NOTICE

**CERTAIN DATA
CONTAINED IN THIS
DOCUMENT MAY BE
DIFFICULT TO READ
IN MICROFICHE
PRODUCTS.**

DECLASSIFIED HW-22 892

1. R. S. Bell
2. T. W. Hamff
3. V. R. Chapman
4. C. T. Groszith
5. A. R. Maguire
6. W. P. Nicklason
7. R. H. Beaton
8. O. H. Greagor
9. W. K. Woods
10. R. J. Richards
11. J. C. Cobb
12. J. C. Cobb

DECLASSIFIED

By Authority of CG-PR-2
RM ITEN 3-17-92
By DM CCK 12-14-92
Verified By J. H. H. 12-15-92

13. O. P. Amacker
14. F. W. Woodfield
15. R. E. Smith
16. G. C. Oberg
17. A. K. Harrison
18. G. J. Reab - J. C. Cobb
19. E. D. Walton
20. - 23. Extra
24. 300 File
25. 700 File
26. Pink Copy
27. Yellow Copy

COPY 1 OF 1

This document consists of

17 pages. No. 24

24

November 13, 1951

DO NOT REPRODUCE!
REFERENCE COPY ONLY

UO₃ PLANT COLD SHUTDOWN RUN PLAN: NO. C-1

I. OBJECTIVES

One complete batch conversion will be made in each of the eighteen Calcining Pots using unirradiated uranium feed solution. The objectives of this series of conversions are as follows:

1. A mechanical test of the conversion units.
2. A shakedown of the unloading, pulverizing and material handling equipment.
3. A functional test of the Nitric Acid Absorber system operating at approximately 5 to 20% of its design capacity.
4. The determination of the optimum temperature and time cycle conditions for 60% UHf feed solution. These conditions may be based upon calcination of the initial charge of 60% UHf solution to form 270% of UO₃, or the addition of 60% UHf make-up charges (boiling off water between successive additions). This latter calcination produces 580% of UO₃ in one pot.
5. A confirmation of the calibration of instruments.
6. To provide operating experience and process know-how.

MASTER

II. BASIS

Prior to the installation of the Product Concentrators in the TUP Plant, the UO₃ Plant will operate on 60% UHf solution (JDU) as received from the Reactor Plant. Consequently after the UO₃ Plant equipment has been finished and calibrated and the operability tests are completed, the first UO₃ conversion scouting runs will start with 60% UHf solution instead of 8% or 100% UHf solution. These runs will provide an integrated test of the Calcining Pots, the vacuum unloading and materials handling equipment, and the Nitric Acid Absorber.

The detailed operating procedures for the operation of this equipment were written by the Separations Section. Copies of these procedures are attached for reference.

DECLASSIFIED

DISTRIBUTION RESTRICTED TO U.S. ONLY

DECLASSIFIED

- 2 -

HW-22892

III. SALIENT PROCESS CONDITIONS

A. Feed Specifications and Volume Requirements

Approximately 2130 gallons of 60% UHM solution is required to operate fifteen Calcining Pots each with a 500% UO_3 charge and three pots each with a 270% UO_3 charge. The solution will be a 60% UHM solution received from 321 Building. The feed solution specifications will be "as received". The approximate composition is expected to be as follows:

<u>Component</u>	<u>Amount*</u>
UHM	60%
UO_3	Nil
H_2O	40%
Impurities	
Na ⁺	14,500
PO_4^{3-}	100
Ni	less than 10
S	Nil
Mo	less than 20
Cr	20
Si	34
B	2
Al	1300

*Impurities are listed as parts per million parts of UO_3 .

B. OPERATING CONDITIONS

1. Pre-startup conditions for the Nitric Acid Absorber System are as follows:
 - a. Negative pressure on the system with the vacuum breaker set to relieve at 5 in. vacuum.
 - b. Cooling water flowing to the Nitric Acid Absorber plates and Gas Cooler.
 - c. Fill Absorber plates with water via the reflux line and then shut off reflux.
2. Three pots will be tested by calcining 60% UHM solution to produce a 270% charge at UO_3 . Operating conditions will be as follows:
 - a. Initial batch size charged to each calcining pot: 60 gallons of 60% UHM solution to give 270% of UO_3

DECLASSIFIED

DECLASSIFIED

- 3 -

- b. Set the automatic temperature controls to limit outside pot skin temperature to 760°C. until the temperature of the reacting mass is raised to 204°C. then reset the outside pot temperature to 427°C. Set the control to shut off the power automatically when the reacting mass reaches 316°C.
- c. Tentative time cycle required after the reacting mass reaches 125°C., corresponding to approximately 100% UNH solution in the pot:

Temperature Range	Time	Material Condition
125 - 204°C.	100 min.	Preheating
204 - 232°C.	90 min.	Decomposing
232°C.	20 min.	Plastic Stage
232 - 316°C.	90 min.	Drying

3. Fifteen pots will be tested by calcining a 580% charge of UO_3 instead of a 270% charge. This will be accomplished in the following manner:
- a. Fill pot to a 60 gallon level with 60% UNH solution
- b. Set the pot skin temperature control pointers at 675°C. and 800°C., and charge temperature control pointers at 60°C. and 125°C. Turn on furnace.
- c. After the charge temperature has reached 125°C. (equivalent to 100% UNH) and the furnace has shut off automatically, refull the pot with 50% UNH to the 60 gallon level. Keep the fume recovery system valve open during filling to remove any steam and fumes arising from the pot. With controls still set as in B-3-b above, turn on furnace.
- d. When the furnace shuts off, repeat step B-3-c except reset the high charge control pointer from 125°C. to 136°C. with the other control pointers set as in B-3-b above. Turn on the furnace.
- e. When the furnace shuts off, reset the charge temperature control pointers at 204°C. and 316°C., and the skin temperature control pointers at 10°C. and 130°C. Turn on the furnace to hold the solution at a temperature of about 125° - 130° C.
- f. After all fifteen pots contain concentrated UN solution as described in B-3-c above, calcination

DECLASSIFIED

DECLASSIFIED

- 4 -

may be started by resetting the skin temperature control pointers at 427°C. and 760°C.

4. During the concentration of the URE solution in the pots the vapors produced will be primarily water, and consequently the condensate from the Gas Cooler, E-A-2, can be routed to the Sump Tank, TK-C-2 (for disposal to the sewer) until the boiling temperature in the pots reaches 136°C. Any condensate from the Gas Cooler at pot temperatures above 136°C. should be routed to TK-C-3 via the Bleacher, C-6.

The HNO₃ Absorber holdup is approximately 530 gallons, and so considerable time will be required to attain steady state if only two pots are operated at one time. Reflux (rate indicated in table below) will be required in the HNO₃ Absorber Tower after steady state is established, and should continue only during the decomposition period when oxides of nitrogen are evolved in large quantities. The reflux should be started when the specific gravity of the nitric acid on the bottom plate of the HNO₃ Absorber is 1.25 and should be adjusted as the pots reach the drying stage.

No. Pots in Decomposition Cycle	Reflux Rate to Absorber, gal./min.
1	.18
2	.36
3	.54
4	.72

IV. DATA

A. Operational Data

For each pot operation record the following data:

1. Initial volume added from the salt header.
2. The number of pot refills after solution temperature reaches 125°C.
3. Temperature indicated on TIC-1 and TIC-2, and pot agitator load every 30 min. after heating is started. Note time control shifts from TIC-1 to TIC-2 and end of cycle.
4. Record the data requested on the 40% Nitric Acid Recovery System data sheet.

B. Sampling Data

Take samples as listed below and request the analysis as indicated:

1. Three samples from loop header. One after first, ninth, and eighteenth pots are charged.

DECLASSIFIED

SECRET

SECRET

DECLASSIFIED

- 5 -

Sample size: 50 ml.

Analysis required: Density, UHM, HNO_3 (also Cl^- on one sample only).

2. A sample from each pot before unloading.

Sample size: 150 gms.

Analysis required: UO_2 (purity), H_2O , HNO_3 , U_2O_8 , particle size, bulk density, and surface area.

3. A sample of UO_2 from composite sampler on packaging machine from each drum.

Sample size: 150 gms.

Analysis required: UO_2 (purity), Na^+ , PO_4^{3-} , Iron, Ni, S, Mo, Cr, W, Si, B, H_2O , HNO_3 , U_2O_8 , particle size, bulk density, and surface area. Request gross beta and gamma on one sample only.

4. A sample of each the plate samplers of T-A-1 after each hour of operation.

Sample size: 100 ml.

Analysis required: Hydrometer density on all samples. UHM and Cl^- on the last complete set (all plates).

V. ADDITIONAL EQUIPMENT

If at the time of these shake down runs Food Tank C-1, which supplies the UHM to the pots via the loop header, is not available for use, a portable pump will be used to pump the UHM solution from the tank trailer (in which it is transported from the 321 Building to the 224-U Building) directly into the loop header via a temporary connection.

VI. RESPONSIBILITY

The operation, including recording of data, will be performed by Separations Section personnel with technical assistance rendered by the Technical Section. Data will be correlated by the Technical Section, and appraised by both the Separations and Technical Sections.

By: *[Signature]*
[Signature]
Approved: *[Signature]*

DECLASSIFIED

[REDACTED] [REDACTED]

DATA SHEET

Date _____

1. Initial volume added from the salt header _____ gals.
2. Number of refills after solution temperature reaches 125°C.
#1 _____
#2 _____
#3 _____
3. Record indicated temperature on TIC-1, TIC-2, the load on the agitator every 30 minutes after heating is started. Note time control shifts from TIC-1 to TIC-2 and at end of cycle.

Time	Temp. TIC-1 °C.	Temp. TIC-2 °C.	Agitator Load Amps.	Remarks

[REDACTED]

[REDACTED]

NITRIC ACID

2000

[REDACTED]

DECLASSIFIED

[illegible]

DECLASSIFIED

224-J STANDARD OPERATING PROCEDURE INNO₃
ABSORBER - SHUTDOWN

Date _____
Operator _____
Shift _____

1. When information is received from the Pot Room operator that reduction is nearing completion, prepare to shut down the absorber system. Time _____
2. Inform the operator of the evaporator-fractionator on line that the absorber is to be shut down and give him an estimated time for shutdown. Time _____
Est. Shutdown _____
3. When all the Pot Room fume ducts are closed and all units shut off, shut off reflux water to absorber. Time _____
4. Shut off cooling water to Heat Exchanger E-00-3. Time _____
5. Allow time for T-A-1 to drain to TK-C-3. Time _____
6. Close valve in line from T-C-6 to TK-C-3. Time _____
7. Shut off jet J-A-3. Time _____
8. Shut off T-A-1 cooling water. Time _____
9. Continue pumping TK-C-3 to fractionator (or TK-X-1, TK-X-2) by putting Wt. F. controller on manual and set flow to 2.27 gpm. Time _____
Flow _____
10. When TK-C-3 is empty, shut off pump and inform evaporator operator that shut down is complete. Time _____

DECLASSIFIED

DECLASSIFIED

- 2 -

11. Set 40% HNO₃ discharge valves for proper routing. (Obtain from your supervisor).
12. Start pump P-C-3-1.
13. Record continuous operating data on 8 hr. sheet.

To X-1 _____
To X-2 _____
To T-B-4 _____
To T-D-4 _____
Time _____

DECLASSIFIED

DECLASSIFIED

224-J STANDARD OPERATING PROCEDURE

HNO₃ ABSORBER - STARTUP

Date _____
Operator _____
Shift _____

Note: THE HNO₃ ABSORBER SYSTEM MUST BE "ON LINE" BEFORE STARTUP OF A REDUCTION POT

1. Turn on cooling water to T-A-1 coils. Adjust flow to 100 gpm, valve to route through Gas Cooler, E-A-2.	Time _____
Flow _____ gpm
2. Turn on steam to jet J-A-3. Close tower bypass line valve. Adjust PICA-A1-1 to control vacuum in T-A-1 at 10 in. Record flow on FR-A1-3.	Time _____
Pressure _____
Flow _____
3. Fill seal pot D-A-3 with water. Maintain small flow of approximately 0.5 g.p.m.	Time _____
Flow _____
4. Start reflux water flow to T-A-1 and adjust flow to 1.25 gpm as indicated on FI-A1-2.	Time _____
Flow _____
5. Turn on cooling water to Heat Exchanger E-OG-3 in tower reflux line. Adjust flow to maintain temperature of T-A-1 reflux at 70°F.	Time _____
Reflux temp. _____ °F.
6. Open valve in T-C-6 discharge line to TK-C-3.	Time _____
7. When WFIC on TK-C-3 indicates that reflux water is being received in that vessel, shut off reflux water to T-A-1.	Time _____
8. Inform reduction pot operator that the absorber system is ready for operation.	Time _____
9. When informed that reduction has been started, check the density of the acid on the bottom plate of T-A-1 and when density approaches 1.25, resume flow of reflux water and adjust to _____ gpm as indicated on FI-A1-2.	Time _____
Density _____

Flow _____ |
| 10. When 400 gallons of weak nitric acid has been received in TK-C-3, set Wt. factor control on automatic. | Time _____
Wt. F. _____
Sp. Gr. _____ |

DECLASSIFIED

DECLASSIFIED

224-J BUILDING - STANDARD OPERATING PROCEDURES

CALCINATION OPERATION

Note: The following operating procedure is written on the presupposition that Fume Recovery, Cyclone and Bag Filter unit and the continuous Ventilation Bag Filter systems are in operation.

A. Charging and Batch Reduction

1. Reset temperature control systems at the control panel button _____°
2. Set TIC-1 control pointers for 427° and 760° C.
3. Set TIC-2 control pointers for 204° and 316° C.
4. Open ventilation header damper to sweep air across the pot opening.
5. Close fume header damper on pot.
6. Open pot lid and draw UNH from the salt header until the UNH level reaches the filling mark on the agitator shaft. Shut off valve in the salt header. Check level in pot to insure that the salt header valve is not leaking. Close pot lid.
7. Energize the heating and control circuits after placing controls on automatic.
8. Open the fume header damper and close the ventilation header damper. Turn on agitator.
9. Watch TIC-1 indicator pointer closely. It should rise slowly to 760° C. in approximately 1 hour. TIC-2 should rise at a slower rate and when the TIC-2 indicator pointer passes 204° C., the indicator light for TIC-1 should show that control is on TIC-2. The temperature of the batch should rise to 316° C. and indicator lights show all current off at that temperature.
10. Cool the batch to 150° C. Cooling may be speeded by opening the dampers on the furnaces to the ventilation header.
11. Close the fume header damper on the pot and open the ventilation header damper.
12. Shut down and lock out the agitator.
13. Open the pot lid.

B. Unloading

1. Using the unloading nozzle, remove the charge from the reduction pot.

DECLASSIFIED

DECLASSIFIED [REDACTED]

- 2 -

2. Inspect agitator and pot walls for evidence of cake buildup
Remove cake and break up with the unloading nozzle until the chips
are small enough to be picked up with the nozzle.
3. Recharge pot after cleaned.

DECLASSIFIED [REDACTED]

DECLASSIFIED

BAG FILTER OPERATION

Dust Recovery System

The multiple-bag dust recovery system X11-1 and X11-2 will be started up before calcination operation begins and shall continue in operation for as long as the calcination process is being carried out. The dust recovery system shall not be shut down during pot-room operations except under emergency conditions resulting from equipment failure.

Bag replacement and blow-ring maintenance will be performed after closing off the affected side of the filter box by means of the damper in the ductwork. The damper shall be locked in position and tagged by operations and maintenance.

Before starting the system have all control and indicating instruments checked for normal function. Check the blow rings for normal operations. See that the damper is in proper position, (open to both sides unless otherwise indicated) start the exhaust fan X-13 by punching the control button MCX-13 on the master panel board. Check the differential pressure across X-11. If it is less than 4" water (est.) shut down X-13 and locate the trouble. A bag may be improperly installed or defective.

During operation of X11-1 and X11-2, routine hourly readings will be taken on all of the control instruments; dust detector, differential pressure recorder, temperature recorder, etc. Any abnormal condition as indicated by the instrument readings or by alarm shall be investigated immediately and the necessary corrective measures taken. (See procedure on Bag Filter Maintenance).

Whenever the dust level in X-11-1 and X-11-2 hoppers builds up to the point of high level alarm, the hoppers shall be emptied into the drums placed under the unloading funnels.

Blow-ring operation in X-11-1 and X-11-2 is automatically controlled by the control point setting in DPRC-X11. The control points are set to energize the blow-ring mechanisms whenever the differential pressure across X11-1 or X-11-2 reaches a pre-determined value.

DECLASSIFIED

DECLASSIFIED [REDACTED]

BAG FILTER OPERATION

Pot Unloading System - Cyclone, Bag Filter, Hammer Mill and Storage Bin

The pot unloading bag filter system will be operated continuously after start-up except when it may be necessary to shut down for maintenance or bag change.

Before starting the system, all units and instrumentation shall be determined to be in proper working condition, and the damper on the inlet side of X-4 exhaustor shall be closed.

To Start: Start the exhaustor X-4 by pushing the control button MCX-4. When the blower is up to speed, open the damper on the inlet side of X-4 slowly over a period of 5 minutes. If the damper is open when the exhaustor blower is started or if the damper is opened too rapidly, the filter bag in X-3 may rupture.

After the exhaustor is in operation and the damper has been opened, start the hammer mill X-6 by pushing the control button MCX-6. Start the rotary valves X3-1 and X3-2 by pushing the control button MCX-3.

When the system is in operation the operator shall take instrument readings on all of the units and record the data on the log sheet provided. If the differential pressure across X-3 is less than 6" water (est.) the exhaustor shall be shut off and the condition corrected.

If the high level alarms indicate that the storage bin X-7 is too full or that the cyclone hopper X-5 is too full, unloading cannot start until these conditions are corrected.

Hourly readings shall be taken on all unloading system instruments and the data recorded on the log sheet provided. Any abnormal condition indicated by the instrument readings or by alarm shall be investigated immediately. (See procedure on Bag Filter Maintenance).

Blow-ring operation in X-3 is automatically controlled by the control point setting in DPRC-X3. The control points are set to energize the blow-ring mechanism, whenever the differential pressure across X-3 reaches a pre-determined value.

DECLASSIFIED [REDACTED]

MATERIAL HANDLING

DECLASSIFIED

I. PACKAGING ROOM OPERATION

A. Drum Inspection

1. Visually inspect all new barrels for defects in workmanship and for cleanliness.
2. Check phenolic resin coating for completeness and uniformity.
3. Check barrel cover and gasket.

B. Tare Weighing

1. After each drum has been inspected, check its weight on the print weigh scale.
2. Be sure that the drum cover and clamp lid are included in the tare weight.
3. Mark the tare weight on the drum cover with a wax pencil. Mark corresponding drums and lids so that one can be identified with the other.
4. Put the tared drums on the roller conveyor upstream of the loading chute.

C. Drum Filling

1. Move the empty drum under the loading chute and lower the funnel snugly over the open drum head.
2. Check the rough weigh scale on which the drum is resting to see that abnormal pressure is not put on the drum by the loading funnel.
3. Open the ventilating line valve and make sure that air is flowing around the drum into the outer section of the loading funnel.
4. Place a sample jar in the sampling mechanism.
5. Start the X-8 screw conveyor and fill the drum with the proper weight of product. (An automatic cut-off will shut down the screw conveyor when the rough weigh scale reads 900-925 pounds.)
6. When the screw conveyor has been shut down, the sampler mechanism will continue to run for a few seconds, then automatically shut off.
7. When the sampler shuts off, remove the sample container and install a new one. Put a cap on the sample jar and label it carefully for later delivery to the laboratory.

DECLASSIFIED

DECLASSIFIED

- 2 -

8. Using the rubber mallet that is provided, strike the loading funnel several times to dislodge any powder that may be held up in the chute.
9. Strike the drum several times with the rubber mallet to jig down the contents of the drum so that the drum cover may be put in place.
10. Raise the loading funnel and put on the drum cover, clamping it securely in place with the closure ring.
11. Shut off the air flow to the loading funnel.
12. Vacuum any UO_3 dust from the drum and clean up any spills at once.
13. Repeat the above procedures until eight drums have been filled.

P. Product Weighing

1. Prior to weighing each lot of drummed UO_3 (eight drums constitute a lot) check the print weight scale and adjust it if it is found to be in error. The method used to check the scale is as follows:
 - (a) A tare drum weighing _____ pounds has been prepared as a standard, and will normally be stored in the packaging room just off the roller conveyor.
 - (b) Set the tare drum on the roller conveyor.
 - (c) Move the tare drum on to the print weigh scale and print its weight.
 - (d) If the scale is in error by greater than 1/10 of one per cent, (one pound in a thousand) make the necessary adjustment.
 - (e) Return the tare drum to its storage place when the scale has been properly adjusted.
2. When the scale has been checked, print the lot number and the tare drum weight on the weigh tape.
3. Weigh each barrel of product and print each barrel number along with its respective gross weight on the weigh tape.
4. Accurate records on lot and drum numbers along with their respective gross, net and tare weights must be maintained.
5. When eight drums of product have been weighed, make up aluminum identification tags on the embossing machine. These tags will identify the drums by code and will carry the lot and tare numbers.

DECLASSIFIED

DECLASSIFIED

- 3 -

6. Fasten the identification tags to their respective drums by means of the lead-wire seal tied through the lid closure ring.
7. Move the sealed and tagged drums to the loading ramp for further handling.
8. Forward all print weigh tape records along with data sheets to the supervisor each shift.

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DECLASSIFIED

DATE

FILMED

4 / 28 / 94
tb

END