNOLOGY TRANSFER

This is a new initiative. Opportunities for collaboration and technology transfer are currently being solicited.

ACCOMPLISHMENTS

Calorimetry design is well established at the Savannah River Site. As mentioned, two calorimeters of this design have been put into production use. Small-scale prototypes have demonstrated less than 0.5% relative standard deviation.

CONTACT

Lee A. ReFalo
Principle Investigator
Savannah River Technology Center
Building 305-A
Aiken, SC 29808
Phone: (803) 725-4473
e-mail: lee.refalo@srs.gov

BIBLIOGRAPHY OF KEY PUBLICATIONS

None at this time.

5.0

TRANSPORTATION AND REMEDIATION INFRASTRUCTURE

The Packaging and Transportation technology baseline products and processes will be developed in concert, meeting published standards (e.g., DOE-STD-3013). The transportation and infrastructure section's research, development, and implementation will provide containers for intra-site transportation of plutonium and other actinide materials for further processing and interim storage. Utilization of modular treatment systems will reduce facility restart requirements and provide stabilization capability on a more predictable schedule.

The Technology Baseline for transportation requires that containers be developed and certified for transport of plutonium solutions and residues in a manner that facilitates stabilization. Transportation technologies will incorporate safeguards and security, emergency planning, surveillance and monitoring, and other applicable requirements.

5.1 MODULAR SYSTEMS

TECHNOLOGY NEED:

Many sites around the DOE complex have facilities that are no longer operating. The restart of major facilities is costly, time consuming, and subject to uncertainties. The availability of modular treatment systems would reduce the restart requirements and provide stabilization capability on a more predictable schedule.

TECHNOLOGY DESCRIPTION

The objective of this effort is to develop and optimize integrated, skid-mounted, modular processing systems to identify where this approach has cost or schedule advantages over the baseline plans outlined in the Rocky Flats Environmental Technology Site (RFETS) Site Integrated Stabilization Management Plan. This study will also determine if there is utility in the DOE complex for using modular portable systems to solve other problems involving special nuclear materials (SNM) for EM-60.

The development team will review and evaluate current plans for stabilizing all backlog residues at RFETS. Based on this analysis, the team will develop and optimize integrated, skid-mounted, modular processing systems to be fielded for three different scenarios to meet the needs of the RFETS Residue Program. These proposed fielded scenarios will be scheduled and costed for direct comparison against the current baseline for residue processing at RFETS. The three scenarios to be developed are:

1. Develop an integrated and optimized, skid-mounted, modular system for all the backlog residues (except sand, slag, and crucible, graphite fines, and wet combustibles, which will be evaluated under the other two scenarios) based on a core capability for unpacking, sorting, assaying, and repackaging and certification. This scope will also address all processing requirements necessary to meet the RFETS commitment to place all residues in a stabilized condition by May 2002.

2. Develop an integrated and optimized, skid-mounted, modular system that addresses processing the wet combustibles (aqueous system plus a pyrolysis or low temperature thermal desorption unit) to meet a Residue Program commitment for stabilization by October 1998.

3. Should it be determined that any residue inventories cannot be sent to Waste Isolation Pilot Plant (WIPP) because of inability to terminate safeguards and security, a method will be developed to deal with the materials at RFETS.

For each of the above scenarios, the team will provide the following: costs, schedule, flow charts, facility/system interface identification, engineering layout plan, design criteria, seismic, fire protection, D&D (decontamination and decommissioning), and those aspects of the authorization basis necessary to ensure implementation success. The cost and schedule of skid-mounted equipment delivery and installation, host facility readiness, procedure development, operator training, equipment start-up, and process operation will be developed so that they can be compared with plans to modify existing process lines. A plan will be developed to achieve successful skid/facility integration, reasonable and dependable criticality limits, and an approved safeguard and security plan, which are integral parts of implementing any plutonium process line.

In a separate task, DOE will identify and document the specific residue processing needs of these other sites that can be met with modular components. Drawing upon the feasibility study for plutonium residues, the study team will review DOE needs involving SNM, including stabilization needs at other sites, transuranic waste certification, and material handling. The team will determine if portable modular systems offer a potential solution to these needs.

BENEFITS

Advantages of the modular concept are as follows:

1. Elimination of the need for new permanent facilities, and mortgage reduction of current facilities
2. Flexibility in changing the process or in processing different materials with minimal impact on the overall system
3. Ability to cold test and train on the system outside a radioactive materials management area
4. Ability to use portions of the modular system to handle other materials solving different problems
5. Portability and the ability to reuse modular systems at different sites
6. Ability for part or all of the authorization basis to move with the modular system

COLLABORATION/TECHNOLOGY TRANSFER

This is a new initiative. Opportunities for collaboration and technology transfer are currently being solicited.

ACCOMPLISHMENTS

- A detailed work plan was developed for the modular systems project.
- Detailed resource loading was negotiated between groups.

CONTACTS

Richard A. Hildner
MS H821
Los Alamos National Laboratory
Los Alamos, NM 87545
Phone: (505) 667-6453
e-mail: rah@lanl.gov

Stanley J. Zygmunt Jr.
MS E517
Los Alamos National Laboratory
Los Alamos, NM 87545
Phone: (505) 667-8978
e-mail: szygmunt@lanl.gov

BIBLIOGRAPHY OF KEY PUBLICATIONS

None at this time.

6.0

CORE TECHNOLOGY PROGRAM INTRODUCTION

In its acceptance of the Defense Nuclear Facilities Safety Board (DNFSB) Recommendation 94-1, the Department of Energy (DOE) developed an Implementation Plan that included a Core Technology Program. The objective of the Core Technology Program is to augment the knowledge base about general chemical and physical processing and about storage behavior of actinide-containing materials and to assure safe interim nuclear material storage until disposition policies are formulated. The 94-1 Research and Development (R&D) Plan recommended that an ongoing Core Technology Program be established to support stabilization technologies, address unforeseen problems associated with long-term storage, and provide technology for predicting the long-term behavior of nuclear materials. A series of R&D tasks has been developed that specifically addresses the technical problems associated with developing a detailed understanding of the processes that affect stabilization and the long-term behavior of plutonium-containing materials. In developing these tasks, the Nuclear Materials Stabilization Task Group (NMSTG) solicited input across all relevant scientific disciplines. The proposed coordinated set of tasks can be seen as a multidisciplinary scientific approach to the understanding and control of these complex materials over a long time.

As materials degrade because of either the reactive nature of plutonium or radiolysis, the chemical description of the remaining material becomes more complex. Understanding the chemical behavior of complex materials requires characterization beyond simple elemental analysis. A detailed description on the molecular level is required, if the chemistry is to be understood and controlled.

There are three tasks relating to stabilization that address the molecular level characterization and chemical behavior of materials. In the Actinide Solution Chemistry task, plutonium in nitric acid and in hydrochloric acid will be studied. The separation of plutonium from these solutions using ion exchange is easily upset by changes in the chemical environment of the plutonium. It is reasonable to assume that solutions sitting for years have changed to such an extent that the solution chemistry is now quite different. Molecular level characterization will allow more informed decisions to be made concerning nuclear materials stabilization. Plutonium can exist within residues either as a molecular species adsorbed onto surfaces and interfaces, or as microcrystallites embedded within the material. The Chemical and Physical Interactions of Actinides with Surfaces task addresses the surface chemistry of plutonium. The Changes in the Chemical State of Plutonium task addresses the physicochemical state of the plutonium compounds found in residues.

Modern analytical techniques that were developed specifically to analyze the complex materials commonly used in industry today are proposed to be used to characterize these two types of plutonium materials. Molecular-level characterization is required to predict the long-term behavior of these materials and to anticipate currently unforeseen problems. Extending the current capabilities in optical spectroscopy and x-ray techniques to the wide variety of plutonium species that have not been studied but that are present in existing residues will lead to insight concerning the chemistry and the long-term behavior of these species. These and other established core technology development activities will fully develop a basic competency in materials science that will have a strong impact on a broad spectrum of stabilization efforts.

6.1 ACTINIDE SOLUTION CHEMISTRY

TECHNOLOGY NEED

This task seeks to describe aspects of the solution chemistry of actinide elements in legacy residues and in proposed residue treatment processes. The intent is to be able to guide stabilization and disposition decisions and to enable efficient optimization of treatment processes.

TECHNOLOGY DESCRIPTION

The accumulation of residues contaminated with plutonium and other transuranic (TRU) elements has resulted from separations processes targeted at recovering plutonium all the way down to an economic discard limit. In some cases, such accumulation has led to the creation of large volumes of slightly contaminated residue that may be physically or thermodynamically unstable. Residue stabilization and treatment processes will need to be efficient and targeted at the present-day issues of safety, stability, and the economics of disposal. Aqueous chemical separations will be required for primary- and post-treatment operations following stabilization of many of the residues. These aqueous separations will have to be carried out under chemical conditions that differ considerably from those used in production of material for weapons. The work proposed here will seek to measure critical thermodynamic parameters governing actinide chemistry under these chemical conditions so that treatment and separation processes can be designed and optimized quickly.

Separations of actinides from other elements and from each other generally rely on partitioning the actinide into a different phase (solid-liquid, liquid-liquid) from the bulk of the material. This partitioning relies on the transformation of the actinide into a chemical form that has a high affinity for one phase while leaving the additional components in the other phase. The chemical interactions of importance in separations processes are (1) the oxidation state, (2) the thermodynamic formation constant, (3) solubility, and/or (4) sorption characteristics. The primary focus during FY96 and the first half of FY97 will be on plutonium solution chemistry, because there are major solution chemistry issues associated with applying traditional acid solution separations processes to largely neutralized and alkaline residues and wastes. The subsequent focus for FY97 and FY98 will be on sorption/desorption reactions between plutonium and model sludge substituents.

Initially, experiments will be completed that are aimed at understanding the chemistry of Pu(IV) in nitric acid, a chemical system of central importance to traditional plutonium separations processes, but one that is surprisingly poorly understood at a molecular level. A significant amount of data is available on this system, but additional data are needed to complete the understanding at high-acid concentrations. Completion of this work will be of broad value in that it will validate the analysis methods prior to application to chemical systems whose macroscopic behavior is only poorly understood. The second part of the experimental work in FY96 through early FY97 will be to study the solution chemistry of plutonium under conditions relevant to post-processing of effluent streams from new decontamination and waste destruction technologies, such as electrolytic decontamination and supercritical water oxidation. The effluent streams from these treatment processes are likely to be neutral or alkaline with high ionic strength. These conditions result in dramatic differences in plutonium chemistry from that observed in acid solutions, and the effects of these differences need to be understood.

Besides stabilizing higher oxidation states, such as Pu(V), the higher pH will allow carbonate complexation of plutonium to dominate over nitrate and phosphate complexation. Thus, the focus will be on measuring carbonate complexation and solubilization of plutonium in alkaline solutions. While there are good room-temperature data for some of these reactions under environmental conditions, little work has been done on the temperature or ionic-strength dependence of these reactions under conditions relevant to existing residues or likely residue treatment processes. Plutonium solution chemistry has been explored using a variety of techniques, including traditional techniques (electronic absorption, potentiometry, bulk solubility); highly structure-specific techniques (x-ray absorption, nuclear magnetic resonance [NMR], and Raman spectroscopies); and highly sensitive techniques capable of speciation even for limited-solubility solutions (such as photoacoustic absorption spectroscopy). These techniques will be applied to high-temperature, high-pH, and high-ionic-strength solution studies.

Chloride complexation of plutonium under conditions relevant to residue treatment is another fundamental area of plutonium chemistry that is poorly understood. Chloride appears to compete with carbonate for plutonium coordination in alkaline solutions, if chloride concentration is high enough. Chloride concentration will be an important consideration connected with any treatment processes for salt residues or for aqueous chloride process residues. Early in FY97, chloride complexation studies will be conducted, focusing on conditions specific to residue treatment.

It is also important to understand how reduction-oxidation (redox) chemistry and solubility of plutonium is affected by coprecipitation with hydroxides of other metals that may be made less soluble due to residue treatment. The limited literature that exists on plutonium under these conditions shows that this condition is poorly understood. Many of the solution techniques previously described to plutonium sorption experiments have been extended. For example, through the use of an optically clear boehmite colloid, the study of Pu(V) sorption onto aluminate surfaces has been initiated. The structure-specific absorption bands of Pu(V) can be observed to shift as the plutonium sorbs to the substrate; in FY97, these changes will be correlated with parallel extended x-ray absorption fine structure (EXAFS) experiments. EXAFS experiments on plutonium sorption onto an iron oxyhydroxide surface, such as goethite, are planned for FY98.

BENEFITS

The benefits of this work can be understood by considering specific cases to which the results could be applied. Electrodecontamination of can-out cans will produce a solution/sludge residue in which most of the actinide component is expected to be in the sludge. This separation must be verified and maximized for this process to operate at high efficiency without generating large amounts of contaminated liquid waste. Furthermore, separation of the actinide component from the sludge, largely iron hydroxide, also may be desirable for waste minimization. Likewise, the organic-free effluent from the supercritical water oxidation of combustible materials will contain plutonium, under solution conditions in which the chemistry is poorly understood. The studies may lead to easy separation of plutonium from the bulk of the material, so that it can be either recycled or discarded readily.

COLLABORATION/TECHNOLOGY TRANSFER

This is a new initiative. Opportunities for collaboration and technology transfer are currently being solicited.

ACCOMPLISHMENTS

This activity was begun in the first quarter of FY96. Accomplishments will be detailed in future revisions of this book.

CONTACT

John M. Berg
Principal Investigator
MS E510
Los Alamos National Laboratory
Los Alamos, NM 87545
Phone: (505) 665-8262
e-mail: jberg@lanl.gov

BIBLIOGRAPHY OF KEY PUBLICATIONS

None at this time.

6.2 CHEMICAL AND PHYSICAL INTERACTIONS OF ACTINIDES WITH SURFACES

TECHNOLOGY NEED

Researchers on this project will study the chemical interactions and surface structural characteristics of plutonium and uranium species adsorbed onto solid-phase materials found in the processing residues throughout the DOE complex. Actinide species interact with residue substrate surfaces by means of a number of mechanisms (surface complexation, physisorption, matrix diffusion, and entrainment) to yield a wide range of surface-bound species. Stabilization of these residues by removal of the actinide species from the matrix, while minimizing secondary waste volumes, is a goal not yet achieved. In many instances, the development and implementation of treatment strategies has been thwarted because of the failure to understand and incorporate fundamental surface chemistry of the actinides into the design of the treatment process. Molecular spectroscopic probes of small spot size will be employed on several prototypical actinides/substrates to obtain detailed characterization and imaging of the nature and extent of the surface interaction between the actinide species and the substrate. This information provides a basic framework of surface interactions of actinides with processing residues that can be incorporated into future R&D stabilization activities.

TECHNOLOGY DESCRIPTION

The interactions of actinides (plutonium and uranium) with three or more prototype solid substrate systems will be investigated. The substrates will be chosen to be representative of actual residues found in ash (silicates), combustibles (cellulosic and plastic materials), and ceramic crucibles (magnesium oxide and Leco®). These three classes of residues provide a diverse range of surface chemical interactions, and they also represent a significant fraction of the total residue mass within the DOE complex. Preparative reactions of the actinides with the prototype solids will be carried out under conditions that reflect as closely as possible the processes responsible for the residues. The chemical and structural characteristics of the resulting actinide-substrate complexes will be determined using a combination of diffuse reflectance, luminescence, photoacoustic, Raman, and x-ray spectroscopies. This suite of spectroscopic tools will provide oxidation state, elemental composition, and molecular or phase composition of the actinide species. By using instrumentation sensitive to small sample sizes, ultimately both elemental and molecular images will be obtained that will graphically illustrate the compositional variance of these species on residues. While the principal focus will be on substrate interactions with plutonium species, the project will also take advantage of the similarity in chemistry and ease of handling of U(IV) and U(VI) O_2^{2+} species to provide baseline information in the initial studies. At the same time, the project will examine actual residues from each of the three residue classes in order to establish the correlation in actinide-substrate interactions between the synthetic controlled samples and the real residues.

BENEFITS

A common theme in the stabilization of most residues is the extent to which the actinide species can be mobilized. In general, if readily implementable schemes can be devised to mobilize the actinide, then residue processing to remove the actinide is a viable strategy for stabilization. Conversely, some actinide-substrate interactions may be of such a tenacious nature that residue processing is intractable. The study will provide necessary fundamental information to assess the probability for successful mobilization of the actinide and sufficiently detailed chemical and physical characterization data to devise the most appropriate mobilization strategies. The characterization protocol is also noteworthy in that all proposed spectroscopic probes can be implemented on challenging samples, such as heterogeneous and mixed-phase residues, with no sample preparation. This approach has been demonstrated successfully on a range of samples, including uranium-contaminated soils from Fernald Environmental Management Project and high-level tank wastes from Westinghouse Hanford Company.

COLLABORATION/TECHNOLOGY TRANSFER

This is a new initiative. Opportunities for collaboration and technology transfer are currently being solicited.

ACCOMPLISHMENTS

This activity was begun in the first quarter of FY96. Accomplishments will be detailed in future revisions of this book.

CONTACT

David Morris
Principal Investigator
MS J586
Los Alamos National Laboratory
Los Alamos, NM 87545
Phone: (505) 665-6487
e-mail: dmorris@lanl.gov

BIBLIOGRAPHY OF KEY PUBLICATIONS

None at this time.

6.3 CHANGES IN THE CHEMICAL STATE OF PLUTONIUM

TECHNOLOGY NEED

The purpose of this work is to identify and characterize changes in the physicochemical state of the myriad of plutonium compounds found in residues. Plutonium-containing materials other than metal and pure oxide will be studied using x-ray diffraction (XRD, powder and single crystal), x-ray absorption spectroscopy (XAS), x-ray photoelectron spectroscopy (XPS), and optical spectroscopies (reflectance absorption and Raman) in order to identify changes in the local chemical environment of the plutonium metal center. Changes in local chemical environment can indicate the onset of deleterious transformations in the waste form that may lead to unsafe storage conditions.

TECHNOLOGY DESCRIPTION

Plutonium-containing materials that are not pure oxide or metal are being considered for interim storage. Interim storage times of decades are anticipated, and the chemical behavior of plutonium-containing materials over this long time scale is not known. These materials contain constituents that can lead to gas generation and corrosion. The chemical behavior of this material, especially the material intimately in contact with plutonium, needs to be studied over time in order to ensure that any chemical changes that do occur do not adversely affect the waste form. The oxidation state of plutonium is especially vulnerable to aging effects, and it has been shown that x-ray techniques are successful in determining plutonium valence. XAS is sensitive to the local chemical environment of a metal, in this case plutonium, and thus is an outstanding analytical technique to study changes in the materials that are directly bonded to plutonium. XAS has been used, which includes the extended x-ray absorption fine structure (EXAFS) and the x-ray absorption near-edge structure (XANES) regions, and optical spectroscopies to study the speciation of plutonium in nitric acid, the local structure of plutonium alloys, the chemical form of plutonium and neptunium in carbonate groundwater at Yucca Mountain, and the form of uranium in soils at the Fernald Environmental Management Project site. XAS has high sensitivity to plutonium embedded into or sorbed onto other material. Los Alamos National Laboratory (LANL) maintains facilities for XAS studies of radioactive materials at the Stanford Synchrotron Radiation Laboratory (SSRL). This work will use this unique expertise to carry out the experimental plan.

The work will concentrate on problem plutonium residues, such as salts, ash, alloys, and carbides. Samples of pertinent residues will be identified from the inventories of the Los Alamos Plutonium Facility and, if possible, from other sites within the DOE complex. These samples will be studied, along with samples of pure compounds, over the length of the project, using diffuse reflectance, XRD, XAS, and XPS techniques. Initial work will focus on determination of the oxidation state of plutonium. XANES and XPS are established techniques for the determination of the oxidation state and the local chemical environment. Actinide oxidation states also can be determined by binding energy shifts in the XPS photoemission spectra, relative to the actinide metal. Pure samples of appropriate plutonium salts, carbides, and alloys will be prepared and characterized using x-ray powder diffraction, reflectance absorption spectroscopy, and vibrational spectroscopy (when appropriate), as well as XAS. Analysis of the XRD and XAS data will yield the necessary database of plutonium solid structures. Absorption spectra of plutonium compounds vary considerably and are very sensitive to the structure and orientation of the ligands surrounding the plutonium. Correlation between the structure obtained by XRD and XAS data and the optical spectra of the pure compounds may be possible. Such information will be useful in determining when chemical changes take place in residues or in other plutonium-containing material.

Sample preparation will take place within facilities at TA-55 and the Chemistry and Metallurgy Research (CMR) Building at LANL. Absorption spectra, x-ray powder, and single-crystal diffraction data will be obtained, using existing equipment at TA-55, at the CMR Building, and at the Los Alamos Radiochemistry Site. XAS experiments will be conducted at SSRL, and the data will be analyzed at existing computing facilities at LANL.

BENEFITS

Early identification of chemical changes in residues in interim storage is necessary for meeting DOE's environment, safety, and health objectives and may be used to guide future disposition technologies. A comprehensive understanding of the chemical changes that occur in residues will ensure the safety of the storage media and will provide for a meaningful environmental assessment of sites where the materials are stored. Early identification of problematic chemical changes will allow sufficient lead time to assess options for the disposition of the material.

COLLABORATION/TECHNOLOGY TRANSFER

This is a new initiative. Opportunities for collaboration and technology transfer are currently being solicited.

ACCOMPLISHMENTS

This activity was begun in the first quarter of FY96. Accomplishments will be detailed in future revisions of this book.

CONTACT

Kirk Veirs
Principal Investigator
MS E510
Los Alamos National Laboratory
Los Alamos, NM 87545
Phone: (505) 667-9291
e-mail: viers@lanl.gov

BIBLIOGRAPHY OF KEY PUBLICATIONS

None at this time.

PFA BUSINESS OPPORTUNITIES

WORKING WITH THE PLUTONIUM FOCUS AREA

DOE provides a range of programs and services to assist industry, universities, and other private-sector organizations and individuals interested in developing or applying plutonium stabilization technologies. At the directions of the NMSTG, the PFA employs a number of mechanisms to identify, integrate, develop, and adapt promising emerging technologies. These mechanisms include contracting and collaborative arrangements, procurement provisions, licensing of technologies, and consulting arrangements. PFA facilitates the development of subcontracts, R&D contracts, and cooperative agreements to work collaboratively with the private sector.

COOPERATIVE RESEARCH AND DEVELOPMENT AGREEMENTS

Cooperative Research and Development Agreements (CRADAs) are mechanisms for collaborative R&D. They are agreements between a DOE R&D laboratory and any non-federal source to conduct cooperative R&D that is consistent with the PFA's mission. The partner may provide funds, facilities, people, or other resources. PFA provides the CRADA partner with access to facilities and expertise; however, external participants receive no federal funds. Rights to inventions and other intellectual property are negotiated between the PFA and the participant. Certain generated data may be protected for up to five years. Several companies may combine their resources to address a common technical problem. Funds can be leveraged to implement a consortium for overall program effectiveness.

PROCUREMENT MECHANISMS

PFA's procurement mechanisms for technology development are in the form of unsolicited proposals and formal solicitations, although the latter are preferable.

For more information about PFA unsolicited proposals and formal solicitations, contact:

William L. Scott, U. S. *Department of Energy - Idaho Operations Office*, 785 DOE *Place, Idaho* Falls, ID 83401-1562, *tel:* (208) 526-8189, e-mail *scottwl@inel.gov, or*

Dr. Finis H. *Southworth, Lockheed* Martin *Idaho* Technologies *Company*, P.O. Box 1625, *Idaho* Falls, ID 83415-3750, *tel:* (208) 526-8150, e-mail *fin@inel.gov.*

LICENSING OF TECHNOLOGIES

DOE contractor-operated laboratories can license PFA developed plutonium stabilization technology. Licensing activities are conducted according to existing DOE intellectual property provisions.

TECHNICAL PERSONNEL EXCHANGE ASSIGNMENTS

Personnel exchanges provide opportunities for scientists from private industry and DOE laboratories to work together at various sites on Focus Area task assignments. Private industry is asked to contribute substantial cost-sharing for these personnel exchanges. To encourage such collaboration, rights to any resulting patents go to the private sector company. These personnel exchanges, which can last from three to six months, result in the transfer of technical skills and knowledge.

CONSULTING ARRANGEMENTS

PFA scientists and engineers are available to consult in their areas of technical expertise. PFA employees who wish to consult with private industry, universities, and other organizations can sign non-disclosure agreements, and are encouraged to do so.

REIMBURSABLE WORK FOR INDUSTRY

The unique resources located at DOE's PFA laboratories are available to perform work for private industry and other federal agencies. The special technical capabilities at DOE laboratories are incentives for the private sector to use PFA facilities and contractor expertise. An advanced class patent waiver gives ownership of any inventions resulting from the research to the participating private sector company.

ACRONYMS

AL	Albuquerque Operations Office
ANL	Argonne National Lab
CH	Chicago Operations Office
CofC	Certificate of Compliance
CRADA	Cooperative Research and Development Agreement
CT	Computed Tomography
DNFSB	Defense Nuclear Facilities Safety Board
DOE	U.S. Department of Energy
EM	Environmental Management
EP	Executive Panel
EPA	Environmental Protection Agency
ES&H	Environment Safety & Health
FGE	Fissile Gram Equivalent
GMODS	Glass Material Oxidation and Dissolution System
HEPA	High-Efficiency Particulate Air
HGMS	High-Gradient Magnetic Separation
INEL	Idaho National Engineering Laboratory
IP	Implementation Plan
IWG	Integration Working Group
LANL	Los Alamos National Laboratory
LLW	Low-Level Waste
LLNL	Lawrence Livermore National Laboratory
LMITCO	Lockheed Martin Idaho Technologies Company
MEO	Mediated Electrochemical Oxidation
MT	Metric Tons
NDA	NonDestructive Assay
NEPA	National Environmental Policy Act

NESHAP	National Emissions Standards for Hazardous Air Pollutants
NMSTG	Nuclear Materials Stabilization Task Group
NRC	Nuclear Regulatory Commission
ORNL	Oak Ridge National Laboratory
PFA	Plutonium Focus Area
PFA MP	Plutonium Focus Area Management Plan
PHP	Plasma Hearth Process
Pu	Plutonium
PuSPS	Plutonium Stabilization and Packaging System
RC	Research Committee
RCRA	Resource Conservation and Recovery Act
R&D	Research and Development
RDP	Research and Development Plan
RFETS	Rocky Flats Environmental Technology Site
RFFO	RFETS Field Office
RL	Richland Operations Office
SAIC	Science Applications International Corporation
SAR	Safety Analysis Report
SRO	Savannah River Operations Office
SRS	Savannah River Site
SSOC	Safe Sites of Colorado
TAP	Technical Advisory Panel
TGS	Tomographic Gamma Scanner
TRD	Technical Requirements Document
TRU	Transuranic
WIPP	Waste Isolation Pilot Plant