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COPY NO. - 100

DATE - February 12, 1962

HRT-Chemical Plant Run 21 Summary

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Abstract

The multiple hydroclone system removed 183 grams of corrosion product solids in 18¹/₄ hours of operation during reactor run 21. The low removal rate was attributed to plugging of multiclone feed ports that presumably occurred during the latter part of run 20. After modifications to the reactor core and removal of the multiclone unit at the end of run 21, the reactor core was backflushed with the flow direction in the core loop reversed. During this period, the single hydroclone removed 205 grams of solids in 10.5 hours of operation.

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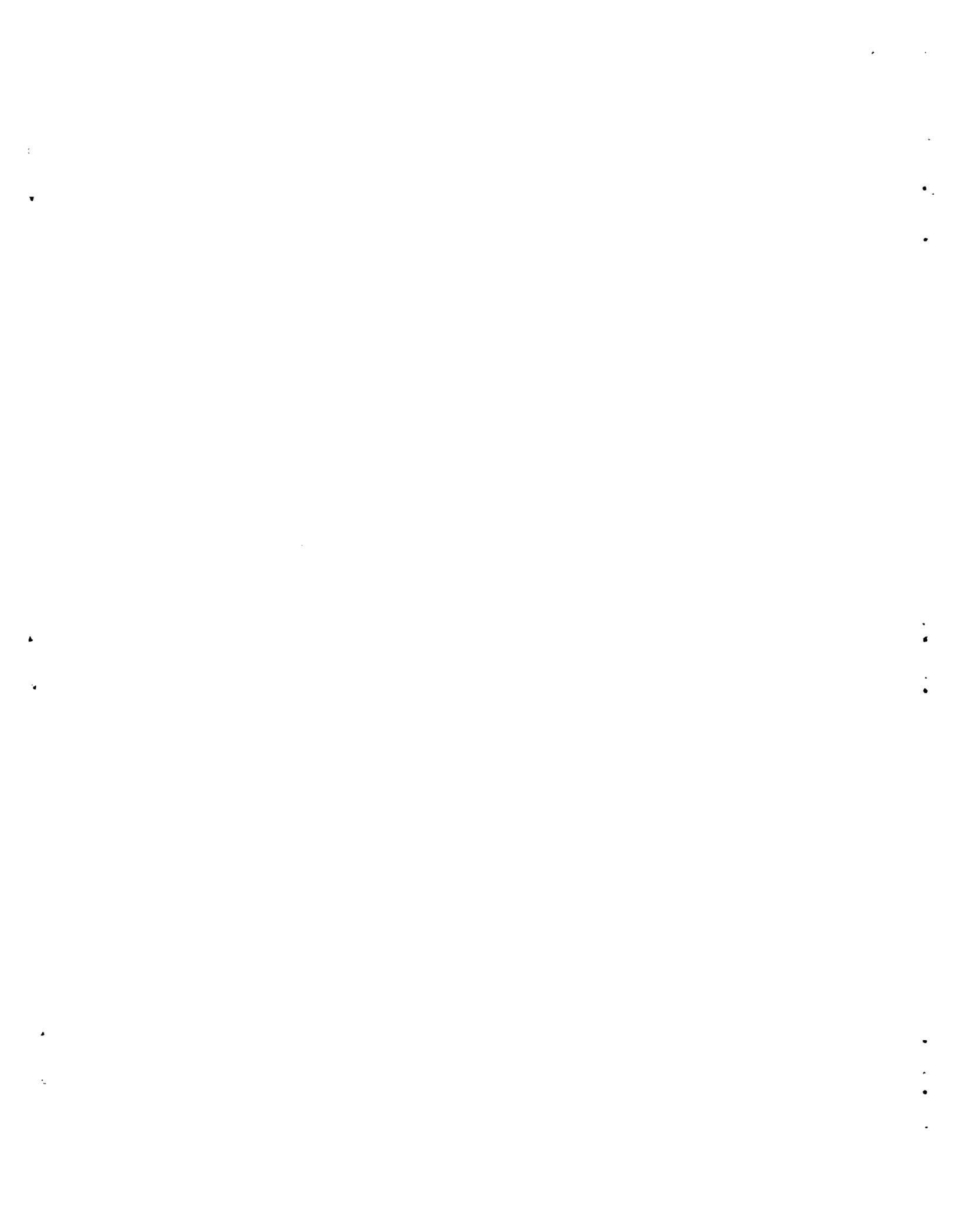
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1.0 SUMMARY

Operation of the multiclone-hydroclone system was continued during reactor run 21 with the multiclone on-stream for 1814 hours. Solids removal during the first two operating periods, with both the multiclone and hydroclone on-stream, averaged 0.35 g/hr. The final operating period included 1384 hr of multiclone operation followed by a 4-hr cleanout of the multiclone underflow pot by the cell C hydroclone. Solids removal during this period was only 35 g, indicating very low efficiency of the multiclone unit. The reduced solids removal rates during run 21 are consistent with those observed during the latter part of run 20. This reduction is attributed to the effects resulting from the plugging of several hydroclone feed ports, as determined by tests performed on the unit after it was removed from the reactor cell.

The composition of solids removed from the reactor system during run 21 averaged 50% zirconium, 14% iron, and 3.7% titanium. The increased zirconium content (solids removed during run 20 averaged 39% zirconium) indicates that about 2-4 kg of zirconium oxide were produced during the run.

Maintenance operations at the end of run 21 included extensive modifications to the reactor core and removal of the multiclone, after which the reactor core system was back flushed. A filter installed in the circulating pump suction removed large particles formed in the screen cutting operations and the hydroclone in cell C was operated to remove fines not collected on the filter. During two periods of operation (10.5 hr), 205 g of corrosion product solids were removed by the hydroclone and approximately 2.5 kg were collected on the filter. The composition of solids removed by the hydroclone during the flushing operation was 56% zirconium and 12% iron.

The Xe-135 poison fraction was determined from mass spectrographic analyses of the Xe-136/Xe-134 ratio in the reactor off-gas, and found to be 0.96 ± 0.15 at a reactor power level of 5 Mw.

2.0 INTRODUCTION

The HRT chemical plant was originally designed to remove corrosion and fission product solids from the reactor system by processing 0.3%

(1.2 gpm) of the circulating reactor fuel through a single hydroclone. In an effort to increase the solids removal rate by increasing the effective fuel processing rate, a multiple hydroclone (Fig. 2.1) was installed in the reactor cell prior to run 20 to process a 10 gpm bypass flow around the fuel heat exchanger. Solids concentrated in the multicclone underflow were routed to the original hydroclone, located in the chemical plant cell, for collection and subsequent dissolution. The hydroclone overflow joined the multicclone overflow and was returned to the reactor circulating stream. With the multiple and single hydroclone operating in series, as described above, the solids removal rate was not a direct function of processing rate but depended on the efficiencies of the two units. A solids removal rate of 0.3 g/hr^(1, 2, 3) after 400-600 hours of reactor operation was demonstrated with the single hydroclone. A removal rate of 0.5 g/hr was demonstrated with the multiple hydroclone system after comparable periods of reactor operation.⁽⁴⁾ With both systems the solids removal rates at the beginning of a reactor run were 4 to 8 times the above values, decreasing to these values after approximately 400 hours of reactor operation.

Extremely low removal rates in the latter portions of run 20 led to the belief that some malfunction of the cell C hydroclone had developed. This single hydroclone was replaced prior to run 21 and was later examined in a hot cell, but no evidence of excessive wear or corrosion was observed.

The HRT was originally constructed as a 2-region reactor, but during the first power run a hole was burned in the Zircaloy core vessel. Since that time the reactor has been operated with fuel in both the core and blanket regions. During run 21, 53% of reactor power was generated in the core region.

Objectives of chemical plant operation during run 21 were as follows:

(1) Determine if replacement of the cell C hydroclone at the end of run 20 increased the low solids removal rate experienced during the latter part of run 20.

(2) Continue to accumulate data on solids composition and their behavior in the reactor system.

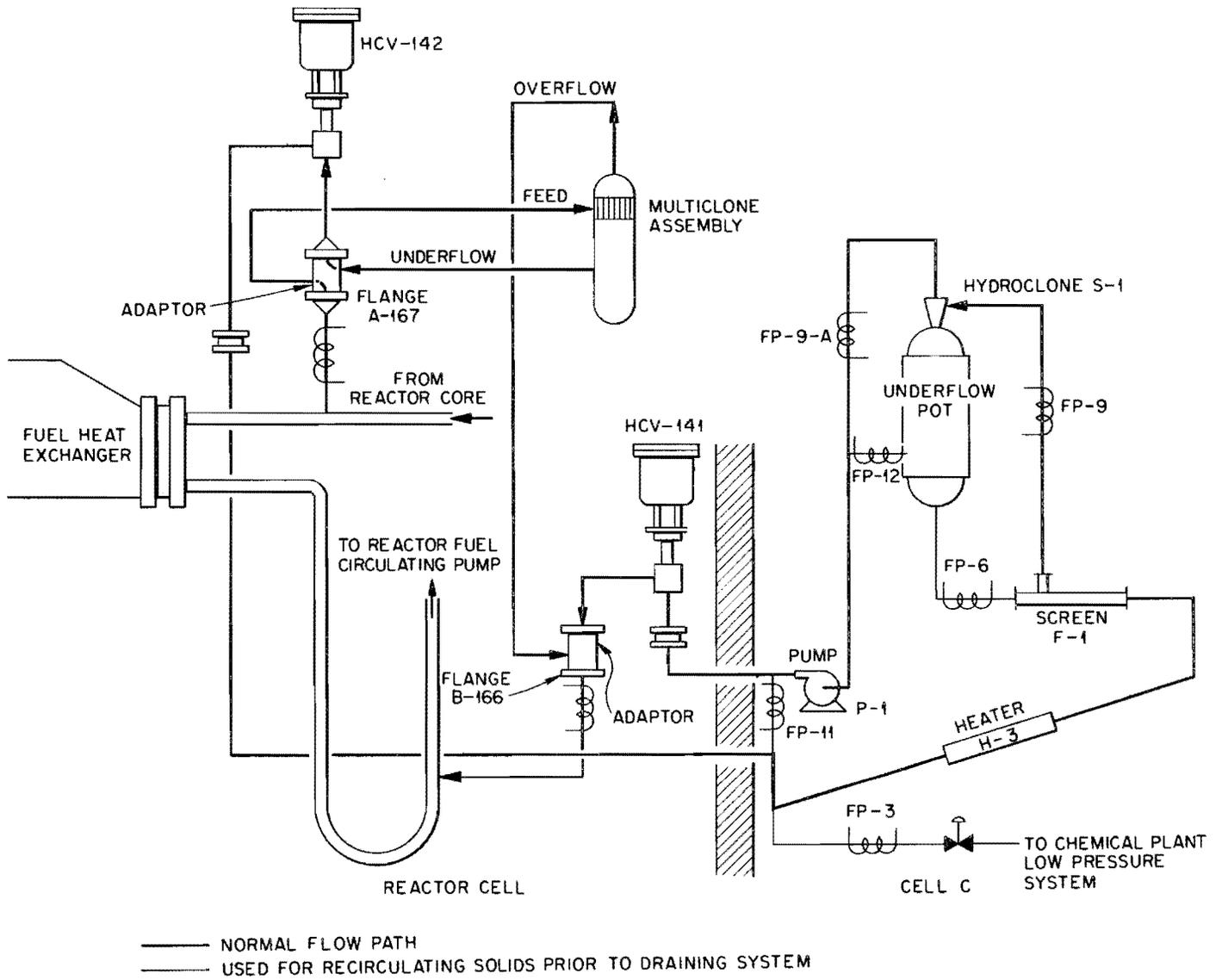


Fig. 2.1. HRT-CP Solids Removal System.

3.0 OPERATION

3.1 Operating Conditions

Operation of the multiclone-hydroclone system installed prior to reactor run 20 was continued during reactor run 21. The multiclone was on stream at 11 gpm feed rate whenever the reactor was in operation. Run 21 was divided into four periods indicated as 21-21, 21-22, 21-23, and 21-24.

The cell C hydroclone was operated (run 21-21) during the first 86 hours of the reactor run. Operating conditions for run 21-21 are presented in Table 3.1 and Fig. 3.1.

Table 3.1 Chemical Plant Operating Conditions

Run No.	Date Run Began	Multiclone On Stream hr	Cell C Hydroclone On Stream hr	Reactor Operation To Start of Run hr	Reactor Power Generation During Run Mw
21-21	10/4/59	86	86	0	84
21-22	11/13/59	344	322	937	467
21-23	11/27/59	1384	4	1280	2935
21-24	8/24/60	^a -	10.5	0	0

^aMulticlone removed prior to run 21-24.

Following chemical plant run 21-21, multiclone operation was continued for 788 hours, after which the multiclone underflow pot was flushed for 63 hours to return the accumulation of solids to the reactor system. The multiclone was flushed free of solids to insure that the removal rate observed during the run which followed was representative of the solids removal rate after approximately 1000 hours of reactor operation. Multiclone operation for run 21-22 began at the end of the flushing period and continued for 344 hours with the cell C hydroclone on stream 94% of this time. Operating conditions for run 21-22 are shown in Table 3.1 and Fig. 3.2.

Multiclone operation for run 21-23 began at the end of run 21-22 and continued until the end of the reactor run (1383 hours). The cell C hydroclone was operated for four hours after the reactor fuel circulating

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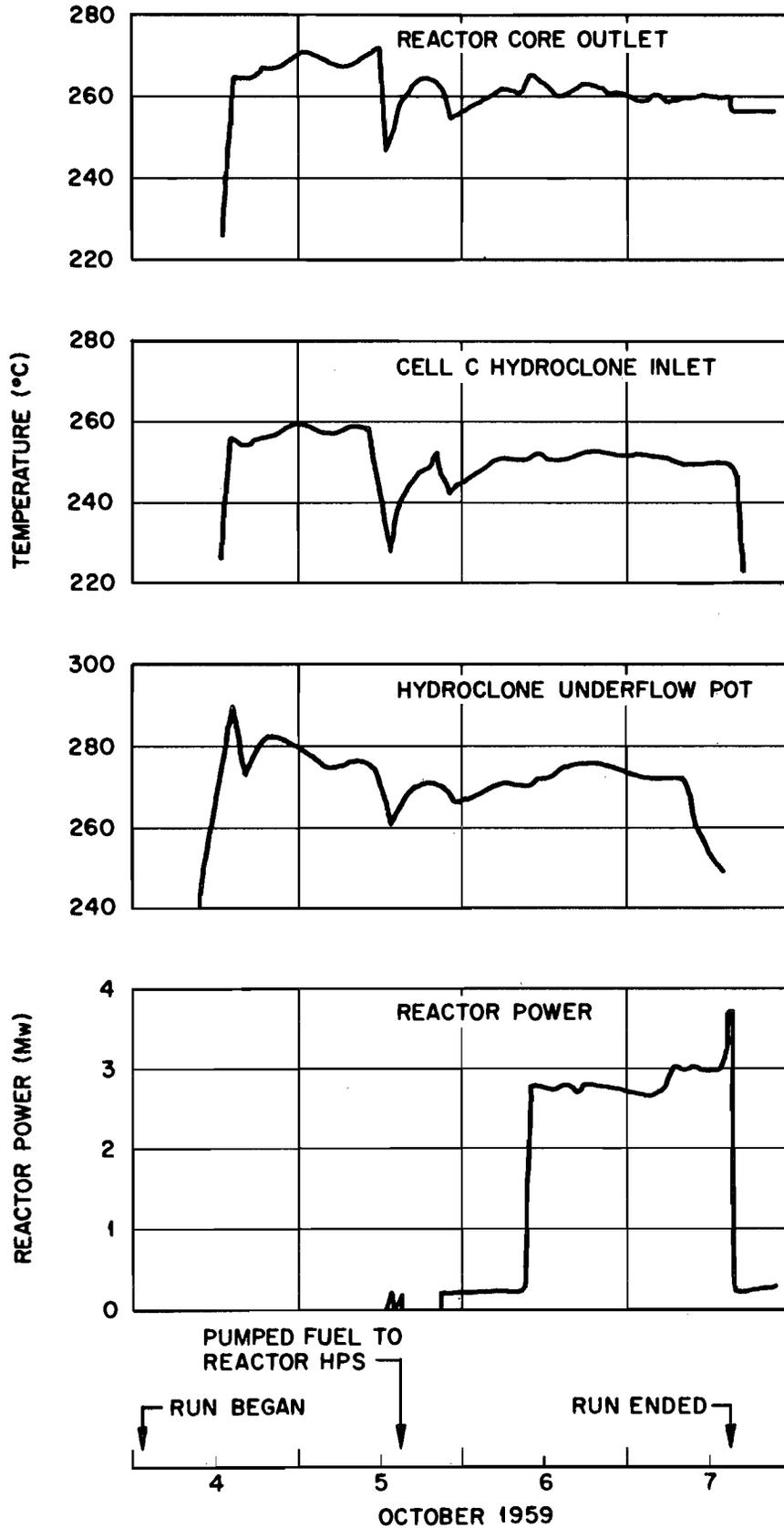


Fig. 3.1. HRT Chemical Plant Operating Conditions, Run 21-21.

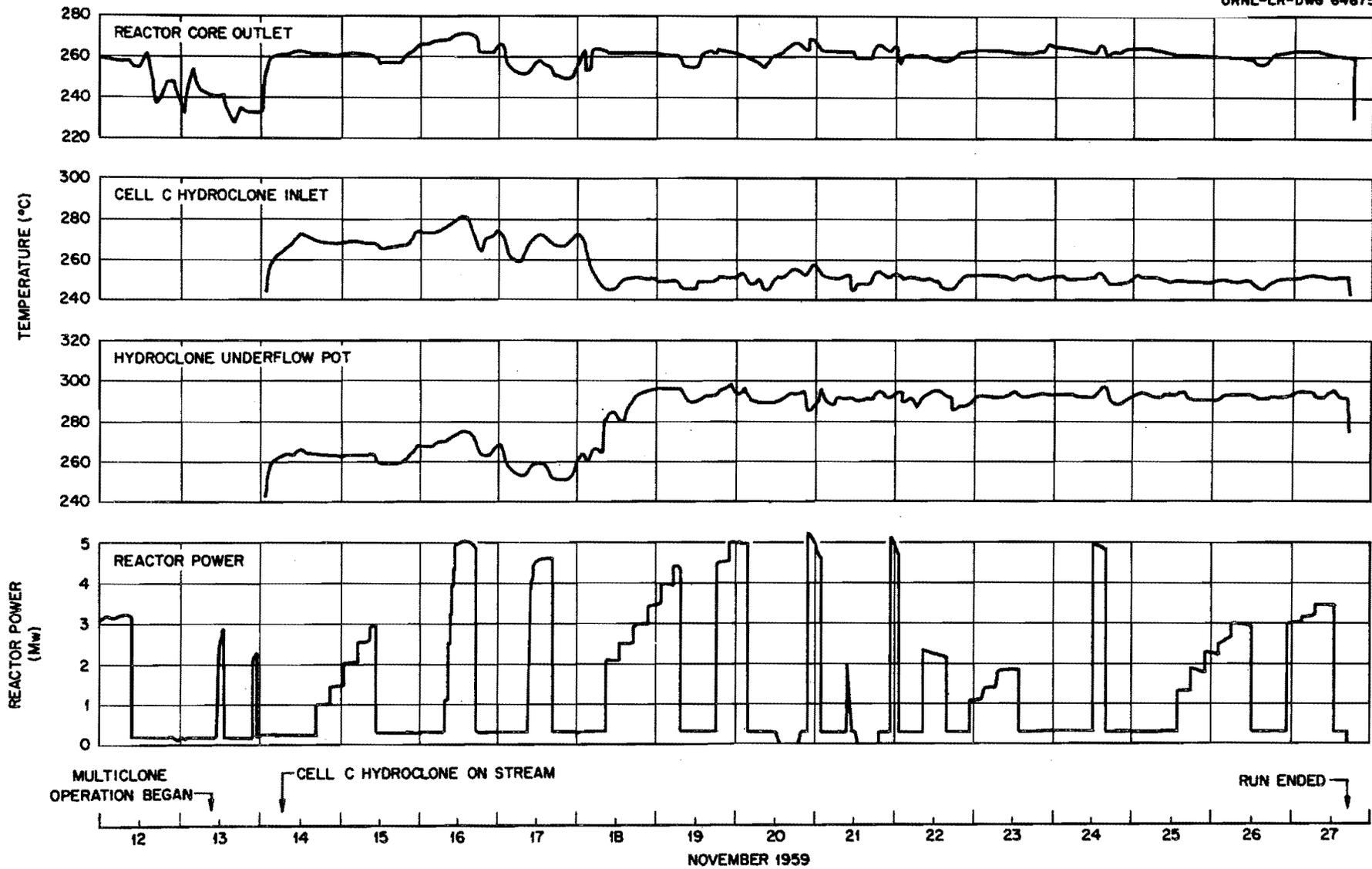


Fig. 3.2. HRT Chemical Plant Operating Conditions, Run 21-22.

pump was stopped, to collect the solids which had been removed by the multiclone. It was intended that freeze plugs isolating the multiclone from the reactor system be frozen prior to putting the chemical plant on stream to prevent loss of solids back to the reactor. With these plugs frozen, the cell C hydroclone overflow would be returned to the multiclone by way of the multiclone overflow line. The freeze plugs could not be established due to thermal convection loops in the reactor system and, therefore, a portion of the solids collected by the multiclone were returned to the reactor system.

No reactor power was generated during chemical plant run 21-24. This run consisted of two periods of operation totaling 10.5 hr with the cell C hydroclone connected directly across the fuel heat exchanger (the multiclone had been removed) and flow in the reactor fuel system reversed from its normal direction. During the first period (9.0 hr) a filter located in the reactor fuel circulating pump suction collected approximately 2.5 kg of solids, and plugged sufficiently to reduce the circulation rate in the core loop by approximately 50%. After replacing the filter with a clean unit, the backflush was continued for 1.5 hr. Accumulation of solids on the second filter was relatively small.⁽⁶⁾ The reactor and hydroclone were operated at 45°C and 100 psi during the backflush operation.

3.2 Dissolution of Solids

After each of the chemical plant runs, the contents of the cell C hydroclone underflow pot were drained and rinsed to the dissolver where D₂O was recovered by evaporation. The solids were dissolved by alternately refluxing in 10.8 M and 4 M sulfuric acid for four hours. The cycle was repeated to insure complete dissolution. After samples of the dissolver solution were obtained, the solution was transferred to storage tanks for future uranium recovery.

3.3 Equipment Performance and Maintenance

The refrigerant lines to freeze coils FP-9 and FP-9A plugged during run 21-21. The inability to freeze FP-9A in the line from the hydroclone overflow to the circulating pump suction prevented sampling of the underflow pot contents and made rinsing of the hydroclone system difficult. The Freon-11 charge in the refrigeration system was replaced after an

oily substance (approximately 2% by volume), which carbonized at temperatures in excess of 250°C, was discovered in the Freon-11. This material was thought to be the source of the plug in the line to FP-9A. After an unsuccessful attempt to unplug the line to FP-9A, by applying 200 psi pressure, operation of the chemical plant was continued. An auxiliary clamp-on freeze plug was installed adjacent to FP-9A following run 21-23, permitting the overflow line to be frozen in subsequent operations.

A plug in the drain line from the hydroclone underflow pot, which developed at the end of run 21-24, prevented draining of the underflow pot contents to the dissolver. Since FP-9 could not be frozen, the pot could not be back-pressured to free the plug (Fig. 2.1). To alleviate the difficulty, the access plug in the shield above the hydroclone was opened and the inlet to the hydroclone frozen with dry ice. With other permanent freeze plugs established and HCV-142 closed, the plug was broken by pressurizing the line to the underflow pot to 100 psi with heater H-3.

Minor difficulties with auxiliary equipment required attention during run 21. These included:

- 1) Excessive leakage necessitated replacement of the shaft seal on the Freon-11 circulating pump (P-14).
- 2) The diaphragm on the operator of the radiation block valve in the steam line to the decay tank (T-22) recombiners ruptured and was replaced.
- 3) The variable frequency motor generator which supplies power to the circulating pump became noisy. It was necessary to replace the drive belt and reface the varidisc surfaces in contact with the belt.

Air leakage into the cell C enclosure averaged 9.6 liters/min at 7.5 psia during run 21. No significant leaks between the cell and operating areas could be located.

3.4 System Inventories

The book uranium inventory in the chemical plant storage tanks was increased from 1401 g to 2082 g during reactor run 21 (Table 3.2). Included in the uranium added to the decay tanks was 537 g from in-pile loop work. In addition to the uranium, the decay tanks contained 1500 moles of sulfuric acid, 189 grams of copper, and 61 grams of nickel.

Table 3.2 Uranium Inventory in Chemical Plant Decay Tanks

Date	Uranium Transferred to Decay Tanks		Total U Inventory g
	Quantity g	Source	
-	-	From previous runs	1400.1
10/16/59	59.6	Dissolver solution from run 21-21	1459.7
1/25/60	71.0	Dissolver solution from run 21-22	1530.7
3/25/60	6.6	Dissolver solution from run 21-23	1537.3
9/21/60	8.4	Dissolver solution from run 21-24	1545.7
8/19/60 to 9/30/60	536.7	From in-pile loop studies	2082.4

4.0 RESULTS AND CONCLUSIONS

4.1 Material Accumulated in Hydroclone Underflow Receiver

The quantity and composition of material removed by the chemical plant is determined from analyses of solution resulting from dissolution of removed solids. The quantity of materials accumulated in the cell C hydroclone underflow pot during run 21 is shown in Table 4.1.

Table 4.1 Material Removed by Chemical Plant

Run No.	Constituent, g								
	U	Cu	Fe	Cr	Ni	Mn	Zr	Ti	Ag
21-21	59.6	8.6	12	3.0	2.7	0.2	40	2.0	1.5
21-22	71.0	9.6	9.4	3.6	3.0	0.3	33	2.1	0.5
21-23	6.6	0.8	3.3	0.1	-	-	19	2.6	-
21-24	8.4	-	24	7.5	0.9	0.2	115	2.5	0.2

The mass and composition of corrosion product solids removed by the chemical plant (Table 4.2) were calculated from the above data by assuming that the corrosion products were removed as oxides. Uranium and copper solids were reported as their ratio to the total mass of corrosion product solids removed. The solids reported as removed by the hydroclone was the amount of material in the dissolver solution after subtracting the amount in solution in the underflow pot at the end of the run.

Table 4.2 Quantity and Composition of Solids Removed by the Chemical Plant

Run No.	Corrosion Product Solids								Ratio to Total Corrosion Product Solids	
	Quantity g	Removal rate, g/hr	Composition %						U	Cu
			Fe	Cr	Ni	Zr	Ti	Ag		
21-21	81	0.94	15	3.8	0	49	2.5	1.9	0.098	0.009
21-22	67	0.19	14	5.4	0	50	3.1	0.8	0.246	0.021
21-23	35	0.025	10	-	-	55	7.5	-	0.189	0.023
21-24	205	23	12	3.7	0.4	56	1.2	0.1	0.041	-

4.2 Corrosion Product Solids Inventory in Reactor System

The data obtained during run 21 on the composition of solids removed by the chemical plant were insufficient to accurately estimate the corrosion product inventory in the reactor system. The increase in zirconium content of solids removed by the chemical plant (50.3% in run 21 as compared to 39.3% in run 20) indicates zirconium corrosion during run 21, which would be expected at the high power levels of this run. An estimated 2-4 kg of zirconium oxide was produced in this run, bringing the total zirconium oxide inventory in the reactor core system to approximately 13 kg. Production of stainless steel corrosion products in the reactor system totaled 1.9 kg during run 21, which gives a total corrosion product inventory in the reactor core system of approximately 20 kg.

4.3 Hydroclone-Multiclone Performance

Run 21 solids removal rates were comparable with those observed during the latter part of run 20. Replacement of the cell C single hydroclone, following run 20, did not improve solids removal, indicating that the reduced solids removal was not due to failure of that unit. The removal rates observed during run 21 were approximately equal to those experienced with single hydroclone operation and are consistent with failure of the multiclone as previously proposed.⁽⁴⁾ Plugging of 1 to 3 hydroclone feed ports was evidenced by tests performed on the multiclone unit after removal from the reactor cell at the end of run 21.⁽⁵⁾ The plugged feed ports significantly reduced the efficiency of the multiclone unit, particularly with the cell C hydroclone off-stream.

The very high solids removal rate during run 21-24, with reversed flow and only the cell C hydroclone on-stream, is indicative of a high concentration of circulating solids during the back-flush operation. Approximately 2.5 kg of solids were removed during the same period by the main line filter installed in the fuel circulating pump suction.

4.4 Xenon-135 Poison Fraction Measurement

Direct measurement of the Xe-135 poison fraction is possible by determination of the Xe-136/Xe-134 ratio in reactor off-gas by mass spectrographic analyses. From this ratio and known fission yields, the Xe-135 poison fraction can be calculated. After a 3-day period of operation at 5 Mw, a portion of the reactor off-gas was routed into a spare charcoal bed. After allowing for decay of Xe-133, a sample was pulled for analysis of the Xe-136/Xe-134 ratio. The poison fraction calculated from this ratio was .0096 ΔK with a standard deviation of ± 0.0005 . The accuracy of the measurement is probably determined by the accuracy of fission yield data for Xe-134 and Xe-136 and is probably no better than $\pm .0015$.

4.5 Iodine Behavior in the Reactor System

Investigation of iodine behavior in the reactor system was continued by observing heating of the iodine removal bed located in the reactor low pressure system.⁽⁷⁾ Startup and shutdown transients were compared with theoretical predictions of heat generation rates calculated with an analog computer. Observations were consistent with previous information on iodine behavior which indicated that only 10% of the iodine in the high pressure system circulates with the fuel.

5.0 RELATED SYSTEMS

5.1 Charcoal Bed Performance

Off-gas from the reactor system was vented through charcoal beds which have sufficient holdup for decay of all radioactive gaseous fission products except Kr-85. Two beds are normally operated in parallel with a third in standby. The system was operated routinely throughout run 21; 112 curies of Kr-85 were released from the 7500 area stack.

5.2 Waste System Operation

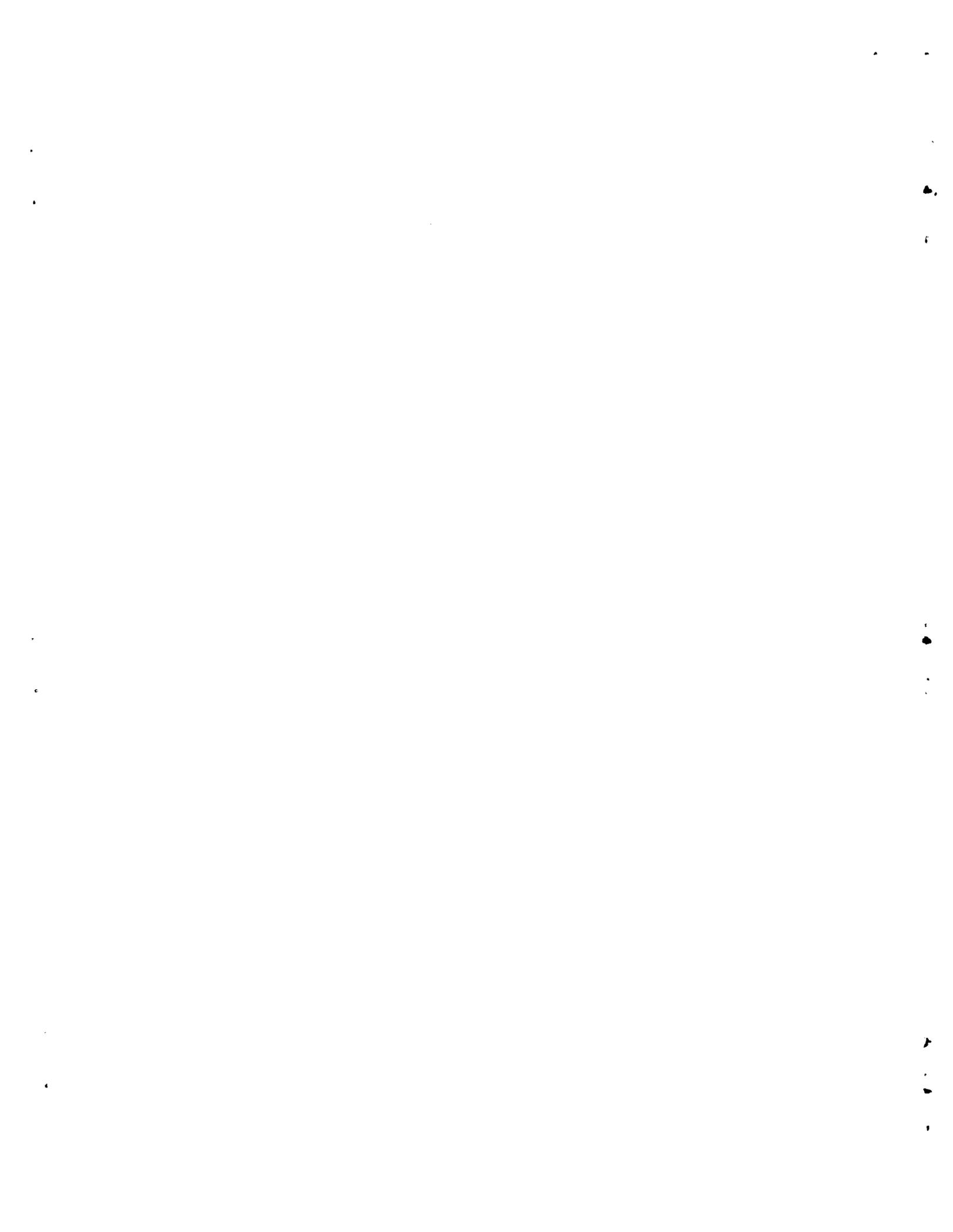
Low level liquid wastes consisting primarily of reactor cell flooding water from maintenance operations are drained to a waste pond where activity is scavenged by adsorption on calcium phosphate precipitate. The calcium phosphate is formed in the waste pond by addition of trisodium phosphate, which forms a precipitate with the calcium normally in process water. Decontaminated water is drained into White Oak Lake. During the period covered by this report (12-1-59 to 4-1-60) 320,000 gallons of water containing 0.4 curies of β activity was released to White Oak Lake. Activity added to the waste pond during the period totaled 2 β curies.

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REFERENCES

- (1) O. O. Yarbrow, "HRT - Chemical Plant Run 16 Summary," ORNL-CF-58-10-111 (October 9, 1958).
- (2) O. O. Yarbrow, "HRT - Chemical Plant Run 17 Summary," ORNL-CF-59-3-62 (March 6, 1959).
- (3) O. O. Yarbrow, "HRT - Chemical Plant Runs 18 and 19 Summary," ORNL-CF-60-3-161 (March 25, 1960).
- (4) O. O. Yarbrow, "HRT - Chemical Plant Run 20 Summary," ORNL-CF-60-7-120 (July 1, 1960).
- (5) O. O. Yarbrow, "Test of HRT-CP Multiclone No. 1 Following Removal from Reactor Cell," ORNL-CF-60-6-105 (June 28, 1960).
- (6) S. E. Beall et al, HRP Quart. Prog. Rep. Nov. 30, 1960, ORNL 3061, p. 9-11.
- (7) S. E. Beall et al, HRP Quart. Prog. Rep. Oct. 31, 1959, ORNL-2879, p. 10-11.



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