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Project Title/Work Order Loss/Gain On Ignition Testing for HC-21C				Date	
				EDT No. 608705	
				ECN No.	
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ENGINEERING DATA TRANSMITTAL

Page 1 of 2
1. EDT 608705

2. To: (Receiving Organization) Distrubtion	3. From: (Originating Organization) PFP Process Engineering	4. Related EDT No.: N/A
5. Proj./Prog./Dept./Div.: HC-21C/15530	6. Cog. Engr.: W. S. Lewis	7. Purchase Order No.: N/A
8. Originator Remarks: No Unreviewed Safety Question exists and the USQ is attached.		9. Equip./Component No.:
		10. System/Bldg./Facility: 73T/234-5Z/2Z
		11. Receiver Remarks:
		12. Major Assm. Dwg. No.:
		13. Permit/Permit Application No.: N/A
		14. Required Response Date:

15. DATA TRANSMITTED					(F)	(G)	(H)	(I)
(A) Item No.	(B) Document/Drawing No.	(C) Sheet No.	(D) Rev. No.	(E) Title or Description of Data Transmitted	Approval Designator	Reason for Transmittal	Originator Disposition	Receiver Disposition
1	WHC-SD-CP-TP-082	1	0	Loss/Gain On Ignition Testing for HC-21C	Q	1	1	

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)	(H)
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1	1	Cog.Eng. W. S. Lewis	<i>[Signature]</i>	12/8/94	T5-55						
1	1	Cog. Mgr. M. W. Gibson	<i>[Signature]</i>	12/8/94	T5-55						
1	1	QA D. R. Groth	<i>[Signature]</i>	12/8/94	T4-19						
		Safety									
		Env.									
1		M. L. De Vries	<i>[Signature]</i>		T5-55						
1	1	Operations G. B Chronister	<i>[Signature]</i>		T5-50						

18. <i>M. L. De Vries</i> 12/8/94 Signature of EDT Originator	19. _____ Authorized Representative for Receiving Organization	20. _____ Cognizant Manager	21. DOE APPROVAL (if required) Ctrl. No. N/A ☐ Approved ☐ Approved w/comments ☐ Disapproved w/comments
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EDT 608705

UNREVIEWED SAFETY QUESTION SAFETY REVIEW FORM

(Per WHC-IP-0842, 15.9)

Page 2 of 2

REFERENCE DOCUMENT(S):

ECN No. N/A

PCA No. N/A

Work Pkg No. N/A

Other (Specify) WHC-SD-CP-TP-082

TITLE: Loss/Gain On Ignition Testing for HC-21C**Does the PROPOSED CHANGE:**

A. Represent a change to the facility as described in the AUTHORIZATION BASIS documentation?

☐ N/A ☒ No ☐ Yes/Maybe

Basis: No modification is being done to the facility as describe in WHC-SD-CP-SAR-021 (SAR).

B. Represent a change to procedures as described in the AUTHORIZATION BASIS?

☐ N/A ☒ No ☐ Yes/Maybe

Basis: The test procedure will use the standard operation for the HC-21C process as described in OSD-Z-184-0006.

C. Represent a test or experiment not described in the AUTHORIZATION BASIS documentation?

☐ N/A ☒ No ☐ Yes/Maybe

Basis: The test being conducted remains within the scope of the basis document and does not exceed any limits in the OSD-Z-184-0006 and st or experiment is being conducted.

D. Does the change impact:

• Implemented OSRs or IOSRs? ☐ N/A ☒ No ☐ Yes/Maybe• Approved IOSR Compliance Implementation Plans? ☐ N/A ☒ No ☐ Yes/Maybe

Basis: No OSRs from WHC-SD-CO-OSR-010 are applicable to the test plan.

Based on the above, a Safety Evaluation ☐ DOES ☒ DOES NOT
need to be performed for this change

USQE No. 1

WS Lewis
Print Name

USQE No. 2

LAPRIEL DAYLEY
Print NameWS Lewis
Signature12/8/94
DateLapriel Dayley
Signature12-12-94
Date

RELEASE AUTHORIZATION

Document Number: WHC-SD-CP-TP-082, REV 0

Document Title: Loss/Gain On Ignition Testing for HC-21C

Release Date: 12/9/94

**This document was reviewed following the
procedures described in WHC-CM-3-4 and is:**

APPROVED FOR PUBLIC RELEASE

WHC Information Release Administration Specialist:

Kara M. Broz

December 9, 1994

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SUPPORTING DOCUMENT

1. Total Pages 15

2. Title

Loss/Gain On Ignition Testing for HC-21C

3. Number

WHC-SD-CP-TP-082

4. Rev No.

0

5. Key Words

LOI, GOI, HC-21C, Muffle Furnaces

6. Author

Name: M. L. De Vries

M. L. De Vries
Signature

Organization/Charge Code 15530/K6018

**APPROVED FOR
PUBLIC RELEASE**

KMB 12/9/94

7. Abstract

Document provides instructions for testing product cans from the HC-21C sludge stabilization process that have received LOI sample results that show a gain on ignition.

8. RELEASE STAMP

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BY WHC
DATE DEC 12 1994
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1 INTRODUCTION

The HC-21C thermal stabilization process stabilizes reactive plutonium bearing material for long term storage. A Loss On Ignition (LOI) analysis is performed on all materials that are stabilized in the muffle furnaces prior to being stored in the vaults to ensure suitability for vault storage. The material is required to have a LOI of less than 1% (OSD-184-00013 Limit). The LOI analysis is performed to assure that all volatiles that could potentially pressurize the sealed containers over long periods of time, like water, have been removed.

It is possible, while performing LOI analysis to see a Gain On Ignition (GOI) due to additional oxidation of materials. In the current stabilization campaign, two items processed have had a positive LOI or GOI result. One concern with a GOI is that the weight gain may actually be masking a weight loss. (Reference 15530-94-WSL-156) Other concerns with a GOI result are that the removal of the oxygen could create a vacuum in the product can causing it to collapse during long term storage or the oxidation of material would increase the volume of the solids potentially bulging or rupturing a the product container. In the past, GOIs have been seen on items that were processed through Hoskin's pot furnaces located in glovebox HA-21I. This test plan provides instruction to test causes of a GOI and to ensure the processed material with a GOI result will not cause a problem during long term storage. Product cans that are sampled for LOI and have results showing a gain of weight will be the test items.

2 OBJECTIVE

The test objectives are to gain data on Loss-On-Ignition and Gain-On-Ignition for material stabilized in the HC-21C Muffle Furnace Operation. Data will verify that the GOI is not masking any weight loss and will help to identify reactions that could potential be the cause of a weight gain during the LOI test. Results from this test plan will be used to provide a technical basis for acceptable GOIs.

3 SCOPE

Several tests will be run during the test plan to determine possible causes of a GOI result on stabilized material from the HC-21C process. The tests will include:

- Reprocessing Material
- Simulated Thermogravimetric Analysis (TGA)
- TGA with Mass Spectroscopy

4 DESCRIPTION OF TEST

4.1 Test Item

4.1.1 TEST 1: Reprocess Material

This procedure provides instruction for reprocessing a charge from a slip-lid can that was sampled for LOI and the results showed a GOI. A 500 gram charge will be taken from the product can identified with a GOI result. The material will be crushed and mixed before reprocessing it. After the charge is fired, the material will be sampled before being placed into a slip-lid can. The LOI test results from the product can and the reprocessed charge will be compared to determine if additional processing time and mixing affect the sample results.

4.1.2 TEST 2: Simulated TGA

This test consists of taking a sample from the item that had a GOI result and send it to the laboratory (analytical or PPSL). Lab scientist will weigh sample; heat to 100° C, cool, weigh sample; heat to 150° C, cool, weigh; repeat at 50° C intervals until 450° C with an option to go to 950° C. Increment size may vary depending on sample data and time. This data will help to verify that the GOI is not masking a LOI.

4.1.3 TEST 3: TGA with Mass Spectroscopy on Off-gas

A sample from a product can with GOI results will be taken and sent to the PPSL lab for TGA with mass spectrum analysis. The test includes heating a sample in a furnace with constant weight monitoring of the sample and any heat generation from reactions occurring. The off-gas will be analyzed to determine any gas being given off as the material reacts from heat. This test will help to determine what the reaction's off-gas constituents are and when this reaction is occurring. This will be helpful to determine what reactions are occurring.

4.2 Equipment and Facilities

The HC-21C muffle furnace will be used to reprocess product items with GOIs. Glovebox HC-21A will be used for furnace charge preparation and sampling of material. The balances that are used to weigh the material are located in HC-21A and should have a current calibration. PFP Analytical Laboratories and Plutonium Processing Support Laboratory (PPSL) facilities will conduct two tests and all the LOI tests. Currently the PPSL facility can not accommodate the TGA with mass spectroscopy, but a maintenance work package should restore their instrumentation to operable condition in the next few months.

4.3 Data

Different data will be collected during the tests and examined. Material type and item number making up a charge from the product can will be recorded. All information on the furnace cycle can be acquired from the data sheets used for each charge from ZO-160-032. Laboratory results will also be collected and any observations made during the processing cycle or lab analysis should be recorded.

4.4 Criteria/Constraints

Product can from HC-21C process has been sampled and LOI results show weight gain (GOI).

5 PREVIOUS RESULTS

Located in the table below are results from the LOI analysis of items processed through HC-21C muffle furnaces. The LOI analysis consists of the sample being weighed, heated to 450° C for two hours and reweighed to determine any weight loss. The first three samples (B3054, B3055, B3056) were heated up to 950° C and the other four were heated up to 450°.

SAMPLE NUMBER	PRODUCT CAN NUMBER	SAMPLE RESULTS (wt%)	Loss/ Gain	ORIGIN OF ITEMS IN PRODUCT CAN
B3054	64-94-11-73	- 0.0002	LOI	5-94-07-13
B3055	64-94-11-75	+ 0.4217	GOI	5-94-07-13 5-94-07-16
B3056	64-94-11-76	+ 0.3994	GOI	5-89-07-55 5-94-07-16
B3057	64-94-11-75	+ 0.1609	GOI	5-94-07-13 5-94-07-16
B3058	64-94-11-76	+ 0.1690	GOI	5-89-07-55 5-94-07-16
B3059	64-94-11-79	- 0.0269	LOI	5-89-07-55 10-88-11-359 19-93-09-35 19-93-09-37 19-93-09-38
B3060	64-94-11-81	- 0.3708	LOI	19-93-09-35 19-93-09-36 19-93-09-38 10-88-11-359 10-94-08-64 21-94-11-88
B3063	64-94-11-82	- 0.2198	LOI	18-88-02-33 19-88-01-10 19-90-07-23 21-89-07-49 62-84-04-214
B3064	64-94-12-90	- 0.2816	LOI	18-88-02-43 18-88-02-48 19-88-12-371

When looking at the results from the previous stabilized items, there were two out of five cans that showed weight gain. The two product cans with GOI results were resampled and the samples run again at 450° C instead of at 950° C. The sample results still showed a weight gain. Both cans with GOI results contained material that had been processed through HA-21I but had failed to be stabilized. The GOI is mostly likely related to the materials that made up item 5-94-07-16. The history of the item is shown below.

5-94-07-16 COMPOSITION

<u>ITEM NUMBER</u>	<u>MATERIAL TYPE</u>
• 5-89-07-65 (made from item 5-87-04-239)	HC-11 Powder Calcium Metal
• 5-89-07-68	Floor sweeps C-Line
• 63-88-06-101	HA-21I Processed Item
- Composed of items: 14-88-05-84-1	PRF Canyon Sludge
14-88-05-84-2	PRF Canyon Sludge
14-82-12-412	PRF Canyon Sludge
12-82-12-414	PRF Canyon Sludge

Expected LOI results on HC-21C processed items generally should be well below 1%. However, there may be an occasional anomaly where a weight gain is seen (GOI) or a greater than 1% LOI. With these product cans, tests will be run to determine the cause of the result and to further stabilize the item or verify its stability for long term storage.

From reviewing the blend plan there are a group of items to be stabilized that could potentially have problems with passing the LOI specifications and should be watched.

The list of items is below:

<u>ITEM NUMBER</u>	<u>MATERIAL TYPE</u>
• 14-82-12-272	PRF Canyon Solids
• 14-82-12-368	PRF Canyon Solids
• 14-82-12-288	PRF Canyon Solids
• 14-82-12-411	PRF Canyon Solids
• 14-82-12-307	PRF Canyon Solids
• 14-82-12-508	PRF Canyon Solids
• 14-82-12-309	PRF Canyon Solids
• 14-82-12-509	PRF Canyon Solids
• 14-82-12-344	PRF Canyon Solids
• 63-89-01-03	Stabilized PRF Scrap
• 62-485	Stabilized Scrap (BNW misc. scrap WG)

6.1 Safety

No changes to operating documents or design configuration are proposed by this test plan. The tests planned fall within the scope of the established safety envelope of the process.

A pre-job meeting shall be held prior to this task. The pre-job meeting shall be documented in Table 1 by Test Engineer (TE) and all persons involved in the meeting.

TEST ENGINEER

PRE-JOB SAFETY/OPERABILITY BRIEFING LIST

[illegible]

7 CHANGE CONTROL

Adjustments to the test procedures may be necessary in the field as the test run progresses. Therefore the Test Engineer is permitted to accommodate minor changes in direction resulting from facility description variants. Each change shall be approved per WHC-CM-3-5, Section 12.7. Change authorizations obtained by the telephone are permitted and shall be noted as such. Major revisions to the test plan can be made by an Engineering Change Notice (ECN).

The following tests can be performed on as many product cans as the TE determine need to be done in order to gain sufficient information. Any number of tests may be run on one item.

8 PROCEDURE

8.1 REPROCESS MATERIAL

- 8.1.1 Product slip-lid can has been sampled for storage and the results show a GOI. Record the following information pertinent to the product can being tested.

Can Number: _____

LOI Sample Number: _____

Sample Results: _____

Item Numbers that made up of the charges that make-up the product can: _____

ITEM NUMBER

MATERIAL TYPE

- 8.1.2 Prepare and process a charge per ZO-160-032 from the product can.

- 8.1.2.1 Test Engineer should record on observation data sheet any problems encountered during the furnace cycle or any observations.

- 8.1.3 When boat has completed the furnace cycle, sample boat before placing into a slip-lid can.

- 8.1.3.1 Stir boat material and mix as well as possible.

- 8.1.3.2 If chunks are in the boat use mortar and pestle to break up chunks.

- 8.1.3.3 Weigh boat and material for gross weight and record.
BOAT WEIGHT BEFORE SAMPLE: _____

- 8.1.3.4 Sample the boat's contents per ZO-200-537.

- 8.1.3.5 Reweigh boat and material for gross weight and record.
BOAT WEIGHT AFTER SAMPLE: _____

- 8.1.3.6 Subtract weights to get sample weight.
SAMPLE WEIGHT: _____

8.1.3.7 Obtain a sample number and ship sample to the lab. Record sample number:

SAMPLE NUMBER: _____

8.1.4 Process boat per Section F of ZO-160-032.

8.1.5 Record reprocessed material LOI result.

SAMPLE RESULT: _____

8.2 SIMULATED TGA

- 8.2.1 Product slip-lid can has been sampled for storage and the results show a GOI. Record the following information pertinent to the product can being tested.

Can Number: _____

LOI Sample Number: _____

Sample Results: _____

Item Numbers that made of the charges that make-up the product can: _____

ITEM NUMBER

MATERIAL TYPE

- 8.2.2 Sample can for simulated TGA analysis per ZO-200-537.

8.2.2.1 Send sample to the lab for the simulated TGA.

- 8.2.3 Collect test results from lab test or laboratory report. Results will be attached to process test report.

8.3 TGA WITH MASS SPECTROSCOPY ON OFF-GAS

- 8.3.1 Product slip-lid can has been sampled for storage and the results show a GOI. Record the following information pertinent to the product can being tested.

Can Number: _____

LOI Sample Number: _____

Sample Results: _____

Item Numbers that made of the charges that make-up the product can.

ITEM NUMBERMATERIAL TYPE_____

- 8.3.2 Sample can for TGA analysis per ZO-200-537 and send to PPSL.

- 8.3.2.1 Collect test results from laboratory test or laboratory report.
Results will be attached to process test report.

9 ORGANIZATION AND FUNCTION RESPONSIBILITIES

The Test Engineer shall be selected from PFP Engineering and has overall responsibility and authority over the Test Plan performance. Testing will be conducted by the Test Engineer (TE), Nuclear Operations, and Laboratory personnel. Each step requiring sign-off shall be initialed by the TE or designee. Data recording will be performed by TE or designee.

10 REPORTS

A report with the test results should be written when sufficient information has been collected.

11 DATA SHEETS

Data sheets for each furnace cycle, FURNACE CHARGE AND CYCLE DATA SHEET, from ZO-160-032 will be copied to provide information on the item processed. Observation sheet for test 8.1, DATA OBSERVATION SHEET, is include and should be filled out during the test.

DATA OBSERVATION SHEET (FOR TEST 8.1)

Item number used for charges: _____

Type of Material: _____

Observations or comments during the cycle:

TIME/DATE

COMMENT

Exceptions: