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POST OFFICE BOX P
OAK RIDGE, TENNESSEE
ORNL
CENTRAL FILES NUMBER

CF-56-8-176

COPY 18

DATE: August 20, 1956

SUBJECT: THOREX PILOT PLANT, SUMMARY OF EQUIPMENT
FAILURES DURING RUN HD-15

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FROM: J. H. Walker

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1.0 INTRODUCTION

Run HD-15 was made in the Thorex Pilot Plant during the period of January 6-18, 1956. The chemical data from this run are evaluated in ORNL CF-56-53, "Thorex Pilot Plant Run HD-15 Summary." This report supplements the chemical data summary report by describing the equipment failures that occurred during the run.

2.0 SUMMARY

Run HD-15 was successfully completed, although during feed preparation and throughout the run mechanical failures and column upsets occurred frequently.

During boildown of three batches of unadjusted feed, the feed adjustment tank pressurized almost continually, requiring that the steam flow to the adjustment tank be reduced to half of flowsheet rate. This lengthened the time cycle for feed preparation considerably, and because Batch C feed was not ready, cold feed was pumped to the columns for about 15 hr.

An electrical wire to the main feed pump shorted out and the spare pump would not operate. Cold feed was pumped for 1.5 hr while the short was repaired.

At the beginning of the run, excessive thorium was lost from the bottom of the extraction column because, during shutdown prior to the start of the run, thorium had settled to the bottom of the column. During the run, water was pumped to the column from the solvent head tank. After the water was drained from the tank and solvent was pumped, the column operated normally.

The stripping column operated poorly throughout the run. The interface consistently rose above the set point. At times the column appeared to be air locked. None of the corrective measures tried, which usually straighten out a poorly operating column, remedied C-column.

The partitioning and the solvent recovery columns gave no trouble throughout the run.

Several check valves in the uranium product isolation system leaked, because the Teflon disks were too hard to seal against the metal seat. The disks were replaced with red rubber and Buna-N. A diaphragm in a relief valve in the CU stand-pipe ruptured and allowed solution to flow back to R-3 tank instead of the ion-exchange columns.

In the thorium product system, DRC-25, which controls the density of the concentrate from the BT evaporator, showed the solution density to be 1.8 when the actual density was 1.9. As a result, the BTC solution which was collected in P-3 and P-4 tanks crystallized.

Several pumps failed, mostly because of dirt in the ball checks, looseness of mechanical seal bolts, and poorly working electrical components.

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Only one jet, the S-2 to S-4 jet, failed to work, delaying feed transfer for 6.5 hr. The feed solution was heated to 100°C, cooled to 75°C, and jetted at 60 psig steam to the jet.

A pin in the S-2 agitator sheared, requiring decontamination of S-2 tank for repair.

The sampler lines from tanks containing concentrated thorium solutions plugged several times. These were unplugged by steaming the lines.

Several instrument probes plugged in concentrated thorium solutions, and one phase separator, N-11, plugged or else the solution in the outlet line from the separator was too dense to allow flow. The instrument probes were unplugged by air or steam pressure and the phase separator was flushed with water.

S-2 cubicle had to be decontaminated to replace a corroded copper tube, which supplied cooling water to N-8-P mechanical seal. The tube was replaced with stainless steel and relocated outside the cubicle.

3.0 FEED PREPARATION

A total of 780 slugs was charged to the dissolver; 240 were charged first and dejacketed to provide a metal heel, and 180 were charged in each of three batches for dissolution. During dejacketing the dissolver temperature rose rapidly and the tank began to pressurize. This was corrected by closing a valve to stop bleeding air into the off-gas system.

Feed preparation was delayed for a considerable length of time, because the feed adjustment tank (S-2) pressurized almost continually during acid boil-off of the three batches. About 1245 liters of feed solution was heated to 150°C in Batches A and B and 980 liters in Batch C. A fourth batch preparation, consisting of 250 liters of solution remaining from Batch C dissolving and 958 liters of rework solution from N-7, was started but was discontinued after about 190 liters had been boiled off because of excessive pressure in the tank. The solution was stored in S-1 and decontamination of S-2 was begun.

When steam flow to the feed adjustment tank jacket was set normally, 90% on FRC-4, the tank would pressurize up to 10 in. of water (PIC-7). The steam controller was set to 45%; this allowed feed preparation to continue at a reduced rate but with about 3 in. of pressure in S-2. After Batch A was adjusted and transferred to the feed tank, dilute nitric acid was boiled for 3 hr in the feed adjustment tank and the acid fractionator (S-9) to clean out the system, but this did not relieve subsequent pressurizing in S-2.

4.0 PULSE COLUMNS AND PULSE GENERATORS

At the beginning of run HD-15, thorium losses in the extraction column aqueous waste stream were excessive because thorium had settled to the bottom of the column during the previous shutdown. Proper thorium distribution in the column was attained by turning off the feed and pumping solvent extractant (AX) and aqueous scrub (AS) solutions to the column. After 2.5 hr with these conditions, radioactive feed was started to the column at a slightly reduced rate, the AX rate was increased to 110% of flowsheet, and the pulse frequency was lowered from 65 to 60 cycles/min.

The extraction column was upset on two other occasions. An electrical wire to the feed pump shorted out, and when the spare pump was turned on, it would not operate. Cold feed was pumped for 1.5 hr while the wire was repaired. The second upset occurred when water was pumped from the AX head tank to the column. After the water was drained from the tank and solvent was pumped, the column operated normally.

The extraction column was operated a total of 225.2 hr, of which 134.5 hr was with radioactive feed.

The partitioning column operated satisfactorily throughout the run.

Prior to the start of the run, the C-column pulser was disassembled and new rings installed. Oil had leaked, which continuously increased, from the bottom of the piston and the pulser had jammed at the extreme up-stroke.

Throughout the run, C-column operation was erratic. The interface consistently rose above the set point, and at times the column appeared to be air locked. Several corrective measures were tried; none of these significantly improved the column performance. The pulse frequency was decreased from 65 to 60 cycles/min and later to 50. For a short period, the frequency was set to 30 cycles/min and then increased to 50 when the interface returned to the set point. The CX (aqueous strip) stream was increased from 24 to 33.6 liters/hr. On 5 occasions when the interface rose above the control point, water was added to the column via R-10 pot and on 3 occasions through the pulse leg to purge air from the column and permit the aqueous phase in the column to flow down the column.

Several times, when the interface was above the control point and then started to lower in the column, organic solution would flow from the bottom of the column through the aqueous (CU) line. The organic phase was separated by a phase separator and was collected in N-8 tank for subsequent recycle and recovery of the uranium.

The solvent recovery column operated satisfactorily throughout the run.

5.0 PRODUCT HANDLING EQUIPMENT

5.1 Uranium Product

The isolation laboratory was operated for 298 hr to isolate and elute the uranium product from this run. Operations were delayed by failure of Meriam check valves and a relief valve in the R-3-P discharge line. Also, a large amount of rework material was reprocessed through C-column and the ion-exchange system. The checks as originally installed had Teflon disks, and apparently this material was too hard to seal against the metal seat. About seven disks were replaced with red rubber and one was replaced with Buna-N. The isolation system was shut down about 2 hr, without interruption of solvent extraction equipment, while the valves were changed.

When the product receiver was air sparged, solution was forced out the vent line into the off-gas header. A Meriam check valve was installed in this line to prevent this situation.

A ruptured diaphragm in the relief valve in the CU standpipe permitted the discharge stream from R-3-P to flow through a bypass line back to R-3 feed tank without flowing to the ion-exchange columns.

5.2 Thorium Product

Throughout the run, DRC-25, which controls the concentration of the evaporator product, was very unsteady, showing a density of 1.8 for the concentrate when chemical analysis showed 1.9. As a result the concentrate which was collected in P-3 and P-4 tanks crystallized and samples could not be obtained. The re-set rate of the instrument was changed so that the instrument would respond faster to density changes.

6.0 PUMPS

6.1 Metering Pumps

- a. The alternate feed pump, S-4-PS, would not pump when switched on after the main feed pump shorted out. The oil was drained from the reservoir, the pump was decontaminated, and the nipples in the hydraulic adapter and all unions and pipe connections were coated with liquid rubber to seal air out or the hydraulic side of the pump. The pump still would not work.
- b. An electrical wire to the main feed pump shorted out, and cold feed was pumped to the columns for 1 hr 40 min while a new wire was pulled through the conduit to the pump. Near the end of the run, the vacuum compensator valve stuck, causing the pump discharge rate to decrease. The valve was depressed and released a few times to free the valve. The pump operated normally thereafter.

- c. Prior to the start of the run, Milton Roy pumps M-13-P (AX) and M-14-P (BS) were replaced with Lapp Pulsafeeder pumps to eliminate solvent leakage. The air-operated control valve which controls the rate of M-14-P stuck, and after the air pressure to the controller was raised and lowered a few times, the valve operated.
- d. Dirt caused two pumps to stop pumping, R-9-P (Milton Roy) and M-14-P (Lapp). The ball checks on both pumps were tapped and the Cuno filter on M-14-P was turned. This was all that was required to dislodge the dirt and get the pumps to operate.

6.2 Transfer Pumps

- a. A wire had become disconnected in the switch box for N-8-P, a Worthington centrifugal. The wire was connected and the pump operated. The bolts that secure the stationary seal face became loosened, allowing the mechanical seal to become loose and noisy. After the bolts were tightened, the pump worked fine.
- b. P-3-P (Worthington) would not start, kicking out the starter. The starter was blocked in for temporary use, and later the switch and wiring were checked. Nothing wrong could be found, and the pump gave no further trouble.
- c. A relay stuck on the intermediate clone pump (Chem-Pump). The relay was jarred loose to correct the trouble. The clones were shut down about 1 hr because of this trouble.
- d. A new mechanical seal had to be installed on M-10-P (Worthington) because a leak from the old seal could not be stopped when the seal was tightened. The floor became contaminated to 1 r/hr from the leakage.
- e. The mechanical seal on M-4-P (Allis-Chalmers) vibrated excessively because the seal face had become loose. The bolts were tightened to stop the vibration.

7.0 JETS AND AIRLIFTS

Transfer of feed solution from the feed adjustment tank (S-2) to the feed tank (S-4) was delayed 6.5 hr when the jet would not work. The solution in S-2 was heated to 100°C, cooled to 75°C, and the solution jetted at 60 psig steam pressure to the jet.

The airlifts worked fine throughout the run.

8.0 AGITATORS AND SPARGERS

A pin sheared in S-2 agitator, requiring decontamination of S-2 tank for repair. All other agitators and spargers operated satisfactorily.

9.0 HYDROCLONES

Before run HD-15, two hydroclones and associated lines and pumps were installed in place of the centrifuge in the solvent recovery system. One clone had 1 gpm capacity. These were tested by Unit Operations Section.

10.0 SAMPLERS

The sample uptake line for the BTC storage tank (P-17) plugged because the concentrated thorium solution (sp. gr. 1.9) had crystallized. By alternate treatments with steam and water for 1 hr, the plug was dislodged. Other sample lines plugged when concentrated thorium solutions (about 500 g/l) were sampled.

The sample conveyor stalled occasionally while it was being moved to the blisters. The clutch was tightened to make the conveyor operable.

During Neptex processing, prior to run HD-15, many orifice disks in the air supply lines to the sampler jets and airlifts were replaced with smaller diameter orifices because the solutions had a lower density. The samplers would not work with these orifices with Thorex solutions. (Table 1 lists in chronological order the sampler failures.)

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Table 1
SAMPLER FAILURES

Date, 1956	SAMPLER	Corrective Measures	MADE OPERABLE
1/4	S-1	Changed orifices ^a .	Yes
1/5	S-2	Steamed lines ^b . Changed orifices.	Yes
1/6	S-1	Changed orifices	Yes
1/7	S-2	Steamed lines. Changed orifices.	Yes
1/9	T-4	Changed orifices.	Yes
1/9	S-2	Steamed lines. Changed orifices.	No
1/10	S-2	Steamed lines. Changed orifices.	No
1/10	S-2	Changed orifices. Cleaned sample needles. Heated to 75°C.	Yes
1/13	P-4	Steamed lines. Changed orifices, cleaned needles. Heated to 75°C.	No
1/13	P-4	Steamed lines.	No
1/14	P-4	Heated solution to 75°C.	No
1/14	P-3	Pulled sample through flange on top of tank. Steamed lines.	No
1/15	P-4	Pulled sample through flange on top of tank. Steamed lines.	No
1/16	P-4	Pulled sample through flange on top of tank.	No
1/16	S-1	Sampler would not operate.	No
1/16	S-1	Steamed lines. Changed needles.	Yes
1/17	P-3	Steamed lines. Blew lines down with gas (300 psi).	Yes
1/17	P-4	Steamed lines. Believe float was stuck.	No

a. The air supply lines to the airlifts and the sampler jets have an orifice plate in the union immediately below the control toggle. Orifices with the desired size were installed.

b. A special block is inserted in the place of the needle block and steam, air, or water can be forced into the uptake or return lines on any sampler that becomes inoperable due to plugged lines.

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11.0 INSTRUMENTATION

Most instrument trouble was experienced from plugged probes (high pressure liquid level) in tanks contained concentrated thorium solution.

The liquid level probe in the dissolver became plugged three times and the density probes once. The plugs in the liquid level probe were dislodged with 60 psig air, but 125 psig steam was required to unplug the density probe. The plugging probably was caused by allowing the solution in the tank to cool to less than 40°C.

The high-pressure density probe in the TNT digester plugged twice. When air pressure was applied to the probe the second time to dislodge the plug, the transmitter was damaged and had to be replaced. Valves were installed to prevent air from getting to the transmitter during blowdown. Also, a mercury seal was placed in the lines so that when excess pressure is put on the lines the seal will blow and protect the transmitter.

The high-pressure density probe on P-17 (BTC storage tank) plugged. The plug was blown out with air, and the solution was heated to 40°C to prevent the probes from plugging.

A copper line to LI-10 on M-10 (cold feed makeup tank) corroded and leaked. An aluminum line was installed to replace the copper line.

The liquid-level, high-pressure probe on T-4 (LRA-10 for CW catch tank) leaked from a sweated fitting. The fitting was re-sweated to repair it.

The re-set rate on DRC-25 was adjusted for better density control of the BTC stream from the thorium evaporator. The instrument indicated a specific gravity of 1.8, but chemical analysis showed 1.9.

The indicator needle on the liquid level dial gauge on C-17 became loosened and indicated 29% when the tank was empty.

The S-10 flow timer low-level alarm (hot feed to S-4-P) sounded off a number of times. The relay assembly was replaced to correct the trouble.

12.0 PHASE SEPARATORS

One phase separator, N-11, either became plugged or the solution density was too high to allow unrestricted flow at the beginning of the run. The phase separator was flushed with water, and proper flow of the aqueous stream from the extraction column was attained.

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13.0 LEAKS AND WELD FAILURES

A copper tube that had been installed to supply cooling water to N-8-P seal was corroded by decontamination reagents in S-2 cubicle, through which the tubing passed. In order to find the source of a steady stream of water to cell 5 sump, S-2 tank was decontaminated and the cubicle was opened, revealing that the tubing was corroded. The tubing was replaced with stainless steel and rerouted so that it did not pass through the cubicle.

A bonnet on the M-11 (cold feed head tank) flow-checker bypass valve leaked, and the drippage from the valve crystallized on the floor, contaminating the floor to 1 r/hr. The valve bonnet was repacked to stop the leakage.

Two other leaks occurred: (1) a union in the condensate line from the BT evaporator leaked (union tightened to stop the leak); and (2) a steam-water supply line to the BTC storage tanks leaked in a weld joint where the steam and water supply lines meet (joint rewelded).

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