DOE/EM-0279

Office of Environmental Management

Nuclear Materials Stabilization

PLUTONIUM FOCUS AREA

Technology Summary

March 1996

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 approach, EM developed a management structure and principles that led to creation of specific focus areas. These organizations were designed to focus scientific and technical talent throughout DOE and the national scientific community on 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), the Nuclear Materials Stabilization Task Group (NMSTG, EM-66) followed EM-50's structure and chartered the Plutonium Focus Area (PFA). NMSTG's charter to the PFA, described in detail later in this book, plays a major role in meeting the EM-66 commitments to the Defense Nuclear Facilities Safety Board (DNFSB).

The PFA is a new program for FY96 and as such, the primary focus of revision 0 of this Technology Summary is an introduction to the Focus Area; its history, development, and management structure, including summaries of selected technologies being developed. Revision 1 to the Plutonium Focus Area Technology Summary is slated to include details on all technologies being developed, and is currently planned for release in August 1996.

The following outlines the scope and mission of the Office of Environmental Management, EM-60, and EM-66 organizations as related to the PFA organizational structure.

EM MISSION

The Office of Environmental Management (EM) is responsible for managing the cleanup of DOE wastes 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.

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

The mission of the Nuclear Material and Facility Stabilization program is to protect the public and environment from the hazards of post-Cold-War nuclear materials, 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 (see Figure 1). As such, the Office provides for the leadership necessary to accomplish the mission and carries out those functions including program planning and budgeting, evaluation and intervention, and representations 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 (EM-66, 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 pending final 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.
- 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 set out 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.

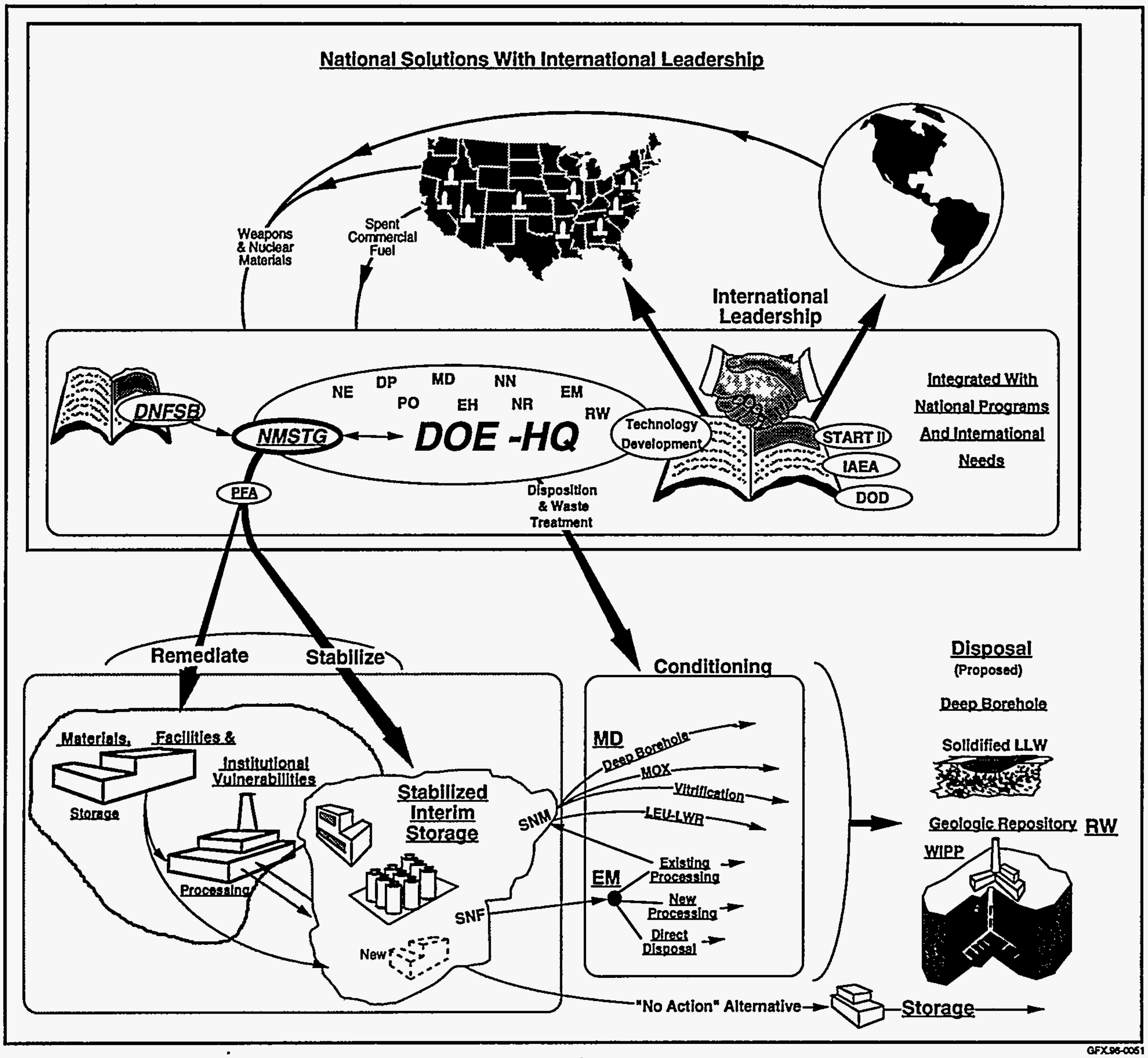

Figure 1. The 94-1 Perspective

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 vast majority of fissile materials scrap and materials from retired weapons was recycled. During this period, it was cost-effective to recover fissile material from high assay scrap and retired weapons. As a result, little fissile material scrap was considered surplus, 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, many materials were left in conditions unsuitable for long-term storage. Consequently, residues and other processing intermediates are presently in various states at several sites under conditions that cannot assure safety. With increasing frequency, the 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 the interim safety issues for the long term. Thus, a focused and concentrated R&D effort is needed to overcome the technical shortcomings associated with these issues.

On May 26, 1994, the Defense Nuclear Facilities Safety Board (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 remediation of these materials. DOE accepted DNFSB Recommendation 94-1 on August 31, 1994. After establishing the Nuclear Materials Stabilization Task Group (NMSTG), DOE issued an implementation plan to address these concerns ("Defense Nuclear Facilities Safety Board Recommendation 94-1 Implementation Plan," February 28, 1995).

Recommendation 94-1, sub-recommendation (2) states "... 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 an R&D plan to address the technology and core program needs of the stabilization program
- Prepare task statements defining R&D activities required to accomplish program objectives.

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

R&D PLAN

The PFA charter includes implementation and maintenance of the R&D Plan developed by the Research Committee. The following summary provides the necessary background to the R&D Plan as an introduction to the PFA.

BACKGROUND

The methodology used by the Research Committee to formulate the R&D Plan included a review of the Implementation Plan and Site Integrated Stabilization Management Plans; walkthroughs of the Savannah River Site (SRS), the Hanford Site (Hanford), Lawrence Livermore National Laboratory (LLNL), and Los Alamos National Laboratory (LANL); and regular meetings of the Research Committee, including ex-officio members and technical advisors.

In developing the R&D Plan, the Committee addressed five of the six material categories discussed in the 94-1 Implementation Plan, which consisted of plutonium solutions, plutonium residues and oxides (<50% Pu), plutonium metals and oxides (>50%), uranium metals, and special isotopes. R&D efforts related to spent nuclear fuel stabilization, the sixth category, were specifically excluded from consideration in the R&D Plan. These efforts are being coordinated though the Technology Integration Plan, Technical Working Group established by the Office of Spent Fuel Management in June 1993. In addition, issues related to funding, schedules, logistics planning, and facilities were not within the scope of the R&D Plan and are being addressed by the Integration Working Group and by other groups as designated by the Director of the Nuclear Materials Stabilization Task Group.

In responding to DNFSB Recommendation 94-1, DOE committed to complete specific nuclear materials stabilization tasks assigned to 3- or 8-year timeframes. The Research Committee focused its review on existing technologies and on technologies currently under development to determine their adequacy relative to the 3-year commitments shown in Table 1. The Committee also outlined R&D requirements to address technologies needed to support the Department's 8-year commitments.

The Research Committee's first objective was to identify technology baseline requirements for all categories of nuclear materials and for related issues that must be addressed by the Plan. These requirements are presented in the R&D Plan and provide the formal basis for all technologies needed to address nuclear materials stabilization, regardless of the status of the required technologies.

Table 1. Three-Year Stabilization Commitments

Commitment	Date
Transfer Purex solutions to tank farms (Hanford)	August 1995
Stabilize plutonium residue sludge (Hanford)	September 1995
Stabilize 220 kgs of residues (LANL)	October 1995
Vent 2,045 drums of residue (RFETS)	October 1995
Process F-Canyon plutonium solutions (SRS)	January 1996
Stabilize 46 packages of ash (Hanford)	March 1996
Repackage all metal in contact with plastics (All)	September 1996
Vent inorganic and wet/miscellaneous residues (RFETS)	October 1996
Remove and ship high enriched uranium solutions (RFETS)	December 1996
Stabilize high-hazard pyrochemical salts (RFETS)	May 1997
Stabilize high-hazard sand, slag, and crucible residues and graphite fines (RFETS)	May 1997
Process H-Canyon Pu-242 solutions (SRS)	November 1997
Covert HEU solutions to stable oxide (SRS)	December 1997
Stabilize remainder of high-hazard pyrochemical salts (RFETS)	December 1997
Stabilize sand, slag, and crucible residues (SRS)	December 1997

PFA CHARTER

To ensure timely development and implementation of the technologies outlined in the R&D Plan, the NMSTG will continuously track the progress of the R&D program through the PFA. If a baseline technology seems unlikely to achieve the desired results or if a single competitive alternative technology must be selected, the NMSTG will, at its discretion, charter a trade study as part of the decision basis for that technology.

The 94-1 Implementation Plan requires research and development to meet short-term needs in a technology-specific program, plus a longer term core program to remediate facilities and stabilize related nuclear materials. To assist the NMSTG in meeting these requirements, the PFA was chartered under the DOE Idaho Operations Office with support from Lockheed Martin Idaho Technologies (LMIT) and Argonne National Laboratory (ANL). This team was selected because of LMIT's demonstrated skill in project management and systems engineering and ANL's experience in plutonium technologies. The PFA will implement and maintain the NMSTG Research & Development Plan, and will follow-on and augment the activities of the former Research Committee .

PFA Management Plan

To support operational needs of the NMSTG and PFA, a PFA Management Plan was written, approved, and published in November 1995 (DOE-Idaho #INEL-96/0004, Rev. O). This document provides direction to Focus Area participants and a detailed description of management, structure, interfaces, reporting relationships, roles and responsibilities. The salient points of the PFA Management Plan are outlined herein, providing the audience with an abstract of the PFA operational structure, objectives, interfaces, and organization.

PFA's Purpose and Scope

The PFA will recommend solutions to site-specific and complex-wide technology issues associated with plutonium remediation, stabilization, and preparation for disposition, in order to:

- Expedite complex-wide progress
- Standardize resolutions, practices, and equipment systems
- Promote integration/interfacing
- Produce cost-effective programmatic results.

The PFA continues to seek opportunities for industry and university participation. The PFA material scope is primarily on Pu-bearing materials (excluding TRU wastes and final-form weapons components), and includes interest in other fissile materials and special isotopes. The technology-specific program is focused on stabilization of materials during the next three to eight years, with concomitant development of treatment options leading to the final disposition state. The core technology program augments the knowledge base about general chemical and physical processing and interim storage behavior to assure safe material management until disposition.

PFA Interfaces

NMSTG:
The PFA Manager reports to the Director of the NMSTG. The PFA assumes the former responsibilities of the Research Committee under the NMSTG, for plutonium bearing materials. The PFA is also the element of the NMSTG to provide technical and peer review of research plans developed and implemented by the 94-1 R&D Lead Laboratory.

Lead Laboratory:
Both the PFA and the 94-1 R&D Lead Laboratory (LANL) receive programmatic guidance from the NMSTG. The PFA addresses technological and core program needs for a viable plutonium stabilization program, while the Lead Laboratory undertakes agreed upon R&D efforts to overcome these technical obstacles.

The PFA analyzes stabilization needs and identifies candidate topics for a concerted R&D effort. These identified roles require close cooperation and interaction between the PFA, Lead Laboratory, and complex-wide resources. This is accomplished on a day-to-day basis by having a representative from the Lead Laboratory participate as a member of the PFA Technical Advisory Panel (TAP) and by having a representative from DOE-AL participate on the PFA Executive Panel (EP). The PFA coordinates periodic peer and technical reviews of research proposals, progress, and products by the Lead Laboratory. Additionally, meetings involving NMSTG, PFA, and Lead Laboratory personnel are held as needed to coordinate activities, and exchange information.

Integration Working Group:

The Integration Working Group (IWG) continues to function as part of the NMSTG organization to integrate the use of facilities and capabilities. The PFA recognizes the need to interface with the IWG on a continuing basis. This coordination and information exchange takes place among the PFA and IWG managers.

PFA OBJECTIVES

The NMSTG is charged with timely achievement of safe and secure remediation and interim storage of plutonium belonging to DOE-EM. Key PFA objectives include:

- Develop plutonium interim storage criteria and surveillance monitoring/requirements that are integrated with follow-on disposal programs.
- Provide appropriate Systems Engineering to the NMSTG.
- Provide a management structure to achieve complex-wide integration, and thereby expedite remediation, standardize resolutions, and minimize cost.
- Encourage industry and university participation in identification and resolution of plutonium stabilization issues.
- Establish effective decision-making processes involving stakeholders.
- Manage implementation and updates of the NMSTG Research and Development Plan.

PFA ORGANIZATION

PFA Management Structure

The PFA organizational structure, functional responsibilities, levels of authority and lines of communication are defined to effectively implement the PFA charter. The PFA organization is shown in Figure 2.

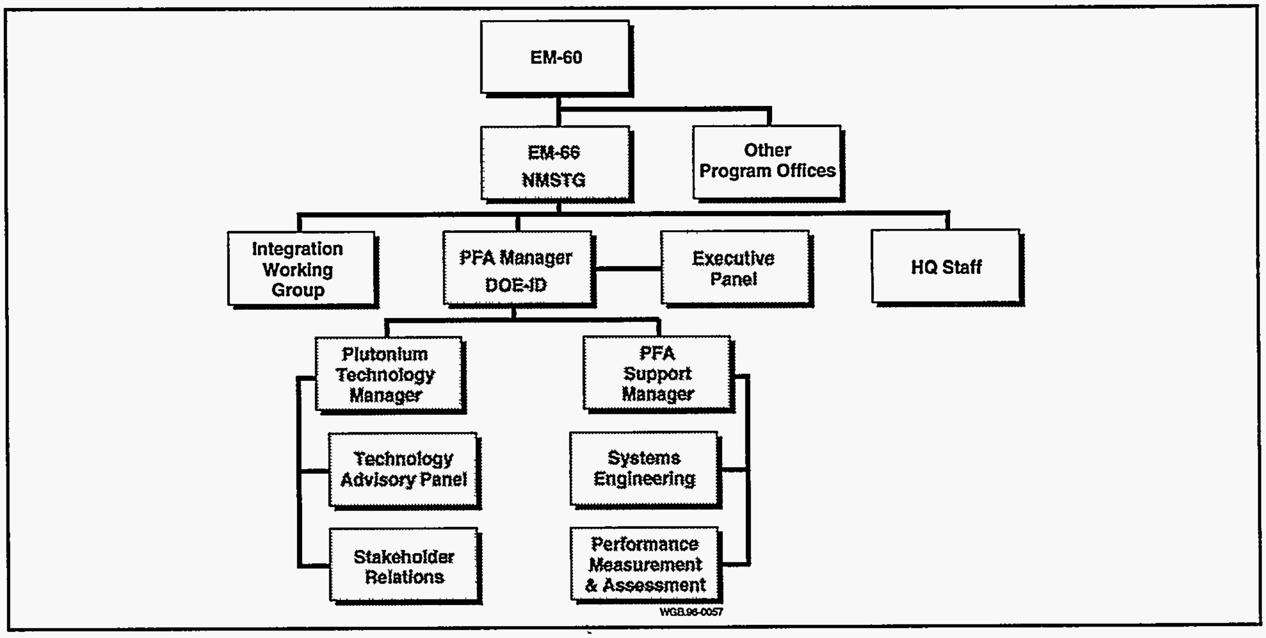

Figure 2. Organization and Interfaces of the Plutonium Focus Area

The PFA Manager is responsible for PFA functions and accountable to the Director of the NMSTG. The PFA Manager has the ultimate responsibility for the quality of the activities of the PFA. The Focus Area is divided into two primary sections, Plutonium Technology and PFA Support. Plutonium Technology is responsible for the PFA technical functions and is comprised of the Technology Advisory Panel and Stakeholder Relations activities. PFA Support is comprised of the Performance Measurement and Analysis, Systems Engineering, and Administrative Support functions.

DOE-ID is responsible for the management and tasking of the PFA, and for providing formal interface with DOE program and field offices. The PFA section managers serve as points of contact, integrate the activities of the groups within the section, and ensure collaboration between the groups and NMSTG management.

Executive Panel:

Members of the PFA's Executive Panel are senior technical and/or programmatic Federal employees. They possess individual and collective expertise with respect to understanding and representing the major plutonium stabilization and management issues of the DOE complex.

The Executive Panel is comprised of representatives from the following organizations:

- Defense Programs (DP)
- Fissile Materials Disposition (MD)
- Albuquerque Operations Office (AL)
- Richland Operations Office (RL)
- Rocky Flats Field Office (RFFO)
- Savannah River Operations Office (SRO)
- Chicago Operations Office (CH)
- PFA Manager (ex-officio)
- Environment Safety & Health (ES&H)

Technical Advisory Panel:

The PFA utilizes a Technical Advisory Panel (TAP) to identify and address technical and operational issues involved in nuclear materials stabilization and management. Although its primary function is to oversee, review, and recommend direction of research activities identified in the Integrated Program Plan, additional activities are also envisioned. Specific activities of the Research Committee identified in the 94-1 Implementation Plan which the TAP assumed include:

- Identification of research and technical requirements for nuclear materials remediation, stabilization, and management
- Evaluation of proposals for addressing these requirements
- Trade Studies and Decision Analysis regarding plutonium stabilization technologies

Principal TAP functions encompass consideration of plutonium stabilization and technical management issues. TAP supports the NMSTG by analyzing stabilization needs and identifying candidate topics for concerted R&D efforts. TAP is also responsible for evaluating the technical progress of NMSTG-sponsored research and development on plutonium stabilization issues. This activity includes review of technical content and approach of research proposals submitted by the Lead Laboratory, input to NMSTG task statements which define research needs to be addressed by the Lead Laboratory, and evaluation of technical progress made by the Lead Laboratory.

TAP is composed of a core group of members with plutonium technical expertise reflecting a wide variety of technical issues and research needs related to plutonium stabilization in preparation for final disposition. The TAP Chairperson was appointed by the PFA Plutonium Technology Manager from among these core members. Areas of plutonium expertise represented in the TAP include chemistry, pyrochemistry, metallurgy, corrosion and pyrophoricity, and process operations. Core members of the TAP represent a cross-section of the DOE complex, including INEL, LANL, RFETS, SRS, LLNL, ORNL, Hanford, and ANL. Ad-hoc members are called upon as needed for specific activities.

The TAP accomplishes its functions and tasks through meetings held at least quarterly. More frequent meetings of selected members of the TAP occur as required to complete specific activities. The meetings provide a forum for resolution of issues and problems and group review of proposals.

Stakeholder Relations:

Successful management of the Plutonium Focus Area requires early and continuous interaction with local, regional, and national stakeholders. Public mistrust places a higher burden of proof on DOE and its contractors and has resulted in increased costs, schedule delays, and lengthy regulatory permitting cycles. Because of reduced funding, aging facilities, and expanding inventories of nuclear materials, stakeholder involvement is encouraged.

A systems approach that considers the total nuclear materials stabilization process, including early and continuous stakeholder and regulatory involvement, is utilized by the PFA. The PFA encourages continued openness with the public regarding plutonium stabilization activities. Additionally, the public is encouraged to participate constructively through existing organizations established at each of the sites. These organizations include Citizens Advisory Boards and other committees that provide the mechanism for public involvement. The PFA established and maintains a working relationship with the appropriate DOE and contractor community relations contacts at each site.

Systems Engineering:

PFA utilizes a Systems Engineering group to perform the following activities:

- Define requirements and flowdown
- Define functional systems
- Develop, analyze, and implement alternatives
- Analyze trade studies using models developed by the NMSTG and other DOE offices
- Evaluate and validate systems performance.

Performance Measurement and Analysis:

Monthly PFA Performance Measurement and Analysis:

PFA's Performance Measurement and Analysis Group provides a comprehensive program for establishing the budget and schedule baselines, measuring performance by comparing actual cost and schedule accomplishments to budget, and monitoring changes to the baseline. The PFA Monthly Performance

Report, identifying monthly progress and status, is sent to NMSTG, members of the PFA Executive Panel, and PFA management by the 15th of each month.

Quality Assurance:

The PFA Quality Program Plan (QPP) established the quality requirements applicable to this Focus Area. The QPP identifies activities important to achieving quality, and establishes management and control systems to assure that focus area objectives meet the objectives of the PFA Management Plan. The PFA Quality Program Plan is developed and implemented by the PFA Support Manager.

PFA Records Management and Document Control:

The objective of PFA records management and document control is to provide the framework and requirements for effective and economic control of project documentation. This documentation can be in any media format (e.g., hardcopy, magnetic, optical, etc.). All records will be legible, traceable, and complete.

PFA MANAGEMENT PLAN

The PFA Management Plan (MP) was prepared under the direction of the PFA Support Manager and covers the entire PFA scope of work. The MP is designed to be a living document, and includes descriptions of the Work Breakdown Structure for each activity within the PFA.

The MP is the guidance document for PFA performance measurement and analysis as agreed to by the NMSTG Director and PFA Manager. Detailed project performance reporting is provided on the following key PFA activities:

- Technical Advisory Panel
- Stakeholder Relations
- Executive Panel
- Systems Engineering

To implement planning, measurement, and analysis functions, the PFA utilizes established EM and LMIT reporting processes. A computerized project management system is used to manage data rollup and distributions from the Summary Cost Account, Cost Account, and, ultimately, Work Packages levels.

PFA PRODUCT LINE OVERVIEW

INTRODUCTION

The primary focus of Revision 0 to this PFA Technology Summary is to introduce the DOE audience to the background, charter, structure, and operations of the Focus Area. This introduction presents methods being utilized by the Technical Advisory Panel to identify and prioritize research needs in the following categories:

- Standards Development
- Stabilization Process Development
- Transportation
- Packaging
- Surveillance
- Core Technologies

As a followup to the introduction, sample technology summaries are presented which provide an overview of tasks being performed. A complete summary of current and planned tasks will be presented in Revision 1 to the PFA Technology Summary, due to be published in the fourth quarter of FY96.

INTEGRATED SURVEILLANCE SYSTEM FOR MATERIAL IN THE PLUTONIUM FOCUS AREA

TASK DESCRIPTION

The objective of this task is to develop a set of requirements for an integrated surveillance system for materials in the PFA. For the purpose of this effort, "surveillance" is any monitoring of material after stabilization and containment, and during interim and long-term disposition. This involves the traditional safeguards effort to manage and maintain accountability of the materials as well as the needs to characterize, identify, and verify the stabilized material and container conditions. Major functions of the system are characterizing stabilized materials after containerization; surveillance of materials in the containers; monitoring the containers and contents during disposition; and tracking containers and contents. An integrated data acquisition, storage, and retrieval system will be developed for use during the life of this material.

TECHNOLOGY NEED

The NMSTG R&D Plan is being implemented by the PFA to survey the entire DOE complex and identify research needs. An additional task was to identify any "gaps" or deficiencies in research to support the needs of NMSTG.

The R&D Plan stated that "..no systematic overall approach has been developed to monitor plutonium materials ....". Three areas of concern related to surveillance and data acquisition were identified below:

Table 2. Surveillance and Data Acquisition Concerns

Recommendations	Comments
Develop analytical methods for determining moisture content, gas composition from radiolysis, and reactive metals present in pyrochemical salts	A process will be required to characterize moisture and reactive metals in salts treated for stabilization and to analyze the effects of radiolysis on stored salts.
Develop a surveillance system for monitoring Am/Cm and Pu-238	Because of the high radiation levels for Am/Cm and the high heat generation rate for Pu-238, additional surveillance measures will be needed for storing materials.
Develop an integrated approach for the disposition and surveillance of plutonium packages using non-intrusive technologies that minimize personnel exposure and maximize safeguards and security	Although surveillance procedures are being developed for specific items, no systematic overall approach has been developed to monitor plutonium materials during disposition. Non-invasive surveillance should be emphasized, minimizing the need for labor-intensive activities.

ACCOMPLISHMENTS

A white paper has been submitted to the PFA TAP identifying the requirements for an Integrated Surveillance System. 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
- Produce System Specification

Figure 3 below is a depiction of the functions required for an integrated approach for the characterization, disposition and surveillance of stabilized plutonium packages after containerization, using non-intrusive technologies that minimize personnel exposures and maximize safeguards and security.

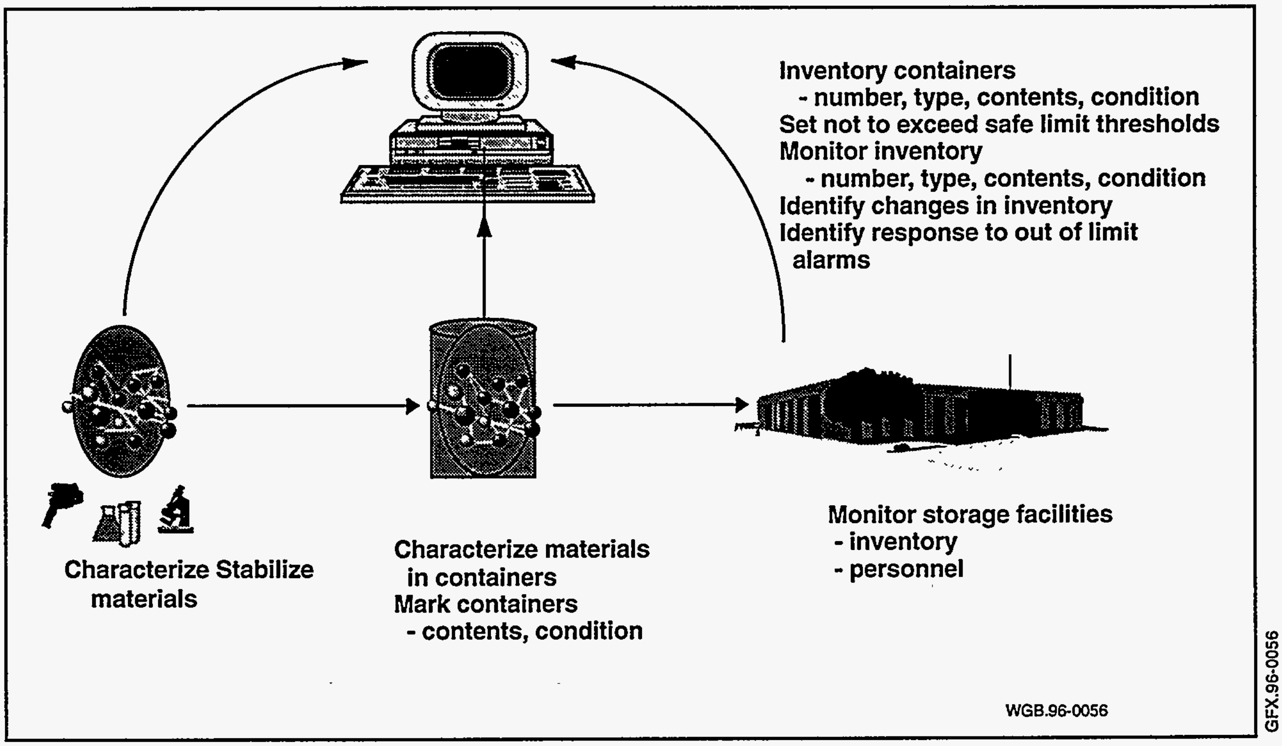

Figure 3. Integrated System for the Monitoring of Stabilized Plutonium Packages after Containerization

A systems engineering approach has been selected to determine the proper balance of requirements for the integrated system. The system engineering process starts with an identification of need, which has been performed and documented in the R&D Plan.

BENEFITS

There are numerous benefits for an integrated surveillance system approach for the characterization, disposition, and surveillance of plutonium packages, using non-intrusive technologies that minimize personnel exposure and maximize safeguards and security. Recognizing the PFA effort is complex wide, the ability to work with a consistent set of requirements and functions will provide the following benefits:

- Identify duplicity and reduce program cost by eliminating these duplications
- Raise public confidence that a consistent and verifiable method of characterizing materials and tracking is being implemented across the complex
- Provide data acquisition, storage, and retrieval for analysis
- Provide a records system that will meet regulatory and quality control requirements for tracking material throughout its lifecycle.
- Provide inventory control and tracking of material within the complex.
- Provide a growth path for upgrading the system as technology improves and increases.

COLLABORATION/TECHNOLOGY TRANSFER

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

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BIBLIOGRAPHY OF KEY PUBLICATIONS

None at this time.

SHELF LIFE STUDIES

TASK DESCRIPTION

Both treated and untreated plutonium metal, oxides, and residues are placed in experimental containers for pressure, volume, and temperature (PVT) measurements over time. Shelf life studies include compatibility testing of plutonium oxides and residues with container materials. A test matrix will be developed from DOE complex requirements and experience. The project supports DOE-STD-3013-94 standard for oxide and development of interim storage standards.

TECHNOLOGY NEEDS

There is a need to understand fundamental and potential problems for the safe storage of plutonium metal, oxides, and residues. The scientific community strives to enhance its basic understanding of stored metal, oxide, and residues over time, including gas-phase generation/recombination, equilibrium effects, corrosion of materials and containment, and thermodynamic and kinetic behavior; close gaps in the present knowledge of water-dependent salt corrosion as a function of concentration, and the effect of radiolysis within salts; develop non-invasive, non-waste producing surveillance techniques for plutonium-containing materials in storage; and develop corrosion-resistant shipable containers and weld closures for use with existing or stabilized residues.

ACCOMPLISHMENTS

Metal A basis for continuing surveillance of metal-filled containers has been established within the Los Alamos Plutonium Packaging project. Each double-welded long-term storage container holds slightly more than 4 kg metal. Approximately one out of five metal-filled packages contains aneroid bellows, which will indicate pressure changes. As part of the Packaging project surveillance program, the boundary containers will be periodically leak-checked, and the total containment system will be weighed.

Oxides and Oxide-Like Materials/Residues

Shelf Life Project--Container Prototype I. The first set of four shelf-life tests (Container prototype I) was completed. In this preliminary set of experiments, PVT studies of residues were conducted on samples of electrorefining salt, combustibles, oxidized electrorefining salt, and incinerated 238Pu-contaminated rags. No significant changes within the four containers were detected in a six-month period. The experimental set-up was not adequate for precise data acquisition over an extended period and the experiments were terminated.

Shelf Life Experiments--Container Prototype II and III. The oxide/ash studies will proceed with a new test set-up. A preliminary test plan has been drafted and is being used in establishing, with representatives from other sites, an experimental matrix to satisfy DOE requirements. For oxide and ash, the design for a container has been approved. A sampling manifold and data acquisition system are available. Experimental goals are well developed. Samples of oxides of various purities and ash must be selected and characterized. Experiments are to be initiated by the end of the second quarter of FY96.

Goals for the experimentation on salts must be developed and candidate materials identified. For salts, a manifold has been designed for installation into an existing glovebox. Containers have been designed and fabricated and await instrumentation. Hardware is to be delivered this quarter. A data acquisition system is available.

Advanced Technologies Testing is proceeding on bellows for detection of pressure changes in long-term storage containers. Sandia-Albuquerque has completed the initial scoping and feasibility study for an electronic hydrogen sensor to be placed within a storage container. Real-time radiography equipment purchased by the Plutonium Packaging project will be available for surveillance studies by the shelf life project beginning in the third quarter of FY96. Laser sampling of gases in storage containers has been found to be feasible. A report evaluating the generation of an explosive mixture in plutonium oxide storage containers is complete; for the container configuration analyzed, calculations show that the container would remain intact during laser sampling under worst-case conditions. Acoustic resonance spectroscopy has been found to be a feasible method for detecting changes in storage containers and warrants further study. A search of the literature reveals a high probability that neutron flux differences in several plutonium compounds can be determined. Investigators are developing Raman and laser-induced-breakdown spectroscopy for use in storage surveillance. Sandia-Livermore is completing conceptual designs for a Pu-238 long-term storage container.

BENEFITS

Technical issues associated with interim storage of plutonium-bearing materials are being identified. Enhancement of surveillance techniques has been demonstrated and development continues. Materials that must be processed to remove the plutonium for safe storage will be identified. Deleterious effects of water will be further defined. Eliminating pressure build-up within containers and corrosion problems will vastly improve the safe interim storage of plutonium-bearing materials.

COLLABORATION/TECHNOLOGY TRANSFER

The shelf life project has set meetings with Rocky Flats Environmental Technology Site (RFETS) and Savannah River Site to obtain their input to the experimental matrix. Los Alamos collaborated on digital radiography and laser sampling with Savannah River, and these interactions will continue. The development of electronic pressure sensitive devices is conducted by Sandia-Albuquerque in cooperation with the shelf life project, and Sandia-Livermore has developed conceptual designs for Pu-238 long-term storage. The bellows development will continue in close coordination with the vendor, Miniflex Corporation of Ventura, California.

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BIBLIOGRAPHY OF KEY PUBLICATIONS

Haschke, J. M. and T. E. Ricketts, *Plutonium Dioxide Storage: Conditions for Preparation and Handling*, report number LA-12999-MS.

CRYOGENIC CRUSHING

TASK DESCRIPTION

The task is to develop an in-line cryogenic filter grinder assembly, a cryogenic crusher for polystyrene cube reduction, and a cryogenic shredder for soft combustibles that will provide feed for further processing. The task includes making polycubes for testing and evaluating size reduction and mixing of combustible materials.

TECHNOLOGY NEEDS

Many combustible residue materials require size reduction before processing to destroy the organic matrix. Among these are contaminated filter units and 1600 polystyrene cubes at Hanford. Technology needs are to understand polystyrene cube pyrolysis and develop a method to produce suitable feed materials for various combustible processing.

ACCOMPLISHMENTS

Assembly of the prototype in-line filter grinder assembly is 90% complete. The assembly is an upgraded concept of a commercial ice shaver. A cold demonstration was performed to determine the particle sizes that can be generated using the unit. A manual method for feeding the filters is being incorporated on the unit to expedite the test for particle size determination. A motor-driven concept is under consideration and may be incorporated at a later date.

Work on the cryogenic crusher for the polycubes also continued during the second quarter of FY96. The unit is about 50% complete. The design requires that a polycube be reduced in size from a 2-in. cube to particles that have a major diameter less than 0.040 in. Processing will be performed with the polycube chilled in liquid nitrogen prior to crushing and fragmentation attempts. The crusher will also be cooled.

Polystyrene test cubes were formed from mixtures of iron particles and polystyrene powder. The need to evaluate chemical changes caused by heat required the use of polystyrene cube composites, as well as cubes of polystyrene. An effort was initiated to form billets of styrene, machine them into cubes, and subject the cubes to pyrolysis. Four cubes were fabricated and delivered to the chemist for processing.

An effort was initiated to fabricate approximately 60 cubes of styrene for future pyrolysis tests. Thirty composite styrene and iron polycubes will also be required for the pyrolysis and the cube crushing efforts. Procurement of these materials was initiated.

BENEFITS

Stabilization of most combustible matrices will require size reduction. Regardless of whether organic matrix destruction, actinide removal, or nitrate removal treatment options are selected, each requires some degree of size reduction to facilitate stabilization. Coupling cryogens that embrittle combustible materials with commercial size reduction equipment produces a feed material that can be effectively processed to a stabilized form. Other benefits include volume reduction and an improved safety envelope for the operator. Utilizing this technology will remove the operator from the glovebox and eliminate potential injuries and contaminations from conventional size reduction techniques such as hacksaws and knives.

COLLABORATION/TECHNOLOGY TRANSFER

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

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BIBLIOGRAPHY OF KEY PUBLICATIONS

McFarlan, James T. and Timothy O. Nelson, "Cryogenic Liquid Introduction System for Gloveboxes," presentation to the 9th Annual American Glovebox Society Conference, Unclassified release number LA-UR-95-2047 (July 19-22, 1995).

RESEARCH AND DEVELOPMENT REQUIREMENTS "GAP" ANALYSIS

TASK DESCRIPTION

As part of PFA's responsibilities to implement the R&D plan and use a systems engineering approach, the PFA has begun an analysis and definition of requirements for research. These requirements will be used to assess the need for proposed research and evaluate progress of on-going research. The requirements will include verification criteria allowing measurement of the level of work completion. The definition of requirements is focused on collection and correlation of sets of requirements from three sources: the NMSTG 94-1 Implementation Plan; the 94-1 Research and Technology Development Plan; and the on-going research currently planned or in-place at DOE sites.

Correlation of these three sets of research 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 Implementation Plan but not identified in the R&D Plan; that called for by the 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 4 below, in a single list of research requirements called the Baseline Research and Development Technical Requirements Document, which will become the baseline specification for research to be done.

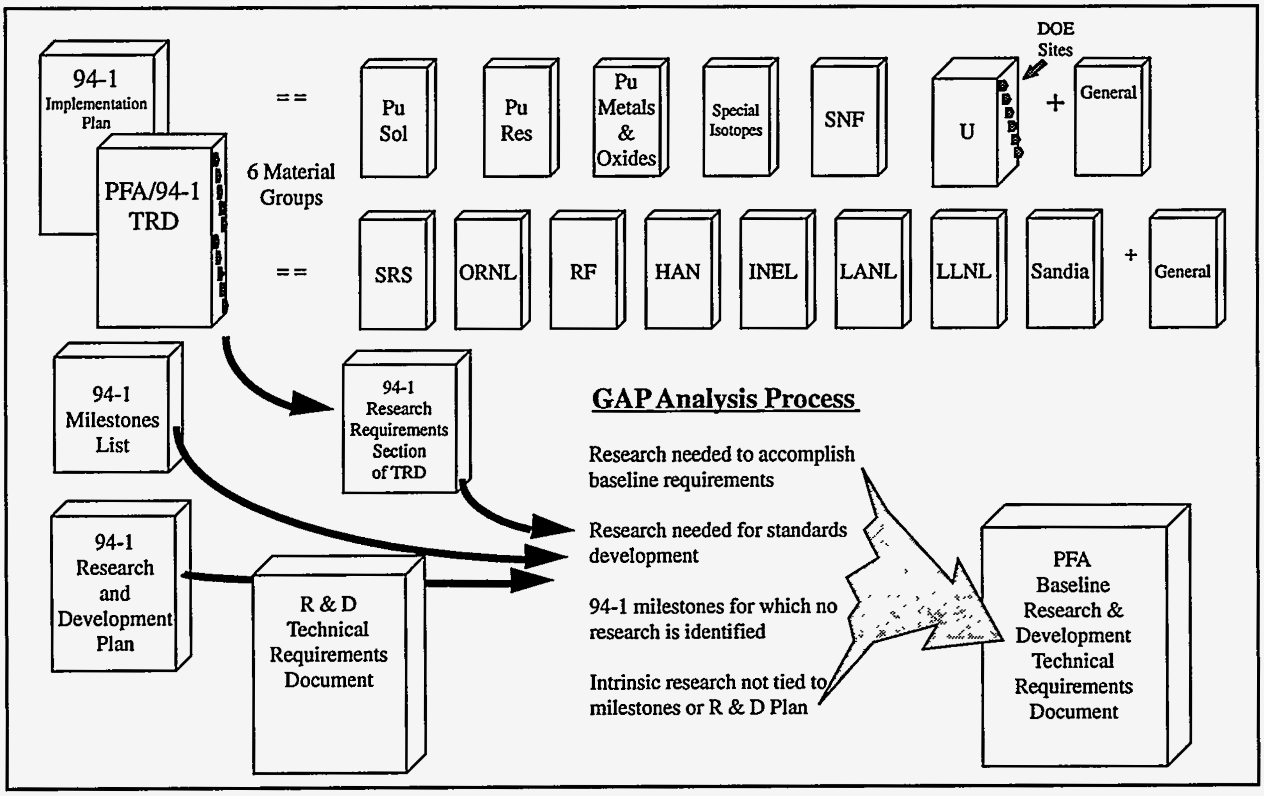

Figure 4. Gap Analysis Process

Accompanying every requirement will be verification criteria establishing the conditions against which successful accomplishment of the research will be assessed. These verification criteria will be measurable, as a scalar quantity, and determined by test, measurement, precedence, audit, inspection or simulation methods.

TECHNOLOGY NEEDS

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

ACCOMPLISHMENTS

In support of gaps definition, 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 Technical Requirements Documents (TRDs).

The first of these, the 94-1/PFA TRD, has been published. It includes a section of only Research and Technology Development Requirements and is separately published as a TRD. Its sister documents list all the Implementation Plan requirements in categories by material type, site, milestone, requirement-type and TRD paragraph number.

The second of these, the R&D Plan TRD, has also been published from a similar analysis of the Research and Technology Development Plan. It lists in "shopping list" format every requirement DOE must meet in doing PFA R&D work, based on the R&D Plan.

A cross check of requirements from the R&D Plan's chapters 4 & 5 has resulted in a gap analysis showing where research requirements are not being met by ongoing technology. A complementary analysis is underway to see how standards for stabilization requirements in chapter 3 compare to R&D requirements in chapter 4. This will expose whether any standards need development or whether research requirements need further analysis to align with needs for standards development.

BENEFITS

The value of the gap analysis is to focus effort and funding on just 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 and to be sure none are missed.

The verification criteria accompanying requirements save time and effort by helping direct technology to specific remediation goals. Having the verification criteria defined enables researchers to apply the right amount of effort, making it is possible to determine percent completion, performance measures, and how to intensify efforts, if needed. It will benefit NMSTG by allowing members of the TAP to uniformly and fairly assess research performance and to rank new research.

Collaboration/Technology Transfer

The gap analysis will provide a baseline for the technology 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.

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Bibliography of Key Publications

Schilling, D. and D. Johnson. *94-1/PFA Technical Requirements Document*, AEDL-PFA-TM-143 (February 1996).

Schilling, D. and H. Heydt. *R&TD Plan TRD*, AEDL-PFA-TM-144 (February 1996).

U.S. Department of Energy. *Nuclear Materialization Stabilization Task Group Research Committee, Research and Development Plan* (November 1995).

TASK 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 3013-94 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 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 5 below, 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 salts 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 number of drums sent to the Waste Isolation Pilot Plant (WIPP).

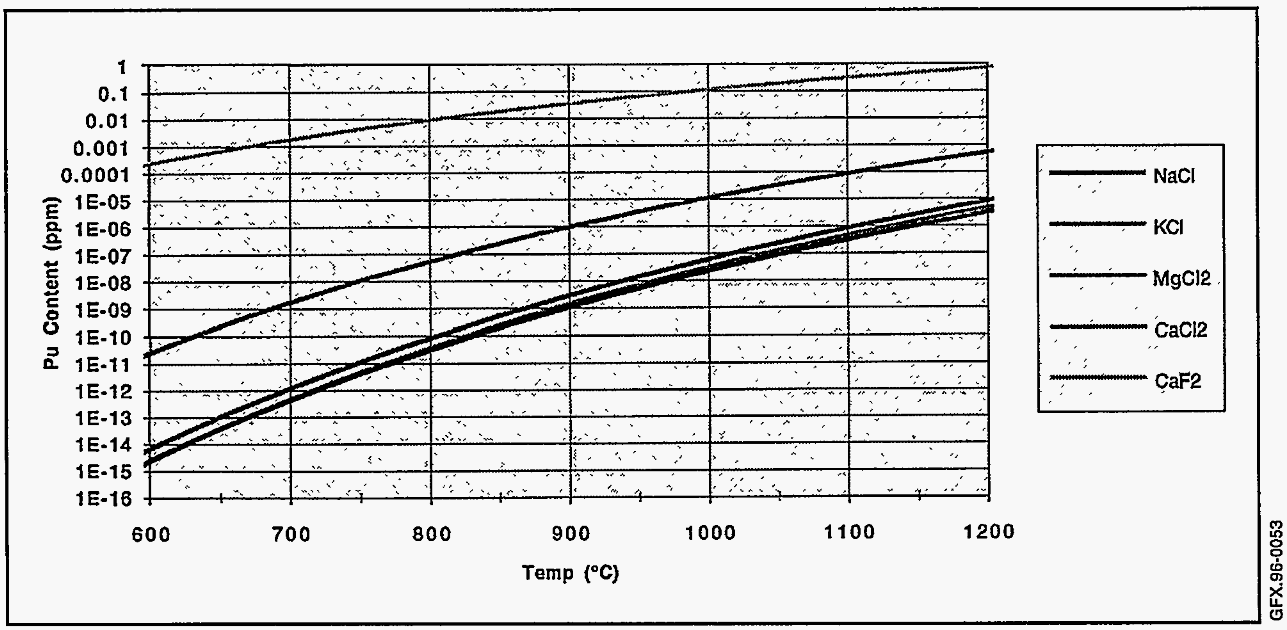

Figure 5. Results of Modeling of Plutonium Concentration in Distilled Salts

TECHNOLOGY NEEDS

The 16 metric tons (MT) of salts at Rocky Flats contain about 1 metric ton of plutonium. Eleven tons 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 Rocky Flats 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.

ACCOMPLISHMENTS

All electrical, thermocouple, and vacuum feedthroughs and fittings needed for glovebox installation were installed. A new cover incorporating the appropriate vacuum feed-through 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. 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 attainable. Distillation rates for calcium chloride were found to be too slow below 1100°C. Calculated distillation rates are shown in Figure 6. 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.

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 demonstration full-scale processing run will be carried out to establish reliability and robustness of the process and equipment in a full-scale processing environment.

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

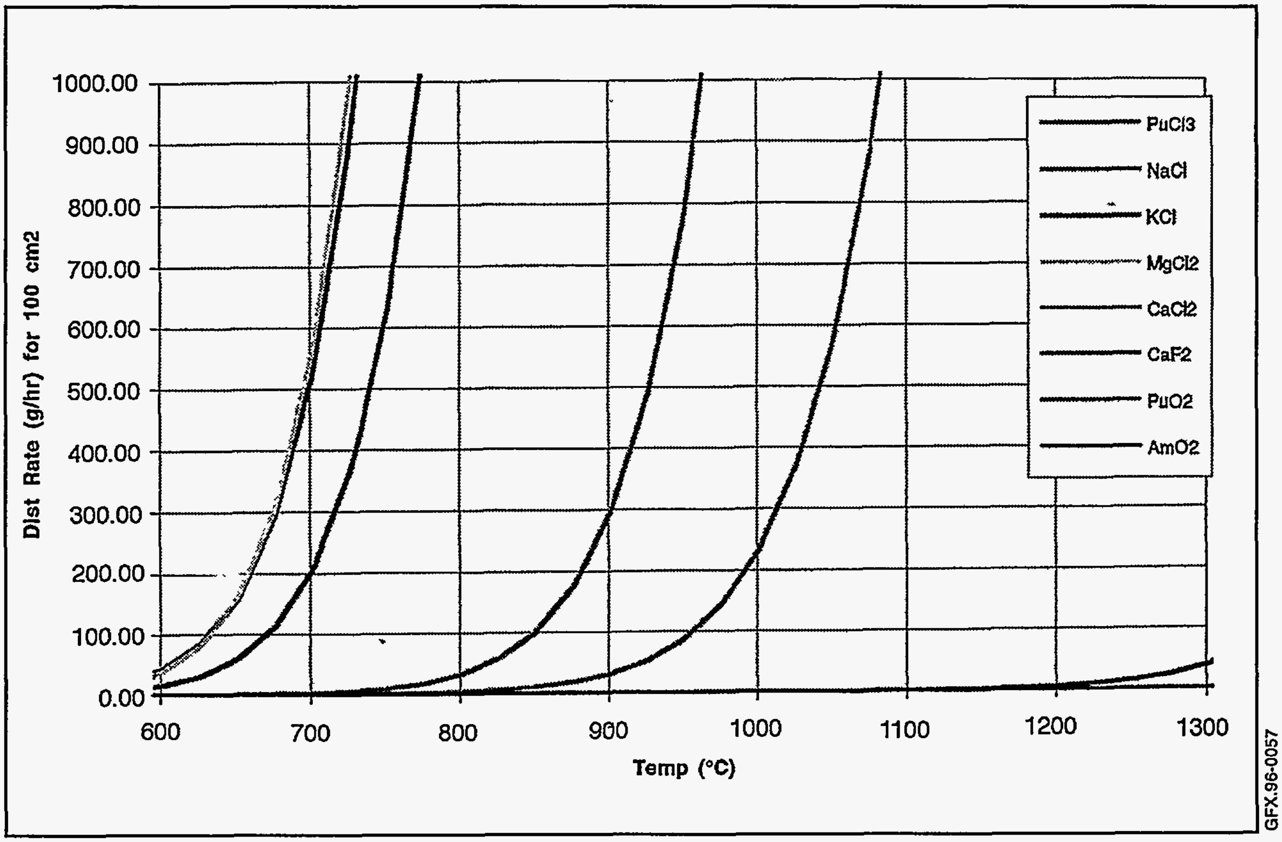

Figure 6. Calculated Distillation Rates for Various Chloride Salts

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 low-level waste disposal criteria. Present WIPP Waste Acceptance Criteria (WAC) would result in a maximum plutonium loading per 55-gal drum of 23 g. In the best possible circumstance, this would lead to over 50,000 drums. Efforts are underway to modify the WIPP WAC to allow 200 g of plutonium per drum. 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 metric ton of plutonium dioxide, could be packaged per DOE-STD-3013-94. Costs incurred by storage of the plutonium oxide would be significantly offset by savings realized from WIPP disposal. Recent estimates of total cost to process the salt inventory at Rocky Flats by distillation are $71 million, compared to $103 million for disposal at WIPP with modified WIPP WAC, and $534 million with present WIPP WAC.

Collaboration/Technology Transfer

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

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Bibliography of Key Publications

Garcia, E., "Vacuum Distillation Separation of Plutonium Waste Salts," *Annual Progress Report to DOE/ EM-50* (June 1994).

Garcia, E., "Vacuum Distillation Separation of Plutonium Waste Salts," *Annual Progress Report to DOE/ EM-50* (June 1995).

ELECTROLYTIC DECONTAMINATION

TASK DESCRIPTION

The task is 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 Rocky Flats, and it has been successfully demonstrated for this application. 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.

TECHNOLOGY NEEDS

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

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. 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 and no swipeable.

After cleaning, these containers were helium leak-tested to 1×10^{-7} std cm^3/s as required by DOE-STD-3013-94, 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 currently used.

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.

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Bibliography of Key Publications

Wedman, Douglas E., Horacio E. Martinez, Timothy O. Nelson, "Electrolytic Decontamination of Stainless Steel Materials in a Sodium Nitrate Electrolyte for Hazardous Waste Management," presentation for Waste Management 95, unclassified release number 95-3241 (February 25-29, 1996).

Nelson, Timothy O., "Electrolytic Decontamination of Conductive Materials for Hazardous Waste Management," presentation to the American Chemical Society, IE&C Division, Seventh Annual Symposium on Emerging Technologies for Waste Management, unclassified release number 95-2474 (September 17-20, 1995).

Glass Material Oxidation And Dissolution System (GMODS)

Task Description

The Glass Material Oxidation and Dissolution System (GMODS) is a general-purpose waste-treatment process to convert ceramics, metals, organics, and amorphous solids to borosilicate glass; oxidize organics with the residue converted to borosilicate glass; and convert chlorides to low-chloride containing borosilicate glass and a secondary clean sodium chloride stream. It is being developed to process those plutonium-containing wastes that cannot be easily and economically processed by other technologies into a high-quality waste glass.

GMODS processes plutonium-containing materials to glass inside a glass melter. The process can operate as a batch (See Figure 7) or continuous process. In 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 metals to metal oxides and organics to carbon oxides. The resultant metal oxides dissolve into the glass, and the carbon oxides exit the melter as gases. The lead metal (Pb) reaction product separates from the glass and forms a separate layer at the bottom of the melter.

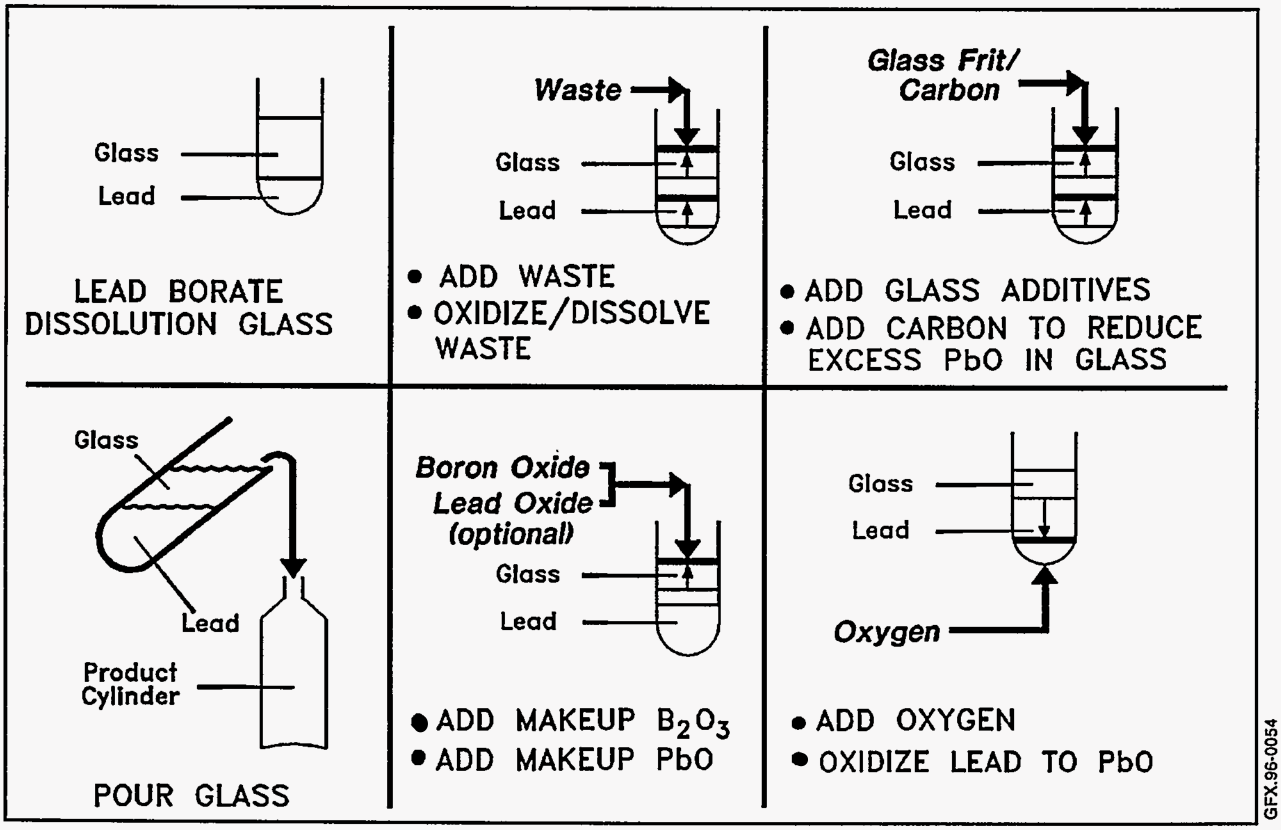

Figure 7. GMODS Batch Processing of Waste to Borosilicate Glass

After dissolution of the wastes, silicon oxide and other glass additives are added to the glass to produce a high-quality product glass. Excess lead oxide is removed from the glass by addition of carbon that converts the lead oxide to lead metal and carbon dioxide. The final glass may have some or no lead oxide depending upon the desired product glass. The product glass is poured 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 with oxygen.

TECHNOLOGY NEED

GMODS addresses the technological need for processes to convert complex waste mixtures containing ceramics, metals, organics, chlorides and amorphous solids with ill-defined chemical compositions into homogeneous high-quality waste forms. There are several processes (plasma torch, etc.) that can convert complex mixtures into semi-homogeneous waste forms; there are many processes (conventional vitrification, synrock processes) that can convert homogenous wastes into high quality waste forms; but no current processes accept almost all wastes and produce a high-quality waste product.

Plutonium containing wastes are both complex and highly hazardous. This creates the need for general purpose processes to convert these wastes into high quality waste forms for disposal.

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.
- Conversion to glass has been demonstrated on a laboratory scale (100-200 g) with a variety of feed materials including: aluminum, zircaloy, stainless steel, carbon, uranium, cerium, aluminum oxide, uranium oxide, cerium oxide, etc. 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.

BENEFITS

Glass is recognized worldwide as a preferred waste form for radioactive and chemically hazardous wastes. However, a major limitation is that 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 direct conversion of oxides, metals, ceramics, organics, chlorides, and amorphous solids to glass. This allows complex waste mixtures (filters, process wastes, laboratory wastes, etc.) to be directly processed to glass.

Collaboration/Technology Transfer

Discussions are underway for industrial cooperation in development of the process.

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Bibliography of Key Publications

Forsberg, C.W, E. C. Beahm, G. W. Parker, J. Rudolph, P. Haas, G. F. Malling, K. R. Elam, L. Ott. *Direct Vitrification of Plutonium-Containing Materials With The Glass Material Oxidation and Dissolution System* (GMODS), ORNL-6825 (October 1995).

Forsberg, C.W., E. C. Beahm, G. W. Parker, J. Rudolph, K. R. Elam, and J. J. Ferrada. "Conversion of Plutonium Scrap and Residue to Borosilicate Glass Using the GMODS Process," *U. S. Department of Energy Stabilization and Immobilization Plutonium Workshop, Wash. DC,* (December 12-14, 1995).

Forsberg, C.W., E. C. Beahm, and G. W. Parker. "Direct Conversion of Plutonium Metal, Scrap, Residue, and Transuranic Waste To Glass," *Proceedings of Waste Management '95, Tucson, Arizona* (March 1, 1995).

TASK DESCRIPTION

The purpose of this task is to design, build, and test a hydrothermal processing unit for the destruction of plutonium-contaminated combustible solid and liquid wastes.

Oxidation of the organic and reduction of the nitrate components of combustible waste will mitigate safety hazards, reduce waste volume, and facilitate separation of radioactive elements. Hydrothermal processing (reactions in hot water) provides high destruction and removal efficiencies for a wide variety of organic and hazardous substances. For aqueous/organic mixtures, pure organic liquids, or contaminated combustible solids (e.g., ion exchange resins, plastic filters, and cellulose rags) hydrothermal processing removes most of the organic and nitrate components (>99.999%) and facilitates the collection and separation of the actinides. A schematic of a laboratory scale process unit is shown below in Figure 8.

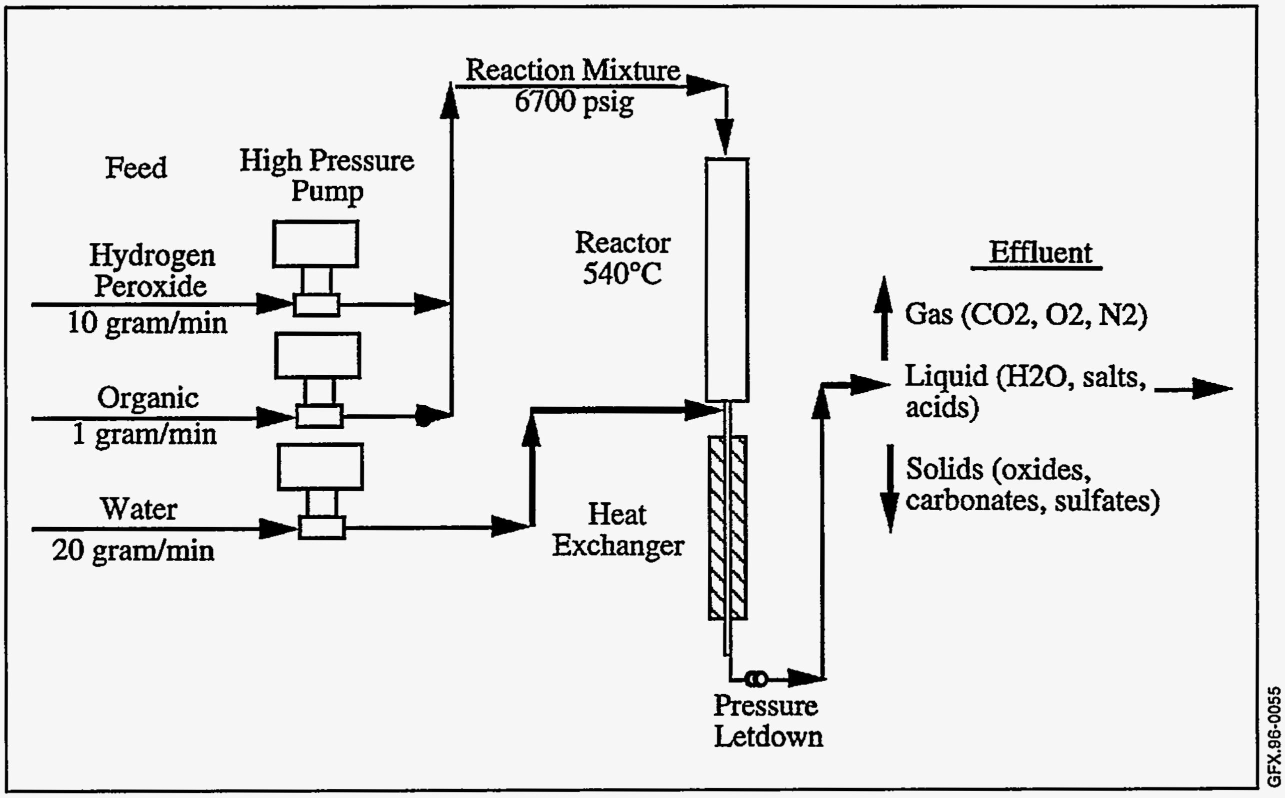

Figure 8. Schematic of Laboratory-Scale Process Unit

Organic material is pressurized and mixed with pressurized hydrogen peroxide (30 wt.%). For pumping solids, the solids are first reduced in size by cryogenic grinding, then mixed with water. The reaction mixture is fed into a high-temperature, high-pressure reactor and allowed to react for 20 to 60 seconds. At the end of the reactor, cold water is added to help cool the mixture and to facilitate transport of any insoluble solid material that may have been formed in the reactor. The mixture is further cooled in a heat exchanger and then depressurized. In the reactor, the organic components of the wastes are oxidized to carbon dioxide by reaction with the water and hydrogen peroxide. Nitrate contaminants also react with the organic material and are converted to nitrogen gas and some nitrous oxide. Heteroatoms such as chlorine, sulfur, and phosphorus are oxidized and converted to acids or salts depending on the pH of the solution. At temperatures above 500°C, reactions are rapid, and greater than 99% conversion can be achieved in seconds. The reactions are carried out entirely in an enclosed pressure vessel and in dilute concentration so that the heat of reaction is absorbed by the water and the temperature can be maintained at any desired level, typically in the range 400 to 550°C. The reactor is fitted with a titanium liner to protect the pressure vessel from corrosion. The speciation of the actinides in the hydrothermal reactor is not yet certain; they most likely will be converted to either small insoluble oxide particles that can be separated by filtration or to water soluble carbonate salts. Since the reactions are rapid, the volume of the reactor is small (200 ml). Consequently, the amount of radioactive material and stored energy are small.

Implementation of testing of hydrothermal processing of TRU-contaminated combustibles will be done in four steps:

- Design and testing of small scale unit (2 grams/min) using nonradioactive simulants
- Design and testing of small scale unit with TRU-contaminated material
- Design and testing of full scale unit (20 grams/min) using nonradioactive simulants
- Design and testing of full scale unit with TRU-contaminated material

At each step, the reactor design will be analyzed and optimized for safety and operability; the reaction kinetics and products will be measured over a range of temperatures, pressures, and residence times; a reactor model will be developed and validated; and the corrosion and wear of reactor components will be evaluated. The effluent streams will analyzed to determine the partitioning of the radionuclides between the solid and liquid phases and the amount of incomplete oxidation products such as carbon monoxide, methane, and hydrogen in the gas phase.

TECHNOLOGY NEEDS

Operations at DOE facilities have created a large legacy of combustible wastes such as rags, plastics, polystyrene cubes, and organic solvents that are contaminated with transuranic materials, other radioactive elements, and strong oxidizers such as nitrates. In some cases, these wastes are an acute safety hazard because of the production of flammable gases from organic decomposition initiated by radioactive decay. Technologies are needed that can mitigate the hazards associated with these wastes. These technologies need to be robust, able to treat a wide variety of waste matrices, produce a minimum amount of secondary wastes, and if possible, compact enough to fit into existing facilities without major modifications.

ACCOMPLISHMENTS

During the final quarter of FY95, hydrothermal processing of a variety of non-radioactive waste simulants was tested. A summary of these tests and results are set out in Table 3. Hydrothermal processing destroyed all organics tested to below detection limits (1 to 5 ppm). Using the results from the simulant experiments, a small-scale hydrothermal unit for the treatment of TRU-contaminated combustible material was designed for glovebox installation in the Plutonium Facility. Components for the reactor were received and assembled, and cold testing is underway. A glovebox is being prepared, and required safety documentation is on schedule. Installation of the reactor in the glovebox is scheduled for the end of March 1996.

Table 3: Summary Waste Treatment Studies

Waste Type	Composition
Hydraulic jack oil	98% hydrocarbons, zinc, sulfur-related compounds, no silicone
Vacuum pump oil	Olefin: $(CH_2)_n$ where 20<n<40
Heavy mineral oil	Paraffin C_nH_{2n+2} where n~34
Tributyl phosphate	$(C_4H_9O)_3PO$
Diesel oil #2	Hydrocarbons: C_{15} to C_{25}
Toluene	C_7H_8
Carbon tetrachloride	CCl_4
Trichloroacetic acid	CCl_3COOH
Trichloroethylene	C_2Cl_3H
1,1,1 Trichloroethane	CCl_3CH_3,
Cation exchange resin	$(C_8H_8SO_3)_n$, 50 - 100 mesh, 50 wt.% water

BENEFITS

This project will provide information on effectiveness, operability, and cost needed to evaluate hydrothermal processing as a technology for the treatment of TRU-contaminated combustible organics. If the technology is successful, the project will produce an operating and tested full-scale treatment unit. This unit will be able to treat approximately 15,000 kg of residue per year.

COLLABORATION/TECHNOLOGY TRANSFER

In addition to building and testing hydrothermal units for TRU contaminated combustible wastes, Los Alamos is building and testing a hydrothermal unit for the treatment of low-level mixed wastes. The laboratory is also collaborating with a consortium led by General Atomics Corporation to build a skid mounted unit for the treatment of U.S. Navy shipboard wastes, and is negotiating a CRADA with an environmental company to develop hydrothermal processing technology for the treatment of industrial wastes. The size of the hydrothermal units for these projects range from 60,000 to 1,000,000 kg of organic waste per year.

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BIBLIOGRAPHY OF KEY PUBLICATIONS

Buelow, S., "Destruction of Hazardous Wastes by Supercritical Water," *Conference Proceedings: Technology for the Nineties, 2nd Annual Symposium, Vanderbilt University* (February 19-21, 1992).

Buelow, S., D. Allen, and G. K. Anderson, "Final Report on the Oxidation of Energetic Materials" *Final Air Force Report*, report OSTI DE95010888, NTIS (1995).

TASK 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 had all of the equipment necessary for completing this task in-place. The Plutonium Stabilization and Packaging project (PuSAP) was initiated by the NMSTG for the purpose of scoping, specifying, and conducting a procurement of a standardized set of packaging and stabilization equipment to be installed at each of the DOE's plutonium sites. Although this effort was initiated and completed by the NMSTG, the PFA contributed necessary technical and systems engineering support.

TECHNOLOGY NEED

The PuSAP was driven by the need for a complex-wide 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 meet the needs of each site. This will provide necessary standardization of stabilized materials and reduce 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. The equipment will be operable under glovebox conditions, automated to reduce exposure, and stabilize and package material to acceptable standards, which will increase safety.

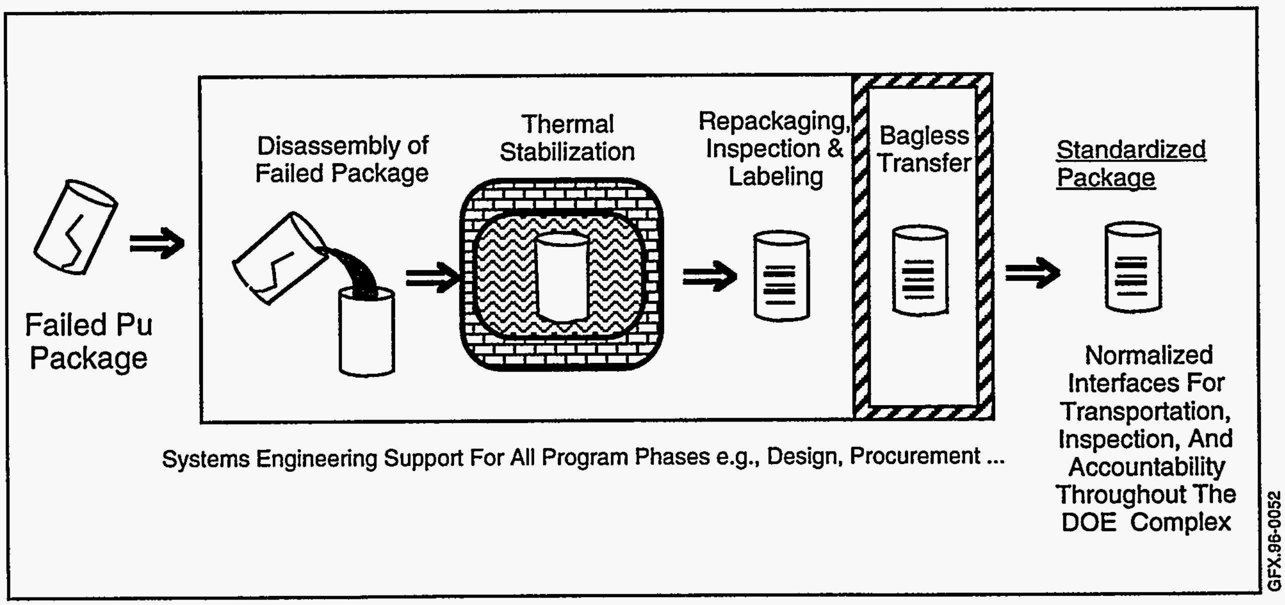

Figure 9. Functional Flow Diagram for Plutonium Stabilization and Packaging

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

Benefits

Utilization of a standardized specification for procurement complex-wide will dramatically reduce total life-cycle costs. These savings will be available for investment in expanded R&D scope.

Collaboration/Technology Transfer

A complex-wide task team with membership from SRS, RFETS, LANL, Hanford, and 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.

For more information, please contact:

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Bibliography of Key Publications

None at this time.

Ceramification for Stabilization and Immobilization of Plutonium Containing Compounds

Technology Description

Ceramification is a new plutonium stabilization/immobilization process under development at Rocky Flats. 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 10 provides a simple process flow diagram for ceramification.

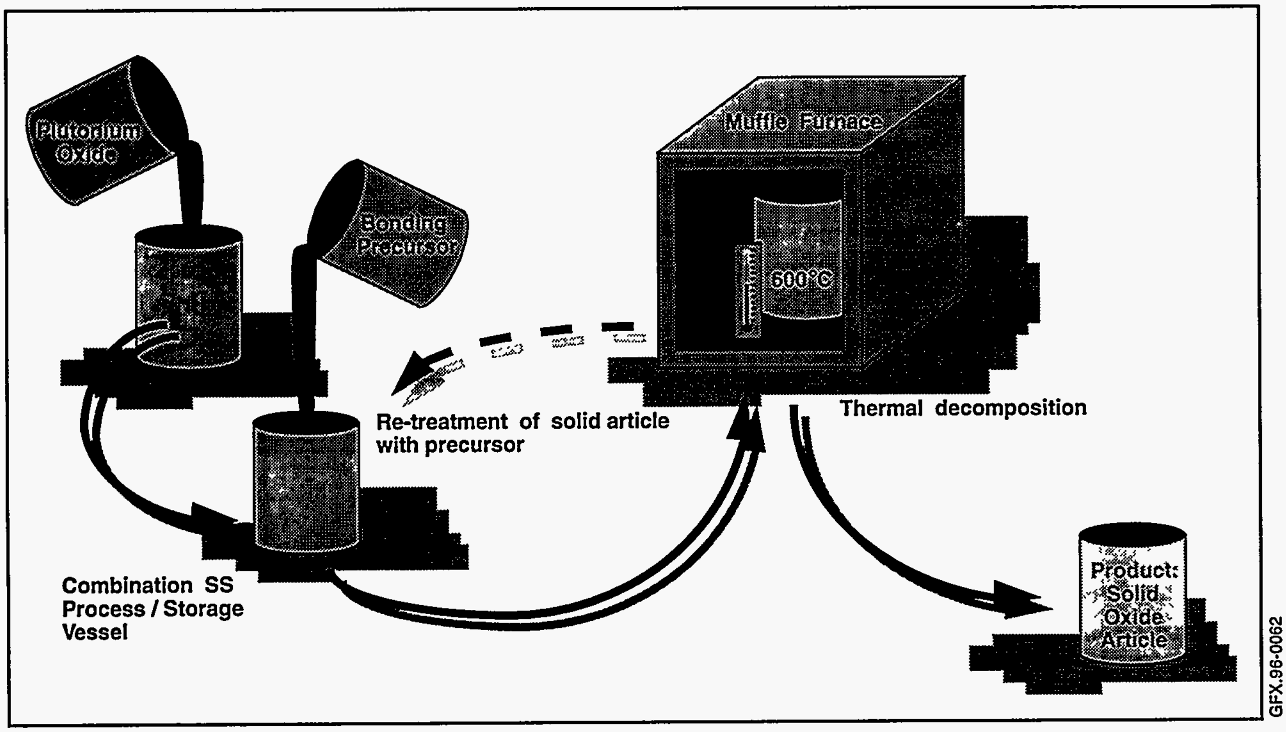

Figure 10. Ceramification Process

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.

TECHNOLOGY NEEDS

The DOE has over 5 MT of plutonium in the form of plutonium oxide across the complex, of which over 3 MT are maintained at Rocky Flats. 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 3 to 10 (Conrad et al. 1995, Rickets 1995). This is a significant risk reduction for the DOE with no additional cost or schedule delay.

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 total treatment time with three re-treatments to provide strength to the article.

Funding to develop ceramification at Rocky Flats 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.

BENEFITS

Ceramification eliminates or reduces 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

Rocky Flats 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. Experiments demonstrating this technology on plutonium compounds will be carried out at Savannah River Site with input from that facility.

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A. G. Phillips
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Phone: (303) 966-4346
no email capability

BIBLIOGRAPHY OF KEY PUBLICATIONS

Conrad, E., R. J. Mattson, J. Stakebake, S. Additon, S. Olinger, *Defense Nuclear Facilities Safety Board Recommendation 94-3 Rocky Flats Environmental Technology Site Implementation Plan: Task 3 Study Site Storage Alternatives*, Rocky Flats Environmental Technology Site (November 22, 1995).

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

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 direction 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 (CRADAS)

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, personnel, 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 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 or

Dr. Finis Southworth, Lockheed Idaho Technologies Company, P.O. Box 1625, Idaho Falls, ID 83415-3750, tel: (208) 526-8150.

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 form the research to the participating private sector company.

ACRONYMS

AL	Albuquerque Operations Office
ANL	Argonne National Lab
CH	Chicago Operations Office
CRADA	Cooperative Research and Development Agreement
DNFSB	Defense Nuclear Facilities Safety Board
DOE	U.S. Department of Energy
DPM	Disintegrations Per Minute
EM	Environmental Management
EP	Executive Panel
ES&H	Environment Safety & Health
GMODS	Glass Material Oxidation and Dissolution System
HEU	High Enriched Uranium
INEL	Idaho National Engineering Laboratory
IP	Implementation Plan
LANL	Los Alamos National Laboratory
LLNL	Lawrence Livermore National Laboratory
LMIT	Lockheed Martin Idaho Technologies
MT	Metric Tons
NMSTG	Nuclear Materials Stabilization Task Group
ORNL	Oak Ridge National Laboratory
PFA	Plutonium Focus Area
PFA	MPPlutonium Focus Area Management Plan
PuSAP	Plutonium Stabilization and Packaging
PVT	Pressure, Volume, Temperature
RC	Research Committee
R&D	Research and Development
RDP	Research and Development Plan

RFETS	Rocky Flats Environmental Technology Site
RFFO	Rocky Flats Field Office
RL	Richland Operations Office
SRO	Savannah River Operations Office
SRS	Savannah River Site
TAP	Technical Advisory Panel
TRD	Technical Requirements Document
TRU	Transuranic
WAC	Waste Acceptance Criteria
WIPP	Waste Isolation Pilot Plant

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