Sta 5
JUN 30 1999

ENGINEERING DATA TRANSMITTAL

Page 1 of 1
1. EDT 626642

2. To: (Receiving Organization) Distribution		3. From: (Originating Organization) PFP Process Engineering		4. Related EDT No.: N/A							
5. Proj./Prog./Dept./Div.: Facility Stabilization/PFP		6. Design Authority/Design Agent/Cog. Engr.: R. W. Szempruch		7. Purchase Order No.: N/A							
8. Originator Remarks: Review and Approve HNF-4762, Revision 0, Material Stabilization Characterization Management Plan				9. Equip./Component No.: N/A							
				10. System/Bldg./Facility: PFP							
				12. Major Assm. Dwg. No.: N/A							
11. Receiver Remarks:		11A. Design Baseline Document? ☐ Yes ☒ No		13. Permit/Permit Application No.: N/A							
				14. Required Response Date: N/A							
15. DATA TRANSMITTED											
(A) Item No.	(B) Document/Drawing No.	(C) Sheet No.	(D) Rev. No.	(E) Title or Description of Data Transmitted	(F) Approval Designator	(G) Reason for Transmittal	(H) Originator Disposition	(I) Receiver Disposition			
1	HNF-4762	-	0	Material Stabilization	N/A	1					
				Characterization							
				Management Plan							
16. KEY											
Approval Designator (F)		Reason for Transmittal (G)			Disposition (H) & (I)						
E, S, Q, D OR N/A (See WHC-CM-3-5, Sec. 12.7)		1. Approval 2. Release 3. Information 4. Review 5. Post-Review 6. Dist. (Receipt Acknow. Required)			1. Approved 2. Approved w/comment 3. Disapproved w/comment		4. Reviewed no/comment 5. Reviewed w/comment 6. Receipt acknowledged				
17. SIGNATURE/DISTRIBUTION (See Approval Designator for required signatures)											
(G) Reason	(H) Disp.	(J) Name	(K) Signature	(L) Date	(M) MSIN	(G) Reason	(H) Disp.	(J) Name	(K) Signature	(L) Date	(M) MSIN
		Design Authority				1	1	P. E. Roeger		6/29/99	75-50
		Design Agent				1	1	K. R. Herzog		6/29/99	74-45
1	1	Cog. Eng. R. W. Szempruch		6/29/99	75-55						
1	1	Cog. Mgr. M. W. Gibson		6/29/99	75-55						
		QA									
		Safety									
		Env.									
18.		19.		20.		21. DOE APPROVAL (if required)					
Signature of EDT Originator		N/A		Design Authority/Cognizant Manager		Ctrl No. N/A					
Date		Date		Date		☐ Approved					
						☐ Approved w/comments					
						☐ Disapproved w/comments					

S

Material Stabilization Characterization Management Plan

M. W. Gibson

Babcock and Wilcox Hanford Company

Richland, WA 99352

U.S. Department of Energy Contract DE-AC06-96RL13200

EDT/ECN: 626642

UC: UC-2050

Org Code: 15530

Charge Code: 100749

B&R Code: EW7040000

Total Pages: 23

Key Words: PFP, SNM, Inventory, 94-1, Characterization

Abstract: This document presents overall direction for characterization needs during stabilization of SNM at the PFP. Technical issues for needed data and equipment are identified. Information on material categories and links to vulnerabilities are given. Comparison data on the material categories is discussed to assist in assessing the relative risks and desired processing priority.

TRADEMARK DISCLAIMER. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof or its contractors or subcontractors.

Printed in the United States of America. To obtain copies of this document, contact: Document Control Services, P.O. Box 950, Mailstop H6-08, Richland WA 99352, Phone (509) 372-2420; Fax (509) 376-4989.

DOES NOT CONTAIN CLASSIFIED OR
UNCLASSIFIED CONTROLLED
NUCLEAR INFORMATION

Reviewing
Official / ADC: *[Signature]*

Date: *6/29/99*

[Signature]
6/30/99
Release Approval

Date

Release Stamp

Approved For Public Release

Contents

1.	INTRODUCTION.....	4
1.1	Background	4
1.2	Scope	4
1.3	Purpose	4
2.	Summary	5
2.1	Overall Summary	5
2.2	Future Characterization Needs	5
2.3	Technology Issues	6
3.	Characterization Requirements	6
3.1	Materials	6
3.1.1	Metals and Alloys	7
3.1.2	Oxides	9
3.1.3	Mixed Oxides and Fuel Pins	11
3.1.4	Solutions	12
3.1.5	Polycubes	13
3.1.6	Residues	14
3.1.7	Fluorides	16
3.1.8	Sources	17
3.2	Container Certification	18
3.2.1	Convenience Can	18
3.2.2	BTS Cans	18
3.2.3	Outer Welded Can	18
3.2.4	Other Containers	19
4.	Measurements	19
4.1	Materials	19
4.1.1	Assay	19
4.1.2	Impurities	19
4.1.3	Moisture	20
4.1.4	Isotopic	20
4.1.5	Mass	21
4.2	Containers	21
4.2.1	Dimensions	21
4.2.2	Leakage	21
4.2.3	Radiography	21
4.2.4	Gas Analysis	21
4.2.5	Visual Observation	22

4.2.6	Other	22
5.	Technical Issues	22
6.	Management of Data	22

1. INTRODUCTION

1.1 Background

In 1994, the Department of Energy (DOE) evaluated vulnerabilities associated with plutonium storage at Hanford's Plutonium Finishing Plant (PFP) as part of a complex wide initiative. The purpose of the assessment was to identify degradation in plutonium materials and packaging, and weaknesses in facilities and administrative controls that could potentially result in inadvertent release of plutonium, exposing workers or the public, or contaminating the environment. The primary concerns from this assessment were: 1) plutonium stored in unstable forms, 2) deterioration of storage containers, and 3) insufficient knowledge of packaging configuration and materials at PFP.

The Defense Nuclear Facilities Safety Board (DNFSB) had similar concerns, and issued Recommendation 94-1. The Board's concern was that the halt in production of materials to be used in nuclear weapons froze the manufacturing pipeline in a state that, for safety reasons, should not be allowed to persist without remediation.

A Plutonium Inventory Characterization and Implementation Plan for the Hanford plutonium inventory was prepared in 1995, and was updated through 1996. The recommendations from these plans were implemented through ongoing monitoring and surveillance, observations during thermal stabilization operations, and special testing (radiography). These plans also provided a basis for prioritizing storage risks and thus the process sequence for stabilization and disposition of the PFP inventory.

The PFP is now operational in a limited capacity, and this Characterization Management Plan reflects new requirements listed in section 1.3 and other inputs since the 1996 time frame.

1.2 Scope

The scope for this Plan includes the plutonium inventory in PFP, a discussion of existing characterization data available, a discussion of future characterization required, and a discussion on packages for storing and shipping plutonium materials. Characterization is defined as activities that result in pertinent data from process history records, ongoing inspection and surveillance, special testing (such as radiography), and information obtained during planned process operations. The inventory has been subdivided into eight categories (with some categories having sub-categories). Each subcategory is identified by groups of items that are listed in Tables in the Plan.

Requirements for each of the categories are listed, and in most cases, are referenced from existing Hanford documents. Measurement systems required are discussed and technical issues. Disposition options are defined, and container certification for each of the options is discussed.

Reference is also made to plutonium vulnerabilities identified in a recently issued Document (HNF-3541, Rev 0) that updates the Hanford vulnerability assessment. This is a continuation of those efforts recommended by Defense Nuclear Facilities Safety Board 94-1

1.3 Purpose

The primary purpose of this plan is to provide a basis for identifying and estimating the need for characterization resources that will be required at PFP. This is in support of the following objectives:

1. Minimize risk for continued storage
2. Development of the technical basis for stabilization processes utilized for the various materials
3. Support development of a final disposition plan for each of the material categories or sub-categories.
4. Provide a technical basis for prioritization of stabilization sequencing.

The Plan also reflects requirements for information, including DOE-MD Acceptance Criteria for dispositioning surplus weapons-usable plutonium bearing materials, the Savannah River Receipt Specification for APSF, and the draft revised DOE 3013 standard for long term storage of plutonium metals and oxides.

2. Summary

2.1 Overall Summary

The PFP inventory consists of 8 major categories of plutonium, with some groups containing sub-categories. Dispositioning these categories must be performed to meet acceptance criteria provided by DOE-MD, to store material at PFP in accordance with Operating Specifications Documents (OSD), and to meet criteria/specifications from a receiving site (e.g., SRS). Most materials currently stored at the PFP can be stabilized and stored to meet applicable OSD's. Characterization of materials will be performed according to the appropriate OSD during processing or preparation for storage/shipping. Ongoing surveillances of stored material and other characterization activities such as inventories and radiography also provide data. Additional OSDs are needed for processing residues such as ash, mixed oxides from fuel fabrication activities, polycubes, and solution stabilization.

Metals and oxides will be the primary products for storage and/or shipment. Those containing 30% or more total actinide (plutonium plus uranium) are acceptable to MD. Fabricated fuel (unirradiated or <200 watt days per MT) is also acceptable to MD. Materials containing less than 30% actinide will primarily be processed and packaged for shipment to WIPP. These include materials such as combustibles, miscellaneous compounds, ash, and sand, slag and crucible. Materials such as fluorides that cannot be accepted by either MD or WIPP, can be processed to an oxide, or if acceptable to SRS, shipped for further processing.

Other materials such as polycubes, mixed oxides, solutions, and miscellaneous metals can be processed to an oxide acceptable for storage at PFP or shipped to MD for disposition. Characterization of the above materials will be performed based on the disposition option selected for each item. Items to be declared as waste must be packaged to WIPP criteria. Materials to be shipped to MD need to be treated and packaged to meet MD Acceptance Criteria. For interim storage within the PFP, the OSD provides the appropriate requirements. The present OSD allows materials packaged prior to January 1, 1998 to be stored in original packaging. Items packaged since October 1, 1979 appear to be in good condition. Items in storage prior to October 1, 1979 pre-date the OSD and every effort should be made to expedite stabilization and repackaging.

Included in the narrative are specific references to those issues identified in the recently issued "Update on the Department of Energy's 1994 Assessment For the Plutonium Finishing Plant" (HNF-3541, REV. 0).

2.2 Future Characterization Needs

In this plan future characterization needs are separately discussed for metals and alloys, oxides, mixed oxides and fuel pins, solutions, polycubes, residues, fluorides, and sources. In proceeding with planning for individual categories of material and processing, the issues described herein are to be considered.

Methods and evaluations to proceed with defining these needs on a case-by-case basis either exist or are in progress. They include:

- Ongoing storage and surveillance per Operating Specification Documents.
- A gap analysis by the "Critical Mass" group to explicitly identify the information and data needed to show conformance with MD criteria.
- Specifications for development of an information management system (ZMITS, see section 6) currently being written.
- Process Flowsheet Documents, Process Control Plans, Blend Plans, and Facility Sample Schedules that are standard technical implementation documents for stabilization process engineering and operations.

This plan provides the bases for proceeding to more detailed estimates and timing of resources needed to conduct characterizations (these include sampling, laboratory analyses, inspections, and engineering analyses). Further detailed planning should address staffing, procurement of equipment, software implementation, and information management.

2.3 Technology Issues

Issues that impact the overall characterization program include the following:

1. Exclusive use of LOI for determining moisture levels is not a valid test for materials that contain metals or other compounds that gain weight (oxidize) upon heating. For these types of materials, special tests for moisture will be required, or a pre-oxidation step to ensure all metal is completely oxidized prior to performing the LOI.
2. Obtaining representative samples may be a problem for heterogeneous materials that are dispositioned to the MD program.
3. Metal that gains little or no weight may be decomposing via a hydriding type reaction.
4. Much of the inventory that also contains uranium does not have a uranium value assigned. In order for an item to be dispositioned to MD, the uranium content will also need to be known. Analytical methods for assaying uranium in the presence of plutonium may be costly.
5. Impurity analysis by the analytical laboratory is very limited. If a large number of analyses need to be performed, the analytical laboratory will need to acquire an Inductively Coupled Plasma Mass Spectrometer. (ICP-MS).
6. The OSD allows use of 7" food pack cans. Any material repackaged and destined to be sent to MD should be placed in a Hanford convenience can (HCC), since this can will fit into a 3013 container.
7. The 9000 plus items in the Hanford inventory can undergo some consolidation during repackaging. Many items within a category or sub-category that will be dispositioned to MD contain similar plutonium assays and isotopic content. Coordination of these consolidation efforts with MD should be encouraged.
8. A few of the issues identified in HNF-3541 may not be applicable to characterization. Those identified are 3.1.3.2.g, 3.1.3.4.m. Materials that are declared waste and must meet WIPP criteria will be shipped in vented drums.

3. Characterization Requirements

3.1 Materials

The PFP plutonium inventory consists of over 9000 items containing almost 4 metric tons plutonium and 4 metric tons uranium. The bulk weight of this inventory is over 17 metric tons of material. These items have been divided into eight categories. These are listed as follows, along with the subcategories:

1. Metals and Alloys
 - Metals
 - Alloys
2. Oxides
3. Mixed Oxides and Fuel Pins
 - Mixed Oxide
 - Fuel Pins
4. Solutions
5. Polycubes
6. Residues
 - Sand, Slag and Crucible
 - Compounds
 - Miscellaneous

- Combustibles
 - Ash
7. Fluorides
 8. Sources

Classified inventory listings are available for each category of material (subcategories are also shown for selected Tables) showing the groups of similar items, the plutonium content, the bulk weight, the uranium content, the ^{240}Pu content, and the heavy metal or actinide assay (Pu + U). The listings are arranged in descending order of the Pu + U content, since MD Acceptance Criteria specifies a minimum limit for total actinide content.

As a bridge between the Standards or basic requirements documents and criteria issued by offsite customers, the PFP Process Engineering group issues Operating Specifications Documents (OSD) that specify requirements that must appear in the implementing procedures. Each OSD lists the implementing procedures associated with a particular function (e.g., storage of nuclear material). Applicable Operating Specifications Documents are referenced in the following discussions of material categories.

3.1.1 *Metals and Alloys*

The bulk of the material in this category is relatively pure metal. Alloys account for an the additional items. Alloy metals include uranium, thorium, zirconium and aluminum. A few items do not identify the alloying metal. A classified listing is available specifying the groups of materials in this inventory. A number of the alloy groups appear to contain less than the 30% actinide limit required by the MD Acceptance Criteria. However, it is possible that some of these alloys contain uranium that is not listed in the inventory (e.g., items 16-17). A number of groups of items do not list the alloying constituent (*Ref. Pu vulnerability 3.1.3.4.b*). Other alloys, such as the aluminum alloys, contain <10% plutonium. These may be oxidized and immobilized for shipment to WIPP. The high ^{240}Pu content (22%) for the bulk of this Pu-Al alloy may prevent shipping this material to Savannah River for chemical processing.

An itemized listing of the metal and alloy inventory, planned during the implementation phase, should help identify items requiring priority attention (e.g., items stored in gloveboxes, items showing degradation in radiographs, weight changes).

3.1.1.1 Existing Characterization Data

The characteristics of metals are known with regard to their original state when placed in storage. Also known is that corrosion has occurred and the nature of the corrosion products, however, the degree of corrosion on all metal items has not been characterized.

Reports have been written describing monitoring activities of the existing metal and alloy inventory. The following reports describe these activities:

- WHC-SD-CD-TRP-067, "Plutonium Inventory Characterization Technical Evaluation Report", G. R. Wittman et al., June 1996. Describes the radiography of selected metal buttons and other metal pieces and chunks. A total of 20 items were examined. The report also describes work on ash; sand, slag and crucible (SS&C); and oxide.
- WHC-SD-CD-TRP-068, "Plutonium Inventory Characterization Technical Evaluation Report 2", G. R. Wittman, October 1996. Describes additional work on materials examined in the inventory. A total of 48 buttons were examined by radiography.
- HNF-SD-CP-TRP-70, "Plutonium Inventory Characterization Technical Evaluation Report 3" (DRAFT), E. P. Bonadie, R. W. Szempruch, May 1999. Describes additional work beyond Report No. 2, and discusses package conditions for five containers of metal that were opened in late 1996.
- HNF-3541, "Department of Energy's 1994 Plutonium Vulnerability Assessment for the Plutonium Finishing Plant" (DRAFT), January 1999. This report updates the vulnerabilities identified in the DOE Vulnerability Assessment (1994). Four vulnerability issues are open. Three of the vulnerabilities contain subsets (identified by a letter following the vulnerability number) that identify specific issues for various material categories.

Operating Specification OSD-Z-184-00013 Rev. J-3 "Special Nuclear Materials Storage", provides the information necessary for surveillance of metals and alloys, and repackaging these materials. The specification also includes a list of procedures that are covered by this specification.

3.1.1.2 Further Characterization Required

Additional process knowledge available in miscellaneous records, along with interviews of personnel knowledgeable in the history of metals and alloys in inventory (e.g., cognizant engineers) needs to be recorded and collected. This includes retrieval of laboratory analysis records, shipping/receiving records, processing records and any other information that may be archived elsewhere.

The monitoring reports referenced above indicate that degradation of plutonium metal during storage has been observed. Also many of the alloys contain uranium metal, which is also known to form hydride with when hydrogen is present. Continued examination of the inventory is necessary to determine the status of packages currently in storage. (*Ref Pu vulnerability 3.1.3.2.c*). As such, a combination of radiography and weighing is planned within the next year to identify the degree of corrosion with priority on those containers that have indications that degradation has occurred.

Many of the Pu-U alloys show no uranium content. Characterization of these alloys needs to be performed to determine if the alloy can be sent to MD (total actinide content >30%) or declared waste and prepared for shipment to WIPP (*Ref. Pu vulnerability 3.1.3.2.j*). Alloy materials eligible for discard to WIPP need to be converted to oxide, and meet WIPP criteria.

A few items listed under alloys do not identify the alloy metal. It will be necessary to identify the alloy for disposition to MD. If an item is to be dispositioned to waste, the alloy must be converted to an oxide and processed to meet WIPP criteria.

With regard to alloys, much of it is not well characterized, in particular alloy scrap. Information required to support decisions to ship to SRS, brush and repackage, thermally stabilize, or send to SRS include the fraction of constituents that are hydridable/non-hydridable, material included in scrap other than Pu and U, and degree of corrosion.

An additional issue identified for metals and alloys is the following:

- 85 items of non-Hanford origin are stored in shipping containers that have not been opened and inspected and/or assayed since receipt. Materials may not be adequately characterized and packaging configurations may vary from accepted practice. A number of these packages may contain reactive metals or semi-metallic compounds (*Ref. Pu vulnerability 3.1.3.4.c*).

Documents that list requirements for storage are:

- DOE-STD-3013-96, "Criteria for Preparing and Packaging Metals and Oxides for Long Term Storage," U.S. Department of Energy, September 1996
- DOE-STD-5XXX-99, "Stabilization, Packaging, and Storage of Plutonium Bearing Materials," (DRAFT) U.S. Department of Energy, March 4, 1999
- "Criteria for Interim Safe Storage of Plutonium (Pu) Bearing Materials," DOE Letter 96-PFP-004 J. E. Mecca, DOE-RL to President, WHC, February 13, 1996
- NMS-APS-98-0436, "APSF Receipt Specification", Interim Version 3 dated February 17, 1999.
- "Acceptance Criteria for Plutonium-Bearing Materials to be Dispositioned by Immobilization", Rev. 0, USDOE, December, 1998

3.1.1.3 Material Stages

Storage

All metal and alloy items are stored within the PFP. Evidence of slow degradation of the materials has been noted, which may be attributed to failure of containers and leakage, or improper atmosphere at the time of packaging (*Ref. Pu vulnerability 3.1.3.2.c*).

Condition of the metal and alloy inventory is reflected in reports referenced in section 3.1.1.1.

During Processing

Plutonium metal containers shall be opened and accumulated powder brushed and removed from the metal, and the powder stabilized as oxide. Oxides containing major impurities (such as from alloys) may be accumulated separately from oxides produced from pure metals.

For oxides containing less than 85% actinide (Pu + U), further characterization may be required to identify major constituents. It should be noted that the uranium content in stoichiometric uranium oxide (U_3O_8) is 84.8%, and items of pure actinide oxide that are principally uranium may have values slightly less than 85%.

If the container contents are found to consist of many small pieces of plutonium metal and potentially other materials (e.g., hydride and nitride) resulting from degradation, the container contents shall be converted to an oxide. This condition may be observed by radiography (e.g., little or no gain in weight, and evidence of large quantities of powder in the container) prior to opening the package.

Metal and alloy items will be repackaged to meet the applicable Operating Specifications Document (OSD) criteria. Radiography or historical process information indicates that a few packages have metal in direct contact with plastic (*Ref. Pu vulnerability 3.1.3.4.e*) contain metal stored in the same airspace as plastic (*Ref. Pu vulnerability 3.1.3.4.f*), or contain convenience cans with organic and/or epoxy coatings (*Ref. Pu vulnerability 3.1.3.4.g*).

Final Packaging

Packaging will be in accordance with the direction provided by the 94-1 Integrated Project Management Plan to use a Bagless Transfer System package, implemented via OSD-Z-184-00013 Rev. J-3, "Special Nuclear Materials Storage".

3.1.2 Oxides

For disposition, the combined plutonium plus uranium content needs to be known for those oxides that also contain percentage quantities of uranium. The categories of oxides can be divided into three sub-categories, as follows:

- Oxides containing 85% actinide or greater. Oxide in this category can be considered pure, and any impurities are within MD specifications (Table A-1 of the MD Acceptance Criteria document). As stated in section 3.1.1.3, pure oxides that are primarily uranium may have an actinide assay slightly less than 85%.
- Oxides containing 30% and up to 85% actinide. Oxide in this category contains impurities that may affect MD immobilization conditions. Impurities that impact immobilization include calcium, fluorine, carbon, and to lesser extent, magnesium. Other constituents in these oxides have often not been identified.
- Oxides containing <30% actinide. Oxide in this category will not meet MD criteria, and shall be prepared for disposal elsewhere (e.g., WIPP). Characterization of this material will be to WIPP requirements (RCRA in addition to radioactive and TRU characteristics).

Many of the inventory listings show only the plutonium content with the above sub-categories. A classified listing is available separately that summarizes the oxide inventory, and provides a listing of the groups of items in descending order of actinide (plutonium plus uranium) assay. A few groups of items show a higher actinide content than the stoichiometric amount for oxide (*Ref. Pu vulnerability 3.1.3.2.f*). This may be due to either weighing errors, or the presence of plutonium metal or semi-metallics in the container. During stabilization of these oxides, some items may increase in actinide concentration due to loss of volatile impurities (e.g., items slightly below 85% assay may increase to 85% or greater, and some items slightly below the 30% limit may increase to above this limit).

3.1.2.1 Existing Characterization Data

In determining disposition, the uranium content may be added to the plutonium value (e.g., items that contain low plutonium values may contain significant quantities of uranium that allows MD to process the material). MD acceptance criteria require the following material information, which is typically available:

- Chemical and physical form
- Best available elemental and isotopic composition

- Quantity (mass) of material
- Specific stabilization processing conditions (including date, process duration, equipment, temperature, and atmosphere).
- Test results (e.g., moisture, etc.)
- Source of material (e.g. PFP).
- Other information that may give insight to impurities present
- Americium content

Oxides are now being stabilized in the PFP, using procedures prepared in accordance to the OSD.

- Operating Specification OSD-Z-184-00013 Rev J-3, "Special Nuclear Material Storage", provides the specification for repackaging these materials. The specification also includes a list of procedures that implement the criteria.
- Operating Specification OSD-Z-184-00006 Rev. D-0, "Thermal Stabilization in Glovebox AC-21C" provides the specification for thermal stabilization of oxides in the glovebox.
- For oxides that are too low in actinide content to go to MD, OSD-Z-184-00020 Rev. C-3, "CH-TRU Waste Disposal" provides the specification for dispositioning oxide as waste. Treatment of the oxide is required to meet WIPP waste acceptance criteria.

Reports WHC-SD-TRP-067 and WHC-SD-CP-TRP-058 also discuss characterization activity on selected oxides in the PFP inventory (Section 3.1.1.1 gives the full reference for these reports).

3.1.2.2 Future Characterization Required.

Oxide lots can be defined based on similar characteristics. For lots that are likely to have them, fluorine, carbon, calcium, and magnesium should be identified. In addition, for oxides that have not been previously thermally stabilized, constituents that could affect furnace operation, such as organics, will need to be identified.

Accurate weights are needed for stabilized materials, and assays that are out of line (e.g., greater than stoichiometric amount) are suspect due to either weighing errors or non-stabilized materials still present (*Ref. Pu vulnerability 3.1.3.2.f*). Three items on the list show a plutonium value of 100% or more, indicating weight errors or mislabeling of material (*Ref. Pu vulnerability 3.1.3.4.a*).

Oxides that are less pure (e.g., <85 % actinide) will also require some knowledge of the impurities for use by Materials Disposition. In particular, the presence of calcium, fluoride, and carbon interfere with the MD immobilization process. Oxides that are <30 % actinide will be immobilized and packaged for shipping to WIPP and will need RCRA characterization.

The use of LOI testing as a measure of moisture content and material stability for oxides is not necessarily always valid. New more sophisticated moisture analyses, addressed later, are required (*Ref. Pu vulnerability 3.1.3.2.k*).

Test packages of oxides produced from the prototype VDC operations have shown evidence of corrosion of the inner container, even though the LOI for the material was within the acceptable specification limit (*Ref. Pu vulnerability 3.1.3.4.f*). These oxides will be surveilled under the existing program in the storage OSD and evaluated for the potential need to be re-stabilized to remove the corrosive component (most likely trace nitrates).

Documents that list requirements for storage are:

- DOE-STD-3013-99, "Criteria for Preparing and Packaging Metals and Oxides for Long Term Storage" (DRAFT), U.S. Department of Energy, 1999
- NMS-APS-98-0436, "APSF Receipt Specification", February 17, 1999.
- "Acceptance Criteria for Plutonium-Bearing Materials to be Dispositioned by Immobilization", Rev. 0, USDOE, December 1998.

3.1.2.3 Material Stages

Storage

All oxides are stored within the PFP and are currently being stabilized in accordance with the thermal stabilization OSD. During stabilization, some consolidation of oxide materials will be accomplished to reduce the number of items and packages in storage. Consolidation may be accomplished for materials having similar characteristics (e.g., isotopic assay, impurity types and concentrations, etc.).

During Processing

Major constituents in the oxide must be determined by either analysis or process knowledge if the material is to be dispositioned to MD. Oxide that is dispositioned to waste needs to meet WIPP waste acceptance criteria, and DOE Guidance for Safeguards Termination Limits (STL).

Final Packaging

Final packaging shall be in accordance with OSD-Z-184-00013 "Special Nuclear Materials Storage" which will implement DOE Standard 3013-96 or successor standards.

3.1.3 *Mixed Oxides and Fuel Pins*

Although much of the mixed oxide and fuel pins contain low percentages plutonium (e.g., <30 %), the uranium content is usually high, and thus the materials can be dispositioned to MD for immobilization. Mixed oxides that are not encapsulated in fuel pins, and contain uranium in the form of UO_2 , should be oxidized to the more stable U_3O_8 . Mixed oxides that contain other constituents (e.g., oxides that contain <82 % actinides assuming that the uranium has been oxidized to U_3O_8) need further characterization.

The list of the groups of mixed oxides and fuel pins is available separately in a classified listing. The listing is in descending order of actinide concentration. A number of items do not list a uranium value, even though the description shows the presence of uranium. Since uranium is usually the major constituent, these items may be relatively high purity once the uranium value is added. Items that contain little or no uranium may be misplaced. An itemized listing of the MOX inventory, planned during the implementation phase, should help identify items requiring priority attention.

3.1.3.1 Existing Characterization Data

Records exist for characterizing the fuel pins. A number of non-FFTF fuel pins have been identified as containing carbides, alloys of unknown metals, possibly some fuel pins bonded with sodium metal, and one item has been identified as possible spent nuclear fuel (*Ref. Pu vulnerability 3.1.3.2.e*). Some MOX items show evidence of corrosion in the inner containers (*Ref. Pu vulnerability 3.1.3.3.e*). For the remainder of the mixed oxides, data is available for a substantial portion of the inventory based on prior stabilization history and potential impurity data.

However, powders and scrap that are exposed directly to air may need to be heated to convert any UO_2 to U_3O_8 (a more stable form of uranium oxide).

Powders and scrap that do not meet the characterization of mixed oxide (MOX) can be handled in the same manner as oxides described in section 3.1.2 (usually MOX is UO_2/PuO_2 mixture sintered at a high temperature, however, exposure to air may result in severe degradation of the UO_2).

3.1.3.2 Future Characterization Required

Any future characterization of fuel pins needs to be based on requirements defined in the MD Acceptance Criteria. Process knowledge should be adequate to meet characterization requirements for much of this inventory. Fuel pins described in the vulnerability referenced above need to be characterized to the greatest extent possible by process knowledge. They will be inspected for integrity of cladding and packaging before disposition. There remains a possibility that these materials may need to be processed for stabilization, and an applicable OSD for the process would then implement characterization requirements to meet the standards.

Mixed oxide lots can be defined based on similar characteristics. Scrap material should be checked for carbon, fluorine, calcium, and magnesium, and the Pu and U should be assayed.

3.1.3.3 Material Stages

Storage

Most fuel pins may be stored with no further stabilization if they have an adequate quality and surveillance history to assure integrity of the fuel cladding.

MOX can be dispositioned as oxides in the same manner as described in section 3.1.2, heeding prior needs for a valid moisture analysis method.

During Processing

MOX processing has the same considerations as oxides.

Final Packaging

Items not in the form of fuel pins may be shipped as oxide per section 3.1.2.

3.1.4 *Solutions*

Solutions must be stabilized by converting plutonium (and in some cases uranium and/or thorium) to a stable solid form. Either precipitation or direct calcination can accomplish this conversion. Depending on the process used, characterization requirements may vary. For pure plutonium nitrate solutions, direct calcination may be employed, producing a pure actinide oxide that can be stabilized for long term storage (and meet MD requirements). For the more impure solutions, direct calcination may not be a viable option, since the process is sensitive to many impurities (e.g., chloride impurity will corrode the equipment).

The magnesium hydroxide co-precipitation process is not as sensitive to the presence of impurities, however, oxides produced by the process contain 60-70 % actinide, with the remainder being magnesium hydroxide (magnesium oxide after stabilization). The precipitate is stabilized to meet DOE storage criteria (DOE-STD-3013-99). Low assay precipitate may not meet minimum actinide content and will need to be processed for disposal at WIPP.

A listing of the groups of plutonium solutions is available separately in a classified form. The solutions are listed in descending order of plutonium concentration (gram per gram). One item on the list has been identified as an organic liquid that will require special treatment. Another item, which is listed as fuel grade, appears to be weapons grade material since the ²⁴⁰Pu content is listed as 5.9%. An itemized listing of the solution inventory has been prepared, and identifies solutions that need priority attention.

3.1.4.1 Existing Characterization Data

Many items have detailed chemical composition data. Flushes, filtrates, and others only have Pu values.

Operating Specifications documents (OSD) have been prepared for removing solutions from storage containers and handling waste solution containing organics. Applicable documents are as follows:

- OSD-Z-184-00012, Rev. D-1, "Load-In of Plutonium Solutions to Hood HC-227-S From Containers"
- OSD-Z-184-00019, Rev. D-0, "Absorption and Packaging of Organic Solutions".

A classified listing is available that itemizes all solutions in the inventory and provides additional characterization information. From this report, as well as information from other sources (e.g., HNF-3541), an order of priority may be established for processing solutions for conversion of plutonium to a stable oxide. The continued storage of solutions shows a number of vulnerabilities. These include the following:

- Recent checks show that not all plutonium solution storage containers have vent clips installed (*Ref Pu vulnerability 3.1.3.1*).
- Solution containers may be corroding, and the design life of the container is not quantified (*Ref Pu vulnerability 3.1.3.3.b*).
- Storage container life for solutions containing chlorides is unknown (*Ref. Pu vulnerability 3.1.3.3.g*).
- Pu bearing solution storage containers are not adequately characterized (*Ref. Pu vulnerability 3.1.3.3.h*).

- Integrity of polyethylene bottles inside 99 of the 431 Pu-bearing solution storage containers is unknown (*Ref. Pu vulnerability 3.1.3.3.i*).
- Not all solution storage cans were designed to the same criteria (*Ref. Pu vulnerability 3.1.3.4.I*).

Completion of solution processing will close out all of the above listed vulnerabilities.

Characterization of the product oxide may be performed using those requirements stated in section 3.1.2. Requirements for stabilization of the oxide are found in OSD-Z-184-00006 Rev. D-0, "Thermal Stabilization Glovebox AC-21C".

3.1.4.2 Future Characterization Data

Additional characterization needed for processing solutions include:

- Identify solution type where not explicitly known (e.g., containing chlorides, organics, etc.)
- Identify magnesium oxide concentration in stabilized product
- Check for fluorine and calcium in products (by lot)
- Identify type and concentration of organic solutions
- Parameters needed for process control.
- Integrity of polyethylene containers

An OSD will be prepared and issued for the magnesium hydroxide process that implements specifications for the needed characterization. The magnesium hydroxide precipitation process requires additional information on acid concentration (and in a few cases, the caustic concentration). The direct calcination process, if used, may require analysis for impurities in many of the solutions to ensure that only acceptable feeds go to the VDC.

For solutions containing chloride and/or caustic, or substantial quantities of organic material, the path forward is not yet defined (*Ref. Pu vulnerability 3.1.3.4.h*). Among others, data will be required for chloride concentration, affect of caustic on precipitation process, and thorium concentration.

3.1.4.3 Material Stages

Storage

Solutions are currently stored in vented containers. The solutions are undergoing slow decomposition by radiolysis, and some evaporation. Solutions stored in plastic liners within the container may have also degraded the plastic by radiolysis, perhaps to failure.

The more dilute solutions are less susceptible to degradation by radiolysis, but may be more prone to degradation from chemical attack, depending on the impurities present (e.g., such as chlorides).

During Processing

Conditions during processing are being developed for processing by magnesium hydroxide precipitation. Experimental work has commenced in accordance with an approved test plan. These experimental results will lead to preparation of an Operating Specifications Document (OSD) and implementing procedures.

Final Packaging

The product from solutions stabilization will be a stabilized oxide that will be handled in the same manner as the oxide described in section 3.1.2 above or per WIPP acceptance criteria when applicable.

3.1.5 Polycubes

Polycubes are polystyrene forms impregnated with plutonium oxide, with some also containing uranium oxide. These surplus materials were used in criticality experiments.

Because of the continuing radiolysis of the polystyrene, the polycubes must be kept in vented containers. Treatment of the polycubes includes calcination, with catalytic reaction scrubbers in the offgas stream. The Los Alamos National Laboratory (LANL) is developing the final treatment method.

A listing of the groups of polycube materials is available separately in a classified format. The list is in descending order of actinide (plutonium plus uranium) concentration. An itemized listing of the polycube inventory, planned during the implementation phase, may help identify items requiring priority attention.

3.1.5.1 Existing Characterization Data

The Los Alamos National Laboratory staff plans to deliver a process unit to the PFP and train PFP operators. Los Alamos process development testing provided detailed information on thermally stabilized polystyrene and offgas constituents. A WHC report, WHC-SD-CP-TI-204, Rev. 0A, "Stabilization of Polycubes Engineering Study", W. H. Lewis, Sept, 1996 also provides characterization information. Although the polycubes are well characterized, they are not well packaged. The generation of hydrogen and hydrocarbons by these items remains a concern (*Ref. Pu vulnerability 3.1.3.2.b*). Recently, the vents on the containers or polycubes had to be replaced due to deterioration of the glue attaching the vent to the container (*Ref. Pu vulnerability 3.1.3.3.m*).

3.1.5.2 Future Characterization Required

No additional characterization of the material should be required. The product from processing the polycubes will be plutonium oxide that can be stabilized in accordance with oxides in section 3.1.2 above. An OSD will be prepared once the LANL work is complete.

3.1.5.3 Material Stages

Storage

Polycubes are currently stored in vented containers, however, the packages do not provide an adequate storage configuration (many are in single, vented food pack cans) (*Ref. Pu vulnerability 3.1.3.2.b*). The vents on the cans are glued on and are prone to failure (*Ref. Pu vulnerability 3.1.3.3.m*). Movement of these containers is currently restricted. Operating Specifications Document OSD-Z-184-00013 Rev. J-3 "Special Nuclear Materials Storage" provides information for storage of polycubes.

During Processing

Conditions during processing are being developed by LANL. The Operating Specifications Document (OSD) and the implementing procedures will be prepared after receiving the necessary information from LANL.

Final Packaging

The product from polycube processing will be an oxide that will be handled in the same manner as the oxide described in section 3.1.2 above.

3.1.6 Residues

Residues are divided into five sub-categories. These are sand, slag and crucible; compounds; miscellaneous; combustibles; and ash. Most of the items in these sub-categories contain low percentages plutonium, and will be dispositioned as waste. A few items are sufficiently high in plutonium content to meet MD criteria. Characterization is difficult because of the diverse nature of materials associated with plutonium.

A listing of the groups of materials are available separately in a classified form. The items are presented in descending order of actinide (plutonium plus uranium) content for each sub-category. An itemized listing of residues inventory, planned during the implementation phase, may help identify items requiring priority attention.

The sand, slag and crucible (SS&C) category also contains miscellaneous scrap and analytical lab residues that may not be directly related to SS&C. By definition, SS&C may contain calcium and plutonium metal, and unreacted fluoride powders. The reactivity of these materials in storage is of concern (*Ref. Pu vulnerability 3.1.3.2.a*). A small portion of this inventory (~9%) has been cemented with no unusual events noted.

The Compounds sub-category contains 13 items of plutonium scrap contaminated with beryllium or thorium. These materials were probably isolated to prevent beryllium or thorium contamination from entering the plutonium

recovery process during former production operations (no longer a concern for stabilization). Disposition of these materials will depend on the plutonium content, and the form of the material (e.g., metal, alloy, oxide, etc.).

The Miscellaneous sub-category contains 28 items. Some of the materials are described as metal and may be pyrophoric. Disposition of these items will depend on the plutonium content after stabilization. Those items identified as metal need to be converted to the oxide (*Ref. Pu vulnerability 3.1.3.2.d*).

The Combustibles sub-category contains 12 items consisting of graphite or plastics and paper. These items can be dispositioned via the appropriate processes after opening the container and deciding the proper stabilization process (stabilize for shipment to MD or package as waste). The presence of plastics and paper in the presence of concentrated plutonium materials may result in generation of flammable hydrogen and hydrocarbons (*Ref. Pu vulnerability 3.1.3.2.g*). Dispositioning these materials as waste results in the materials being dispositioned in vented containers. The material waste package would need to meet WIPP Acceptance Criteria.

The Ash sub-category contains 990 items. It consists of residue collected after combustible materials have been treated in an incinerator. Characterization of the ash can be very difficult because of the diverse nature of the feed materials to the incinerator. Ash is not an acceptable material for disposition to MD and must be dispositioned as waste. Approximately 5% of the ash has been cemented. As waste, the material packaging must meet WIPP criteria. In lieu of characterization, WIPP allows the use of Acceptable Knowledge for waste disposition.

3.1.6.1 Existing Characterization Data

Materials in the Residue category will be difficult to characterize, and most will be dispositioned as waste. Operating Specification OSD-Z-184-00013, Rev J-3, "Special Nuclear Material Storage", contains information for storage until the materials are treated and packaged as waste.

Operating Specification Document OSD-Z-184-00006 "Thermal Stabilization in Glovebox AC-21C", provides information on thermal stabilization.

Operating Specifications Document OSD-Z-184-00039 provides information for processing sand slag and crucible (SS&C) for dispositioning to waste.

A large portion of the ash sub-category is ash from Rocky Flats. Data on ash still held at Rocky Flats may help characterize this material. A recent paper has compared characteristics of ash from Rocky Flats with ash from LANL. The ash samples were found to be remarkably similar.

Reports WHC-SD-TRP-067 and WHC-SD-CP-TRP-058 also discuss characterization activity on selected ash and SS&C in the PFP inventory (Section 3.1.1.1 gives the full reference for these reports).

3.1.6.2 Future Characterization Required

Residues will be either thermally stabilized if >30wt% plutonium, or discarded via cementation or "pipe and go. Further characterization may be required depending on the choice for final disposition (e.g., cementation, "pipe and go") to WIPP. Similar to the other material categories, much of the characterization required will be implemented through the processing, storage, or disposal OSD's. Except for SSC, which has no additional needs, the following tabulation indicates characterization needed for residues:

	Scrap	Compounds	Miscellaneous	Combustibles	Ash
Assay to determine actinide %	Also check for Pu metal	Also check for Th & U	X	X	X
Process knowledge & prior stabilization history, LOI, etc.		X	X	X	
Review nature of material for decisions on treatment	X			X	
If > 30% review suitability for furnace stabilization	X	Also check for C, Ca, F, Mg	Also check for C, Ca, F, Mg	Also check for C, Ca, F, Mg, organics	

	Scrap	Compounds	Miscellaneous	Combustibles	Ash
If < 30%, RCRA characterization for WIPP disposal	X	Also reactivity in water for cementation			X

3.1.6.3 Material Stages

Storage

Material storage is covered by OSD-Z-184-00013 Rev. J-3, "Special Nuclear Materials Storage". If items are identified as reactive, corrosive, or degradable through radiolysis, and/or contain finely divided actinide metal, other pyrophoric materials, or explosive or shock sensitive materials, storage is not allowed, in accordance with the OSD.

During Processing

Conditions during processing have been developed for SS&C (see OSD-Z-184-00039 Rev. B-0, "Sand, Slag and Crucible Cementation Process, PFP"). Processing of other materials may be accomplished by use of procedures such as those for metal and oxide. Some materials (e.g., incinerator ash) may require special procedures for stabilization and treatment.

Final Packaging

Final packaging will be accomplished either for dispositioning to MD or to waste.

Interim storage will also be affected by the disposition option selected. Materials in these sub-categories, if not declared waste, may be processed and packaged to meet the OSD-Z-184-00013 Rev J-3 "Special Nuclear Materials Storage".

3.1.7 Fluorides

The Fluoride category contains 13 items. This material form is not acceptable for either disposition to MD (fluoride compounds are not acceptable) or waste (plutonium levels too high to meet waste acceptance criteria). These materials may require treatment to remove the fluoride component. A possible treatment is metathesis of the fluoride to a hydroxide using potassium hydroxide.

An alternative is to ship the fluorides to SRS for treatment (process through FB Line). A listing of the groups of fluorides is available in a separate classified form. Most of the items contain a high plutonium assay. An itemized listing of the fluoride inventory, planned during the implementation phase, may help identify items requiring priority attention.

3.1.7.1 Existing Characterization Data

Plutonium fluorides are covered by an exception to OSD-Z-184-00013 for special nuclear materials storage. The plutonium content in PuF_4 is approximately 76%, thus those items containing higher percentages of plutonium may contain a significant percentage of oxide. Oxide containing fluoride as a lesser constituent may be acceptable to MD, if the quantity is manageable through mixing with non-fluoride containing oxides.

3.1.7.2 Future Characterization Required

Items containing less than 76% plutonium also contain unknown impurities. These may include calcium metal, magnesium oxide, or other materials used in metal reduction operations. If conversion to an oxide is necessary, characterization can be accomplished on the material after conversion is complete. Direct conversion of fluorides to metal is not currently allowed by the EIS.

If this material is converted to oxide, it can then be handled like material in section 3.1.1, and acceptable to MD. Disposition of fluorides needs to be coordinated with MD. Further discussion of fluorides is found in section 4.1.2.

3.1.7.3 Material Stages

Storage

Although this material has not been fully characterized, both trivalent and tetravalent fluorides are stable materials. One item is identified as PuF_3/UF_6 , and should be characterized to determine if the more reactive UF_6 component is actually present (*Ref. Pu vulnerability 3.1.3.4.a*).

During Processing

Conditions for processing fluorides have not yet been identified. The high neutron level associated with handling fluorides is the principal issue in processing these materials. Conversion to an oxide will substantially reduce the neutron radiation, particularly for those items that are high % ^{240}Pu .

Final Packaging

Final storage of the material will be in the oxide form. Storage conditions will be the same as described in section 3.1.2.

3.1.8 Sources

A listing of radiation sources is available separately in a classified form. These sources may be metal, oxide, fluoride, and possibly other materials. Most of the listed sources show a high plutonium concentration indicating that they are mostly oxides. One group of sources also contains a substantial amount of uranium. An itemized listing of the sources inventory, planned during the implementation phase, may help identify items requiring priority attention.

3.1.8.1 Existing Characterization Data

Many of the sources were received from off site. Existing data may be available from the shipping papers and original registration documents for each of the sources, if still available. Sources are usually encapsulated (sealed) and contain a stable form of plutonium.

3.1.8.2 Future Characterization Data

Older sources that are sealed may begin to leak due to pressure from helium generation caused by radioactive decay, or from slow corrosion over many years caused by trace impurities. Sources may be surveyed, and older sources can be processed early whenever processing begins.

Characterization needs include:

- Integrity of the casing
- Integrity of Pu metal in the source (radiograph)
- Assay, composition and form of "Encapsulated Pu Compounds and/or Carbides"
- Assay, composition, and RCRA or carbon content of "General Combustible/Graphite Scrap" source

3.1.8.3 Material Stages

Storage

Storage of sources, even though they may not have been completely characterized, represents a low risk. Leaking sources have a higher risk, and can be selected for processing first.

During Processing

Sources when processed will most likely become the same as materials in the oxide category. Stabilized sources would meet the requirements found in section 3.1.2. This includes metals and semi-metallic compounds that would be converted to oxide for disposition.

Final Packaging

Final packaging would be the same as for section 3.1.2.

3.2 Container Certification

Those data required by the MD Acceptance Criteria will be recorded. These criteria include the following:

- Fill gas composition of each container
- Leak test data
- Package configuration
- Date of packaging
- Initial measured radiation field
- Package weights (both tare and net)
- Measured external dimensions
- Unique identification numbers (e.g., bar code TID, etc.)
- Lot number
- Site location

3.2.1 Convenience Can

Specifications for the Hanford Convenience Can are found in OSD-Z-184-00013, Rev J-3 "Special Nuclear Materials Storage". The Hanford convenience can package consists of four cans; a gasket free crimp sealed material can overpacked by a second crimp sealed can which is bagged out of the glovebox in polyethylene. The package is completed outside the glovebox by application of the third and fourth crimp sealed cans.

The Hanford convenience can is designed to fit into a 3013 can for long term storage. This arrangement meets OSD-Z-184-00013 Rev J-3, "Special Nuclear Materials Storage".

A number of packages in the inventory do not meet present storage requirements.

Approximately 5% of the metal and alloy packages have gained more than 5 grams, indicating air leakage (*Ref. Pu vulnerability 3.1.3.3.i*), and seals on containers may be failing based on the observed weight gains (*Ref. Pu vulnerability 3.1.3.3.c*). These failures may also be due to high temperatures and radiation conditions from high activity (high ²⁴⁰Pu) material (*Ref. Pu vulnerability 3.1.3.3.d*).

As determined by radiography, a number of inner storage containers have paneled (partially buckled), indicating a reaction of metal with the container atmosphere (*Ref. Pu vulnerability 3.1.3.3.a*). The VSIS system will not detect storage containers that are paneled (*Ref. Pu vulnerability 3.1.3.3.k*). It should be noted that only straight walled cans have paneled. Those with a ribbed wall have not exhibited this phenomenon.

A few containers packaged prior to 1983 provide only a single contamination barrier, and a few containers may weigh more than the maximum mass analyzed for the technical basis on gas generation (*Ref. Pu vulnerability 3.1.3.4.k*). Four containers may contain metal in direct contact with plastic (*Ref. Pu vulnerability 3.1.3.4.e*).

3.2.2 BTS Cans

The inner storage can is based on a SRS system that is being adapted to PFP storage constraints.

3.2.3 Outer Welded Can

The outer welded can is currently envisioned as being implemented as a "just-in-time" application prior to shipping product to SRS. Investigation is currently underway for use of TIG welding in lieu of the laser welding used by SRS. Specifications for the outer welded can are currently provided in DOE-STD-3013-96, which is being revised and taken over by DOE-EM. The standard provides the specifications for long-term storage (e.g., 50 years) of stabilized plutonium materials. The can is fully welded, with no plastic or organic materials in the can.

3.2.4 *Other Containers*

A variety of other containers such as 55 gallon drums will be used for transfer and storage of material. In most cases, only visual inspection will be required.

4. **Measurements**

4.1 **Materials**

Measurements consist of assay of the materials for plutonium (and uranium if present), impurity analysis (major impurities only), moisture analysis, and mass measurements. For each material, there may be more than one option available for determining the measurement to be used.

4.1.1 *Assay*

The precision and accuracy of any assay analysis is not of great concern, as long as the results are within a couple of percentage points (e.g., mixtures containing 40% Pu and 40% U as oxide are adequate if the assay shows a range of 39-41%). Nuclear Materials Management may request higher precision if the analyses are used for inventory purposes.

Chemical Assay

Although there are plutonium values assigned to each of the inventory items, many do not have a uranium value assigned. However, the uranium content may be available from other records. Assuming that uranium assay is needed, this can be done via wet chemical analysis, particularly if percentage quantities of plutonium are present. This particular analysis is labor intensive, and would be used sparingly.

Nondestructive Assay

Non-destructive assay methods are the easiest and least costly methods for determining plutonium and uranium content. Because of the weak signature of uranium isotopes, this could not be easily accomplished in materials with percentage quantities of plutonium present. For those materials containing a mixture of plutonium and uranium, a separation may need to be made in order to analyze for the uranium via NDA.

Other

Mass Spectroscopy may also be used for assay, using isotopic dilution. Selection of this method would depend on the precision and accuracy required, and the availability of equipment. The PFP analytical laboratory has very limited capability, and samples may need to be prepared for analysis by other laboratories on site.

4.1.2 *Impurities*

Impurities in minor concentrations (e.g., <1%) should be of little concern. Table A-1 of the MD Acceptance Criteria lists specification limits for selected blended feed impurities.

Uranium and Other Actinides

Uranium assay is discussed in section 4.1.1.

Americium content is required per section 4.4.2 (8) of the MD Acceptance Criteria. This may be calculated by determining (or knowing) the ^{241}Pu content and calculating americium growth from the last date of separation. Confirmatory analyses can be performed via radioassay.

There may be additional items that contain other actinides (e.g., neptunium) that may require special analytical laboratory support. These will be evaluated after review of each item in a material category, and if present in significant quantities, may require additional analyses.

Emission Spec or ICP

Analysis of impure materials for determining other constituents will be necessary. At the present time, the Analytical Laboratory does not have the capability for analyzing materials for significant impurities (defined as impurities of >1%). If only a few sample are required for determining these constituents, then the lab can separate the plutonium, and send the sample to the 222-S lab for ICP analysis. If a large number of samples require analysis,

this may not be practical. The separation and shipment of samples to another lab may be costly in terms of labor required for performing the separation, performing the analysis, and delays in receiving the results from the analysis. Depending on the number of items requiring analyses, an ICP may be required in the PFP analytical laboratory.

Carbon Analysis

Capability to perform carbon analysis on inventory materials does not presently exist. Existing equipment has not been operated in many years, and needs spare parts, which may not be available. Although carbon is a specific impurity of concern to MD, stabilization of most of the materials will remove essentially all carbon. Materials containing high carbon, such as incinerator ash and graphite, should be treated as waste and prepared for shipment to WIPP. Some smaller quantities of graphite may be thermally stabilized for removal of carbon.

Other Analyses

Analyses for fluorine and calcium may be required, however, process knowledge may also be used for identifying those materials containing significant amount of these impurities. Table A-1 of the MD Acceptance Criteria lists the specification limits for calcium. For oxides that may contain small amount of fluoride, further consultation with MD is necessary. For small quantities, MD may be able to blend small quantities of oxides containing fluorides. Materials such as sand, slag and crucible and plutonium fluoride are specifically excluded from dispositioning to MD.

Chloride analysis on solutions may be performed using a specific ion electrode. Analysis of solids for chlorides will depend on solubility of the chloride and level of detection required.

Alloys and oxide mixtures from alloys may need to be analyzed to determine the amount of alloying agent present. In many cases this information may be available from existing records, however, there may be items that the quantity of alloying agent is not known.

4.1.3 Moisture

The determination of moisture in plutonium oxides has been performed using loss on ignition (LOI), however, this method does not necessarily result in providing stable materials for storage (*Ref. Pu vulnerability 3.1.3.2.k*). Development work being conducted at LANL and elsewhere is evaluating alternative methods for determining moisture content. Depending on the method selected as a DOE standard, additional equipment may be necessary for determining moisture content of stabilized plutonium oxides.

The technique at the moment for moisture analysis is LOI. The analytical laboratory is equipped to perform this analysis.

Thermogravimetric analysis (TGA) may also be used for determining moisture content, however, the precision of the analysis may not be sufficient to meet the DOE 3013 requirements. A TGA capability will be available in the PFP (plutonium laboratory).

A supercritical CO₂ extraction method is being implemented in FY 2000. In addition, neutron moderation is being considered, but it requires development before being deployed for production support.

4.1.4 Isotopic

All or almost all of the inventory items have a plutonium isotopic ratio assigned. If confirmatory analyses are required for any lot or group of items, an analysis can be performed using mass spectroscopy, radiochemistry or NDA. The precision and accuracy of the analysis required may decide the method selected.

Analysis of uranium for isotopic content may be performed by NDA or mass spectroscopy. Analysis by NDA may be limited, if percentage quantities of plutonium are mixed with uranium.

If an ICP-MS instrument is purchased for the analytical laboratory, it also has the capability of performing isotopic analysis for both uranium and plutonium.

4.1.5 Mass

Mass measurements will be required for a significant portion of the inventory. These include the net weight of the material, the weight of each container holding the material, and the gross weight of the package. All measurements will need to be to the nearest gram, with a precision of ± 0.1 gram.

Pu Weight

Plutonium weight should be available for each item in the inventory. If confirmatory analyses are required, the plutonium weight will be determined by multiplying the net weight by the plutonium assay. All values will be reported to the nearest gram. Plutonium weight limits are found in OSD-Z-184-00013 Rev. J-3, "Special Nuclear Material Storage".

U Weight

Uranium weight may not always be available, and uranium analysis may be required for items containing normal or depleted uranium. For enriched uranium, inventory records should provide the necessary information. Enrichments may vary, and uranium assay may be required for enrichments of less than 90% ^{235}U .

Thorium and Other Actinides

Considerations are the same as for uranium.

Net Weight

Net weights shall be determined using a balance that is calibrated to traceable standards (usually to NIST standards), with a precision of ± 0.1 gram.

Gross Weight

Gross weights for all convenience cans shall be determined using a balance that is calibrated to traceable standards (usually to NIST standards), with a precision of ± 0.1 gram.

4.2 Containers

The food pack can SNM storage system used at the PFP is intended to provide two contamination free barriers to support interim storage in vaults, and the majority of the stored material is packaged in this configuration. Inventory items packaged before October 1, 1979 are allowed to remain in storage per OSD-Z-184-00013, Rev. J-3 "Special Nuclear Materials Storage," but may not be packaged in accordance with the specifications for material packaged today.

4.2.1 Dimensions

For material that is stabilized and/or repackaged, the container shall meet conditions provided in OSD-Z-184-00013 Rev. J-3, "Special Nuclear Material Storage".

4.2.2 Leakage

For material that is stabilized and/or repackaged, the container shall meet conditions provided in OSD-Z-184-00013 Rev. J-3, "Special Nuclear Material Storage".

4.2.3 Radiography

Packages may be radiographed to determine status of the package and detect unusual conditions. For example, it can determine if a package has the minimum number of barriers, if corrosion is apparent to one or more of the containers, and if bulging or paneling is occurring to one or more of the inner cans.

4.2.4 Gas Analysis

The analytical laboratory does not have the capability to perform sampling and analysis of gases from the head space of SNM packages.

4.2.5 *Visual Observation*

Upon opening packages in the glovebox, operations personnel need to be alerted to look for unusual features of both the inner cans and the material itself. This may include sparking of the material (indicating finely divided plutonium metal or hydride), corrosion of the can (usually indicating the presence of acid), gas release when the can is opened, and degradation of the material (color and color change upon opening, coarse or fine powder, etc.).

4.2.6 *Other*

Heat generation may be calculated, using the isotopic composition of plutonium. Other heat producing radionuclides are assumed to be absent. OSD-Z-184-00013 Rev.J-3 provides instructions for calculating the heat generation rate and a specification on maximum thermal loading.

5. **Technical Issues**

Issues that impact the overall characterization program include the following:

1. Exclusive use of LOI for determining moisture levels is not a valid test for materials that contain metals or other compounds that gain weight (oxidize) upon heating. For these types of materials, special tests for moisture may be required, or a pre-oxidation step to ensure all metal is completely oxidized prior to performing the LOI.
2. Obtaining representative samples of may be a problem for heterogeneous materials that are dispositioned to MD.
3. Metal that gains little or no weight may be decomposing via a hydriding type reaction.
4. Much of the inventory that also contains uranium does not have a uranium value assigned. In order for an item to be dispositioned to MD, the uranium content will also need to be known. Analytical methods for assaying uranium in the presence of plutonium may be costly.
5. Impurity analysis by the analytical laboratory is very limited. If a large number of analyses need to be performed, the analytical laboratory will need to acquire an Inductively Coupled Plasma Mass Spectrometer. (ICP-MS).
6. The OSD allows use of 7" cans. Any material repackaged and destined to MD should be placed in a Hanford convenience can (HCC), since this can will fit into a 3013 container.
7. The 9000 plus items in the Hanford inventory can undergo some consolidation during repackaging. Many items within a category or sub-category that will be dispositioned to MD contain similar plutonium assays and isotopic content. Coordination of these consolidation efforts with MD should be encouraged.
8. A few of the issues identified in HNF-3541 may not be applicable to characterization. Those identified are 3.1.3.2.g, 3.1.3.4.m. Materials that are declared waste and must meet WIPP criteria will be shipped in vented drums. Head space gas analysis is performed on each drum to confirm absence or existence of volatile components that are RCRA regulated.

6. **Management of Data**

Three information management systems that are important to information management are:

- LANMAS is a classified system for material safeguards and security has been implemented. It contains some, but not all, of the information needed by the DNFSB 94-1 program requirements.
- ZMITS is a classified system that is in the initial steps of definition. It is designed to import information from LANMAS and include other data needed to implement DNFSB 94-1 program requirements. Funding for implementation of ZMITS has yet to be authorized.
- LABCORE is a system for managing laboratory analyses and the results. It is in the process of implementation. Some of these results are needed by ZMITS.

LANMAS and ZMITS databases will provide information on each inventory item required by the MD Acceptance Criteria. These include item identification number, location, plutonium content (mass), time in storage (age of the

material may be longer if the material was received from off-site, or repackaged at the PFP), isotopic ratios, net weights, chemical and physical form, specific stabilization processing conditions and test results, americium content, origin (e.g., metal button or casting from HNF, alloy from ANL, etc.), alloying agent (if any), and purity. Additional fields are available for other attributes as required.

Extensive information on each item in the inventory will eventually be available from databases such as LANMAS and ZMITS (described later). In the meantime, existing information can be used such as the Safeguards Inventory Database and archived documents that contain shipper/receiver information and process knowledge. Information on oxides produced during the stabilization campaigns will be available from the LANMAS and ZMITS databases. Process knowledge information available from other records, such as analytical laboratory and shipper/receiver papers is also available.

DISTRIBUTION SHEET

To	From	Page 1 of 1
Distribution	R. W. Szempruch/PFP Process Eng.	Date June 28, 1999
Project Title/Work Order Material Stabilization Characterization Management Plan		EDT No. 626642
		ECN No. N/A

[illegible]