1 of 1

DISTRIBUTION SHEET

To PFP Process Engineering	From Plutonium Process Support Laboratories	Page 1 of 1
		Date 3/30/95
Project Title/Work Order Test Plan for Non-Radioactive Testing of a Vertical Calciner for Development of Direct Denitration Conversion of Pu-Bearing Liquors to Stable, Storable Solids		EDT No. 610903
		ECN No. N/A

Name	MSIN	Text With All Attach.	Text Only	Attach./ Appendix Only	EDT/ECN Only
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 APR 05 1995	ENGINEERING DATA TRANSMITTAL	Page 1 of 1 1. EDT Nº 610903
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2. To: (Receiving Organization) PFP Process Engineering	3. From: (Originating Organization) Plutonium Process Support Laboratories	4. Related EDT No.: 608701
5. Proj./Prog./Dept./Div.: PSS	6. Cog. Engr.: Fred D. Fisher	7. Purchase Order No.: N/A
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		12. Major Assm. Dwg. No.: SK-2-300303
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		14. Required Response Date: 2/24/95

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1	WHC-SD-CP-TP-083		0	Test Plan for Non-Radioactive Testing of a Vertical Calcliner Development for Direct Denitration Conversion of Pu-Bearing Liquors to Stable, Storable Solids	SQE	1		

16. KEY					
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		Safety	<i>[Signature]</i>	2-16-95	TS-14				
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18. Signature of EDT Originator <i>[Signature]</i> 3/30/95	19. Authorized Representative Date for Receiving Organization <i>[Signature]</i> 3/31/95	20. Cognizant Manager Date <i>[Signature]</i> 2/16/95	21. DOE APPROVAL (if required) Ctrl. No. ☐ Approved ☐ Approved w/comments ☐ Disapproved w/comments
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RELEASE AUTHORIZATION

Document Number: WHC-SD-CP-TP-083, REV 0

Document Title: Test Plan for Non-Radioactive Testing of Vertical
Calciner for Development of Direct Denitration
Conversion of Pu-Bearing Liquors to Stable, Storage
Solids

Release Date: 4/4/95

**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

April 4, 1995

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SUPPORTING DOCUMENT		1. Total Pages 25
2. Title Test Plan for Non-Radioactive Testing of a Vertical Calciner for Development of Direct Denitration Conversion of Pu-Bearing Liquors to Stable, Storable Solids	3. Number WHC-SD-CP-TP-083	4. Rev No. 0
5. Key Words Test Plan, Direct Denitration, Vertical Calciner, Pu-Bearing Liquors	6. Author Name: Fred D. Fisher Signature Organization/Charge Code 8E120 K6032	
7. Abstract Plutonium-bearing liquors, including ANL scrap liquors, will be used for development and demonstration of a vertical calciner direct denitration process for conversion of those liquors to stable, storable PuO ₂ -rich solids. This test plan is to test with non-radioactive stand-in materials to demonstrate adequate performance of the vertical calciner and ancillary equipment.		
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<p style="text-align: center; font-size: 2em; font-weight: bold;">MASTER</p>		<p>8. RELEASE STAMP</p> <div style="border: 1px solid black; padding: 5px; text-align: center;"> <p>OFFICIAL RELEASE BY WHC</p> <p>DATE APR 05 1995</p> <p>21A 4</p> </div>
A-6400-073 (08/94) WEF124		

TEST PLAN FOR NON-RADIOACTIVE TESTING OF A VERTICAL CALCINER FOR DEVELOPMENT OF DIRECT DENITRATION CONVERSION OF PU-BEARING LIQUORS TO STABLE, STORABLE SOLIDS

1.0 INTRODUCTION

Plutonium-bearing liquors, including ANL scrap liquors, containing plutonium and nitric acid and, possibly, uranium and thorium and hydrochloric, sulfuric, phosphoric and hydrofluoric acids and minor amounts of other substances will be used for development and demonstration of a vertical calciner direct denitration process for conversion of those to stable, storable PuO_2 -rich solids. Some of those liquors are quite dilute and very impure and may first be subjected to various processes for beneficiation, concentration, and purification before direct denitration conversion to PuO_2 -rich solids suitable for long-term vault storage. Untreated ANL scrap liquors containing some amounts of chloride and, possibly, fluoride, sulfate, phosphate, sodium and/or potassium may also be tested for suitability of direct denitration for conversion directly to PuO_2 -rich solids.

In the vertical calciner direct denitration process to be examined, small additions of liquid feed are metered into a continuously heated and stirred bed of previously generated product solids. The liquid feed is rapidly evaporated, then, more slowly, undergoes drying and denitration and final heat treatment to stable PuO_2 . The PuO_2 product may contain some residual sulfate and/or phosphate-derived impurities, but will be substantially free of chloride, fluoride, and other volatile acid impurities. Offgas condensates are expected to be non-TRU. The process is known to work with plutonium, thorium, uranium, and mixtures of those elements in concentrations ranging from 15 to 500 g/L. That range will be extended down to ca. 5 g/L.

Problems include materials of construction resistant to abrasion and to HCl and HF at 500°-800°C. Since there is no continuous liquid phase, corrosion is limited to vapor phase attack. Ordinary stainless steel may be adequate for the short term although it would exhibit some oxide scaling on the hotter portions of the calciner. A somewhat higher alloy will be more resistant to oxide scaling. The abrasion problem is not extremely severe and since shaft seals submerged in the powder bed are avoided by design, alloy construction will be adequate. Hard facing of impellers is not believed to be necessary. Suitable bearings for the impeller shaft present formidable materials problems.

The process is not particularly energy efficient from very dilute feeds and cannot handle feeds high in sodium and potassium or other constituents which form nitrates which are molten and refractory at $\leq 800^\circ\text{C}$. Organic impurities are largely consumed and only partly report to the offgas.

A vertical calciner has been built and will be used along with associated ancillary equipment in GB 188-1 to demonstrate direct denitration processing of various plutonium solutions to stable, storable PuO_2 -rich solids. Prior to that, it will be temporarily installed in Hood 202-1 and operated "cold," i.e., with non-radioactive stand-in materials to demonstrate adequate performance of the calciner and most of its associated ancillary equipment items

and systems. Deficiencies noted during that cold testing will be corrected and the adequacy of those corrections demonstrated by further cold testing in Hood 202-1. That cold testing program is the subject of this test plan.

2.0 OBJECTIVE

1. To demonstrate, prior to commitment to plutonium, the adequacy of performance of the critical components of a vertical calciner and ancillaries system including the identification and correction of any performance deficiencies.
2. To familiarize personnel in calciner and scrubber operation prior to operation with plutonium in a glovebox.

3.0 SCOPE

A production-scale vertical calciner and most of its ancillary equipment will be operated by the Plutonium Process Support Laboratories for several hours to days with non-radioactive materials in Hood 202-1. Operation will include periodic disassembly to examine equipment condition. Many of the process controls to be used later for radioactive testing will also be employed in this testing.

4.0 DESCRIPTION OF TEST

4.1 Test Items

The vertical calciner, built from high chromium, high nickel Type 310 stainless steel according to Drawings SK-2-300303, SK-2-300305, SK-2-300309, and field insulated, will be installed in a large fume hood in Rm 202 of the 234-5 Building, 200-West Area, along with the critical supporting equipment intended for eventual use with the calciner when it is operated on plutonium-bearing solutions in GB 188-1. That supporting equipment includes an offgas chiller-scrubber, Drawing SK-2-300306, a Feed Tank, Drawing SK-300307, and the 10-L Denitration Calciner Control Drawing SK-2-824342. Also used will be temporary mounting supports and wiring and instrument lines, temporary interconnecting piping and service lines, a feed metering pump, and vacuum, compressed air, and cold water utilities. Electrical services will be via a temporary load center tapped into existing 480-volt utility service.

The test items will include an about 4-inch deep magnesium oxide starter bed in the vertical calciner and non-radioactive stand-in feedstreams of pure water and water solutions of magnesium nitrate. Magnesium nitrate solution undergoes evaporation and thermal decomposition in the heated stirred bed vertical calciner largely according to:

in a fashion analogous to that anticipated with plutonium-bearing solutions. The magnesium oxide increases in the bed overflow to a product receiver vessel below the calciner.

The chiller-scrubber will be used during this test program with once through tap water in the cooling coil rather than recirculating chilled water. The inlet temperature will be about the same and the performance expected from the unit in plutonium service will be adequately simulated. The chiller-scrubber will be operated with sodium hydroxide scrubbing reagent introduced as discrete batches with periodic total discharge of spent scrub liquor rather than with continuous sodium hydroxide introduction with constant overflow of spent scrub liquor. This is done to enable disposal of spent scrub liquors as Hazwaste in batches since the drains in Rm 202 cannot accept chemical wastes. The heat transfer adequacy of the chiller-scrubber was demonstrated to be satisfactory in a full-scale full-temperature mockup by the Chemical Engineering Laboratory. See Appendix A for Internal Letter OM540-95-013. By analogy, the mass transfer capability should be more than adequate. Gross deficiencies in the mass transfer will manifest themselves as visible N_2O_4 fumes in the vapor space above the scrub liquor in the chiller-scrubber. The feed injection pump will be calibrated with 100 g/L magnesium nitrate hexahydrate solution in this cold testing. Other items to be tested include the vacuum regulator, one six point thermocouple recorder, both calciner heaters and their power supplies and controllers.

4.2 Test Environment

The testing will be performed in a large fume hood in Rm 202 of the 234-5 Building, 200-West Area. Control and measurement equipment will be arranged around that hood, as appropriate. The hood provides confinement of magnesium oxide, magnesium nitrate, and nitrogen oxide releases and simulates, to some degree, the environment expected during radioactive testing.

4.3 Equipment and Facilities

Equipment, other than that being tested, will include a vacuum pump system and compressed air supply. A Digistrobe¹ will be used for calibration of the calciner agitator drive controller. Various vessels will be used for reagent preparation and storage, and several permanent and movable benches will be used for temporary location of test equipment and for conduct of supporting operations.

4.4 Data

The calciner and ancillary equipment being tested will be ultimately operated for several hours at simulated full load and at design temperatures of 1000°C furnace(s) temperatures and 800°C lower shell temperature. Other temperatures will be measured. Time-temperature profiles will be inferred for product powders as a function of feed rate, air rate, and stirring rate. The performance of the filters and associated blowback system will be

¹ Digistrobe is a registered trademark of Cole-Parmer Instrument Co.

assessed from pressure differential determinations and, as appropriate, visual inspections. The sequence of tests will bring the calciner and critical ancillary equipment to full simulated operation in the following stages:

1. Bring shell to 450°C with atomizing air at 2 SCFM. Hold for one hour. No bed, no stirring.
2. Bring shell to 800°C with atomizing air at 2 SCFM. Stirrer at 60 RPM. Hold for one hour. No bed. Measure temperatures.
3. Bring shell to 800°C with atomizing air at 2 SCFM, 4-inch MgO bed, 3 L/hr water, stirrer at 60 RPM, hold for one hour. Measure temperatures.
4. Bring shell to 800°C, 4-inch MgO bed, stirrer at 60 RPM, 100 g/L $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ feed solution at 3L/hr, hold for two hours, measure temperatures. Observe chiller/scrubber overheads for N_2O_4 . Do a material balance around the calciner and scrubber for magnesium and nitrate-nitrite.
5. Same as Step 4 except $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ solution at 4 L/hr.
6. Repeat any of above steps as indicated by observations.
7. After each test, visual examination of accessible major components will be conducted to determine any wear, material fatigue, apparatus failure, and overall condition of the system. After the completion of testing, Step 6, disassemble and examine the calciner for the following:
 - a. Visual examination of accessible welds for cracking.
 - b. Visual examination of stirrer bearings, journals for cracking, scoring, perceptible wear.
 - c. Micrometer measurement of bearing journals to quantify wear rates.

4.5 Criteria/Constraints

These tests do not require revision to the PFP FSAR. A USQ review, USQ-PFP-94-05, was performed on the plutonium testing to follow and this cold testing.

5.0 EXPECTED RESULTS

Hoped for results are that the calciner and its associated ancillaries function smoothly and adequately and that adequate throughput capability is exhibited. Expected results are that some deficiencies will become apparent and that some remedial modifications will be required. Those will also be tested. This test plan will be amended, reviewed, and reissued

as appropriate in accordance with WHC-CM-6-1, Standard Engineering Practices, EP-1.12, Rev. 8, Change 2, "Supporting Document Requirements."

6.0 TEST PROCEDURE

I. Calibration of Feed Pump

1. Calibrate the feed injection pump for 3 and 4 Liter per hour rates using water and the calibration procedure in Section 7.0 of Manual, Electronic Metering Pumps, Installation Maintenance and Troubleshooting, LMI Milton Roy.
2. Repeat 1 above with 100 g/L $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ solution.

II. Calibrate the Stirrer Drive Assembly

1. Calibrate the stirrer drive assembly using the instructions of Manual, Instructions for Installation and Operation Type-FPM Controls with Driver Board Only or with Electronic F-B-R Board, Bodine Electric Co.
2. Measure RPM with Digistrobe unit and four marks at 90° apart on the upper stirrer shaft collar. Actual RPM is one-fourth of the Digistrobe indication.
3. Calibrate for 40, 60, 80, 100 RPM.
4. Visually observe the set screw repeat frequency to be about once per second to avoid a misleading reading by having the Digistrobe set for twice or one-half the desired RPM x 4.

III. Calibration of Temperature Controller

NOTE: Thermocouples and the temperature controller need not actually be calibrated.

1. Program temperature controller using those instructions in Manual, Eurotherm 818 Controller, Programmer, Operating Instructions, Eurotherm Corporation.

IV. Test #1 Low Temperature Normalization Run

1. Turn on main power switch.
2. Program the temperature controller for a 5°C/min ramp up to 450°C, one hour soak at 450°C, ramp down at 7°C/min.
3. Feed pump off.
4. No bed.

5. Fill scrubber with water. Turn on scrubber water flow at about 1 gpm.
6. Set atomizing air flow at 2 SCFM.
7. Initiate preprogrammed temperature cycle. Turn on temperature recorder. Temperature traces should all be coincident. Replace any temperature element, i.e., thermocouple, whose trace differs from the other traces by more than 5°C.
8. Turn on vacuum pump. Adjust for positive bubble rate in scrubber.
9. Identify temperature element (TE) numbers on the temperature traces near the beginning of the run. Make and label a time mark on the chart.
10. Turn off furnace power, atomizing air, vacuum scrubber water flow when temperature controller $\leq 200^{\circ}\text{C}$.
11. When cold, examine accessible components for wear, fatigue, apparatus failure, overall condition of the system.

V. Test #2 High Temperature Normalization Run

1. Turn on main power switch.
2. Program the temperature controller for ramp up at 8°C/min to 800°C, one hour soak at 800°C, ramp down at 10°C/min.
3. Set atomizing air at 2 SCFM.
4. Turn on temperature recorder. Temperature traces should all be coincident. Replace any temperature element, i.e., thermocouple, whose trace differs from the other traces by more than 5°C.
5. Initiate preprogrammed temperature cycle. Turn on temperature recorder.
6. Fill scrubber with water to within one foot of top. Turn on scrubber chilled water at about 1 GPM.
7. Feed pump OFF.
8. No bed.
9. Turn on vacuum pump. Adjust valve for positive bubble rate in scrubber.
10. Identify temperature element (TE) numbers on traces near beginning of run. Make and label a time mark on chart.
11. Turn on stirrer at 60 RPM when temperature controller indicates 400°C.

12. Turn off furnace power, atomizing air, vacuum pump, scrubber chilled water, stirrer, when temperature ramp down is $\leq 200^{\circ}\text{C}$.
13. When cold, examine accessible components for wear, fatigue, apparatus failure, overall condition of the system.

VI. Test #3. Water Feed Simulation Run

1. Fill the feed tank with water.
2. Turn on main power switch.

NOTE: Controller may already be programmed from Test #2.

3. Confirm the program for temperature controller for ramp up at $8^{\circ}\text{C}/\text{min}$ to 800°C , soak for one hour at 800°C , ramp down at 10°C minute.
4. Turn on temperature recorder. Temperature traces should all be coincident. Replace any temperature element, i.e., thermocouple, whose trace differs from the other traces by more than 5°C .
5. Charge water to the scrubber to about one foot from the top flange.
6. Turn on vacuum pump. Adjust valve for positive bubble rate in scrubber.
7. Remove the pressure relief valve floater on the calciner head and insert a plastic funnel.
 - a. Feed 0.8 Kg of MgO through that funnel into the calciner.
 - b. Replace PRV floater.
8. Turn on cold water flow to scrubber coil. Set at about 1 gal/min.
9. Turn on atomizing air at 2 SCFM.
10. Initiate preprogrammed heating, soaking, cooling cycle.
11. Turn on blowback air. Set pressure regulator to 40 psig.
 - a. Set automatic backflush timer KT188A to 30 seconds OFF and 150 seconds ON.
 - b. Set pulse timer KT188B to 0.5 seconds ON, 78 seconds OFF.
 - c. Set pulse timer KT188C to 0.5 seconds ON, 113 seconds OFF.

- d. Set pulse timer KT188D to 0.5 seconds ON, 148 seconds OFF.

NOTE: That will give 0.5 seconds duration blowback pulses to each filter every 3 minutes with the second filter blowback pulse lagging the first by 35 seconds and the third lagging the second by 35 seconds.

12. Should this be inadequate, as indicated by actuation of the high differential pressure alarm:
 - a. Reset the KT188A timer to 30 seconds OFF and 90 seconds ON.
 - b. Reset the KT188B timer to 0.5 seconds ON and 46 seconds OFF.
 - c. Reset the KT188C timer to 0.5 seconds ON and 67 seconds OFF.
 - d. Reset the KT188D timer to 0.5 seconds ON and 88 seconds OFF.

NOTE: That will blowback each filter once each two minutes with 21 seconds between blowback pulses to the first and second filters and to the second and third filters.

13. If still inadequate, raise blowback air pressure to 60 psig.
14. Identify various temperature traces vs. the associated temperature element identities on the recorder chart. Label recorder chart as Test #3. Make and label a time mark on the chart.
15. When the controller temperature indication is about 400°C, turn on the stirrer drive at 60 RPM. Check that with the Digistrobe. Adjust to 60 RPM if necessary. Note and record time and speed controller setpoint.
16. When controller temperature indication is 800°C, turn on the feed pump and set to 3 L/hr. Note and record time and feed tank level.
17. Turn off feed pump when furnace controller begins temperature ramp down. Note and record time and feed tank level.
18. When furnace controller temperature indication is $\leq 400^{\circ}\text{C}$, check stirrer RPM with Digistrobe. Note and record actual RPM and drive controller setpoint. Turn off stirrer drive. Record time.
19. When furnace controller temperature indication is $\leq 200^{\circ}\text{C}$, turn off atomizing air, blowback air, vacuum, cold water to scrubber coil, furnace controller, recorder, main power switch.
20. When cold, examine accessible components for wear, fatigue, apparatus failure, overall condition of the system. Record findings.

21. Empty MgO receiver, replace.

VII. Test #4. Low Rate Magnesium Nitrate Simulation Run

1. Make up 4 liters of 100 g/L $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ solution. Put into feed tank.
2. Turn on main power switch.

NOTE: Controller may already be programmed from Test #3.

3. Confirm the program for temperature controller for ramp up at $8^\circ\text{C}/\text{min}$ to 800°C , soak for one hour at 800°C , ramp down at 10°C minute.
4. Turn on temperature recorder. Temperature traces should all be coincident. Replace any temperature element, i.e., thermocouple, whose trace differs from the other traces by more than 5°C .
5. Turn on vacuum pump. Adjust valve for positive bubble rate in scrubber
6. Turn on cold water flow to scrubber coil. Set at about 1 gal/min.
7. Turn on atomizing air at 2 SCFM. Turn on blowback air supply. Set as in Run #3.
8. Initiate preprogrammed heating, soaking, cooling cycle.
9. Identify various temperature traces vs. the associated temperature element identities on the recorder chart. Label recorder chart as Test #4. Make and label a time mark on the chart.
10. When the controller temperature indication is about 400°C , turn on the stirrer drive at 60 PFM. Check that with the Digistrobe. Adjust to 60 RPM if necessary. Note and record time and speed controller setpoint.
11. When controller temperature indication is 800°C , turn on the feed pump and set to 3 L/hr. Note and record time and feed tank level.
12. Turn off feed pump when furnace controller begins temperature ramp down. Note and record time and feed tank level.
13. When furnace controller temperature indication is $\leq 400^\circ\text{C}$, check stirrer RPM with Digistrobe. Note and record actual RPM and drive controller setpoint. Turn off stirrer drive. Record time.

14. When furnace controller temperature indication is $\leq 200^{\circ}\text{C}$, turn off atomizing air, blowback air, vacuum, cold water to scrubber coil, furnace controller, recorder, main power switch.
15. When cold, examine accessible components for wear, fatigue, apparatus failure, overall condition of the system. Record findings.
16. Empty MgO receiver, weigh MgO, replace receiver.
17. Drain scrubber liquor to Hazmat carboy. Measure volume. Sample for pH, nitrate, and nitrite.

VII. Test #5. Full Flow Rate Magnesium Nitrate Simulation Run

1. Repeat steps 1 through 17 of Test #4 procedure except feed pump setting to be 4 L/hr and Recorder Chart to be labeled Test #5.
2. Disassemble calciner into subassemblies. Examine accessible welds, bearings, bearing journals, sprockets and chain, filters, gaskets, heating elements for wear, scoring, general condition. Measure bearing journals.

7.0 SAFETY

This testing involves high-temperature, electrically heated chemical processing equipment. The thermally hot equipment is extensively insulated, but warm surfaces may still be accessible. Temperature-resistant Kevlar² gloves will be worn around the hot equipment.

Bare electrical connections will not be accessible to casual contact.

Chemical safety will be assured by rigid adherence to the PPSL Chemical Hygiene Plan. The MSDSs for MgO, $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ are available in Rm 202.

The stirrer of the vertical calciner is chain driven. That drive train is extensively shrouded and is inaccessibly high in the hood. It will be subjected to Lock and Tag procedures for maintenance or inspections as will the furnaces and their power supplies and the feed pump.

8.0 QUALITY ASSURANCE

Test plans, detailed procedures, equipment acquisition documents, work control documents, equipment items, and equipment installations, have been and will continue to be afforded QA participation as required for Safety Class 3 equipment.

² Kevlar is a registered trademark of E. I. duPont de Nemours and Co.

9.0 ORGANIZATION AND FUNCTION RESPONSIBILITIES

These tests and examinations will be performed by PPSL personnel. The cognizant engineer/test director has reviewed this test plan, vis-a-vis facility requirements. Facility Operations will supply hood ventilation and utilities services. There are no safeguards or security implications. PFP Safety will review test facilities and test plans and procedures; PFP Environmental Management will provide disposal of spent scrub liquors and excess reagents. Quality assurance will continue to review engineering documents, purchases, and test plans and procedures. PFP Design Engineering will provide sketches and other design documents for the fabrication and installation of equipment and the connections to utilities.

10. SCHEDULE

These tests are to begin upon completion of equipment acquisition and installation. That will not occur prior to April 1, 1995. Tests are anticipated to require three weeks if no deficiencies are uncovered. Correction of deficiencies and retesting could take up to five weeks.

11.0 REPORTS

A final test report will be issued shortly after completion of these tests. It will be issued as a supporting document in accordance with EP-1.12. Progress of the testing may also be reported weekly in the PPSL Weekly Report.

12.0 REFERENCES

Drawings:

SK-2-300303		10L/Calciner Assembly
SK-2-300304		10L/Calciner Glovebox 188-1 Assembly
SK-2-300305		10L/Calciner Glovebox 188-1 Misc Details
SK-2-300306		10L/Calciner Scrubber Assembly
SK-2-300307		10L/Calciner Glovebox 188-1 Feed Tank
SK-2-300308		10L/Calciner Glovebox 188-1 Waste Tank
SK-2-824342	sh 1 of 8	10-L Denitrate Calciner Control Arrangement
SK-2-824342	sh 2 of 8	10-L Denitrate Calciner Control Panel Details
SK-2-824342	sh 3 of 8	10-L Denitrate Calciner Control Ladder Diagram
SK-2-824342	sh 4 of 8	10-L Denitrate Calciner Control Wiring Diagram
SK-2-824342	sh 5 of 8	10-L Denitrate Calciner Control Connection Diagram
SK-2-824342	sh 6 of 8	10-L Denitrate Calciner Control Details
SK-2-824342	sh 7 of 8	10-L Denitrate Calciner Control Wire Run and Diagrams
SK-2-824342	sh 8 of 8	10-L Denitrate Calciner Control Views and Details

Manuals:

Agastat Specification Grade, Catalog 55D-1, Amerace Electronic Components.

Commander PR100 Advanced Process Recorder, ABB Kent-Taylor, Inc.

Electronic Metering Pumps, Installation, Maintenance, Troubleshooting, with LE-71S, LE-72S, LE-75S, Liquid Handling Assembly Supplement and Series C7 Split Board Unipulser Models Instruction Supplement, LMI Milton-Roy.

Eurotherm 818 Controller/Programmer Operating Instructions, Eurotherm Controls, Inc.

Instructions for Installation and Operation, Type-FPM Controldds with Driver Board Only or with Electronic F-B-R Board, Bodine Electric Company.

13.0 DATA SHEETS

Data shall be recorded in Controlled Laboratory Notebook, WHC-N-967-1, with recorder traces or any other mechanically produced data stored separately as noted in that notebook. Those data items will be identified on the PPSL RIDS plan as record material.

HANFORD JOB HAZARD ANALYSIS CHECKLIST										Page 1 of 2	
Prepared By Fred D. Fisher					Date 02/13/95		Area 200W		Bldg. 234-5Z		
Scope/Description: Non-Radioactive Testing of a Vertical Calciner for Direct Denitration Processing of Plutonium Solution.										☒ New ☐ Revised	
Emergency Contact Person(s): Primary: Fred D. Fisher Secondary: Caroline S. Sutter Emergency Radio/Phone Number: 33721 32757										JHA Number (not required):	
Specific Work Location(s): Rm 202, Bldg 234-5Z, 200-West Area											
KNOWN OR POTENTIAL HAZARDS											
	Yes	No	✓	●	Reference		Yes	No	✓	●	Reference
1. Radiation Area Work		X	✓	●		10. Respiratory Hazards		X	✓	●	
2. Hazardous Waste Operations	X		✓	●		11. Electrical Hazards	X		✓	●	
3. Confined Space Entry		X	✓	●		12. Lock and Tag	X		✓	●	
4. Cutting/Welding		X		●		13. Scaffolding		X		●	
5. Roof Work		X		●		14. Aerial Lifts		X	✓	●	
6. Fall Hazards (> = 10')		X		●		15. Asbestos Removal		X	✓	●	
7. Excavation/Trenching		X		●		16. Other (see JHA Sht. 2):	X				
8. Asbestos Inspection Report		X		●		✓ = Formal training required. ● = Items that require a permit/form/report.					
9. Hazardous Materials	X		✓	●							
Other Hazards					Yes	No	Control Measures				
1. Temperature Extremes					X		Furnace elements 1000oC, Calciner shell 800oC, See PG 2 #1				
2. Noise					X		Noise from hood fan/stirrer drive; ear plugs, if necessary				
3. Poor Lighting						X					
4. Animals/Insects						X					
5. Process Chemicals/Steam					X		Mg(NO3)2.6H2O, N2O4 fumes, Hot air and steam				
6. Dust						X					
7. Flammable/Combustible Materials						X					
8. Ladders						X					
9. Wet/Slippery Floors						X					
10. Uneven Terrain						X					
11. Open Excavations/Trenches						X					
12. Adjacent Water Hazard						X					
13. Vehicle Traffic						X					
14. Heavy Equipment						X					
15. Rigging Operation						X					
16. Manual Lifting					X		Unit weighs about 100 lbs, heaviest subass'y about 25 lbs				
17. Power Tools						X					
18. Pinch Points					X		Chain drive is heavily shrouded and inaccessible				
19. Falling Objects						X					
20. Sharp Objects						X					
21. Overhead Obstructions						X					
22. Site Control (Signs/Barricades)						X					
23. Remote Work Area						X					
24. Other (see JHA Sht. 2):					X						
MINIMUM DRESS REQUIREMENTS: Long sleeved blue coveralls, Kevlar gloves											
APPROVALS											
Does further evaluation of the job steps, associated hazards, or safety measures need to be performed? ☒ Yes ☐ No If Yes, continue job hazard analysis on the following pages.											
Supervisor, Person in Charge (Signature)						Industrial Safety/Hygiene (Signature)					

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Appendix A: Test Report for PFP Calciner Scrubber Water Hammer Test

From: Chemical Engineering Laboratory OM540-95-013
Phone: 3-5014 S4-25
Date: February 6, 1995
Subject: TEST REPORT FOR PFP CALCINER SCRUBBER WATER HAMMER TEST

To: James A. Compton T5-12
cc: L. C. Brown L5-62
G. B. Chronister T5-50
S. B. Merrick T4-20
J. E. Sherling T4-20
M. J. Schliebe S4-25 (Original initialized by
C. S. Sutter T5-12 M. J. Schliebe)

MDB File/CEL LB

REFERENCES:

- 1) King, Craig V. and Michael D. Britton, 1994, *Test Plan For PFP Calciner Scrubber Water Hammer Test*, (internal letter 8A400-94-044, to James A. Compton, December 6), Westinghouse Hanford Company, Richland, Washington.
- 2) Compton, James A., 1994, *Letter of Instruction for Calciner Scrubber Heat Transfer Testing*, (internal letter 8E120-94-099, to M. D. Britton, November 17), Westinghouse Hanford Company, Richland, Washington.
- 3) Britton, Michael D., 1994, *PFP Calciner Scrubber Heat Transfer Test*, Notebook number WHC-N-1102 1, Westinghouse Hanford Company, Richland, Washington.

INTRODUCTION:

The Plutonium Finishing Plant (PFP) currently holds approximately 250 liters of dilute plutonium solutions in 10-liter polyethylene bottles from Argonne National Laboratory (i.e., "10-L Solutions"). The solutions also contain nitric acid and hydrochloric acid. These solutions must be removed from the polyethylene containers and stabilized. Solution removal is required to prevent eventual leakage. These solutions will then be used to demonstrate various methods of stabilizing other plutonium solutions inside the PFP.

A direct denitration calciner has been designed as one method for stabilizing solutions. The calciner heats the plutonium nitrate solution directly (i.e., without adding additional chemicals) to boil away the nitric acid solution and decompose the nitrate ions associated with the plutonium and nitric acid. Crystalline plutonium dioxide is the calciner's product while an off-gas stream of nitrous or nitric acid decomposition gases is also produced. The hydrochloric acid in the 10-L solutions also vaporizes into the off-gases.

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The nitric acid decomposition gases (assumed to be mostly NO_2 after exiting the calciner) and the hydrogen chloride vapors must be scrubbed from the calciner off-gas stream to prevent unnecessary corrosion to the building vacuum system. A scrubber is being designed to remove the NO_2 and HCl vapors and to cool the gases as much as possible given the space limitations inside the glovebox. The scrubber design is hampered by a lack of information regarding heat transfer rates from ascending bubbles in a moving liquid stream and scrubbing efficiency.

The Plutonium Process Support Laboratories and Plutonium Finishing Plant Design Engineering requested the Chemical Engineering Laboratory to furnish relevant information regarding the performance of the proposed scrubber design.

OBJECTIVES:

The objectives of this test were: 1) to identify whether water hammering will occur under the operating conditions; 2) to determine the temperature of the gas exiting the system and the temperature profile in the scrubber during operation; and 3) to determine the effects of raising and lowering the feed tube in the scrubber. This test was performed according to the test plan (reference 1) with the exception that only two feed tube positions were used rather than seven different positions.

SCOPE:

This test was designed to investigate part of the heat transfer capabilities of the proposed scrubber design. Gas scrubbing efficiency was assumed to be similar to the heat transfer efficiency. This test was a simple demonstration of an air/steam mixture exiting a pipe into a flowing water bath.

TEST SCRUBBER:

The test scrubber, shown in Figure 1, was constructed nearly identically to the design of the actual scrubber to be used in the PFP calcining operation. Information on the calciner scrubber were described in reference 2. The outer casing of the scrubber was constructed of a 6-inch glass pipe, 120 cm in length, and was flanged shut at both ends using

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Figure 1. Scrubber Design.

stainless steel flanges and Teflon³ gaskets. The feed tube was a 1-inch schedule 40 stainless steel pipe with a long 10- μ m sintered stainless-steel dispersion filter on the end. The filter was 2.54 cm in diameter and 5.08 cm

³Teflon is a registered trademark of E.I. DuPont de Nemours & Co.

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in length. The draft-tube cooling-coil was constructed of $\frac{1}{4}$ -inch copper tubing and had a 9.53 cm inner diameter and a 76 cm height.

There were two primary differences between the test scrubber and the PFP calciner scrubber. The first difference was that the draft-tube cooling-coil was constructed from copper tubing rather than stainless steel. This was done because a stainless-steel tubing coil is much more difficult and expensive to construct and the difference in heat transfer rates between copper and steel are small and not important for this test.

The second difference was that the feed pipe in the middle of the scrubber was a stainless-steel pipe with a 10- μ m sintered stainless-steel dispersion filter rather than a quartz pipe with a glass dispersion filter. It is believed that the bubbles formed by the stainless-steel filter are similar in size to the bubbles that will be made by the glass dispersion filter. This deviation from the PFP design was made to cut the cost of this test. The dispersion feed pipe was also constructed so that its height in the scrubber was variable.

To simulate the feed to the calciner scrubber, a 50/50 (by volume) air/steam gas mixture was used. This gas mixture was above 102°C and fed to the scrubber at a rate of approximately 0.139 normal m³/min (Nm³/min). Other conditions used to mimic the calciner scrubber operating conditions were a 4 L/h flowrate for the scrub solution into the scrubber and a calm water depth of the scrub solution in the scrubber of 1 m.

EXPERIMENTAL APPARATUS:

Figure 2 shows a flow diagram of the experimental apparatus that was constructed and operated in the Chemical Engineering Laboratory. The steam generator was a Coats Boiler (model number 12S). The steam flow was controlled by adjusting a pressure relief valve (V-2). The steam flowrate was measured by diverting the steam flow into a condenser cooled by a refrigerated recirculator (chiller) and measuring the retrieved condensate per time. Valves V-3 and V-5 controlled whether the steam flow was directed towards the condenser or the superheater.

Compressed air was metered through a variable area rotameter and then through a tubular heater to elevate the temperature. The hot air was then mixed with the steam and fed to the superheater.

The superheater was constructed from an 8-inch stainless steel pipe, 33.6 cm (14 inches) in length with plates of stainless steel welded to each

Figure 2. Calciner Scrubber Test Apparatus Flow Diagram.

end. A 1-inch inlet pipe was connected to the side of the pipe. The 1-inch outlet pipe was attached to the bottom end of the superheater. Six type CIR cartridge heaters were installed in the top end of the superheater. Each heater was 30.5 cm (12 inches) in length, 550 W, and 120 V. Three of the heaters were wired for constant full power. The other three heaters were attached to a variable autotransformer to provide manual temperature

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adjustment.

The superheated air/steam mixture then flowed into the scrubber through a 1-inch feed tube. The offgas exited from the scrubber through a 1-inch tube and was routed to a sump. The scrub solution (water) was metered into the scrubber through a $\frac{1}{4}$ -inch tube connection using a peristaltic pump. To maintain the height of the scrub solution inside the scrubber at 1 m, a Tygon⁴ tube on the scrub solution outlet was elevated to form a gravity decanter. Tap water was valved to flow through the draft tube cooling coils and into a sump.

As a safety precaution, a sheet of clear Lexan⁵ was installed around the scrubber in case severe water hammering occurred and caused the glass pipe to shatter.

⁴Tygon is a registered trademark of Norton Company.

⁵Lexan is a registered trademark of the General Electric Company.

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PROCEDURES:Scrub Solution Flowrate Calibration Procedure

The scrub solution flowrate was calibrated to 4 L/h by measuring the mass of water pumped per time. The adjustable flowrate dial was then locked in place for the remainder of the test.

With scrub solution feeding into the scrubber, the scrub solution drain tube was raised to keep the calm liquid depth in the scrubber at 1 m.

Steam Flowrate Calibration Procedure

1. Open valve V-5 and close valve V-3 so the steam will pass through the condenser.
2. Start the chiller and set it for 2°C.
3. Check the steam lines for moisture and drain any moisture that may be present before starting the steam generator.
4. Startup the steam generator per procedure WHC-IP-0874-6.1.
5. Partially open the steam feed valve V-2.
6. Begin timing the collection of the water using a tared collection container.
7. After several minutes weigh the collected water. If the mass flowrate is not 50.6 g/min, adjust the steam feed valve V-2 to move it closer to that quantity and repeat steps 6 and 7 until it is within 50%.
8. Once the proper setting of valve V-2 is achieved, shutdown the steam generator as required by the steam generator operating procedure WHC-IP-0874-6.1. Label valve V-2 with "Do Not Touch." It is not necessary to shut the generator down if operation is to be performed immediately following the completion of this test.

Startup Procedure

1. Start the peristaltic pump. The flowrate should be 4 L/h.
2. Check that the liquid level in the scrubber is being maintained at 4 L/h.
3. Open valves V-3 and V-5, let any condensate drain from the system, and close these valves.
4. Start the video camera with it pointed at the scrubber.
5. Set the air flow at 0.0695 Nm³/min by adjusting valve V-1.
6. Start the air preheater and the heaters in the superheater.

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7. Start water flowing through the draft-tube cooling-coil.
8. Start the steam generator per procedure WHC-IP-0874-6.1 (unless it is already operating) and check the steam flowrate. Calibrate if necessary. (See Steam Flowrate Calibration Procedure)

Operation Procedure

Note: Do not begin until the superheater exit gas (thermocouple T-12) exceeds 100°C.

1. Open valve V-3 and close valve V-5 so that the steam flow is directed into the preheated air stream.
2. Allow the system time to reach equilibrium. This will occur when the temperatures measured by the system thermocouples have stabilized.
3. Record the temperatures and pressures in the laboratory notebook.

Note: If at any time the pressure in the low pressure side exceeds 103 kPa or water hammer occurs, shut the system down by first turning off the boiler, then the hot air stream and power to the superheater and preheater, otherwise continue.

Shutdown Procedure

1. Close valve V-3.
2. Shut down the steam generator (procedure WHC-IP-0874-6.1).
3. Shutoff all the heaters.
4. Continue to pass air through the system until it has cooled to below 200°C.

Allow the scrubber feed pipe to cool to room temperature then change out the feed tube length to another depth and repeat the startup, operation, and shutdown procedures.

RESULTS

All the data gathered during the test was recorded in the laboratory logbook (reference 3). Table 1 lists measurement from the two runs performed in this test. In the first run, the feed tube was extended to the floor of the scrubber so that there was 100 cm of water above the bottom of the feed tube. In the second run, the feed tube was raised to 50 cm above the scrubber floor so that there was only 30 cm of water above the feed tube. These two

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positions presented the maximum and minimum water headspace available for scrubbing.

Using vendor literature on the variable area rotameter and assuming standard atmospheric pressure, the air flowrate was set at $0.0695 \text{ Nm}^3/\text{min}$. The actual air pressure during the high and low feed tube tests was 128.9 kPa absolute (4 psig) and 115.1 kPa absolute (2 psig), respectively.

During the steam flowrate calibration for the first run, 2247 grams of steam condensate were collected in 32 minutes. This puts the steam flowrate calibration at 70.2 g/min. The chiller fluid temperature was 23 to 25°C. For the second run, the steam flowrate was measured at 77 g/min.

During both runs, the superheater exit gas temperature (T-12) reached 490°C. At no time during the test did the scrubber offgas temperature (T-4) exceed 25°C.

The temperature of the scrub solution was measured with four thermocouples (T-5, T-6, T-8, and T-9). Measurements from these thermocouples indicate that the scrub solution temperature was maintained between 8 and 11°C in both of the runs. Also, there were no significant differences in scrub solution temperature at different locations in the scrubber.

Throughout the test, no water hammering occurred.

CONCLUSION:

The calciner scrubber, as built and operated in the Chemical Engineering Laboratory, can effectively cool a 490°C air/steam feed gas to below 25°C. Also, no water hammering effects were noticed in this test. The draft tube design worked effectively in circulating the scrub solution up through the center of the tube and down the outside and was vigorous enough to overcome the buoyant force of all but the largest gas bubbles and pull them down the sides of the scrubber. This was true for both the high and low feed tube

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Table 1: Calciner Scrubber Test Data.

Measurement	Measurement Location	Units	Measurement at Feed Tube Position	
			High	Low
Temperature				
Superheater	T-1	°C	910.5	909.6
Preheated Air	T-2	°C	417.2	444.7
Steam	T-3	°C	83.6	98.3
Scrubber Offgas	T-4	°C	23.4	22.9
Scrub Solution - top	T-5	°C	10.7	8.8
Scrub Solution - top	T-6	°C	10.7	8.8
Scrub Solution - in	T-7	°C	20.0	20.5
Scrub Solution - bottom	T-8	°C	10.8	8.8
Scrub Solution - bottom	T-9	°C	10.8	8.9
Cooling Water - out	T-10	°C	10.7	8.6
Cooling Water - in	T-11	°C	7.5	6.1
Scrubber Feed Gas	T-12	°C	496.6	492.0
Pressure				
Air Line Pressure	P-1	psig	4	2
Steam Line Pressure	P-2	psig	4	2
Scrubber Feed Pressure	P-3	psig	2.5	0.5
Flowrate				
Cooling Water		L/min	1.98	3.59
Steam		g/min	70.2	77.0
Air		Nm ³ /min	0.0884	0.0790

positions used (0 cm and 50 cm above the floor of the scrubber) although the velocity did appear slightly slower when the feed tube was in the high position. More detailed data can be found in the laboratory notebook (Reference 3). An edited video tape containing an explanation of the

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experimental apparatus and representative samples of the scrubber in operation will be transmitted to James Compton with this test report.

(Original signed by M. D. Britton)

Michael D. Britton, Advanced Chemical Engineer
Chemical Engineering Laboratory

(Original signed by C. V. King)

Craig V. King, Senior Engineer
Chemical Engineering Laboratory

MDB

AIM

Association for Information and Image Management

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