*Nuclear Materials Stabilization and Packaging
End-of-Year Status Report*

April 1–September 30, 1996

*Compiled by:
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NUCLEAR MATERIALS STABILIZATION AND PACKAGING END-OF-YEAR STATUS REPORT

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ABSTRACT

This report documents progress on the Los Alamos Nuclear Materials Stabilization and Packaging projects for the third and fourth quarters of fiscal year 1996. It covers development and production activities for the Plutonium Packaging project, the Plutonium Recovery and Stabilization project, and the Uranium Recovery and Stabilization project. In addition, it reports on quality assurance activities for the Plutonium Packaging project.

INTRODUCTION

This report documents technical progress on and plans for the Los Alamos National Laboratory Nuclear Materials Stabilization and Packaging projects. Project leaders are listed below:

Nuclear Material Stabilization and Packaging Project	Keith W. Fife (505-667-2353)
Plutonium Packaging	Carl W. Hoth (505-667-2354)
Plutonium Recovery and Stabilization	Keith W. Fife (505-667-2353)
Uranium Recovery and Stabilization	Jon B. Nielsen (505-665-8763)

Quarterly progress reports for these projects began in the second quarter of fiscal year (FY) 94. This report covers the third and fourth quarters of FY96 and is the ninth progress report to be issued.

Los Alamos project leaders and the Nuclear Materials and Stockpile Management program office are working to bring subprojects of the nuclear materials and stabilization effort into an integrated budgeting and reporting system. They will also clarify the interface between the Nuclear Materials Stabilization and Packaging operational projects, which are funded by the Department of Energy-Defense Programs (DOE-DP), and the Los Alamos 94-1 Research and Development Lead Laboratory Support projects, which are funded by DOE/Environmental Management (DOE/EM-60). Thus, plutonium oxide studies, in the section of this paper entitled DOE Storage Criteria, are included in this report for the final time. Beginning in FY97, oxide studies will be included under the Material Identification and Surveillance project in the 94-1 Research and Development (R&D) reporting. Risk-based prioritization of materials in storage is no longer reported in this document. The subject has been expanded in scope to include Rocky Flats materials and is now reported in the 94-1 R&D report under Risk Status of Residue Holdings. We have added a section entitled Plutonium Disposition Methodology to this report. Most stabilization R&D work will naturally fall within the 94-1 R&D project. However, some R&D that is specific to Los Alamos needs will be charged to the Nuclear Materials Stabilization and Packaging project and reported in all subsequent progress reports.

Los Alamos representatives on the DOE Working Group for the Plutonium Storage Standard (DOE-STD-3013)¹ participated in the discussions, telephone conferences, and meetings to assist in a major revision to the standard. Los Alamos staff met with DOE Headquarters personnel in June to discuss the impact for Los Alamos of any potential nondestructive assay (NDA)/analytic requirements for packaged containers. Concurrence was reached to reduce reliance on analytical chemistry within the Los Alamos Plutonium Packaging project and permit the material to be verified by NDA. A requirement for chemical analysis or NDA is not specified in the standard. The working group considered possible expansion of the standard to include storage of mixed plutonium-uranium oxides (MOX) <50 wt % plutonium. This change would allow existing MOX fuel to be stored under DOE-STD-3013. In

response to a request from the working group, Venetz et al. prepared a technical evaluation of issues related to such a change entitled "White Paper on Possible Inclusion of Mixed Plutonium-Uranium Oxides in DOE-STD-3013."² This paper has been submitted as a Los Alamos (LA) series report and will be published shortly. It addresses stabilization procedures, specific surface area effects, moisture readsorption kinetics, criticality issues, and loss-on-ignition (LOI) analysis. Difficulties arise from mass changes caused by the formation of higher uranium oxides during LOI measurements. Inclusion of MOX in the criteria was recommended only if the accuracy of the LOI results can be "rigorously demonstrated" by showing that mass losses during stabilization are not masked by mass gains from oxidation. The working group did not include MOX materials in DOE-STD-3013-96.¹ Imposing American Society of Mechanical Engineers pressure-vessel code on the boundary container was discussed but not adopted. The working group met again in July in Richland, Washington. Issues were resolved and DOE published the revised DOE-STD-3013-96 in September 1996.¹ The major changes to the document are that

- the packaging configuration was clarified to include two sealed containers—both to be externally contamination free—and an optional third inner (convenience) container,
- the primary containment vessel (PCV) was eliminated, and
- the inner container(s) shall contain features to allow for a nondestructive indication of internal pressure buildup at less than 690 kPa (100 psig), such as a pressure-deflectable lid or bellows, that would be observable by radiography.

Results of experimental studies to define the chemistry and stabilization procedures of metal and oxide were presented at meetings with United States and United Kingdom personnel. Work on the chemistry and kinetics of moisture-enhanced plutonium corrosion by air was described at the Twentieth Compatibility, Aging, and Stockpile Stewardship Conference held at Allied Signal in Kansas City from April 30–May 2 and at the joint working group (JOWOG) 22 Focus Meeting on Plutonium held at Lawrence Livermore National Laboratory on May 6–7. Topics covered in criteria-related presentations at the JOWOG 22 Focus Meeting on Actinide Stabilization, Packaging, and Storage held at Los Alamos May 21–22 include known failure mechanisms, parameters for stabilization of metal and oxide, pressurization and condensation in long-term storage containers, and recent findings in storage-related chemistry. Revised chapters from the International Atomic Energy Agency's (IAEA) *Safe Handling and Storage of Plutonium* were prepared for external review and comment. The chapters, entitled "Nuclear, Physical, and Chemical Properties of Plutonium" and "Plutonium in the Environment," were also published as LA Reports LA-UR-96-1228³ and LA-UR-96-1261,⁴ respectively.

PLUTONIUM PACKAGING PROJECT

DOE Storage Criteria

Investigation of Pressurization Processes. Efforts continue to define the chemistry and kinetics of the plutonium dioxide-water reaction. Knowledge of this process is essential for defining the fate of moisture that remains on stored oxide and the rate at which hydrogen is generated during extended storage. As reported in the preceding quarterly report, microbalance and x-ray diffraction measurements demonstrate that a superstoichiometric plutonium oxide, PuO_{2+x} , and hydrogen are formed by the reaction at temperatures up to 350°C .⁵ However, further evaluation of the data shows that the long-term stability of the balance is insufficient for accurate determination of kinetic parameters.

An alternative experimental approach based on pressure, volume, temperature (PVT) methods is being developed in an effort to accurately measure kinetics of the oxide-water reaction. Fabrication of the stainless steel vessels and manifold systems is complete. Internal surfaces of the devices have been gold coated to minimize interaction of water with the container. After operational procedures are completed and approved, simultaneous measurements will be made with two PVT systems at several temperatures.

Evaluation of Data Relevant to Storage. A formal report entitled "Reaction Kinetics of Plutonium with Oxygen, Water and Humid Air: Moisture Enhancement of the Corrosion Rate"⁶ has been accepted for publication in *The Journal of Alloys and Compounds*. Corrosion of plutonium by oxygen is accelerated in the presence of moisture because the water reaction is inherently rapid. The corrosion rate of unalloyed metal by water at 110°C and 160 torr is 6×10^4 greater than the corrosion rate by oxygen at the same temperature and pressure. Moisture enhancement of the plutonium-oxygen reaction is driven by the catalyzed combination of oxygen and hydrogen on the oxide-coated surface of metal. The catalytic reaction, which reverses the radiolytic dissociation of adsorbed water, is evident at humidity levels as low as 1 ppm.

Evaluation of equilibrium and kinetic results shows that adsorption of water on clean surfaces of plutonium dioxide occurs by a sequence of distinct processes. Surfaces are cleaned by firing the oxide in air at the temperature recommended for stabilization (950°C) prior to storage. Kinetic results for readsorption of water on the oxide show that the first two steps of the sequence involve a half-molecular layer of water. These processes are consistent with dissociative adsorption of water as hydroxyl, OH. In subsequent steps, water adsorbs in step-wise manner that involves nine molecular layers at room temperature and 100% relative humidity. Equilibrium data also show that water forms a tenacious water layer on the oxide. Both measurements suggest that a $\text{PuO}(\text{OH})_2$ surface composition is formed when oxide is exposed to water.

Results suggest that hydroxylation of the oxide surface may be a particularly important process for handling and storage of metal and oxide. Formation of PuO_{2+x} by the dioxide-water reaction and moisture-accelerated corrosion of plutonium are apparent consequences of the process. Observation of a square-root pressure dependence for reaction of water with oxide-coated plutonium metal provides additional evidence that the corrosion rate is controlled by the dissociative adsorption process.

Surveillance Experiments. Surveillance experiments were initiated approximately 16 months ago and reported in the previous quarterly report.⁵ Contents of the containers are shown in Table I.

Table I. Contents of Containers

Container No.	Material	Quantity (kg)	LOI (wt %)	Pu Factor (%)
1	Pure Metal	2.0	—	99.93
2	Oxide A ^a (as received)	1.0	0.60	87.7
3	Oxide A (600°C for 12 h)	1.0	0.11	87.7
4	Oxide A (980°C for 2 h)	1.0	0.05	87.7
5	Oxide B ^b (980°C for 2 h)	1.3	0.07	87.9

^a Oxide A was obtained by decomposition of Pu^{+3} oxalate precipitated from a plutonium nitrate solution.

^b Oxide B was obtained by hydride-catalyzed oxidation of plutonium metal in air at room temperature.

The materials were sealed in a helium atmosphere, in containers fitted with thermocouples, pressure and vacuum gauges, and with the exception of container number 3, a gas-sampling port. A quantitative baseline gas sample was obtained on containers 1 and 5, and a qualitative baseline gas sample was obtained on containers 2 and 4. Quantitative results are shown in Table II.

Table II. Composition of Gas Samples (vol %)

Container No.	Helium	Nitrogen	Oxygen	Argon
1	96.8	2.8	0.4	— ^a
5	84.9	12.5	2.4	0.1

^a Nondetect.

These containers were monitored daily for temperature. (See Table III.) Thermocouple number 1 measured the gas temperature at the top of the container, and thermocouple number 2 measured the temperature above the surface of the material. The equilibrium temperature values obtained in each container are reported.

Table III. Equilibrium Temperature Values Obtained in the Surveillance Containers

Container No.	Thermocouple No. 1 (°C)	Thermocouple No. 2 (°C)
1	35.6	41.1
2	27.8	28.6
3	27.5	28.3
4	26.9	27.8
5	28.1	28.9

During the monitoring of the surveillance experiments, daily pressure and vacuum readings were obtained. At the beginning of these experiments, there were slight fluctuations in the gauge readings that appeared to be consistent with measured temperature fluctuations. But during the last six months of these experiments, both pressure and vacuum gauges read zero with no daily or weekly fluctuations. Both containers 1 and 5 were resampled, with the results shown in Table IV.

Table IV. Composition of Gas Sample After Sixteen Months of Surveillance (vol %)

Container No.	Helium	Nitrogen	Oxygen	Argon	CO ₂
1	None	99.02	None	0.98	None
5	None	80.1	17.5	0.96	1.49

It was discovered that even though the containers were tested before they were placed in service, a small leak had developed. Apparently, with time, temperature fluctuation, and handling in the glovebox, some of the screws securing the lid had loosened, providing a pathway for glovebox air to enter. This would explain the gas sampling results obtained for container number 5, which are close to that of air. Container number 1 was a different situation. The gas in that container was almost completely nitrogen. The fact that after the container had apparently leaked for approximately six months, there was zero percent oxygen indicates that there is a strong affinity for oxygen reacting with plutonium metal and substoichiometric oxides. This reaction also indicates that the surveillance container is under a slight vacuum. The metal pieces were found to have a significant amount of oxide growth, which appeared to be formed in layers with significant fractures throughout the coherent oxide layer. Spallation had occurred, and there was loose oxide on the bottom of the container.

In conclusion, because of apparent leaks in the surveillance containers, the results of this study in regard to container pressurization or vacuum are inconclusive. However, other pertinent information was collected: the equilibrium temperatures inside the surveillance containers were determined for these quantities of material, and the fixtures on the surveillance containers should be welded, or if screws are used, they should be checked on a routine basis and retightened as necessary. In addition, the information collected from container number 1 provides some insight to the phenomena that would likely occur if there were ever a breach in a long-term storage container holding plutonium metal.

Evaluation of Material in Long-Term Storage—Material Shipped to Lawrence Livermore National Laboratory (LLNL). To satisfy a requirement to transfer material to LLNL for a casting demonstration, four long-term storage containers were shipped to LLNL. These items were chosen based on the purity of the material and their packaging configuration: sealed stainless steel containers, helium fill gas, and controlled oxygen and moisture level.

In preparation for shipping, the outer (boundary) containers were removed inside a hood with a standard-issue pipe cutter. The contaminated outer surfaces of the inner containers were decontaminated before these items were transferred to the shipping

and receiving section. At LLNL, two of the long-term storage inner containers were opened in a glovebox with an argon atmosphere.

The metal pieces were a shiny metallic (silver) color when they were placed into the long-term storage container roughly 15 months previously. Upon removal and inspection of the individual pieces, the metal appeared to be slightly discolored. The weight of the metal in one container after approximately 6 months of storage was 4 400.5 g compared with 4 400.4 g as packaged. Since a slight discoloration of the plutonium metal was noted, we can conclude that a small degree of oxidation occurred. During our inspection and evaluation of the plutonium metal pieces, no loose oxide was noted. This would indicate that the thickness of the oxide is probably much less than the equilibrium oxide thickness of 4–5 μm . (See "A Mechanism for Plutonium Pyrophoricity.")⁷ It should be emphasized that a thin layer of oxide actually acts as a protective layer on the metal surface. This layer of oxide acts as a diffusion barrier and could decrease the rate of oxidation.

When the inner long-term storage containers were being opened, there was no evidence of anomalies or unusual conditions.

Metal and Oxide Packaging

Processing and Packaging. From the time of the first demonstration container through the fourth quarter of FY96, processing personnel completed 81 plutonium metal items. These items were sent to the packaging section to be weld-sealed in approved inner and outer containers, then were delivered to the vault for long-term storage. Eighty items were completed in FY96, meeting the Los Alamos Site Materials Stabilization Management Plan commitment to the Nuclear Materials Stabilization Task Group for long-term packaging.

Section personnel also packaged two demonstration plutonium oxide containers during the fourth quarter of FY96, completing a Los Alamos internal milestone. The oxide material used was that brushed from metal during the metal packaging operations over the last several months. After the oxide was calcined at 950°C for 2 hours, LOI on this batch of oxide was 0.0125 wt %. Processing personnel placed the conditioned oxide into two aluminum convenience containers developed by packaging personnel to control contamination in the packaging gloveboxes. These convenience containers include a slip-lid design with a filtered lid to permit the helium backfill. The convenience containers were placed within the stainless steel DOE-STD-3013 material container, then, in the same manner as metal packages, weld-sealed and placed within the boundary container for the second weld seal. The containers were successfully removed from the glovebox line with no detectable alpha count on the outer surface of the boundary container. Processing and packaging developed for oxide was thus demonstrated for production.

Every fifth plutonium metal container and each plutonium oxide container included the pressure-measuring bellows to provide detailed data on the pressurization rate over a range of 0-50 psig.

Welding and Bellows Development. The initial lot of 10 sets of second-generation inner containers and lids manufactured by Spin Forge International has been evaluated, and a second order for 50 sets has been placed. In the first lot processing, 0.250-in. plate was spun to 0.065-in. wall thickness. After forming, the ends were machined down to the final thickness of 0.125-in. The first lot had a high degree of work hardening, which produced a very strong and unyielding container with the high burst strength of 4 500 psi. The work hardening also produced undesirable surface microfissures.

For the second lot of containers, the thickness has been reduced and the processing has been changed to reduce work hardening and produce a container that yields more readily at low pressure, thus giving obvious visual signs of pressurization well before rupture. A preform cup will be drawn from 0.090-in.-thick sheet prior to spin forming the container to the final 0.050-in. wall thickness. An intermediate annealing step was added to reduce work hardening. The lid was draw-formed from 0.050-in.-thick sheet. The second lot was delivered in September.

The new Miller Electric Co. welding equipment has been installed and tested. A weld fixture for aligning the container, lid, and torch at the weld joint has been designed and fabricated. Welding trials are currently being conducted, and test containers will be fabricated to verify the container strength.

Development and testing of the pressure measurement bellows is continuing with the Mini-Flex Corporation. Currently bellows are installed vertically and unprotected at the top in every fifth metal storage container. Future plans call for incorporating some protection for the bellows and for positioning the bellows horizontally to allow increased loading of oxide. Mini-Flex is currently producing prototype bellow assemblies in protective tubing capped with metal filters for use in the horizontal position. Mini-Flex is also developing and testing a shorter, larger-diameter bellows that may be suitable for vertical mounting in the container. Additionally, development has begun on a magnetically coupled sensor to read the bellows motion and pressure changes from outside of the double container system.

Plutonium Packaging Quality Assurance

All peer review action items reported previously are now complete. An internal quality assurance review of the Plutonium Packaging project is being scheduled during the second quarter of FY97.

Document Status. Work on quality assurance documents this quarter is summarized below.

1. Documents approved:
 - 611-NMT4-R00, NMT-4 Inventory Control for Bellows and Long-Term Storage Container Components Procedure
 - PAK-DR-023A and -23B-R00, Long-Term Storage Container Components Inventory Tracking Sheet
 - 609-NMT4-R00, Safe Operation of the Real-Time Radiography System
 - 608-NMT4-R00, Safe Operation of the Lumonics LightWriter™ Laser Marking System
 - PAK-DR-026-R00, Laser Marking Data Record
 - 610-NMT4-R00, Laser Welding of Bellows to End Piece
 - PAK-DR-027-R00, Laser Welding of Bellows Data Record
2. Documents under review:
 - PAK-QP-021-R00, Data Base Plan
 - 604-NMT4-R01, Container Fabrication Procedure
3. Documents being prepared:
 - PAK-QP-007-R00, Weld Qualification Plan/Weld Specification
 - PAK-DR-015-R00, Radiography Data Record (baseline surveillance)
 - PAK-DR-009-R00, Bellows Measurement Data Record
 - PAK-QP-022-R00, Container Weld Destructive Test Plan
 - Safe Operation of the Miller Intellitig 4/Maxstar™ 175 Welding System
 - Weld Development Data Record
 - Oxide Data Record
 - Real-Time Radiography Qualification Plan
 - Radiographic Inspection and Surveillance Procedure
4. Revised documents under review:
 - PAK-QP-004-R01, Record Management Plan
 - PAK-QP-003-R01, Document Control Plan
 - PAK-QP-003-R00, Appendix, Distribution List for Quality Assurance Documents
 - PAK-DR-019-R03, Container Fabrication Data Record
 - PAK-QP-006-R01, Calibration and Measurement Control Plan
 - PAK-QP-09-R01, Control of Special Processes
 - PAK-QP-005-R01, Procedure for Controlling Nonconformance Activities and Documents
5. Document being archived:
 - PAK-QP-008-R01, Procurement Plan

Nonconformance Reports. Nonconformance report number PAK-101 was initiated on container number LM0033. The outer container top weld did not melt uniformly due to gas pressure venting during the second welding pass. A third welding pass was performed to repair the weld. The weld passed the helium leak-rate test but was rejected because of visual inspection considerations. The outer container was removed, replaced with a new outer container per 604-NMT4-R01, Container Fabrication Procedure, and tested per 597-NMT4-R00, Helium Leak Test Procedure.

Nonconformance report number PAK-102 was initiated on container number LM0038. The outer container did not pass the helium leak-rate test. The deficiency was a pinhole in the outer container bottom. The outer container was removed, replaced with a new outer container per 604-NMT4-R01, and tested for the helium leak-rate per 597-NMT4-R00. The container was then approved for storage.

Nonconformance report number PAK-103 was initiated on containers numbered LM0055 through LM0059. These five containers were loaded and sealed with a material type different from that marked on the containers. The material type number identifies the specific isotopic category and the element. The material type number was originally included on the laser-marked labels to assist vault personnel. However, it became difficult to coordinate laser marking with the selection of feed material. The marking procedure was changed so that the material type number will no longer be marked on the container. The existing material-type marking on these outer containers will be blanked out at a later date, possibly using a laser marker that is currently scheduled to be installed in the Plutonium Facility for another project. The marking on the inner container will not be changed. The ID matrix will also not be changed because it is not human readable. The database and the data package will record the correct material type. The affected containers will be identified in the vault with a paper label until the disposition is complete.

PLUTONIUM RECOVERY AND STABILIZATION PROJECT

Project Management. We completed changes to the Defense Nuclear Facilities Safety Board Recommendation 94-1 Implementation Plan (IP) Milestones⁸ and a second revision of the Los Alamos National Laboratory Site Integrated Stabilization Management Plan (SISMP). Milestone IP-3.2-014, Thermally Stabilize and Repackage All Plutonium Oxide to Meet the Metal and Oxide Storage Standard, has been changed to include stabilizing the remaining residue inventory and recovering the plutonium as oxide, and repackaging the existing plutonium metal and oxide inventory (>50% plutonium assay) to meet the long-term storage standard by May 2002. Milestone IP-3.2-035, Stabilize and Repackage High Risk Vault Items to Meet Long-Term Storage Standards, has been changed to include stabilization of high-risk vault items and recovery of the plutonium as oxide for packaging to meet the long-term storage standard by September 1997. Neither of the two Los Alamos milestones are expected to incur schedule slippage as a result of these changes.

The SISMP revision reflects two significant changes Los Alamos is integrating into its 94-1 response. The first change is a redefinition of the legacy 94-1 inventory to reflect an overall decrease in the number of items under the auspices of the recommendation because items have been moved into active or planned programmatic use ADP-xxx projects. The second change is that the ADP-xxxx materials will no longer be packaged for long-term storage, but will now be packaged for availability to new or expanding DOE programmatic activities at the Laboratory.

Recovery and Stabilization Operations. Overall, the project continues to be on schedule from an item-processing standpoint. As of October 1, 1996, we have stabilized approximately 25% of the legacy plutonium items subject to the 94-1 Recommendation. Overall progress includes packaging 326 kg of plutonium as metal in containers meeting DOE-STD-3013 for long-term storage,¹ packaging 6 kg of plutonium as oxide to meet that standard, and stabilizing 130 kg of plutonium from residue sources. During the last half of FY96, however, stabilization totals were adversely affected by two unexpected situations: unscheduled downtime and maintenance requirements on our facility evaporator system and preparation for a facility stand-down to cross over safety systems as part of the Operations Center Upgrade project.

Both of these activities caused our aqueous nitrate operations to operate intermittently for the last half of FY96. In addition to completing all required maintenance and preparing our operations to be down for portions of the first quarter of FY97, we have instituted process changes designed to improve the lifetime of various evaporator components, as well as completed a more efficient integration of cement immobilization with evaporator operation. Completion of these activities is expected to produce a much more efficient and reliable aqueous nitrate process sequence when operations are restarted in January 1997.

Because the Operations Center Upgrade project was accelerated and the unscheduled evaporator downtime occurred, specific internal Los Alamos milestones were not met in FY96. These internal milestones consisted of maintaining pace with stabilization and recovery of the high-risk material categories of sand, slag, and crucible and hydroxide precipitate items. These are internal milestones, however, and have not affected the projection for cumulative items worked off since the beginning of the project. It is too early to project the impact of this activity on our ability to meet the 94-1 Implementation Plan⁸ milestone of having high-risk categories stabilized by September 1997 (Los Alamos milestone IP-3.2-035).

As a result of the focused statistical sampling of our vault inventory completed in July, we have identified two subcategories of items (silica solids and cellulose cleanup rags) that we feel cause undue worker safety concerns for vault personnel. We observed high failure rates of the samples we inspected, ~ 60% of the silica solids and ~ 20% of the cellulose cleanup rag samples failed. These two subcategories have been moved into the high-risk category, along with legacy

hydroxide solids and the sand, slag, and crucible categories. These new items do not replace existing high-risk items but are added to the inventory of high-risk materials requiring stabilization by September 1997.

A summary of risk-based prioritization methodology development and application at Los Alamos is found in the report "Risk-Based Analysis for Prioritization and Processing in the Los Alamos National Laboratory 94-1 Program."⁹

In addition to processing legacy items as the focus of the recommendation, we strive to coordinate and stabilize currently generated residues to avoid their accumulation in process areas or in the vault. These materials include machine turnings and chips from surveillance and manufacturing operations, hydroxide cakes from aqueous chloride operations, and analytical chemistry solution returns. We will also pyrolyze cellulose rags contaminated with significant amounts of ^{238}Pu . This type of operational flexibility is encouraged and will be maintained for the safe operation of our facility.

Our FY96 accomplishments in response to the recommendation for legacy material stabilization are shown in Table V.

Table V. Legacy Items Stabilized to Date

Material Category	Legacy Items Stabilized in FY95	Legacy Items Stabilized in FY96	Total Legacy Items Stabilized to Date
High-Risk Sand, Slag and Crucible	106	69	175
High-Risk Hydroxide Precipitate	159	55	214
High-Risk Silica Solids (added 7/96)	0	3	3
High-Risk Cellulose Rags (added 7/96)	15	5	20
Impure Metal	100	309	409
High-Priority Process Residues	0	0	0
Analytical Chemistry Sample Returns	13	110	123
Analytical Chemistry Solution Returns	286	0	286
High-Priority Compounds	0	24	24
Other Combustibles	0	0	0
Other Compounds	135	35	170
Other Process Residues	119	97	216
Noncombustibles	63	32	95
Unsheltered Containers	0	1	1
Gases	1	0	1
Total Items Stabilized	997	740	1737

In response to the recommendation for legacy material packaging to meet DOE-STD-3013 for long-term storage,¹ our accomplishments in FY96 consisted of consolidating 199 plutonium metal items into 79 packages containing a total of 326 kg metal and consolidating 5 plutonium oxide items into 2 packages with a total of 5.7 kg of oxide.

Other items of interest for FY96 include completing the processing and recovery of one unsheltered container and Los Alamos beginning to receive material from Mound Laboratory. The Mound material is essentially a small quantity of nuclear material but it consists of numerous small items. Currently, the material is embroiled in shipper/receiver negotiations that may take some time to reconcile.

Research and Development Activities. Because of facility priority conflicts, the installation and demonstration schedule for the salt distillation prototype for NaCl-KCl salts slipped about three months. We were, however, able to maintain our original goal of demonstrating the technology by the end of FY96. The extent of the demonstration was shortened; however, demonstration will continue in FY97.

Deployment of a pyrolysis unit for treating certain categories of noncombustibles has also slipped due to additional development required by the 94-1 R&D project. Demonstration of waste-stream polishing for acidic chloride streams, however, was successfully completed during the third quarter of the fiscal year. We continue to evaluate more selective ligands for americium and plutonium extraction from solution destined for waste processing. Hydrothermal processing of mixed-waste solution items has been determined to be the most appropriate technology for completing the stabilization of these legacy items. The equipment has been installed in an active glovebox in the facility, and we are currently awaiting DOE approval and a readiness assessment prior to demonstration with actual materials. This activity will hopefully be completed early in calendar year 1997.

During the third quarter of FY96, we initiated a small development project to evaluate selected ultrafiltration modules for particulate removal from caustic waste solutions. This technology will ultimately be used to effect a more complete actinide removal from the caustic waste prior to its transfer to the Los Alamos Liquid Waste Effluent Treatment Facility. We also completed modification of a shielded blending apparatus for the homogeneous mixing of small quantities of actinide oxide. This apparatus will be used for high-exposure actinide-oxide mixtures requiring analytical chemistry analysis for material control and accountability (MC&A) needs prior to packaging for long-term storage.

PLUTONIUM DISPOSITION METHODOLOGY

The Plutonium Disposition Methodology (PDM)¹⁰ is being implemented in a manner that complements residue inventory stabilization efforts at TA-55. Under the PDM, potential inventory dispositions and discard limits are quantitatively evaluated to project the consequences of implementing candidate dispositions in terms of waste

generation rates, personnel exposure, special nuclear materials (SNM) recovered/disposed, overall costs, and equipment/personnel allocation (e.g., how long resources are dedicated and therefore unavailable for processing of other materials). Evaluated in conjunction with the criteria for Attractiveness Level E material (discardable low-grade SNM-bearing material) recently issued by the DOE Office of Safeguards and Security (OSS), these projected consequence data provide a solid basis for dispositioning individual residue matrices.

For example, we have recently used the PDM to evaluate the graphite residue inventory at TA-55. We determined it would be far less costly, result in significantly reduced personnel exposures, and minimize waste generation to discard all but the richest materials in inventory rather than to recover down to plutonium concentrations discardable under the 1989 Economic Discard Limits. Under this disposition, Attractiveness Level D materials must be converted to a form acceptable for safeguards termination in accordance with the previously mentioned OSS criteria. This disposition also reduces the time required for residue processing from 140 man-weeks to 20 man-weeks, thereby freeing up resources for recovery/stabilization of other residue matrices. Plans are to ultimately evaluate the entire residue inventory using this methodology.

It should also be noted that the DOE Albuquerque Operations Office has presented the PDM to the New Mexico Environment Department (NMED), the state's Resource Conservation and Recovery Act (RCRA) enforcement agency. NMED has informally indicated concurrence with the overall process and therefore the salient PDM rationale that SNM-bearing materials are not waste until they are determined discardable by DOE.

URANIUM RECOVERY AND STABILIZATION PROJECT

Approval for Temporary Material Access Area (TMAA) was granted for Wing 5 of the Chemistry and Metallurgy Research (CMR) Building at TA-3. Activities involving large items of UF_6 can now be scheduled.

TMAA was granted for Wing 4 and Wing 1 of the CMR Building. Repackaging and NDA measurements of large items can now be conducted in these wings.

Construction of the room air system in Wing 9 of the CMR Building is approximately 90% complete.

A 60% review of the room air system was conducted with Architectural Engineer Lockwood Greene on September 23, 1996. Revisions are being conducted and they are working toward a 90% review.

The transfer of the last UF_6 item from Los Alamos to the Portsmouth Gaseous Diffusion Plant in Portsmouth, Ohio, was completed and other unknowns were identified as non- UF_6 items.

One technician was hired for the 94-1 project.

During the last quarter of FY96, 63 items were repackaged and NDA measurements taken.

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