*Nuclear Materials Stabilization
and Packaging*

*Quarterly Progress Report
October 1–December 31, 1995*

*Compiled by
Kenneth M. Chidester*

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NUCLEAR MATERIALS STABILIZATION AND PACKAGING

QUARTERLY PROGRESS REPORT October 1–December 31, 1995

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Kenneth M. Chidester

ABSTRACT

Progress is reported for Los Alamos Nuclear Materials Stabilization and Packaging projects for the first quarter of Fiscal Year 1996. Development and production activities in Plutonium Recovery and Processing, Plutonium Packaging, and Uranium Recovery and Processing are covered. Packaging quality assurance activities are reported.

INTRODUCTION

This document reports technical progress and plans for the Los Alamos National Laboratory Nuclear Materials Stabilization and Packaging project operations. Project leaders are as follows:

Kenneth M. Chidester, 94-1 Operations (505-667-2358);
Carl W. Hoth, Plutonium Packaging (Packaging) (505-667-2354);
Keith W. Fife, Plutonium Recovery and Processing (Processing) (505-667-2353); and
Jon B. Nielsen, Uranium Operations (505-665-8763).

This report continues quarterly progress reporting begun in the second quarter of FY 1994.

The Laboratory's Research and Development portion of the 94-1 response is organized under the Nuclear Material and Stockpile Management Transition Technologies program manager. Progress in these tasks is reported separately. The first report of progress on 94-1 Research and Development Project Lead Laboratory Support will be issued this month.

OPERATIONS PROJECT STATUS

Project Management

In December, Defense Programs management at DOE Headquarters and the DOE Albuquerque Operations Office (DOE-AL) agreed to provide support for an 8-year schedule for completion of 94-1 stabilization activities at Los Alamos. The Los Alamos National Laboratory Site-Integrated Stabilization Management Plan (SISMP) for Residue Remediation and Material Management, Volumes I and II, was submitted to DOE in November and is now being revised to reflect the change from a 15-year to an 8-year schedule.

Presentations and Informational Reporting

Nuclear Materials Technology Division Review. Recent work in developing the technology base needed for implementing DOE-STD-3013-94 was reviewed in November at the Science and Technology Assessment of the Nuclear Materials Technology (NMT) Division. Progress in defining the conditions for preparing metal and oxide for compliant storage were described in an oral presentation entitled "Plutonium Storage: Requirement and Response." Results of studies that define the chemistry and kinetics of the interactions between oxide and residual water in storage were described in a poster presentation on "Characterization of the Reaction Between Plutonium Dioxide and Water." These results were of particular interest to certain members of the review committee because of the potential application in elucidating the mechanism by which the oxide forms solutions of high-valence-state plutonium ions under environmental conditions. Discussions also included the relevance of the results in interpreting the dissolution behavior of high-fired oxide, the sintering characteristics of oxide compacts, and the marked enhancement of plutonium corrosion rates by moisture. Progress was reported in qualifying a double-welded container for long-term storage and placing the first several quality-assured packages in the Plutonium Facility vault.

Plutonium Focus Area Site Visit. A presentation outlining the technical base for storage of metal and oxide was also made during the Plutonium Focus Area Site Visit in December. By

describing the effort required to achieve compliance with well-characterized materials such as metal and oxide, an indication was given about the anticipated difficulties of stabilizing poorly characterized and variable residues.

Publications. An extensive review article on the storage of weapons- and reactor-grade plutonium was prepared and submitted to the *Encyclopedia of Environmental Analysis and Remediation*. Topics discussed include plutonium production, the worldwide plutonium inventory, historical packaging methods, storage options, relevant properties of plutonium, potential hazards, documented accidents, and storage recommendations. This paper is issued for publication as LA-UR-95-4415.

Participation in DOE Task Teams—International Atomic Energy Agency Standard

Development of a safe practices document for the International Atomic Energy Agency (IAEA) continues. Additional revisions to the chapter on "Plutonium in the Environment" were completed. The document has not been released for review by member states.

PACKAGING PROJECT STATUS

Packaging/Welding Operations

Packaging Operations

Eight metal-filled containers were completed this quarter and placed in the vault. Quality Assurance documentation was completed and submitted for archival purposes. Two containers included a bellows welded onto the inner surface of the material container lid. It is anticipated that every fifth metal-filled container will include a bellows. Although pressure increase is not expected in metal-filled containers, this opportunity is being taken to evaluate the ability to read the bellows length in a filled container application. Effective next quarter, the throughput goal is two completed containers per week. Packaging operations personnel have demonstrated this rate, although it may be difficult to sustain at the present resource level.

Laser Marking System

The Lumonics laser marking system was delivered and installed in Building PF-41. A vendor representative was scheduled for check-out of the equipment and training of Packaging project personnel in January. After this visit, the Robotics Laboratory personnel from Sandia National Laboratories/New Mexico (Sandia/NM) will provide training and orientation regarding the optimum laser parameters as they impact corrosion resistance and machine readability.

Weld Equipment Improvement and Container Design

Project personnel are presently working on the design and fabrication of an improved material container consisting of a one-piece formed bottom and a one-piece formed top joined with an autogenous square butt gas-tungsten-arc (GTA) weld. The container is 4.5-in. inside diameter with a 0.065-in. wall thickness and 0.125-in.-thick flat ends. The bottom is 8 in. long and the top is 1.75 in. long. The single square butt weld is inherently stronger than the top and bottom

standing welds of the existing container, and the butt weld is more suitable for ultrasonic or radiographic nondestructive examination methods.

Spin Forge International of Long Beach, California, is under contract to produce 80 sets of the prototype container. These will be evaluated for roundness, wall-thickness variation, and surface finish. We have requested 40 containers made from low-sulfur ($S < 10$ ppm)-type 304L stainless steel and 40 made from high-sulfur ($80 < S < 160$ ppm)-type 304L stainless steel. The two material types will be evaluated for weldability, electropolishing, and electrolytic decontamination characteristics. Characterization of the two materials will enable us to specify the more desirable material type or to adjust the container cleaning and welding processes, as varying materials are encountered in production. The vendor is presently working on the tooling for the spin-forming process and expects to deliver some first-run cans by mid-February 1996.

Weld Logic, Inc., was contacted in regard to an integrated weld station, power supply, and programmable controller for the new container design. The vendor is investigating the necessary modifications to reduce the overall height of their standard system so that it will fit into the

31-in.-high glovebox space. We expect a price quote from Weld Logic on the modified weld station by the first week of February 1996.

Container Testing

At the request of the Laboratory Packaging project leader, a 30-ft drop test was conducted on the Packaging project container. Because the 100-ft drop tower that would normally be used by Engineering Sciences and Applications Division (ESA) testing personnel is out of service, a temporary drop test setup was put in place at the Plutonium Facility site, TA-55. Container engineering staff from the Design Engineering Group (ESA-DE) advised in the setup to meet their general testing requirements; and Johnson Controls, Inc., (JCI) provided equipment and personnel to conduct the test under the direction of the TA-55 crafts supervisor. A cherry-picker personnel lift was placed on a concrete pad and a 1/2-in. steel plate was positioned beneath it. A JCI employee certified in use of the personnel lift dropped the container. A photographer from TA-55 Chemical Science and Technology Materials Characterization and Analytical Chemistry Group (CST-15) took video and still films of the test. For the test container system, a material container was filled with 10 lb of metal pieces, welded closed according to Packaging project procedure, and placed within the boundary container, which was also welded. The container was dropped three times—on end, on the lengthwise axis, and at a 45° angle on an edge. It was leak-tested after each drop and found to meet the required DOE-STD-3013-94 leak rate each time.

Quality Assurance

Project Review. The Packaging project was reviewed for the NMT Division and the Nuclear Material and Stockpile Management (NMSM) program office managers on December 11, 1995. The presentation focused on quality issues and the status of the project operations in regard to the work authorization basis. Since no new technologies or equipment are employed by the project and the scope of its materials handling is within the defined safety envelope of the facility, the consensus of the meeting was that no further environmental or safety assessments will be required. It was decided by the division and project managers at that time to request that DOE-AL also review the project. The NMSM program office and DOE-AL will arrange this review to be conducted in February.

The status of August peer review action items that were open at the last quarterly report is listed in Table I.

Table I. Project Internal Peer Review Action Items

The Quality Assurance (QA) Group (ESH-14) will provide an issues paper on the DOE-STD-3013-94.	Open. Due in January.
Processing personnel will provide statements for their procedure 433-REC stating criteria for selection of metal pieces for brushing or calcining by size and for acceptable brushing of the metal to remove oxide.	Draft revision complete.
The project QA representative and technical personnel will make minor adjustments to data package documents, particularly to correct and update the oxide processing data record PAK-DR-018 and the welding data record PAK-DR-019.	Complete.
Project personnel will prepare an administrative procedure to direct Materials Science and Technology (MST) Division welding of bellows to the container lids using Metallurgy Group (MST-6)-approved safe operating procedures.	Open. Due February.
Project personnel will provide a procedure for the container package data base and implement the data base operation.	Draft complete. Final due in January.
Concurrent with the procurement of the radiography equipment, project personnel will prepare, review, and approve procedures for its use.	Open.
Project QA representative and the project leader will complete Surveillance and Inspection Procedure PAK-QP-020 and obtain signatures.	Complete.
Project personnel will procure and install necessary shelving in QA cage in Building 5, the TA-55 warehouse.	Open. Status of cage uncertain.
The project leader will determine the number of weld quality test containers to be set aside for destructive tests of the welds. The procedures for quality destructive testing must be documented.	Draft destructive test plan complete. Due March.
Project personnel and the project QA representative will revise 604-NMT4 to state the frequency of checking the weld setup when multiple welds are done on one day.	Weld setup will be checked with each weld. Complete.
Processing personnel will determine the possibility and technical consequences of varying humidity in processing gloveboxes in PF-4, Room 208, and will take appropriate action, if necessary. [Although the relative humidity of the boxes is not regularly monitored directly, processing personnel believe that atmospheric conditions coupled with plant intake air driers will not allow air moisture content to reach a level of concern.]	After lengthy discussion with facilities personnel, it was agreed that the possibility for significant relative humidity fluctuation is extremely low; however, the decision was made to record relative humidity for each container. Complete.
Processing and Packaging personnel will conduct experiments with plutonium oxide to determine the procedure needed to replace oxygen atmosphere in containers with helium atmosphere before welding.	Open. This task will be conducted by the Shelf-Life R&D project.

Container Marking. Project personnel attended a meeting in Washington, D.C., November 17-18, 1995, called by the Nuclear Materials Stabilization Task Group (NMSTG) to determine container marking requirements for the consolidated packaging line procurement. The procurement specifications will state requirements for human- and machine-readable container marking and will require that a manufacturer's model number, a container serial number, and a reference mark for repeatability of radiography should be placed on each container in specified locations. The spacing on the can will allow for site-specific information to be added locally.

Nonconformance Report. Nonconformance report PAK-100 was written on the top weld of the boundary container for the second long-term storage container demonstration. Gas pressure vented through the weld bead during the second pass, causing the weld bead not to seal. Although at the time, an additional pass was made to repair the weld, the boundary container was later replaced. Analysis of metallography samples showed that the repaired weld appeared to be acceptable. The use of a heat sink placed around the container near the weld area was proposed to prevent future failures of this nature. Subsequently, it was decided that with sufficient cool down between the material container weld and the boundary container weld, the use of the cumbersome heat sink would not be necessary. There has been no repetition of this type of failure to date.

Documents. The following quality assurance documents were approved this quarter:

PAK-DR-018 (Rev. 01)	Repackaging Record, Oxide and Metal;
PAK-DR-019 (Rev. 02)	Container Fabrication Data Record;
PAK-DR-020 (Rev. 00)	Inspection and Surveillance Plan; and
PAK-DR-024 (Rev. 00)	Supervisor Approval Checklist (Metal).

The Data Base Plan, the Container Weld Destructive Test Plan, and the Inventory Plan and Inventory Data Record are in preparation.

DOE Storage Criteria

Investigation of Pressurization Processes

Pressure-volume-temperature (PVT) studies of the oxide-water and catalyzed oxygen-hydrogen combination reactions continue. Although important questions have been answered regarding the chemical fate of residual water on stored oxide, further measurements are needed to address issues concerning possible effects of initial oxide stoichiometry on the observations and to establish the long-term kinetic behavior of the chemical system.

Investigation of the oxide-water reaction by microbalance techniques has provided data that strongly support PVT results showing that a superstoichiometric oxide, PuO_{2+x} , is formed. After completion of experiments with aluminum oxide to demonstrate that the balance system is functioning properly, a series of experiments was initiated in which substoichiometric plutonium dioxide, PuO_{2-x} , was exposed to 15 torr of water vapor for extended periods at temperatures in the 100–300°C range. Samples taken at different points were analyzed by x-ray diffraction. The total mass gain corresponding to a bulk composition of $\text{PuO}_{2.07}$ was observed after reaction at 300°C. Diffraction results show that the cubic lattice parameter decreases as PuO_{2-x} is oxidized to PuO_2 and increases in the superstoichiometric region. Efforts to define the temperature dependence of the reaction rate are continuing.

Evaluation of Data Relevant to Oxide Storage

Questions about the consequences of residual moisture in storage containers for metal are being addressed. This effort is promoted by results showing that the combination of oxygen and hydrogen is catalyzed by plutonium oxide and by suggestions that this reaction may participate in acceleration of the plutonium-oxygen reaction by moisture. Preliminary evaluation of literature reports and unpublished data show that the plutonium-water reaction is inherently faster than the plutonium-oxygen reaction. The apparent mechanism for enhanced corrosion of plutonium in moist air or oxygen is a process that involves rapid reaction of water to form oxide and hydrogen and subsequent combination of the product hydrogen with gaseous oxygen. The reaction of oxygen is catalyzed by formation of water as a reactive intermediate on the oxide-coated surface of the metal. Efforts to further define the kinetic effects of moisture are in progress.

Processing Metal and Oxide

Special Projects

Oxide Particle Size Study. In response to a joint agreement between Los Alamos and Rocky Flats Environmental Technology Site (RFETS), a memorandum "Mass Fraction of Respirable and Dispersible Particles of Pure Plutonium Oxide as a Function of Processing History and Thermal Treatment," was developed by Tom Ricketts and submitted to Jerry Stakebake, Scientific Applications International Corporation (SAIC)/RFETS.

Pelletizing Plutonium Oxide. Tom Ricketts of the Processing section and Tom Blair of group NMT-9, the Heat Source Technology group, fulfilled commitments to RFETS in regard to pressing green pellets of pure plutonium oxide. The Processing section provided characterized pure plutonium oxide to NMT-9 to be pressed into pellets and assisted in interpretation and evaluation of the particle size data obtained from the experiments conducted on the green pressed pellets. A letter report was submitted to RFETS in November 1995.

94-1 Research and Development Proposal. A proposal was developed for the 94-1 Research and Development Lead Laboratory Technical Program Plan to characterize plutonium oxide having plutonium content between 50 wt % and 88 wt %. Major areas of emphasis are loss-on-ignition (LOI), surface area, and weight gain of representative items with varying calcination temperatures.

Aluminum Cans for Plutonium Transport

An aluminum can is under development for use in placing oxide into the material container. Although the double stainless steel can configuration used in the Packaging project meets the DOE-STD-3013-94 standard and there is a successful protocol for packaging metal, some handling issues have not been addressed for plutonium oxide. The low-density oxide readily becomes airborne and rapidly and thoroughly contaminates surfaces. By wrapping all containers before they are entered into the glovebox line and removing these contamination barriers at crucial steps, Los Alamos has successfully packaged plutonium metal and removed boundary containers with no detectable external contamination. In this manner the boundary container remains essentially uncontaminated throughout the welding operation, with the possibility of easily removing any contamination that is found on the container. The movement of plutonium oxide will require more care and sophistication to ensure that materials are not lost by inadvertent spills during transport, welds are not compromised by oxide contamination, or

that the boundary container is so unacceptably contaminated that it can not be successfully handled during the cold (with nonradioactive materials) welding operation. Use of an aluminum can to transport plutonium oxide to the material container may alleviate this potential problem. The aluminum can under development can be inserted into the material container leaving sufficient head space to secure a pressure sensing bellows. It can be closed mechanically without the use of a sealing compound. The lid of the aluminum container can be pierced before the material container is welded, allowing for a pressure change to be recorded by the bellows. Use of the aluminum container in this manner would reduce particle interference of bellows compression and alpha contamination of the solid-state signal devices currently under development at Sandia National Laboratories/New Mexico (Sandia/NM). It would make possible the packaging of oxide before full implementation of either the electrolytic decontamination system or a bagless can-out system. Initial design with vendor participation will begin next quarter with prototype containers to be fabricated and tested by April.

Loss-On-Ignition Analysis

A series of LOI runs was conducted to evaluate the impact on the LOI of the post-heating cool-down procedure. Questions regarding the effect of the cool-down procedure on the LOI have been expressed over the past several months. A cursory review of the literature did not reveal specific information regarding the impact of the cool-down procedure on the LOI.

One hundred grams of PuO_2 from oxidized plutonium metal was obtained from the NMT-2 Pyrochemical Operations team for use in this evaluation. The oxide was calcined at 950°C for 6 h in a muffle furnace. The calcined oxide was then riffled down into eight approximately 12.5-g samples that were sealed in sample jars with phenolic cap seals.

Each sample was split into two aliquots and processed for LOI determination according to the LOI procedure 283-MRD. The only deviation from the procedure was in the post-heating cool-down procedure as described in Table II.

Table II. LOI Post-Heating Cool-Down Procedure

Sample	Cool-Down Procedure	Total Aliquots
1	30 min in desiccator under ~1 scfm argon flow.	4
2		
3	30 min in desiccator under 24 in. of water vacuum.	4
4		
5	30 min in desiccator under static head of argon.	4
6		
7	30 min in glovebox atmosphere.	4
8		

These cool-down procedures were selected to provide a dramatic difference in technique in an effort to magnify the impact of the cool-down procedure on the LOI.

The LOI results are shown in Table III. This study indicates that the cool-down procedure does impact the overall LOI; however, this impact is small and does not appear to significantly impact the LOI results in the range of interest. The mean LOI for the four aliquots processed for each cool-down technique ranged from a low of 0.00294 to a high of 0.0209. The lowest mean LOI was obtained when the cool down was conducted under a flow of argon and the

highest obtained when a static head of argon was maintained over the sample. The coefficient of variation is an appropriate technique to use when evaluating samples from different sample populations. Coefficients of variation (CVs) for the samples cooled under vacuum and a static head of argon were 90.81% and 93.97%, respectively, while the coefficient of variation for the flowing argon and glovebox atmosphere cool downs were 27.89% and 48.72%.

Table III. LOI Cool-Down Results

Sample	#1 LOI	#2 LOI	Mean LOI	Batch Mean LOI	Standard Deviation	CV (%)
1	0.00333	0.00333	0.00333	0.00294	0.00082	27.89
2	0.00337	0.00171	0.00254			
3	0.00304	0.01454	0.00879	0.01328	0.01206	90.81
4	0.02980	0.00573	0.01776			
5	0.00982	0.01019	0.01001	0.0209	0.01964	93.97
6	0.01332	0.05027	0.03180			
7	0.00504	0.00346	0.00425	0.00587	0.00286	48.72
8	0.01002		0.00748			

While this study is not definitive, it indicates that the post-heating cycle cool-down procedure does have an impact on the total LOI. Cooling of the sample under vacuum and a static head of argon should probably be avoided because of the large variability associated with those techniques. In addition, it is recommended that until additional studies have been conducted, the cooling of the sample in the glovebox atmosphere should not be used because of the potential susceptibility of the sample to perturbations in the glovebox atmosphere, such as humidity. Therefore, it is recommended that the LOI samples be cooled in a dessicator, using a low flow of argon (~1 standard cubic feet per minute) for 30 min prior to final weight determination. The fact that this technique was used in the LOI determination should be communicated to the sample requester. Additional evaluation of this phenomenon using various oxide feed lots, cooling techniques, and moisture monitoring equipment is required to establish the validity of this study and its application to other feed lots.

Inspection and Surveillance of Containers

Bellows Development

Four prototype bellows were tested at Miniflex Corporation. In these tests, the set was removed and the precision of the compression was evaluated using shadowgraph data, as was reported last quarter. Miniflex has tested and shipped 46 production bellows to Los Alamos using specifications derived from this testing. The four prototype bellows were returned to Los Alamos and are undergoing further testing. A simulated plutonium material container having a screw lid and an O-ring seal was fabricated for the tests. The bellows is held in place inside the lid in a bracket (Fig. 1) making it easier to assure that the entire length of the bellows can be seen on film; and bellows deflection is measured using radiography. The completion of comparison evaluation testing using the Miniflex shadowgraph data and the Los Alamos radiography is expected in February. A report on the bellows repeatability and specifications derived from their performance will be issued by the Technical and Safety Assessment Division Statistics Group (TSA-1). Currently there are two long-term metal storage containers utilizing

bellows in the vault. A statistical sampling of metal containers containing bellows will be used to further evaluate bellows performance.

Fig. 1. Simulated material container for bellows testing.

Los Alamos is supporting the development of several new bellows designs at Miniflex. A design using a 10-ml stainless steel sheath with filtering material on the ends will be tested. This design should prevent the plutonium oxide from interfering with the bellows during compression. A 2-in.-diam bellows utilizing only three or four convolutions is being developed for stability and strength. The pressure range of this bellows will be from atmospheric to 100 pounds per square inch (psi). New materials such as Berylco will also be tested to determine if there is an increased linearity. Also, suspending the bellows from the center of its sheath to the can lid will be evaluated to determine the effect of this configuration on accuracy. A horizontal suspension of the bellows would allow some increase in the amount of oxide in each can.

Radiographic Capability

In November, Packaging project and Engineering Sciences and Applications (ESA) Division radiographic personnel traveled to VJ Technologies in Bohemia, New York, for acceptance testing of the real-time radiography system. They checked out quality of construction and operated all components. The system operated satisfactorily and was accepted. It was then dismantled and shipped to Los Alamos where it was received in January. Assorted unused control panels and equipment were removed from Room B-38 in the Building PF-4 basement

and other items were rearranged to make room for the radiographic unit. A work order was written to provide the necessary power supply. The real-time unit can then be set in place.

Automated Glovebox Can-Out System—Sandia Collaboration

The Packaging project has followed with interest the development of automated can-out systems at Savannah River Site (SRS) and Sandia/NM. Cooperation between Los Alamos and Savannah River on the SRS Bagless Transfer System was proposed by both laboratories, but sufficient funding was not provided for FY 1996. However, a collaboration between Sandia and Los Alamos that began some years ago has been revived. Sandia's Robotic and Intelligent Systems Center (ISRC) was originally tasked to develop a bag-out system for Rocky Flats. This led to Los Alamos cooperation in an automatic glovebox can-out (AGC) system designed to meet Los Alamos needs, with the intent that demonstrated technology would be made available to the DOE complex. The can-out system incorporates the electrolytic decontamination equipment described below. A robot inside a glovebox is used to transfer a cleaned welded material container from the decontamination chamber to an air lock. A second robot then retrieves the container from the air lock and places it into a boundary container to be welded. An automated orbital welder is also part of the system design. A design requirements document on the Los Alamos installation was approved in 1994. Installation and demonstration of this system in the TA-55 Plutonium Facility, Building PF-4, was delayed when funding was lost in FY 1995. With the restoration of funding for FY 1996, the Packaging project is working with ISRC personnel to update the design requirements and proceed with a hot demonstration (with radioactive materials) in PF-4, Room 124, using the project's sealed containers. Meetings have been held at Los Alamos and Sandia including ISRC staff and Packaging project welding and processing personnel to review the AGC operation and design. The system is to be installed in PF-4, Room 208, adjacent to the present processing gloveboxes.

Electrolytic Decontamination

Electrolytic decontamination is a technology that has been under development for several applications. The metal to be decontaminated (the anode), a stainless steel containment fixture (the cathode), and the sodium nitrate aqueous solution (the electrolyte) are configured as an electrolytic cell. 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 these applications. The potential for electrolytic decontamination to provide contamination-free containers as part of an automated system led to its integration with the Sandia can-out system, as described above. It is also being applied as the means of container decontamination in the ARIES pit conversion project. This fiscal year, electrolytic decontamination will receive funding from the Packaging project to provide a demonstration of its utility on Packaging project containers.

Preliminary demonstration tests of electrolytic decontamination of storage containers have been completed. Three empty welded material containers were contaminated in the glovebox line of the Packaging project and moved to the hood where the electrolytic decontamination equipment is demonstrated. There the containers were decontaminated to below the specifications for release into the laboratory room. For the sides of the containers, contamination was reduced from an average direct alpha contamination reading of 25,000 disintegrations per minute (dpm)/100 cm² and an average swipable alpha contamination reading of 5,000 dpm/100 cm² to no detectable and no swipable. 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 cm² and an average swipable alpha contamination reading of 10,000 dpm/100 cm² to less than the required 500 dpm/100 cm² direct and no swipable. The limit for release to the room is a 20-dpm swipable. After cleaning, these containers were helium leak-tested to 1×10^{-7} std cm³/s, as they had been prior to cleaning, and no leaks were detected.

Precipitate formed from contaminants was easily separated from the electrolyte. Also, the recycled electrolyte was analyzed by liquid scintillation and gas proportional alpha counting, and no plutonium was detected. Precipitate analysis verified plutonium contamination in the precipitate, as expected from other electrolytic decontamination experiments using sodium nitrate electrolyte at Los Alamos. These results mean that the electrolyte does not become a source of recontamination of the can.

Process development and improvement will continue on Packaging project containers. Tests are also being run on samples of flat welds such as are being considered for the next generation of Packaging containers.

Resource Conservation and Recovery Act (RCRA) Issues—Plutonium Disposition Methodology and Related Issues

The DOE-AL issued the Plutonium Disposition Methodology (PDM) in October 1995. The PDM mandates that plutonium-bearing materials with no present or planned programmatic use be dispositioned using 12 environmental, safety, health, nonproliferation, and economic criteria established by DP-22. Los Alamos is working with DOE-AL to develop a PDM Implementation Plan, which is tentatively scheduled for publication by March 31, 1996. As part of this implementation effort, Los Alamos is developing a quantitative method for optimizing recovery limits in relation to associated negative processing parameters, e.g., waste generation, personnel exposure, waste management and recovery costs, as a basis for establishing new discard limits. This process is being developed in conjunction with an effort by the DOE Office of Safeguards and Security (OSS) to develop policy on Safeguards Termination Limits that are derivative of the degree of difficulty of recovery (an aspect of "Attractiveness") of special nuclear material (SNM) from low-grade SNM-bearing materials. Discard limits established by Los Alamos will necessarily be below the Safeguards Termination Limits. Having discard limits in place is essential to the processing required for stabilization of plutonium for long-term storage. DOE-AL approval of the Los Alamos PDM Implementation Plan will cancel authorization to use the 1989 Economic Discard Limits currently in effect.

It is worthy of note that quantitative analyses performed in development of the first straw-man discard limit under Los Alamos's proposed methodology have confirmed an intuitive truth related to recovery and stabilization of plutonium-bearing materials. For hydroxide cake, two recovery passes, e.g., two dissolution episodes, result in a 34-fold reduction in transuranic (TRU) waste and an associated savings in waste management costs of \$400 K. These analyses compared the disposal cost of hydroxide cake containing 5205 g of plutonium to the disposal cost of dissolution process residuals containing only 143 g of plutonium subsequent to recovery. Estimated recovery costs of \$52 K in this instance were negligible in relation to the waste management cost savings. Of course, recovery also reduces other negative consequences of waste management such as environmental insult of land disposal and radiation exposure associated with waste management activities.

In addition to the development of discard limits, the PDM Implementation Plan will also provide for disposition of plutonium-bearing materials having no planned programmatic use and that are not discardable under DOE Order 5633.3B. Anticipated dispositions include (1) set aside for IAEA safeguarding, (2) ship to other sites for programmatic use or inventory consolidation, and (3) hold for further evaluation or resolution of individual inventory problems such as assay difficulties or shipper-receiver differences.

Finally, both Los Alamos and DOE-AL expect the PDM Implementation Plan to establish the point in processing where SNM-bearing materials become subject to the Resource Conservation and Recovery Act (RCRA), if they exhibit hazardous characteristics or contain hazardous constituents. It is the NMT Division's contention that SNM-bearing materials only become solid waste, and therefore potentially hazardous waste, when (1) they have no planned programmatic use *and* (2) they are determined discardable under DOE Order 5633.3B. This is particularly true at TA-55 where SNM recovery has been one of our primary missions since commissioning the facility.

Vault Prioritization

The report, "Analysis of LANL Options for Processing Plutonium Legacy Materials," number LA-UR-95-4301, by Stephen T. Boerigter and Nelson DeMuth, was published in January by TSA Division. The risk-assessment-methodology meeting for presentation of this information to the complex was set by the NMSTG for January 1996.

PROCESSING PROJECT STATUS

Project Management

During the first quarter of FY 1996, we felt the NMT Division growing pains and, as a result, fell behind on our projected work-off schedule. We attribute the slowdown to two specific areas:

- Redistribution of our already extremely lean staff to cover new FY 1996 programmatic growth opportunities arriving in the Division and
- Priority conflicts that caused our facility evaporator to operate at much lower level than was acceptable during the first quarter. This kept our process liquid effluent surge tanks full and limited our ability to process additional items.

We are refocusing and reprioritizing resources on this project and expect a full recovery to our advertised schedule before the end of the second quarter of operation.

The continued discussion on the Los Alamos 8-year versus 15-year remediation schedule was resolved in late December with a commitment by the DOE to find the extra funding to accelerate us to the 8-year schedule recommended by the Defense Nuclear Facilities Safety Board (DNFSB). It will likely take at least the remainder of the current fiscal year to advertise, interview, select, and train the appropriate personnel to affect a real acceleration to the 8-year schedule. As a result of our need to increase staff resources, we have completed a review of

our processing schedule and have issued a revision that reflects the slowdown necessary to hire and train new personnel.

Processing Operations

From an operational standpoint, we continue to process high-risk plutonium metal items through thermal stabilization (oxidation) and corrosive and reactive items through aqueous nitrate and chloride operations. As in the past, we recover the plutonium as an oxide (>50% Pu) and place it in a queue for packaging to the DOE long-term packaging standard. In addition, we continue to stabilize cellulose rags contaminated with significant amounts of Pu-238 by thermal decomposition (pyrolysis). This particular activity continues to eliminate a real fire-safety hazard in the facility and was not part of the original scope of the 94-1 response. This type of operational flexibility is encouraged and will be maintained for the safe operation of our facility. It further illustrates the need to factor into our current program the ongoing stabilization of currently generated materials.

In response to the DNFSB Recommendation for legacy material stabilization, our accomplishments this quarter consisted of the following:

- Oxidizing 26 reactive high-surface-area metal items. Our projected goal for the quarter was 73 items.
- Processing 23 high-priority process residues. Our projected goal for the quarter was 62 items.
- Completing the stabilization of the solutions category, with the exception of mixed-waste analytical solution items. We will continue to report activity, however, as we process currently-generated items.
- Processing 20 combustible items. These items were entirely Pu-238-contaminated rags although our goal was to process 3 legacy items this quarter.
- Processing 10 items from the miscellaneous process residue list. The first quarter goal is 5 items.
- Introducing an unsheltered container into the facility, thus meeting our first quarter goal in this area.

We processed no items from the categories of high-priority compounds, other compounds, and noncombustibles. The first quarter goals for these categories were 13 items, 4 items, and 4 items respectively. Year-end milestones for these categories are not considered to be at risk at this time.

Process Development and Demonstration

We are currently on schedule with the installation of a salt distillation prototype for NaCl-KCl salts, with the deployment of a pyrolysis unit for treating certain categories of noncombustibles, and for completing the demonstration of waste stream polishing for acidic chloride streams. We have not made much progress yet in the areas of developing and

demonstrating ultrafiltration for treating caustic effluents or for developing shielded equipment for handling high-exposure materials. Salt distillation, pyrolysis, and ultrafiltration will be funded under the 94-1 Lead Laboratory Technical Program Plan for research and development; and progress will be reported in that project quarterly.

URANIUM PROJECT STATUS

During the first quarter of FY 1996, we completed the ventilation construction project supporting the glovebox system in Wing 4 basement of the Chemistry and Metallurgy Research (CMR) Building at TA-3. We also completed a readiness assessment during this quarter. The facility management team provided authorization to operate the glovebox system. We now have a small-scale uranium packaging and recovery operation to meet goals set out in the 94-1 integrated program plan.

We also completed the first fit-up inspection for the glovebox procurement contract that was let in the fourth quarter of FY 1995. The schedule indicates that the boxes will begin to arrive next quarter.

The following items were stabilized and repackaged during the first quarter of FY 1996.

- Forty-five high-priority enriched-uranium residues were completed; 13 items were scheduled for FY 1996.
- One high-priority depleted-uranium item was processed; six items are scheduled for FY 1996.
- Two enriched-solution samples were processed; one was scheduled.
- Thirteen enriched-uranium gas items were processed; 28 items are scheduled.
- Four enriched-uranium combustible items were processed; none was scheduled.
- Three noncombustible enriched-uranium items were processed; one was scheduled.
- Ten enriched-uranium oxide items were processed; 154 are scheduled.
- One enriched-uranium metal item was processed; none was scheduled.