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FY94/95 Thermal Stabilization Campaign Report

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Abstract: The report provides a synopsis of the FY94/95 Thermal
Stabilization Campaign.

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1. SUMMARY

The FY94 Thermal Stabilization Campaign ran from November 1, 1994 to June 13, 1995 and stabilized 236 items (22 kg) of reactive plutonium bearing material. A total of 49 cans of stabilized material was moved to the vaults for interim storage.

While the initial downtime was high, with an Operating Efficiency (OE) of 27% over the first 3 months, the OE improved to 78% during the last 5 months for an overall OE of 58%. The campaign was completed 85 days ahead of schedule thus exceeding the "Excellent" date for the Performance Base Incentive (PBI) for this campaign by 25 business days.

2. INTRODUCTION

This report summarizes the FY94 Thermal Stabilization Campaign in terms of system performance, downtime, problems, and product quality. The Thermal Stabilization performance documentation will provide a baseline for system performance standards, as well as a basis for measuring the effect of proposed system changes. Establishment of performance standards through documentation and evaluation of actual operating data will aid in making flowsheet changes, design of system and cost savings analysis. It will also document design improvements that will be used for the installation of additional furnaces to support thermally stabilizing materials so they can be repackaged to meet the DOE Long Term Storage Criteria (94-1).

3. DISCUSSION

3.1. Process Description

The thermal stabilization process is operated in gloveboxes HC-21A and HC-21C which are located in rooms 230-B and A respectively. Glovebox HC-21C contains 2 muffle furnaces and 2 desiccators. Glovebox HC-21A contains a sieve shaker, a desiccator and some miscellaneous scrap screening and handling equipment. Reactive solid scrap items are sealed into glovebox HC-21A or transferred from HC-18BS through the conveyer hood HC-2 to glovebox HC-21A. Materials are placed in roughly a 1.5 liter boat and heated up in one of the two muffle furnaces. This drives off any volatiles and high fires the plutonium to an oxide form. Neither of the two gloveboxes have a dry air supply at this time so stabilized items are temporarily stored in desiccators while cooling or waiting sample results. The materials, once heated, are scraped out of the boat and placed in a 0.8 liter (PUREX) slip lid can. Once enough material is collected to fill the can, the material is sieved with a 4 mesh sieve and shaker and the larger chunks crushed with a mortar and pestle. It is then sampled for loss on ignition (LOI). If the LOI specifications are met, the material is canned for vault storage.

3.2. System Analysis

3.2.1. Performance

Table 1 summarizes the Thermal Stabilization process in terms of potential charges, actual charges, lost charges due to downtime and efficiency determinations.

Table 1 System Performance Overview

Potential Charges	307
Actual Charges	177
Downtime Due to HC-21C Problems (Lost Charges)	85
Downtime Due to Plant (Lost Charges)	45
Total Downtime (Lost Charges)	130
Recycle Charges	15
Operating Efficiency	58%

The Operating Efficiency for the FY94 campaign was generated as a baseline for future campaigns. The monthly progressions of the operating efficiency is shown in Figure 1. Although not reflected in the operating efficiency, an important indicator of performance is the number of product cans that require recycle. Figure 2 shows the percent of product cans that required reprocessing per month of operation. The large number of cans requiring reprocessing towards the end of the campaign is directly related to the change of performing LOI analysis at 1000°C rather than 450°C. The reasons for this are discussed in section 3.3.

A total of 236 items were stabilized during the campaign. These items are detailed in table 2. Of the original 236 items on the blend plan, 23 items had to be replaced with other material because they did not meet the feed criteria in OSD-Z-184-00006. These items that were not processed included Plutonium Fluoride and partially burned polycubes. The replacement items were obtained from material that had been taken out of processing gloveboxes during on going remediation activities and items that had originally been left off the blend plan because they were listed as plutonium metal turnings. Upon inspection of these items it was determined that all of the metal had oxidized and therefore they would meet the feed specifications.

Table 2 Detailed Feed Input For the FY94 Campaign

<u>Feed Type</u>	<u># of Items</u>	<u>Kgs Pu</u>	<u>% (Pu) of total</u>
Oxide	51	4.0	18%
PRF Sludge	144	11.5	52%
Oxalate	41	6.5	30%
Total	236	22	100%

Figure 1 Thermal Stabilization Operating Efficiency

Figure 2 Thermal Stabilization Recycle

3.2.2. Downtime Analysis

The sludge stabilization downtime was measured in lost charges. Table 3 and Figure 3 ranks the downtime items along with the associated lost charges and percent of total downtime.

The seismic over pack incident resulted in the single largest downtime item (44%). This item was due to problems in conduct of operations rather than actual processing problems. By far the largest source of downtime related to the process was difficulties in the off gas system. Problems with the off gas system accounted for 29% of the downtime. Once the off gas system problems were solved, downtime from the process was virtually eliminated. The off gas system problems are further discussed in section 3.2.3.1. Downtime from the plant (not related to Thermal Stabilization) accounted for roughly 27% of the downtime. Future operations can probably expect similar plant downtime. Downtime due to processing problems can be expected to be low.

Table 3 Thermal Stabilization Downtime Items

	FREQUENCY	LOST CHARGES	% DT
COP - Seismic Over Pack Event	1	44	33.7
MEC - Plugged Filters/Off Gas Line	4	19	14.6
MEC - Replace Rotameters	5	14	10.8
COP - RAD Con use of On/Off Mask	1	12	9.2
MEC - Loss of 26" Vacuum	3	8	6.2
SDT - Steam Outage	1	8	6.2
SDT - Training & Time Out Sessions	3	8	6.2
MEC - Install Orifices/Dp Gauges	1	4	3.1
MEC - Loss of Ventilation	2	4	3.1
SDT - Inventory	2	4	3.1
SDT - CAP Outage	1	2	1.5
MEC - RMC Fire Dampers	1	2	1.5
MEC - Deviation Alarm	1	1	0.8
	26	130	100.0%

SUMMARY	CHARGES	% DT
COP - Conduct of Operations	56	43
MEC - Mechanical	52	40
SDT - Scheduled Downtime	22	17
PDT - Process Downtime	0	0

% of Down Time

Thermal Stabilization

Down Time Items

Figure 3 Thermal Stabilization Ranked Downtime Items

3.2.3. Problems and Downtime Items

3.2.3.1. Off Gas System

Problems with the off gas system accounted for the majority of the process related downtime. The formation of liquid in the off gas system was first seen during the performance of the Operational Test Procedure (WHC-SD-CP-OTP-151). Formations of liquids continued during much of the first part of the campaign. A temperature profile of the off gas line (generated as part of a process test₁) indicated that the heat losses in the line were much greater than originally anticipated and that condensation in the lines with the original piping configuration was inevitable (see Figure 4 and 5 for temperature profiles of the two furnace's off gas lines). As a result of the lower line temperatures the heat exchangers were removed. While this helped the condensation problem in the lines, condensation persisted, leading to additional process downtime. A process test₂ was prepared to systematically trouble shoot the problem. A number of modifications were made to the off gas system as a result of this test plan. The most important modifications was to use differential pressure gauges across an orifice to measure off gas flow rates rather than rotameters.

Another off gas problem which resulted in system downtime was plugging of the off gas filters. The plugging was primarily caused by either boil over of material (oxalate) or entrainment of particulate fines. Problems with filters plugging was encountered within the first 10 days of actual operation. A lower soak temperature was used when processing oxalate and a baffle was installed in each of the furnaces. After these changes the rate of filters change out was reduced to roughly 1 every couple months for the types of material being processed. Occasional filter plugging can be expected in the future. However, by developing a Pre Approved maintenance Procedure (PAP) to clean or replace the filters, downtime should be minimized.

Figure 4 Off Gas Line Temperature Profile Furnace 1

Temperature Profile

For Furnace 1 Operation on 2/8/95

Figure 5 Off Gas Line Temperature Profile Furnace 2

3.2.3.2. Boat Material

The boat carriers were constructed out of 304L stainless steel. This material is subject to high temperature oxidation and corrosion. During the campaign, 10 boats failed. As a result, a process test₄ was prepared to determine a material that was more compatible with the process conditions. A number of potential materials were identified and tested. These materials included 310S Stainless Steel (SS), Cobalt Protective Metal Alloys (PMA) 6B, Cobalt PMA 25, Hastelloy X, Platinum, and Ceramic (rebonded fused silica).

Coupons of each material were first heated to 1000°C without contacting any of the material in the boats. All of the test material showed little if any degradation during this test (Figure 6). The coupons were then placed in contact with the corrosive material inside of the boats and repeatedly heated to 1000°C. All of the materials except for platinum demonstrated varying degrees of degradation (Figure 6).

Platinum is cost prohibitive, therefore Hastelloy-X, the next best performing material, will be used to fabricate future boats. When stabilizing corrosive materials, such as sludge scraped from PRF gloveboxes, a platinum liner may be used in the boat. The platinum liner could be obtained from the spare RMC Line fluorinator tube and therefore would incur minimal extra cost.

Figure 5 Boat Material Corrosion Testing

PERCENT WEIGHT LOSS VS. CHARGE

BOAT MATERIAL

3.2.4. Off-Standard Conditions

Tables 4 and 5 list the Unusual Occurrence Reports and the Off Normals which took place in the Thermal Stabilization area during the FY94 campaign. Refer to the specific report for incident details.

Table 4 Unusual Occurrence Reports

<u>Report #</u>	<u>Date</u>	<u>Subject</u>
94-0056	12/13/94	Plutonium Leak from Double Bagged Container Resulting in Personnel Contamination

Table 5 Off Normals

<u>Report #</u>	<u>Date</u>	<u>Subject</u>
94-0057	12/13/94	CPS infraction when polyjars were left in seismic overpacks
95-0004	2/8/95	Fire alarm caused evacuation of 234-5Z
95-0005	2/9/95	Temporary loss of power to building suspected to cause failed electrical cable
95-0009	2/23/95	Fire alarm caused evacuation of 234-5Z
95-0015	3/22/95	Fire alarm caused evacuation of 234-5Z
95-0018	4/6/95	Fire alarm caused evacuation of 234-5Z
95-0025	5/27/95	Loss of power to turbine control panel caused ventilation to shut down and emergency turbines to come on line.
95-0028	6/7/95	Fire alarm caused evacuation of 234-5Z

3.3. Product Quality

During the campaign a number of items that were stabilized did not meet the vault storage criteria for Loss On Ignition (LOI). A total of 3 items had a LOI of greater than 1% and 6 items showed a Gain On Ignition (GOI). The majority the product quality problems occurred when the LOI temperature was raised from 450°C to 1000°C. A process test was prepared to troubleshoot the cause for the gains or higher losses. The results from this testing is documented in a process test report₂. Three primary tests were performed using the test plan.

The first test performed a simulated thermogravimetric analysis (TGA). Samples from both high LOI material and GOI material were periodically weighed while being heated to 1000°C. The results (Figure 7) show that both the losses and the gains occur primarily at temperatures over 750°C. The gains are potentially from additional oxidation of material. This

additional oxidation does not cause a problem as long as it does not mask a loss at the lower temperatures. The higher losses seem to be from material, such as salts, that are volatilizing and being removed in the off gas stream at the higher temperatures. This is reinforced by the results of the second and third tests.

The second test involved placing a thermocouple in contact with the material in the boat. During the first run, with sludge type material, the boat temperature tracked within 50°C of the furnace temperature during the soak period (Figure 8). During the second run, when recycling high LOI material, the temperature in the boat only reached about 850°C. This reinforces that TGA testing which indicated that some material was being driven off at a temperature between 750°C and 1000°C (Figure 8). The temperature data after these first two tests was suspect due to continued degradation of the thermocouples. Additional testing will be required to verify the results.

The third test involved holding the sample material at 1000°C for several hours while periodically measuring the weight loss/gain. The data (Figure 9) indicates that the weight changes, for the most part, level off after about 3 hours. Based on this data, the material with high LOI was recycled and held at 1000°C for 3 hours. Upon resampling, the material met the LOI specification of less than 1% weight loss. The data being collected during these tests is being used in the development of the long term storage criteria for less than 50% Pu material. It is clear from the results that an LOI may not be the best method of determining the stability of material with high impurities for long term storage. A TGA, for instance, can provide information to verify that any volatiles that could react under vault storage conditions have been removed.

Figure 7 Simulated TGA

SIMULATED TGA

BOAT TEMPERATURE VS. FURNACE TEMPERATURE

Boat Temperature vs. Furnace Temperature

Figure 3 Boat Temperature Profile

Figure 9 Simulated TGA for Extended 1000°C Soak

SIMULATED TGA

Simulated TGA

Cum % mass change vs. time @ 1000C

3.4. Radiation Exposure Analysis

The amount of dose received by operating personnel was a major concern. Many of the items that were being stabilized had been stored for a number of years and had a high dose rate. A number of recommendations by the ALARA committee were implemented during the campaign. This greatly reduced the dose rate. Figure 10 shows the gamma and neutron dose rates per month for Operators, Lab Personnel and Radiation Control Technicians for Thermal Stabilization. The average dose per item processed at the beginning of the campaign was close to 50 mRem. By the end of the campaign the dose was reduced to around 15 mRem for an overall average dose rate of 27 mRem per item. Part of the reduction can be attributed to smoother operations at the end of the campaign (less maintenance) but the following ALARA enhancements certainly contributed greatly.

- The product cans were wrapped with 3 layers of lead foil. This reduced the gamma exposure by 75%
- Lead plastic shielding, 18 mm thick, was added to the windows of glovebox HC-21A, where most of the material handling occurs. This reduced the head and shoulder dose rate from 30 to 1 mRem/hr.
- A lead curtain was hung on the conveyor in room 230A to help reduce dose to the control room operators. The dose at the conveyor was reduced from 10 to < 0.5 mRem/hr.
- A lead blanket was placed over the top of the product containers when they were temporarily stored in a wagon in room 230B. This reduced the dose from the tops of the containers by 75%.
- A monthly status of operator dose rates was posted. This helped keep personnel aware of their accumulative dose as well as their co-workers. This also aided management in rotating workers through high exposure tasks.
- Lead shields were placed over the 8" and 16" seal out ports when they were not in use. This reduced the dose in front of the port by 90%.
- The water walls surrounding glovebox HA-23S were filled with water. The water had been drained from the walls some time in the past and had never been refilled, possibly because of the low occupancy rate in room 235B. Over 75% of the items stabilized during the campaign were from HA-23S. Filling the walls with water reduced the dose rate from 18 to < 0.5 mRem/hr.

SLUDGE STABILIZATION EXPOSURE

TOTAL BY MONTH WITH NUMBER OF POLY JARS COMPLETED

NEUTRON	640	370	138	746	628	245	223
GAMMA	310	320	149	635	925	413	566

TOTAL-6308 mrem AVERAGE PER JAR- 27 mrem
 EXPOSURES INCLUDE OPERATIONS, LABS AND RAD CON

Figure 10 Thermal Stabilization Exposure

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4. RECOMMENDATIONS

The following section gives recommendations for changes to be made in future campaigns based on observation made in this campaign. Changes are divided into maintenance items and operational improvements. Recommendations are also included for the installation of the additional 6 furnaces under the 94-1 planning.

4.1. Maintenance items

The most obvious way to improve the overall efficiency is to reduce the percent of downtime that occurs. The following is a list of troublesome items which need to have proper repairs made in order to operate more effectively during campaigns.

- Prepare a Pre Approved Procedure (PAP) for cleaning and/or changing out of filters. Filter pluggage can be expected to continue at a rate of roughly 1-2 per month. A PAP will eliminate the need to prepare a complete JCS package each time.
- Improve air flow rates in glovebox HC-21C. The glovebox exhaust HEPA filter needs to be replaced and the dry air supply fan needs repair. The low air flow in the glovebox has caused higher temperatures in the glovebox. Failure to obtain higher air flow will result in reduced process efficiency when the process is operated on an XYZ shift rotation.
- Make the boats out of Hastelloy-X material. As described in section 3.2.3.2, the boats used in the future should be made from a material that is more resistant to high temperature oxidation and corrosion. A platinum liner is recommended when processing "wet" materials.
- Place a screen on the boats when stabilizing flighty material to minimize plugging of filters. When the filters do plug with this type of material a soft bristle brush should be used to clean them.

4.2. Operational or Programmatic Changes

The following is a list of recommended operational or programmatic type changes that should be implemented before the new campaign.

- Increase charge size. The charge size was based upon limiting the amount of TBP in a charge. Future campaigns that do not process PRF sludge material should utilize a larger charge size. Some testing will be required to determine the maximum charge size without affecting the oxidation of material. As discussed in section 4.4 boats should be redesigned to maximize surface area of feed.

- Optimize off gas flow rate. When stabilizing "dry" material that does not contain any TBP (is not reactive material that originated in PRF) or corrosive components, use the off gas only system during the cool down portion of the cycle. This will reduce the time required to ramp up to 1000°C and hence reduce the overall cycle time. Testing will be required to determine the effect this would have on glovebox temperatures.
- Increase the plutonium limit on glovebox HC-21C to allow 5000 grams. The current limit of 900 grams was used to allow a readiness assessment be performed for original startup as opposed to a ORR. The 900 gram limit may hinder future operations when feed that has higher percentages of plutonium (oxide from the vaults) is stabilized.

4.3. ALARA Considerations

Many of the ALARA improvements that were discussed in section 3.4 should be continued during future HC-21C operations and should be evaluated for use with the installation and operation of the 6 new furnaces under 94-1. In addition, means need to be developed to further reduce direct handling of material (especially high dose material). Methods of reducing handling include:

- Stabilize low dose material first then develop remote handling for higher dose material.
- Repair conveyor system to minimize direct handling of material.
- Use an electric can opener to open cans from the vault that require stabilization.
- Use a V-Blender to blend larger amounts of material prior to sampling. This will minimize the number of samples that have to be taken, thus reducing handling.

4.4. 94-1 Furnace Installation

The follow is a list of recommendations that should be implemented into the design and operation of the 6 new muffle furnaces that are being installed in gloveboxes HA-20MB and HA-21I as part of the 94-1 project.

- Provide backup vacuum pump on off gas system. The 26" vacuum system was the fifth leading cause of downtime during the campaign. The off gas system for the new furnaces should have a backup vacuum capability.
- Reduce the size and weight of the off gas filter housings. The housings used in the campaign weighed 125 pounds each. This not only made them difficult to get into the glovebox but also made replacing filters difficult. A number of design modifications should be made to reduce the weight of the housings. Modifications should also allow for easy material removal from the housings.
- Modify the boat design. The boats should be designed more like a cake pan. The material that will be stabilized in these furnaces will not boil over. The new design would provide more surface area, thus allowing a larger charge size while maintaining sufficient oxidation of material.
- Similar to section 4.2 the off gas should only be operated during the cool down section of the cycle when stabilizing materials that have no corrosive off gases. Testing would need to be performed on each new feed type.
- A PC type control system should be used instead of individual controllers. This will not only be more cost effective but will allow more flexibility in the future for adding other processes (like the vertical calciner).
- Provide the capability to allow cans from the vault to be introduced into the gloveboxes with sealing them in (ie. can sphincter port or air lock). This would minimize waste and direct handling of materials.

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5. GLOSSARY

Charge -	500 grams of material charged to HC-21C furnace for stabilization. May consist of one or multiple items.
Cycle -	Processing of a charge in HC-21C furnace.
Design Rate -	40 charges/5 day week; equivalent to 2 charges/shift.
Downtime(Conduct of Operations) -	Downtime attributed to management decision not to operate, i.e, CPS violation, work control, lock & tag, etc.
Downtime(Mechanical) -	Downtime attributed to mechanical problems, i.e., failed furnaces, thermocouples, plugged lines, filters etc.
Downtime(Process) -	Downtime that occurs when the process is mechanically able to operate, but does not due to processing problems, i.e., lack of sample results, etc.
Loss on Ignition (LOI) -	Percentage of mass loss measured when a representative sample of material is heated to a specified temperature (450°C or 1000°C) for a specified time (1 hour) to verify removal of moisture and other volatiles.
Lost Charges -	A measure of downtime and operating efficiency. Cycles when the furnaces was not operated due to Conduct of Operations, Process or Maintenance downtime.
Operating Efficiency -	Actual charges/potential charges.
Thermal Stabilization -	A process that exposes a material to a high temperature (1000°C) for a period of time (1 hour minimum) required to convert reactive constituents to an oxide while reducing the amount of adsorbed water and other chemical species to a level compliant with the LOI storage specification (<0.5% for material with >50% Pu).

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6. REFERENCES

1. Heat Exchanger Bypass Test Report, WHC-SD-CP-TRP-060.
2. Loss/Gain On Ignition Test Report WHC-SD-CP-TRP-062.
3. HC-21C Off Gas Test Report, WHC-SD-CP-TRP-063.
4. Boat Material Test Report, WHC-SD-CP-TRP-061.

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