

UCRL-JC-129966
PREPRINT

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**This paper was prepared for and presented at the
Waste Management '98
Albuquerque, NM
March 1-5, 1998**

February 13, 1998

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Surplus Plutonium Immobilization Feed Materials Requirements and Blending Strategy

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ABSTRACT

The Excess Fissile Materials Disposition Program's Record of Decision (ROD) published in January of 1997 by DOE/MD describes three potential pathways for the disposition of excess fissile materials: burning as MOX fuel rods, and two can-in-canister immobilization candidates: glass and ceramics. In addition, the ROD introduced processing schedules for MD disposition program. Prior to the ROD, the only acceptance specification that AMD had for incoming materials was DOE STD-3013. However, STD-3013 is a specification aimed at maintaining safety for long term storage (approximately 100 years) and was never intended to act as an acceptance specification.

An effort has begun to examine all of the technical issues associated with the processing and transfer of materials from EM to MD. Since that time, several related initiatives have begun to deal with the many issues, including the EM Material Stewardship Program, the latest EM-66 sponsored trade studies, and a new storage standard.

A draft of feed material requirements for the ceramic Immobilization Facility that will be used for the disposition of surplus plutonium has been developed for discussion. It establishes impurity limits for feed materials to the immobilization process, identifies impurities in feed materials that may have an adverse effect on the immobilization process, and indicates how these materials can be further processed and blended at the Immobilization Facility to ensure manufacture of an acceptable product.

Work performed under the auspices of the U.S. Department of Energy by the Lawrence Livermore National Laboratory under contract number W-7405-ENG-48.

INTRODUCTION

This paper describes surplus plutonium Immobilization Facility feed materials, documents the current state of knowledge of these materials, identifies impurities that can have an adverse effect on the product produced by the Immobilization Facility, and indicates how these materials can be further processed and blended at the Immobilization Facility to ensure manufacture of an acceptable product. This paper summarizes material in Ref. 1.

Sections below:

- Give background and states assumptions.
- Describes post-blend requirements that must be met in order to ensure an acceptable product.
- Describes the current state of knowledge of the feed materials, how the material is expected to be processed before blending, and the expected pre-blend composition of these feeds.
- Describes a blending campaign that could be used to dilute impurities.
- Gives other Immobilization Facility acceptance specifications and their technical basis.

FEED MATERIALS REQUIREMENTS FOR IMMOBILIZATION

The immobilized form is a high temperature melting crystalline ceramic that is formed by pressing a powder at ambient temperatures followed by sintering at high temperatures. The ceramic product contains three primary phases; pyrochlore, zirconolite, and brannerite that incorporate the actinides, neutron absorbers, and feed impurities. Pyrochlore is the dominant of the three phases. The product is also buffered with excess rutile, which helps maintain the desired product mineralogy and hence the durability of the form.

The ceramic product can tolerate significant variations in the feed composition without significantly affecting the overall mineralogical composition. A number of feed compositions have been tested (See Table 1). Compositions 1 through 6 simulate various feed categories expected by the Immobilization Facility. Composition 7 is an overall average composition and composition 8 is an extreme case. Composition 9 is an intermediate between 7 and 8 that corresponds closely to a composition that was tested in the glass form development. Additional testing on the effects of impurities is in progress.

Based on the information from the 9 impurity test suites, preliminary feed impurity limits for the ceramic immobilization process can be established. Preliminary durability tests indicate that the products from all the tests are extremely durable. In each test suite, pyrochlore was the dominant phase, but the relative abundances of the constituent phases varied with the impurity levels. The largest variation from the baseline assemblage occurred in composition 8, which contains the highest impurity level (approximately 13 wt%). Composition 9 contained considerably less impurities (about 5 wt%) and deviated relatively little in mineralogy from the baseline formulation. As a result, the impurity levels for composition 9 were used to establish most of the acceptance criteria for the ceramic immobilization process. Impurity levels for composition 8 represent, in general, the most extreme levels tested and a basis for believing that higher impurity levels can be accommodated with further ceramic form development. The actual feed composition to the Immobilization Facility can be different from the post-blend feed criteria because the various feeds will be blended before being immobilized. The limits of known acceptability are presented in Table 2. If the feed compositions fall under the specified limits, acceptable ceramic product can be made. If the feed compositions are in excess of the limits, it is not yet known whether acceptable ceramic product can be made. Further impurity testing is expected to allow significantly less stringent limits. The limits are reported in Table 2 as moles of impurity category per mole of plutonium oxide (PuO_2). This unit was chosen instead of weight percent because the impurities compete for sites in the crystalline lattice on a molar basis and not a weight basis. Acceptance criteria for the ceramic process are largely developed from the derived mole ratios of test composition 9, and to a lesser extent from composition 8.

Table 1 Goes Here

Table 2 Goes Here

FEED COMPOSITIONS FOR BLENDING

The surplus plutonium is currently stored at various sites in various forms. Before it is immobilized, much of this material will be processed for safe storage. The first processing would be for material stabilization. The planned stabilization processing steps include: calcining, pyro-oxidation, pyrolysis, salt distillation, and salt washing. At the Immobilization Facility, further processing includes: Converting metals to oxides by the HYDOX process, decladding unirradiated fuel elements, grinding materials to the proper size, calcining materials, and a very limited amount of leaching of soluble salts.

Current Feed Compositions

The available data are from one of four sources: engineered materials data, material specifications, sampling data, and engineering knowledge. The level of knowledge decreases as one moves down the list. Engineered materials are well known because they have been designed to meet certain criteria. The feed streams that fit into this group are the alloy reactor and oxide reactor fuels. The alloy reactor fuel is dominated by the ZPPR fuel. The oxide reactor fuel is dominated by the FFTF fuel.

The material specification data are given as a range of allowable values for the impurities. Material specification values were available for the clean metal and clean oxide categories. The specification for the clean oxide is that the impurities are less than 3 wt%.

The sample data comes from sampling of the streams. Some of these data pertain to individual samples, while some of it is a composite of several samples. The composite data generally provide information on maximum, minimum, and average concentrations. Composite data are used for Rocky Flat's Ash, Ash Heels, and Rocky Flat's Chlorinated Oxides.

The fourth type of data is based on engineering judgment. Most of the data are of this type. This type of data was used to estimate the composition of Hanford impure oxide, DOR residue, ER Residue, and MSE Residue.

The material compositions describe materials as they are currently stored. Many of these materials will be processed before they are staged for blending. This processing will occur to stabilize the material before shipping it to the Immobilization Facility and in the Pu Conversion portion of the Immobilization Facility. The feed compositions were modified to account for this processing.

How Individual Streams Meet Criteria

A comparison of the average of each stream to the criteria in Table 2 is given in Table 3. Bold numbers exceed the criteria limits shown at the top of the table. As can be seen from the table, fewer than half of the streams meet the criteria. The following streams require blending to meet the criteria: Pu Alloys, Hanford impure oxides, chlorinated oxides, ash, ash heels, ER salts, DOR salts, ZPPR fuel, and FFTF fuel.

The table also shows that these streams exceed different criteria. Therefore, by blending streams, an acceptable stream can be formed. The average of all the streams is shown at the bottom of Table 3.

Table 4 Goes Here

BLENDING CAMPAIGN

Need for Blending

If all of the feed streams were blended in one large batch, the impurity levels in that large batch would be acceptable for the immobilization process. This provides confidence that a feasible blending strategy could ultimately be implemented. However, criticality and other concerns make generation of one homogenous batch in one blending operation impractical. Cans must be blended in batches of 10 to 30 cans at a time.

As indicated by the feed stream impurity data in Table 3, the average impurity levels in some of the feed streams do not meet the requirements of the ceramic form specified in Table 2. Different feed streams must be mixed in each batch. In addition, due to can-to-can variation within a feed stream, an unacceptable batch might be generated from a feed stream that, *on average*, has acceptable properties but the particular can used in the batch was outside of the criteria. There is a need to mix cans from different feed streams in order to dilute high levels of impurities in different streams and in different cans within a stream.

The objective of the blending analysis is to identify combinations of feed streams (recipes) that are likely to yield acceptable batch properties. Because not all feed streams contain the same amount of material, some streams will be depleted before others. Therefore, the recipe for a batch will vary over time as various feed streams are depleted.

Simulation model for evaluation of blending strategies

A discrete-event simulation model was developed in order to evaluate different blending strategies (can permutations). After each batch is blended, the properties of the batch are examined. Batches that do not meet requirements must be stored and reblended with relatively high-purity feeds in order to reduce the impurity content to acceptable levels. One objective of the simulation effort is to identify blending strategies that minimize the number of reblending operations required. Another objective of the simulation effort is to estimate the value of obtaining additional information about the feed streams.

Some general assumptions that were used in the developing the simulation model are:

- a) The blending problem is decoupled from storage problem. The available storage facilities at the Savannah River Site can be used to stage materials that are fed to the Immobilization Facility. Thus, it is assumed that all materials are available for blending at the time of plant startup.
- b) In general, the blending strategy will be aggressive, and attempt to process high-impurity feed streams early in the campaign (after a suitable plant shake down period with relatively clean materials).
- c) A can of feed material cannot be used in more than one batch (the costs of repackaging and storing materials are high). This assumes no reblending of cans.
- d) The statistical properties (mean and standard deviation) of each feed stream are known, but the properties of individual cans are not well known. This is a conservative assumption that does not fully utilize available information.
- e) The total amount of Pu in each stream is known.

In the simulation model, the properties of each can from each feed stream are generated in the following manner. First, the mass of Pu in the can is randomly generated from a triangular distribution using the minimum, mode (most likely), and maximum Pu content for that stream. Second, the Pu-239 atom percent is generated using a second triangular distribution for that feed stream. Next, the molar ratio of volatiles to Pu is sampled from a lognormal distribution for that impurity in that feed stream. Molar ratios of other impurities are then sampled independently using other lognormal distributions unique to the impurity and the feed stream¹. Where standard deviation values are not available, they are estimated by assuming that the maximum impurity levels specified correspond to the 99th percentile of a lognormal distribution. The standard deviation is then derived from the 99th percentile using a mathematical relationship. Cans sampled from these distributions are generated for each of the feed streams and placed in queues to be accessed by the blending logic in the simulation model.

¹ Impurities may be positively or negatively correlated with each other. At this time data are insufficient to support the development of joint distributions needed to account for these potential dependencies.

The discrete event simulation model was built using a commercially available simulation modeling system (Extend®). It includes approximately 1500 nodes.

Results of Blending Run

The blending model was used to evaluate alternative production schedules. The production schedule shown in Table 4 was found to be fairly effective in diluting impurities. The schedule can be further optimized.

Table 4 Goes Here

The 18 MT immobilization campaign was simulated using this production schedule. In all, 7678 cans of material were generated and blended into 477 batches. Each batch contained approximately 40 kg Pu. In the model, the properties of the batches were monitored and compared to the immobilization process feed requirements specified in Table 2. Of the 477 batches, 80 (17%) did not initially meet the feed material requirements, and would have to be reblended. The 80 batches exceed at least one of the criteria.

Conclusions of the blending simulation analysis

The simulation model currently incorporates relatively simple logic for blending feed streams and does not rely upon knowledge of the contents of individual cans prior to blending. It is encouraging that with this simple blending logic and limited use of information, only 17% of the blended batches require a second blending step. It is anticipated that additional analyses, using linear programming models for blend optimization, can lead to some reduction in the need for reblending. This would further reduce operational costs and the need for in-line storage. However, it is unlikely that a blending strategy can be devised to completely eliminate the need for reblending of the ash and DOR salts feed streams.

Currently, some of the feed streams are poorly characterized, relying on sparsely documented engineering judgment. As more information becomes available about feed stream characteristics, the inputs to the simulation model will be refined and additional runs made.

The simulation model currently operates in an open loop mode, in which it is assumed that the contents of individual cans are unknown, and the properties of the batch are revealed only after all cans have been blended in the batch. Use of information about contents of individual cans shipped to the immobilization facility, and information derived from non-destructive examination (e.g., X-ray fluorescence) of cans at the facility may lead to improved blending and significant cost reductions. Future analysis may identify the value of additional information about contents of individual

cans in order to optimize design and implementation of NDE and process monitoring equipment.

SPECIFICATIONS

This paper does not cover requirements related to policy, radiological protection or shipping requirements. These specifications are written primarily for large lots of material, for example, 100 kg or more of plutonium in the lot. Small lots of material, such is frequently the case with CSMO materials, will have to be handled on a case-by-case basis.

Excluded Materials

The following materials can not be processed in the Immobilization Facility because of high impurities or lack of information about the streams. These materials must be either pre-processed in other facilities to yield a product acceptable for transfer to MD-Immobilization or prepared for shipment to WIPP.

1. Materials blended across points of origin (glovebox lines, MBAs, IDCs, facilities, ANSI codes, etc.) unless these materials are fully (chemically and physically) re-characterized.
2. Plutonium materials with excessive amounts of elements added during the stabilization processing including: Vanadium contents greater than 2.5 wt% V, and Calcium contents greater than 2.5 wt% Ca.
3. Unreacted PuF_3 or PuF_4 , failed runs, misfires, or floor-sweepings from the glove-boxes between fluoride precipitation or fluorination and bomb reduction.
4. Molten salt solvent residues: Alkali and alkaline earth halide salts used as solvents for DOR, MSE, and ER, and Calcium fluoride solvent salt from bomb reduction, usually called sand, slag, and crucible residues.
5. Plutonium alloys in which the non-actinide content is greater than 27 atomic percent, i.e., scrub alloy generated by MSE salt residue scrubbing with aluminum and magnesium.

Known Acceptable Materials

Based upon the present state of knowledge of the plutonium residues, and the ceramic immobilization form impurity experiments so far completed and analyzed some materials appear to be sufficiently characterizable by process history that they can be blended into acceptable immobilization feed. These include:

1. Fast Flux Test Facility (FFTF) unirradiated fuel elements, pins, reject pellets, and loose blended powders.
2. Zero Power Plutonium (now referred to as Physics) Reactor (ZPPR) fuel elements (irradiated to about 50 watts).
3. Declassified weapons returns (Pit Disassembly and Conversion oxide product).
4. Clean plutonium metal.
5. Clean plutonium oxides.
6. Plutonium oxide materials in which the plutonium content is >30 wt%.
7. Mixed plutonium-uranium oxide in which the combined uranium plus plutonium content is greater than 60 wt% and the plutonium content is greater than 5 wt%.

Other Requirements

Accurate impurity information exists for only a small portion of the material to be immobilized. However, much information is available from process knowledge. For each area of the process, it is generally known what the primary elemental impurities are or can be. It is therefore imperative that this knowledge be preserved in the form of item description codes of the origin of the material, MBAs of origin of the material, etc.

SUMMARY AND CONCLUSIONS

This paper identifies and documents the basis for the impurity tolerances for feed materials to the immobilization process. Based upon experiments performed to date with materials in the ceramic matrix, allowable levels of 13 categories of impurities in feed streams to the proposed immobilization plant are established. It is currently believed that an acceptable ceramic form can be fabricated if the impurities in plutonium feed streams are maintained below these levels.

In general, the feed materials can be blended to produce an acceptable feed to the Immobilization Facility. The blending campaign requires staging of feed materials so that problematic feed streams are fed in with pure feed streams in order to dilute impurities. In the model 17% of the batches did not initially meet specifications and had to be reblended with pure feed materials. In-line hot storage of cans from batches that did not meet specification would be needed.

The ash materials, chlorinated oxides and DOR salts were particularly problematic. It is possible that each can of these feed materials would have to

be split between two batches to achieve sufficient dilution of impurities. In-line hot storage would be needed to store the opened cans.

REFERENCES

- 1) Ebbinghaus, Bart, Thomas A. Edmunds, Leonard W. Gray, David C. Riley, "Preliminary Feed Materials Requirements and Blending Strategy," Lawrence Livermore National Laboratory - PIP-98008 (January 16, 1998).
- 2) Office of Fissile Materials Disposition, "Feeding Materials Planning Basis for Surplus Weapons-Usable Plutonium Disposition," US Department of Energy, April 2, 1997.

Table 1 Suite Impurities

	Suite #1 (wt%)	Suite #2 (wt%)	Suite #3 (wt%)	Suite #4 (wt%)	Suite #5 (wt%)	Suite #6 (wt%)	Suite #7 (wt%)	Suite #8 (wt%)	Suite #9 (wt%)
Category									
Typical Impure Oxide									
ZPPR Reactor Fuel									
Atypical Impure Metal									
Atypical Clean Metal									
U/Pu Oxides									
Pu Alloys									
Average									
Extreme									
Kg Pu	4,980	2,860	1,740	1,200	1,180	920	19,120	930	
Base Feed Materials									
CaO	9.67	9.89	9.73	9.92	9.85	9.83	9.80	8.65	9.44
TiO₂	34.88	35.64	35.08	35.77	35.50	35.43	35.34	31.20	34.04
HfO₂	10.35	10.58	10.41	10.62	10.54	10.52	10.49	9.26	10.11
Gd₂O₃	7.73	7.90	7.77	7.93	7.87	7.85	7.83	6.91	7.54
UO₂	23.03	23.54	23.17	23.63	23.45	23.40	23.34	20.60	22.48
PuO₂	11.56	11.81	11.63	11.86	11.77	11.75	11.71	10.34	11.28
Impurities									
Al₂O₃	0.63	0.2	0.22		0.11	1.04	0.32	1.59	0.50
MgO	0.19		0.23	0.02	0.46	0.18	0.13	0.87	0.44
CaCl₂	0.37						0.16	2.19	0.66
Ga₂O₃			1.27	0.14			0.14		0.57
Fe₂O₃	0.17		0.14		0.16		0.08	0.50	0.15
Cr₂O₃	0.04		0.02				0.02	0.13	0.08
NiO	0.08		0.09				0.04	0.33	0.13
CaF₂	0.21						0.12	1.30	0.44
K₂O	0.15		0.04				0.07	1.05	0.32
Na₂O	0.16						0.06	0.47	0.14
MoO₃	0.05	0.44			0.30		0.11	0.47	0.28
SiO₂	0.51						0.19	1.50	0.46
Ta₂O₅	0.05		0.15				0.06	0.64	0.19
B₂O₃	0.04							0.34	0.17
WO₃	0.14		0.06					1.64	0.49
ZnO				0.11			0.01		0.07
Total	2.76	0.64	2.22	0.27	1.03	1.22	1.51	13.02	5.09

Table 2 Impurity Composition Envelope for the Ceramic Form

	Category	Moles per mole PuO₂	Impurities in Category
1	Volatiles	0.60	NaCl, KCl, CaCl ₂ , CaF ₂ , MgF ₂ , Carbon, etc. plus CuO _{0.5} , KO _{0.5} , NaO _{0.5} , HgO _{0.5} , ZnO, etc.
2	Zirconolite stabilizers	0.75	AlO _{1.5} , FeO _{1.5} , GaO _{1.5} , CrO _{1.5} , MgO, ZrO ₂ , HfO ₂ , VO _x etc.
3	Pyrochlore stabilizers	0.40	WO ₂ , MoO ₂ , TaO _{2.5} , etc.
4	SiO ₂ + BO _{1.5} (Si > B)	0.30	SiO ₂ , BO _{1.5}
5	PO _{2.5}	0.10	PO _{2.5}
6	BaO	0.45	BaO
7	NiO	0.10	NiO
8	Total of all impurities included in 1 to 7	1.75	All in 1 to 7
9	"Rare earth" oxides	X + 1.40	LaO _{1.5} , GdO _{1.5} , AmO _{1.5} , etc.
10	CaO	X + 0.25	CaO
11	"Actinide oxides" excluding UO ₂	1.00	ThO ₂ , NpO ₂ , CeO ₂ , etc. (no UO _{2+x})
12	Total of all impurities included in 1 to 11	3.00	All in 1 to 11
13	"Actinide oxides" including UO ₂	2.00	All in 11 plus UO _{2+x}

Table 3 Summary of Feed Composition for Blending

	#	1	2	3	4	5	6	7	8	9	10	11	12	13	# Above Criteria
	Cat	Volatiles	Zirconolite Stabilizers	Pyrochlore Stabilizers	SiO ₂ + BO _{1.5}	PO _{2.5}	BaO	NiO	Sum 1-7	RE Oxides	CaO	AcO-UO ₂	Sum 1-7 +9-11	AcO+UO ₂	
	Limit	0.60	0.75	0.40	0.30	0.10	0.45	0.10	1.75	1.40	0.25	1.00	3.00	2.00	
Stream	MT Pu														
Pure Metal Converted to Oxide	0.000	0.00	0.01	0.00	0.00	0.00		0.00	0.01	0.00	0.00	0.00	0.01	0.00	0
Hanford Pure Oxides	1.700	0.04	0.07					0.00	0.11	0.04	0.01	0.04	0.21	0.05	0
Hanford Off Spec Metal-Oxides	3.400	0.01	0.04	0.00				0.00	0.06	0.04	0.04	0.00	0.14	0.00	0
Pu Alloys	1.000		0.81	0.01					0.82	0.00	0.40		1.22	0.30	2
Hanford Impure Oxides	3.481	0.57	0.92		0.06		0.01	0.02	1.59		0.21		1.80	0.00	1
RF Oxides at Hanford	0.000	0.57	0.92		0.06		0.01	0.02	1.59		0.21		1.80	0.00	1
Chlorinated Oxides	1.040	0.56	1.38	0.02	1.11		0.05	0.16	3.29	0.00	2.43	0.01	5.73	0.01	6
Ash	0.131	0.66	1.45	0.01	4.81		0.03	0.03	7.00	0.00	0.41		7.40	0.00	6
Ash Heels	0.003	0.66	1.18	0.02	3.69	0.09	0.01	0.03	5.69	0.00	0.33		6.02	0.00	6
ER Salts (NaCl/KCl)	0.326	0.25	1.39	0.00				0.00	1.64	0.00	0.00	0.01	1.66	0.01	1
ER Salts (CaCl ₂)	0.326	0.03	1.37	0.00	0.00			0.00	1.41	0.00	0.03	0.01	1.46	0.01	1
DOR Salts	0.130	5.20	0.91					0.00	6.11	0.04	7.23		13.38	0.00	5
MSE Salts (CaCl ₂)	0.203	0.12	0.12	0.26					0.49	0.11	0.09	0.01	0.70	0.01	0
MSE Salts (NaCl/KCl)	0.203	0.10	0.16	0.26					0.52	0.11	0.07	0.01	0.72	0.01	0
Anode Heels	0.558	0.00	0.20	0.00	0.00			0.00	0.21	0.00	0.00	0.01	0.22	0.01	0
Pu/U Oxides	0.900		0.26	0.00					0.27	0.01	0.12		0.40	0.92	0
ZPPR Fuel	3.500		0.02	0.19					0.21	0.01			0.22	2.18	1
FFTF Fuel	1.300								0.00				0.00	3.15	1
													Total number of Streams above Criteria	11	
Average	18.200	0.20	0.41	0.04	0.11	0.00	0.01	0.01	0.78	0.02	0.27	0.01	1.07	0.01	1
% Margin		67.30%	45.62%	89.03%	63.12%	99.99%	98.97%	85.82%	55.52%	98.77%	-8.93%	99.42%	64.21%	99.71%	

Table 4 Production schedule used by the simulation model

Index	Feed Stream	Start time	Feed rate	Completion time	Comments
1	Pure metal	N/A	N/A	N/A	
2	Hanford pure oxides	0	1	450	plant start up
3	Hanford off spec metal	350	1	1306	
4	Pu alloys	350	1	757	
5	Hanford impure oxides	200	1	1400	
6	FFTF fuel	0	1	1336	plant start up
7	Rocky Flats oxide at Hanford	N/A	N/A	N/A	
8	Rocky Flats chlorinated oxides	350	1	989	
9	Rocky Flats ash	200	1	259	problematic
10	Rocky Flats ash heels	350	1	351	
11	ZPPR fuel	0	1	1500	plant start up
12	ER salts (Na, K)	350	1	572	
13	ER salts (Ca)	450	1	672	stagger
14	DOR salts	275	1	353	problematic
15	MSE salts (Ca)	550	1	874	stagger
16	MSE salts (Na, K)	650	1	1174	stagger
17	Anode heels	275	5	303	dilute feed #14
18	Pu/U oxides	200	8	245	dilute feed #9

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