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CHEMICAL TECHNOLOGY DIVISION
UNIT OPERATIONS SECTION
MONTHLY PROGRESS REPORT
JUNE 1956

OAK RIDGE NATIONAL LABORATORY
OPERATED BY
UNION CARBIDE NUCLEAR COMPANY
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AEC RESEARCH AND DEVELOPMENT REPORT

CHEMICAL TECHNOLOGY DIVISION

UNIT OPERATIONS SECTION

MONTHLY PROGRESS REPORT

June 1956

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W. K. Eister
J. T. Long
E. O. Nurmi
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Note: The primary objective of this report is the monthly distribution of experimental data as acquired during that period in the course of engineering studies. Much of the data is of preliminary nature and, therefore, reasonable care should be exercised in its application. You are invited to contact the individual authors for substantiation of the data.

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SUMMARY

Chemical Engineering Research

Dual Clone System. The experimental dual clone apparatus was found to give lower separation factors (100 vs 1000) at high water feed concentrations (~30%). It appears that for the best separation the optimum feed concentration is approximately 13% water when two 0.4 inch clones are incorporated in the system.

Jet Pump System. The jet pump experimental apparatus is located in Cell 3 of Building 4505. At present only a few runs have been completed with the results correlating quite well with those found in the literature.

Excer

The UNOP Excer electrolytic cell operates satisfactorily except that the product fails to meet the product specification of 3.0 gm/cc tap density; the highest obtained was 2.6 gm/cc.

The Excer ribbon flite dehydrator has shown that the water concentration of UF_4 can be reduced to 0.37% in 90 minutes with exit temperature of 390°C.

Fluorox

In preparation for future integrated operation of the R-H MB reactor and the Oxidation MB reactor, plans have been formulated for evaluation of pelleting procedures for appropriate mixtures of feed and recycle streams.

Several short test runs oxidizing carbon from a mixture of 50% UO_2F_2 pellets and 50% graphite pellets were completed.

Equipment modifications on both the R-H and Oxidation MBR systems are still under way.

HR Fuel Reprocessing

A-Loop. Operation at reactor conditions for a period of 311 hours through 25 thermal cycles showed removal of solids by the hydroclone and good recovery of heat transfer in the preheater used to remove neodymium.

Chemical Plant-Hydroclone and Underflow Pot Test. Heat transfer coefficients of 24-28 BTU/hr ft² °F were determined for the underflow pot air cooling system. Heat-up and cool-down times were determined for various air flow rates.

Flange Tests. Tests using octagonal O-ring gaskets showed these to be superior to oval O-ring gaskets.

Hydroclone Development Tests. Corrosion or erosion of titanium hydroclones in 10 g U/kg H_2O UO_2SO_4 solution was found to be less than 10 mils per year.

Component Tests for Loop P-1. A water-cooled sample valve showed no attack after 20 cycles in 300 g U/kg H_2O UO_2SO_4 .

Loop T-1. Solids addition as $Zr(SO_4)_2$ to Loop T-1 showed only 8% of the γ activity ($Zr^{95} + Nb^{95}$) was recovered.

Loop P-1. Components are being fabricated and construction of the loop operating area in 4501 was continued.

Metallex

The installation of the Interim Facility of the Dry Salt flowsheet was completed.

Ore Processing

The fabrication of 36-in. diameter resin test loop is 40% completed and the field installation should be completed by July 15, 1956. The 36-in. diameter resin loop will be used to study resin flow characteristics and pulse power requirements.

The 12-in. diameter slurry contactor at Grand Junction, Colorado, may be tested further, starting in 60 to 90 days.

Power Reactor Fuel Reprocessing Studies

A spiral vibratory conveyor for dissolution of fuels with solids discharge has been designed which permits the connection of piping to the unit without the use of flexible connections.

An experimental hydrochlorinator for the dissolution of 3-in. lengths of Zr-U STR-type fuels has been designed and is being installed. This unit is being installed chiefly to determine the reaction temperature of Zr-U with HCl gas versus reaction rate.

A flowsheet for the aqua regia continuous dissolution of stainless steel-U fuel elements has been prepared and the dissolver designed.

A correlation of the vapor-liquid equilibrium of the $\text{HCl-HNO}_3\text{-H}_2\text{O}$ system has been completed. The volatility of HCl relative to HNO_3 increases as the total acid concentration increases and as the HCl concentration decreases.

Corrosion tests show tantalum to be superior in the aqua regia system and inconel and nickel for the anhydrous HCl system.

A work order has been written for the construction of the enclosure and manipulator carriers for the experimental mechanical handling facility.

Protective coatings and glass have been mounted on stringers for irradiation outside the HRT biological shield.

The 3-in. diameter ion exchange contactor for the isolation of plutonium from uranium has been completed and checked hydraulically. Minor alterations are to be made.

1.0 CHEMICAL ENGINEERING RESEARCH

J. C. Bresee

1.1 Dual Clone System
D. F. Bruley, D. W. Leigh

The dual clone experimental apparatus is being investigated to determine the feasibility of applying to liquid-liquid phase separation for extraction processes. This system was proposed because of the short hold-up time required thus making it desirable for the separation of radioactive materials.

The object of this month's work was to find out if the dual clone system could be operated effectively at high water concentrations in the feed tank (~30%). At present the experimental data indicates that a perfect separation cannot be attained at high water concentrations in the feed. Whether the system will be applicable as a practical unit for liquid-liquid separation processes will probably be dependent on the individual process separation efficiency required.

Because of the lower separation factors obtained at the high water feed concentrations the low range was rechecked (10-20% water in the feed). Results from the tests carried out supported previous findings as can be seen by comparing run number 29 of this report (Table 1-1) with run number 16 in the March 1956 monthly progress report.

Comparison:

Run No.	FC Vol. % H ₂ O	FR cc/min	R cc/min	FR/R	$\frac{FR}{FC}$ B'	SF_w	SF_s
16	13	500	1500	.33	.9	5000+	1000+
29	13.3	540	1400	.39	1.1	∞	∞

It appears that the present system of two 0.4-in. hydroclones operates most efficiently at an optimum concentration of approximately 13% water in the feed solution. It can be seen that the system is quite sensitive to alterations in flow rates, etc., even when at 13% by examining runs 30, 31, and 32. (Table 1-1) These runs were made by adjusting the system to perfect separation and then proceeding to readjust the system until either the underflow or overflow samples showed signs of mixed-phase. In this way a range in the 13% water feed concentration zone was established, the optimum conditions being those cited in run number 29.

As the feed concentration was varied above and below 13% water (a range from 10 to 20%) it was found that the separation factors decreased when determined at the optimum conditions within the specific feed concentration

Table 1-1

DUAL CLONE SEPARATION STUDIES
Summary of Data and Calculated Values

Run No.	Average Operating Conditions								ΔP F to B'	Sol Clarity Vol % Hexone	Aq Clarity Vol % H ₂ O	SF _W	SF _S
	FC Vol % H ₂ O	FR cc/min	R cc/min	FR/R	$\frac{FR}{FC}$ B'	C _B Vol % H ₂ O	C _R Vol % H ₂ O	C _F Vol % H ₂ O					
17	29.5	234	1800	0.13	0.79	41.7	24.6	26.5	15.8	97.23	86.5	10.6	5.2
18	29.5	355	1320	0.27	0.64	35.0	14.4	18.6	12.4	99	78	29.5	3.2
19	29.0	804	1040	0.77	0.96	73.0	58.0	45.5	10	95.7	85	6.7	4.75
20	29.0	1003	680	1.52	0.84	61.0	54.0	38.2	8.75	98.7	80	22.3	3.5
21	29.0	1284	560	2.30	0.97	71.0	67.6	41.0	9	98.6	93.0	20.5	10
23	28.3	522	2050	.254	0.84	40.8	27.7	28.4	28	96	90	7.1	7
24	28.3	1112	1720	.648	1.09	50	43.3	34.8	31.5	96.4	83.5	7.75	4.35
25	29.2	1342	1340	1.0	1.0	58.1	50.2	32	25	96.8	92.5	9.3	9.5
26	29.2	2234	700	3.2	1.12	73	66	37.6	24	96.7	99.99	9	7100
27	16.8	866	1460	.594	2.2	---	---	---	31.5	99.7	83.5	62	5
28	11.7	524	1400	.388	.88	6.2	1.9	16	34	99.96	84.5	26.6	6
29	13.3	540	1400	.386	1.07	---	---	---	33	100	100	∞	∞
30	13.3	1148	1440	.80	2.0	87	84	53.5	40.5	94.3	100	2.3	∞
31	13.3	1118	1180	.95	.81	47	47	29.3	34	99.4	82	22.5	4.8
32	13.3	1144	1020	1.12	.76	21.8	3.3	8.3	33.5	100	71	∞	3
33	14.7	802	1300	.615	.91	32	14.5	14.5	33.2	99.7	100	55	∞

under investigation (this can be seen in runs 28, 33). The optimum conditions were found by trial and error prior to data recording, thus, where it was possible to determine that the run was not acceptable by visual examination, data was not recorded.

Since the system is quite sensitive to alterations in flow rates, it might be advisable to install automatic controllers on the feed and recycle streams as well as having automatic control on the underflow from the second clone. The Fischer and Porter Automatic Controller now being used on the underflow stream has operated very efficiently in the past.

The information accumulated at this time indicates that substituting different clones in the dual clone system is probably the only solution to obtaining good separation at the high feed concentrations. At the present time the first 0.4-inch clone can give fairly good separation at the high feed concentrations ($\sim 30\%$ H_2O) if the recycle stream is mainly organic ($\geq 80\%$), however, the underflow from the first clone is then too concentrated in organic to give good separation in the second clone.

The clones being used in the system were originally designed for solid-liquid separation processes where an appreciable specific gravity difference was present. Since liquid-liquid separations are now being performed where the specific gravity of the two liquids is almost identical (difference of ~ 0.2) it is felt that a modified clone might improve the separation. The suggested modifications for separations at high water feed concentrations are (a) introduce the inlet stream to the clone slightly below the vortex finder, and (b) leave the core surface unpolished after machining.

1.2 Jet Pump System

D. F. Bruley, D. W. Leigh

Construction has been completed on the experimental jet pump system located in Cell 3 of Building 4505. As can be seen from Figs. the major equipment consists of a 1/2-in. Schutte and Koerting jet pump for mixing the hexone and water being used in the system, and a 0.4-inch hydroclone for phase separation so that mixing efficiencies* can be calculated. A Roth centrifugal pump of approximately 4 gpm capacity is being used to move the primary fluid (hexone) which is being pumped from a 50 gallon feed drum through the jet and clone into a 50 gallon collection tank.

It has been proposed that jet pumps be used in a multiple stage jet-clone system for liquid-liquid separation.

$$\text{*Thermal mixing efficiency} = \frac{(T_1 - t_1) - (T_o - t_o)}{(T_1 - t_1)}$$

NOTE: LETTERS REFER TO TABLE 1.1

The diagram illustrates a water supply system for a building. It features a main supply line with a valve (A) and a pressure-reducing valve (B). The main line branches into two parallel distribution lines. The left distribution line has a valve (C) and a pressure-reducing valve (D). The right distribution line has a valve (E) and a pressure-reducing valve (F). The left distribution line also has a valve (G) and a pressure-reducing valve (H). The right distribution line has a valve (I) and a pressure-reducing valve (J). The system is shown with various components labeled with letters A through J.

FIGURE 1.1
EXPERIMENTAL JET PUMP SYSTEM CHEMICAL ENGINEERING RESEARCH LOCATION
BUILDING 4505 - CELL 4

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Fig. 1-2. Experimental Jet Pump System (Refer to Table 1-1).
(Uninsulated)

TABLE 1.1

EXPERIMENTAL JET PUMP SYSTEM

Chemical Engineering Research Location, Bldg. 4505, Cell 4

Equipment on Hand:

- A. 50 gal. solvent feed tank
- B. Centrifugal pump (Roth - 4 gpm)
- C. Rotometer
- D. Rotometer
- E. Rotometer
- F. Jet pump (Schutte and Koerting, 1/2-inch)
- G. Clone (0.4-inch)
- H. 50 gallon catch tank
- I. Thermocouple
- J. Thermocouple
- K. Pressure gauge (0, 60 psi)
- L. Pressure gauge (-30, +30 psi)
- M. Pressure gauge (0, 100 psi)
- N. Hot water inlet (70-75°C), cold water inlet (20-25°C)
- O. Metal stand
- P. Metal stand

Note: Cross hatch represents insulation.

T_1 = temperature of inlet water t_1 = temperature of inlet organic
 T_0 = temperature of outlet water t_0 = temperature of outlet organic

Since the present jets have a large net driving head to net jet pump heat ratio (~ 3), they are not practical for use in multiple stage extraction systems. For instance, the initial pressure would have to be as high as 2300 psi for a four stage unit if the pressure drop across the clones is assumed to be 10 psi and $1/N$ equal to 3.

Assume:

$$\frac{1}{N} = \frac{H_1 - H_d}{H_d - H_s} = \frac{\text{net driving head}}{\text{net jet pump head}} = 3$$

ΔP across a clone = 10 psi

H_1 = driving head on nozzle
 H_d = discharge head of jet pump
 H_s = head on liquid being pumped

The primary goal of the experimental work with the jet pump system will be to reduce the $1/N$ ratio to unity. In an attempt to do this a range of R values (area of nozzle/area of throat) will be investigated as it is indicated in the literature (Centrifugal and Axial Flow Pumps, Stepanoff, p. 415) that as the area ratio is increased the $1/N$ value decreases.

Five runs were made without taking temperature readings due to the lack of a potentiometer. The data and results from these runs are presented in Table 1-2 and are found to check quite closely with the information available in the literature (Centrifugal and Axial Flow Pumps, Stepanoff, p. 416).

Runs 2 and 5 indicate that it might be possible to reach higher N values at similar* M values if lower flow rates are used. This will be investigated in future runs along with the effects of R value variation.

$$* M = \frac{Q_2}{Q_1} = \frac{\text{liquid being pumped (flow rate)}}{\text{liquid pumping (flow rate)}}$$

Table 1-2

JET PUMP EXPERIMENTAL APPARATUS
Summary of Data and Calculated Values

$$R = \frac{\text{Area Nozzle}}{\text{Area Throat}} = 0.25$$

Run No.	H_1 Pressure Rdg. #1 psig	H_2 Pressure Rdg. #2 psig	H_d Pressure Rdg. #3 psig	Q_1 Organic Flow ml/sec	Q_2 Water Flow ml/sec	Underflow Rate ml/sec	N $\frac{H_d - H_2}{H_1 - H_d}$	$1/N$	M $\frac{Q_2}{Q_1}$	Efficiency % $(MN)(100)$
1	11	5	7	4850	1800	2500	0.50	2.00	0.370	18.5
2	17	7.5	10.25	5750	1800	3200	0.41	2.44	0.313	12.8
3	23	10	14	6650	1800	3800	0.45	2.23	0.270	12.1
4	23.5	11	15	6300	2200	4000	0.47	2.12	0.350	16.5
5	23.25	11	14.5	6450	2020	4000	0.34	2.50	0.314	10.7

1.3 Steam Pulse Column

D. F. Bruley, D. W. Leigh

Construction of the modified steam pulse column is to be started on July 2, 1956. A 240 volt, 2 kw Chromalox immersion heater (catalog No. MTS-220-3B or MTI-220-3) was recommended as a hot finger for the pulsing unit. A 230 volt, 60 cycle Chromalox input controller (catalog No. VCS-202) is being considered for regulation of heat to the system.

2.0 EXCER

C. W. Hancher

The purpose of the Excer process program is to develop and test a method for the reduction of uranium oxides (UO_2 and U_3O_8) to UF_4 by a low temperature-aqueous chemistry procedure. Specification grade UF_4 is needed for feed for metal and UF_6 production. The Excer process consists of four main sections:

1. Feed preparation and decontamination.
2. Electrolytic reduction.
3. Filtration and drying.
4. Dehydration.

Electrolytic reduction and dehydration are now under investigation.

2.1 Electrolytic Cell

C. W. Hancher

The UNOP Excer Cell II, see Figure 2-1, has a 128 sq in. cathode, anode and membrane area. The cell is fed from the top of cell in a parallel manner. The catholyte and resulting $UF_4 \cdot 3/4 H_2O$ slurry are removed together from the bottom of cell and are separated outside of the cell. The anolyte is fed in series to the anolyte compartments.

Four satisfactory tests have been completed using the modified UNOP Cell I and redesign UNOP Cell II, see Table 2-1. The tap density of the filtered and dried product was not up to the specification (3 gm/cc). The average tap density of the four tests was 2.3 gm/cc. The electrolyte concentration was raised to obtain dense salt, from 0.5 M to 1.0 M with no change in salt density. It is now thought that salt density, which is directly related to crystal size, which is a function of current density, cell resistance, temperature, and electrolyte concentration. The conditions required to obtain the correct combination will be studied by additional tests.

The UNOP Cell II has capacity for uranium reduction to $UF_4 \cdot 3/4 H_2O$ of 65 pounds per day at 95% current efficiency. Ionics, Incorporated is designing a cell similar to the UNOP Cell II with a 450 sq in. cathode. This cell has a rated capacity, at 95% current efficiency, of 240 pounds UF_4 /day at 2.0 amps per sq in. For a 5000 ton/year plant 160 cells of this design would be needed with total current load of 144,000 amps.

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Figure 2-1 UNOP Excerpt Electrolytic Cell II

TABLE 2-1
KICER ELECTROLYTIC CELL TEST

Run No.	FEED TO CATHOLYTE				FEED TO ANOLYTE			ELECTRICAL			CATHODE	ANODE	% Uranium Reduction	PRODUCT	WASTE	
	Conc U G/L	SO ₄ G/L	F G/L	Rate cc/Min	Conc SO ₄ G/L	Rate cc/Min	Discharge Conc F G/L	Cathode = 128 in. ² Voltage	Amperage	% Current Efficiency	Temp °C	Temp °C		U%	U G/L	SO ₄ G/L
Ex-R-1	59.7	24.0	28.5	320	24.0	200	0.20	12.4	300	52	90-100	90-100	76%	73.9	12.0	38.0
Ex-R-2	51.6	40.0	21.6	320	24.0	200	0.14	10.4	220	39	90-95	90-95	28		37.4	14.0
Ex-R-3				130		150		10.2	240							
Ex-R-4	Mercury Pump Broke		--	--	--	--	--	--	--	--	--	--	--	--	--	--
Ex-R-5	120	100	42	150	100	150		12.0	250		90-95	85-90				

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2.2 Dehydration Study

C. W. Hancher

The dehydration of $UF_4 \cdot H_2O$ has been continued using the 3" diameter by 6 foot nickel ribbon flite dehydrator. To date, the highest % of dehydration was 89.4% or 0.37% water, on wet basis, see Figure 2-2 and Table 2-2, at an exit temperature of $390^\circ C$ and a heating time of 90 minutes. The high percentage of UO_2 and UO_2F_2 may have been caused by insufficient washing of hydrate after filtration and incomplete sweeping of oxygen from dehydrator before starting of dehydration test.

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FIGURE 2-2

% DEHYDRATION OF UF_4 VS HOLD-UP TIME

TABLE 2-2

EXCER DEHYDRATION STUDY

Fixed Conditions: Feed $\text{UF}_4 \cdot 3/4 \text{H}_2\text{O}$ as 3.5% water

Dehydration Temperature: 1 ft at 125°C ; 2 ft at 250°C ; 3 ft at 380°C

Dehydrator: Nickel Tube: 6 ft by 3" Dia. - Ribbon Flite Screw

Run No.	Feed Rate #/hour	Sweep Gas Co-current cc N_2 /Min/gm UF_4	Sweep Gas Counter-current cc N_2 /Min/gm UF_4	Hold-up Min	% H_2O	% Dehydration	% UF_4	% UO_2	% UO_2F_2	Tap Density cfm/cc
DR-1	3.42	76	115	35	1.47	66.0	96.70	0.58	3.71	2.50
DR-2	1.50	248	374	50	0.45	87.1	90.45	2.00	7.11	2.30
DR-3	1.40	268	400	50	0.51	85.4	86.05	7.50	7.93	2.35
DR-4	3.20	210	317	90	0.37	89.4				2.30
AEC Specifications							96.0	1.9	0-2.5	3.00

3.0 FLUOROX *

J. C. Bresee

Plans have been formulated for evaluation of pelleting procedures for appropriate mixtures of UO_3 , UO_2F_2 , starch and hydrated UO_3 fines.

The 4-in. i.d. Oxidation MBR has been under test on a dummy feed material containing 50% graphite.

Equipment modifications on both R-H and Oxidation MBR systems continue.

3.1 Pelleting

J. B. Adams, D. H. Newman

Plans for evaluation of pelleting procedures for future integrated operation of the Reduction-Hydrofluorination MBR and the Oxidation MBR have been formulated. Initially, optimum water content for pelleting of UO_3 , UO_2F_2 , starch, and hydrated UO_3 fines in appropriate ratios will be estimated. Further, relative strength, dusting properties, and setting time of test pellets will be evaluated. Finally, test runs in the present pelleting equipment or, if necessary, in a more suitable unit will be performed.

3.2 Reduction-Hydrofluorination Equipment

J. C. Bresee, J. B. Adams

Installation of new heaters and new insulation on the R-H Moving Bed Reactor is near completion. Modification of heater controls and drying of the insulation will be required before the reactor is ready for use.

3.3 Oxidation Equipment

J. C. Bresee, J. B. Adams, D. H. Newman, J. S. Taylor

Several short test runs oxidizing carbon from a mixture of 50% UO_2F_2 pellets and 50% graphite pellets were completed. Purpose of the runs was to dry out, test, and calibrate component parts of the system. Ignition of the feed was obtained easily by dropping feed material preheated to 500-600°C into dry oxygen preheated to 100-200°C. Temperatures in the bed proved very sensitive to oxygen flow rate and could be controlled fairly accurately by a manual control valve. To facilitate response to rapid temperature changes, however, the bed probe-thermocouple was connected to an individual indicator-recorder rather than to the multipoint instrument used for all other thermocouples measuring various temperatures in the same unit. The screw feeder used to meter feed from the preheat section into the reactor proper was calibrated. A delivery rate of 1/15 of a pound per

*Refer to ORNL-2117, "Fluorox Moving-bed Process for Producing UO_3 , UF_4 and UF_6 : Bibliography", by J. E. Moore.

revolution was found to be approximately constant in the range of absolute rates of from 4 to 13 pounds per hour. To permit lower absolute feed rates with sufficient power to maintain a constant delivery rate the pulley drive on the feed screw shaft was increased from 3 in. to 5 in. in diameter. Difficulty in starting bed flow in both preheat and oxidation sections after a static preheat and startup period have precluded runs of sufficient length to heat up completely and dry out the reactor system. In future runs the bed will be kept moving at all times in an attempt to prevent bridging and partial fusion from local overheating that causes flow stoppages.

The UF_6 hydrolysis column for recovery of cold trap contents as a solution of UO_2F_2 is scheduled for construction during the next month.

4.0 HR PROCESSING

E. O. Nurmi

Engineering evaluation of HR components, i.e., flanges, underflow pot, and hydroclone, and development studies on the behavior of solids and retrograde solubility fission products in high temperature circulating loops was continued. The results of these studies for the period ending June 15, 1956 are reported.

4.1 HR Fuel Processing Study in A-Loop

R. W. Horton, R. D. Arthur, V. L. Fowler, G. Woodall,
F. McLain, J. C. Rose, J. S. Taylor

Neodymium sulfate was precipitated and redissolved with reproducible results through 25 temperature cycles in an A-loop test of 311 hr duration. The position of the hydroclone (ahead of the preheater) had no noticeable effect on neodymium sulfate behavior. Zirconium oxide was concentrated in the underflow pot to 4560 mg Zr/liter, with a total zirconium recovery of 35%. Copper sulfate added to the loop feed solution to bring the concentration to 0.01 M remained in solution at all temperatures (4% was recovered from loop solids). An average loop overall corrosion rate of 0.002 in. per year was estimated for this test.

The temperature cycling test (LR 48) covered by this report was made to determine the effect of placing the hydroclone ahead of the preheater, particularly with respect to the behavior of zirconium oxide and neodymium sulfate. The stability of CuSO_4 in the solution at high temperatures was also to be investigated.

Placing the hydroclone ahead of the preheater (i.e., on the low temperature side) was considered desirable as a means of separating insoluble products (corrosion or fission) before the stream entered the heater, thus allowing the heater surfaces to remain clean except for the precipitation of inverse solubility fission products, and thereby preventing the preheater from being plugged.

The test extended over a four-week period, with the loop shut down 48 hours (approximately) during the intervening week ends. Several hours of operation at each temperature level allowed steady state conditions to be reached. A condensed log of the test (LR 48), with pertinent analyses, is shown in Table 4-1. The loop solutions used were at about the same zirconium concentration as the reactor is expected to produce in one day of operation, and contained only enough neodymium sulfate to maintain a system concentration greater than the solubility for the high temperature cycles. At the end of the test the loop was allowed to cool down before the solution was drained. This is contrary to previous procedures for A-loop in that the loop was drained at high temperature and pressure in other work reported. The analyses

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Table 4-1

CONDENSED LOG OF TIME, TEMPERATURES, AND OPERATING DATA FOR LR 48

Fill:

0.04 M UO_2SO_4
 0.01 M H_2SO_4
 50 mg $\text{Nd}_2(\text{SO}_4)_3/\text{kg H}_2\text{O}$
 0.167 gm Zr/kg H_2O
 0.003 M CuSO_4 (0.191 mg/ml)

Feed:

0.04 M UO_2SO_4
 0.01 M H_2SO_4
 150 mg $\text{Nd}_2(\text{SO}_4)_3/\text{kg H}_2\text{O}$
 0.5 gm Zr/kg H_2O
 0.01 M CuSO_4 (0.635 mg/ml)

Hydroclone: 0.25-in. T1 lined, 160 ml
 underflow pot, ahead of preheater
 Temperature differential (preheater
 over loop): 20°C
 Oxygen overpressure: 200 psi
 Loop flow rate: 6.4 gpm (80 cps
 power supply to pump motor)
 Total running time: 311 hr

Hours Operated	Preheater Temp. °C	$\text{Nd}_2(\text{SO}_4)_3$ Conc. mg/kg H_2O	Cu Conc. mg/ml	Zr Conc. mg/ml	U_1 BTU/hr/sq ft/°F	Preheater Flow Rate gpm		Volume of Feed ml	Remarks
						Calc. from Heat Transfer	From DP Cell		
6:35	30-315	50	0.19	0.167	---	---	0.19	0	Start-up, 4-23-1240
24	315	68	0.41	0.012	337	0.21	0.19	1150	
8	300	104	0.50	0.006	346	0.22	0.19	400	
12	315	91	0.51	0.007	333	0.21	0.18	600	
8	260	140	0.56	0.008	371	0.16	0.20	350	
12	315	99	0.53	0.009	336	0.22	0.19	550	
8	220	162	0.53	0.009	363	0.18	0.21	350	
8	315	102	0.52	0.013	339	0.21	0.19	350	
8	180	176	0.60	0.016	363	0.18	0.21	200	
4:30	180-30	180	0.65	0.018	---	---	---	50	Shut-down, 4-27-1545
6:15	30-315	178	0.64	0.018	---	---	---	35	Start-up, 4-30-1245
8	315	95	0.62	0.005	339	0.21	0.18	50	
11:15	265	151	0.59	0.005	350	0.19	0.18	50	
8	315	94	0.59	0.005	329	0.20	0.17	50	
12	215	164	0.63	0.005	364	0.18	0.19	100	
8	315	95	0.65	0.005	330	0.21	0.18	50	
13	165	172	0.67	0.005	371	0.19	0.20	100	
8:35	315	97	0.64	0.005	327	0.20	0.18	50	
12:10	145	175	0.71	0.005	365	0.18	0.21	50	
7	315	95	0.61	0.005	341	0.21	0.18	50	
4	315-30	178	0.67	0.005	---	---	---	---	Shut-down, 5-4-1500

Table 4-1 (Cont.)

Hours Operated	Preheater Temp. °C	Nd ₂ (SO ₄) ₃ Conc. mg/kg H ₂ O	Cu Conc. mg/ml	Zr Conc. mg/ml	U ₁ BTU/hr/sq ft/°F	Preheater Flow Rate gpm		Volume of Feed ml	Remarks
						Calc. from Heat Transfer	From DP Cell		
5:50	30-315	180	0.69		---			100	Start-up, 5-7-0910
8	315	95	0.61	0.005	339	0.20	0.17	125	
16	290	139	0.65	0.005	335	0.20	0.18	50	
8	315	95	0.61	0.006	323	0.20	0.16	100	
16:15	265	153	0.60	0.006	330	0.17	0.17	50	
7:45	315	98	0.61	---	301	0.17	0.13	100	
9:50	240	171	0.68	---	320	0.13	0.13	50	
8	240-30	---		---				---	
14	30-315	168			329	0.20	0.17	150	Shut-down, 5-10-1650
5:05	315	106			319-257	0.18-0.11	0.19		Start-up,* 5-14-1815
11	290	134			259-299	0.10-0.14	0.20	40	Flow rate falling
4:10	290-30	168							Final shut-down, 5-16-0430

*Loop solution was drained and measured before this start-up to determine amount lost through a defective valve packing during the shut-down period. 0.56 liters of new feed were added to make up the volume and the charge was returned to the loop.

of material drained and washed from the system is shown in Table 4-2, and material balances for uranium, zirconium, neodymium sulfate, and copper are shown in Tables 4-3, 4-4, 4-5, and 4-6, respectively.

The neodymium sulfate concentration, heat transfer coefficient, and preheater flow rate varied with operating temperature in a very consistent manner, as shown in Figures 4-1, 4-2 and 4-3. During the first two weeks of the test, it appeared that the preheater returned to original conditions each time that the temperature was reduced. During the third and fourth weeks of operation there was a tendency toward plugging the preheater, and this is reflected in heat transfer coefficients and flow rates. It is probable that zirconium oxide is the material causing the system to plug, since the amount in the system increased throughout the test. It will be noted that the zirconium concentration in samples drawn from the loop during the test is very low (0.005 mg/ml). The amount of zirconium removed from the underflow pot at the end of the test (Table 4-2) was about 30% of the amount added to the system. Previous experience with B2-loop (CF-56-5-197) has shown that zirconium oxide tends to adhere to all loop surfaces without respect to temperature or velocity. The same condition probably prevails in A-loop. The condition might be aggravated by the 48 hr shutdowns for week ends during the test.

The possibility that copper might be removed from solution at high temperature was also investigated in this test. Since the use of copper in small amounts as a catalyst for recombination of radiolytically produced hydrogen and oxygen is important to reactor operation, its stability in solution is of importance. The loop samples taken during the run showed that copper concentration soon reached that of the feed material and remained at that level for the duration of the test (Table 4-1). The amount of copper trapped in loop solids was about 4% of the total amount recovered (Tables 4-4 and 4-6) verifying the results indicated by loop sampling.

A corrosion rate for the loop was calculated from the amount of iron removed from the loop at the end of the test plus the amount assumed to be tied up with the zirconium oxide film remaining in the loop (this amount was taken to be equal to 10% of the weight of zirconium remaining in the loop, as shown by analyses of scale from B2-loop, CF-56-5-197). This calculates to a corrosion rate of 1.3×10^{-3} in. per year of continuous operation. The average temperature for this period was below 300°C because of temperature cycling.

Figure 4-1

Figure 4-2

Figure 4-3

Table 4-2

ANALYSES OF SOLUTIONS AND SOLIDS RECOVERED FROM SYSTEM AT THE END OF IR 48

Sample Designation	Volume ml	Total Solids mg	U in Solution gm	U in Solids mg	$\text{Na}_2(\text{SO}_4)_3$ in Solution mg	Cu in Solution mg	Cu in Solids mg	Zr in Solution mg	Zr in Solids mg	Fe in Solids mg
Loop drain	1930	30.5	19.50	4.6	322.0	1350.0	0.85	9.6	7.8	5.5
Loop wash, No. 1	2040	187.8	0.51	3.7	5.2	38.8	3.60	10.2	17.0	78.2
Loop wash, No. 2	2060	449.3	---	---	---	---	---	---	64.8	188.0
Clone pot drain	163.0	3653.5	1.74	47.8	27.4	114.0	188.0	0.8	654.0	1445.0
Clone pot wash #1	630.0	806.8	0.13	6.9	3.1	9.5	11.6	3.1	65.9	255.0
Clone pot wash #2	94.0	73.6	0.02	1.4	0.5	1.6	1.6	0.5	6.3	32.5
Lower filter wash	240	51.9	0.02	1.3	0.5	3.6	0.6	1.2	9.7	19.0
Top filter and preheater wash	600	299.0	0.35	5.1	7.9	18.6	2.1	3.0	26.3	127.5
Total		5552.4	21.27	70.8	366.6	1536.1	208.3	28.4	851.8	2150.7

Clone pot concentration:

A. Zirconium

731 mg recovered from 160 ml (nominal volume) = 4560 mg/liter.

B. $\text{Na}_2(\text{SO}_4)_3$

31 mg recovered from 160 ml = 193 mg/liter.

Table 4-3

URANIUM MATERIAL BALANCE FOR LR 48

Added to System:

Fill: 2.0 liters at 10 gm U/liter	20.0 gm
Feed and make-up: 5.96 liters at 9.8 gm U/liter	<u>58.4 gm</u>
Total Added	78.4 gm

Recovered from System:

Samples: 5.45 liters, avg. 9.89 gm U/liter	53.9 gm
Drain and wash solutions	21.27 gm
Loop solids	<u>.07 gm</u>
Total Recovered	75.24 gm

$$\frac{75.24}{78.40} = 0.96 \text{ or } 96\% \text{ recovery}$$

Table 4-4

ZIRCONIUM MATERIAL BALANCE FOR LR 48

Added to System:

Fill: 2.0 liters at 0.167 gm Zr/liter	0.334 gm
Feed and make-up: 5.96 liters at 0.38 g Zr/liter	<u>2.261 gm</u>
Total Added	2.595 gm

Recovered from System:

Samples: 5.45 liters at 0.005 gm Zr/liter	0.027 gm
Drain solutions	0.028 gm
Solids	
Loop	0.090 gm
Filters and preheater	0.036 gm
Underflow pot	<u>0.726 gm</u>
Total Recovered	0.907 gm

$$\frac{0.907}{2.595} = 0.349 \text{ or } 34.9\% \text{ recovery}$$

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Table 4-5

 $\text{Nd}_2(\text{SO}_4)_3$ MATERIAL BALANCE FOR IR 48Added to System:

Fill: 210 liters at 50 mg $\text{Nd}_2(\text{SO}_4)_3/\text{kg H}_2\text{O}$	100.0 mg
Feed and make-up: 5.96 liters at 150 mg $\text{Nd}_2(\text{SO}_4)_3/\text{kg H}_2\text{O}$	<u>894.0 mg</u>
Total Added	994.0 mg

Recovered from System:

Samples: 5.45 liters at various concentrations	605.2 mg
Drain solutions: 2.09 liters at 168 mg $\text{Nd}_2(\text{SO}_4)_3/\text{kg H}_2\text{O}$	351.1 mg
Wash solutions	<u>17.2 mg</u>
Total Recovered	973.5 mg

$$\frac{973.5}{994.0} = 0.98 \text{ or } 98\% \text{ recovery}$$

Table 4-6

COPPER MATERIAL BALANCE FOR IR 48

Added to System:

Fill: 2.0 liters at 0.19 gm Cu/kg H_2O	0.380 gm
Feed and make-up: 5.96 liters at 0.635 gm Cu/kg H_2O	<u>3.785 gm</u>
Total Added	4.165 gm

Recovered from System:

Samples: 5.45 liters at avg. conc. = 0.550 gm Cu/kg H_2O	3.000 gm
Drain solutions	1.536 gm
Solids	<u>0.208 gm</u>
Total Recovered	4.744 gm

$$\frac{4.744}{4.165} = 1.13 \text{ or } 113\% \text{ recovery}$$

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4.2 Hydroclone and Underflow Pot Test

N. A. Brown, C. C. Haws, Jr., F. N. McLain, J. S. Taylor,
J. C. Rose, R. D. Arthur, G. E. Woodall

The operating characteristics and dependability of the hydroclone and underflow pot circuits were tested prior to CPP installation. The testing program consisted of the determination of:

1. Heat transfer characteristics of the underflow pot heating jacket.
2. Hydroclone characteristics.
3. Effectiveness of the sampler and the agitation required for good sampling.
4. Effectiveness of the freeze plug on the bottom outlet of the underflow pot.
5. Remote tools required for removing the hydroclone and the clone retainer plug.

Heat transfer characteristics for the underflow pot have been determined. Heat-up and cool-off times and heat transfer coefficients have been obtained. In determining these values, the pot was filled with H₂O and air was passed through the underflow pot jacket. During the heating up period, heat was supplied by four 3.4 kw electric furnaces mounted on the air pipe. As the H₂O was heated the pressure in the pot was allowed to build up to 2000 psig, and was then maintained in the range of 1900-2100 psig by bleeding off the required amount of H₂O.

It was found that heating the pot from ambient temperature ($\sim 20^{\circ}\text{C}$) to 300°C required approximately 6 hr when air was supplied at 40-50 scfm (see Fig. 4-4) and that cooling the pot from 300 to 100°C required ~ 2.5 hr with the same air flow (see Fig. 4-5). The pot was cooled (from 300 to 100°C) in 1.5 hr by increasing the air flow to ~ 87 scfm. The heat transfer coefficient was determined to be 24 to 28 BTU/hr/sq ft/ $^{\circ}\text{F}$ for air flows of 40 to 50 scfm and 34 to 37 BTU/hr/sq ft/ $^{\circ}\text{F}$ for 86 to 88 scfm of air. These values were based on the internal area of that portion of the underflow pot which contacted the heating jacket. The data used to arrive at these values are shown in Table 4-7.

The line to the bottom of the underflow pot is to be used in the recirculation of the contents of the pot to provide a uniform slurry of the fission and corrosion products. The line is to be blocked (during operation of the pot to remove fission and corrosion products) with a freeze plug located immediately adjacent to the pot. It was feared that this freeze plug, being located so near to a large source of heat, would not hold. In order to check this plug an additional freeze plug was installed approximately 1 ft away on the same line. A nipple was welded into the space between these freeze plugs and an autoclave valve was installed at this point.

The freeze plug in question was checked by bringing the underflow pot

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Table 4-7

HEAT TRANSFER DATA FOR UNDERFLOW POT

Conditions: Overall coefficient based on inside wall area of underflow pot (2.265 ft²)
 For construction details of underflow pot see Dwg. E-24205

Heating Air				Liquid Temperature °F	Heat Transferred BTU/hr	Log Mean Temperature Difference (Air to Liquid) °F	Overall Coefficient of Heat Transfer BTU/hr/sq ft/°F
Flow cfm	Temperature °F						
	Inlet	Outlet	Difference				
37.2	655	565	90	531	3716	69.6	23.6
37.2	660	576	84	545	3469	64.1	23.9
37.2	660	585	75	559	3097	55.3	24.7
38.1	660	545	115	511	4863	77.6	27.7
42.6	574	482	92	444	4350	74.8	25.7
42.6	595	505	90	473	4256	67.2	28.0
42.6	608	525	83	491	3925	67.2	25.8
46.4	406	266	140	190	7211	134.0	23.8
47.1	502	352	150	271	7842	143.1	24.2
47.1	604	536	68	505	3555	58.6	26.8
47.1	606	558	48	536	2509	41.5	26.7
47.1	608	547	61	520	3189	51.6	27.3
47.1	608	554	54	531	2823	44.7	27.9
47.3	554	421	133	354	6983	121.6	25.4
47.3	572	464	108	410	5670	98.3	25.5
47.3	586	496	90	453	4725	79.7	26.2
47.3	594	518	76	480	3990	69.2	25.5
48.0	532	442	90	401	4795	77.5	27.3
48.2	464	338	126	277	6741	112.5	26.5
48.2	511	399	112	345	5992	99.7	26.5
50.9	360	259	101	201	5706	100.2	25.1
Cooling Air				(Liquid to Air)			
47.9	100	192	92	237	4892	82.6	26.1
48.2	154	302	148	387	7918	146.8	23.8
86.5	93	117	24	136	2304	29.4	34.6
87.7	97	133	36	163	3504	45.7	33.9
88.0	106	172	66	223	6447	79.5	35.8
88.0	126	234	108	322	10549	134.9	34.5
88.3	189	347	158	466	15486	187.0	36.6

to 300°C and 2000 psig, after which the plug was allowed to freeze. The back-up plug was then frozen and the valve was opened. There was no flow from the line, nor was there any loss of pressure from the system. This test was made several times. It is concluded that the freeze plug at the bottom of the pot will be effective.

Following the tests to determine the heat transfer characteristics and those of the freeze plugs, a test was made to determine the hydroclone characteristics. In this test the B-loop and underflow pot were charged with 10 gm U/kg H₂O soup and the system was brought to operating conditions. Although normal conditions would be 2000 psia at 300°C, it was possible to raise the temperature to only 270°C due to inadequate heater capacity in the B-loop.

The material added to test the hydroclone was composed of Fe₂(SO₄)₃, Cr₂(SO₄)₃·K₂SO₄, and Zr(SO₄)₂ in the proportions the Fe, Cr, and Zr will theoretically be present in the HRT-CP feed stream. A solution was prepared which contained 5 gm/l of Fe, Cr, and Zr (total) and 10 gm of U/kg H₂O. The solution was prepared in small lots to prevent hydrolysis of the Zr before the solution could be injected into the loop. A Lapp Pulsafeeder was used to add the solution to the loop, and another was used to introduce a 0.5 M solution of NaOH to maintain the loop pH at 1.9-2.1. As these materials were added, an equivalent volume of liquid was removed through the clone overflow sampler in order to maintain the operating pressure at 2000 psi.

Operation of the two Lapp pumps was not satisfactory and after two days of almost fruitless effort, the loop was shut down and cooled off. The remainder of the Fe-Cr-Zr was dissolved in about 1 liter of soup and was injected into the loop using air pressure. Circulation was started, heat was applied, and a temperature of about 250°C was attained. Circulation was continued for about 30 hours in an attempt to hydrolyze the Fe-Cr-Zr sulphates to oxides and to have the hydroclone remove them. Samples of the clone overflow and the pot contents will be submitted for analysis.

Following this test the remote tools for the removal of the clone retainer plug and the clone will be tested.

4.3 High Pressure Flange Tests

N. A. Brown, C. C. Haws, Jr., F. N. McLain, J. S. Taylor,
J. C. Rose, R. D. Arthur, G. E. Woodall

Heat losses affect the efficiency of the CPP hydroclone and thus the operation of the pilot plant. Insulation can limit heat losses, but may furnish other problems in the long term leak tightness and safety of the flanged joint. These tests were set up to determine the extent of the heat losses, type and quantity of insulation required, effects of thermal

cycling, and the best materials and method for assembly and disassembly of the flanges.

Work during the past month has been devoted almost entirely to thermal cycling of octagonal gasketed flanged joints. This series of tests shows the octagonal ring to be a marked improvement over the oval ring previously tested. No leaks were obtained in any of the cycles and the only principal remaining concern is the high bolt stress (65,000 psi) found at reactor temperatures. Forthcoming work will be directed toward lowering this stress.

Tests have shown that torque wrench readings are adequate for accurately stressing the bolts of the flanged joints. Also, the cadmium-plated bolts purchased for this use may be used repeatedly (within their elastic limit) without encountering a change in their torque-load relationship.

Two flange pairs, assembled with standard octagonal rings, were tested as shown in Table 4-8. An attempt was made to start with relatively long heating and cooling cycles and then shorten these as the run progressed, but loop operating difficulties prevented any reasonable control over the cycles. It was possible, however, to shorten these cycles to the desired minimum values by the end of the run as shown in Cycle 9. The times shown in Cycle 9 represent the minimum obtainable and compare with previous shock tests on the oval ringed joints. No leaks were obtained on any of these cycles, while similar tests on oval rings produced leaks in all closures.

One of the gaskets used in this series of tests was the standard 347 SS octagonal type ring with a 0.003 in. gold plate. While a pressure-tight seal was obtained at 4000 lb per bolt with this gasket (its bare companion required 6000 lb per bolt), the hot loading was the same for both gaskets (20,000 lb per bolt, or 65,000 psi). Thus gold plating offers no advantage and is not recommended.

High bolt stresses (65,000 psi) found in these tests should be lowered to control creep and to provide a greater safety factor for the joint. Belleville springs have been ordered for testing and are scheduled for shipment late in July. A modified flange-ferrule design has been completed and the unit is ready for assembly and testing. Either of these methods should lower the above stress to ~30,000 psi.

Inspection of the octagonal gasket and the flange grooves after the test showed no perceptible deformation, only a brightening of the contacting surfaces. This contrasts with actual grooving, swaging, and galling of these same surfaces when the oval ring was used.

The absence of leaks in the cycles of this series, plus the negligible marring of the metal mating surfaces represents a considerable improvement

improvement over the previously tested oval gasket. While it is recognized that this series is lacking in depth, the improved performance of the closure is so great that octagonal ring gaskets can be recommended for CP use. An adequate supply of these octagonal rings should be obtained (per latest HRT specification) for the high pressure circuit of the CP.

Bolt loadings and equivalent stresses obtained at various torque values are given in Table 4-9. Stresses were calculated from measured bolt elongation. Tests No. 1 and 3 (using new bolts) show that the precision obtained in torque wrench loading is $\pm 5\%$. This is excellent. Test No. 2 used the bolt from Test No. 1, and since this bolt had been badly overstressed in the previous test, a different torque load relationship was obtained (see Fig. 4-6). Other new bolts were tested with equally precise results and no change in the torque load relationship was found upon their repeated reuse.

In the April issue of this report, (CF-56-4-210) comment was made that the hot loading of bolts on the oval gasket closure always reached, but did not exceed, 15,000 lb (equiv. to 50,000 psi). Gasket slip (or penetration of gasket into the opposing flange grooves) was stated at that time to be a more serious problem than overstressing of the bolts. The serious nature of excessive gasket slip in the oval gasketed joint is recognized only when it is considered as flange groove and gasket deformation - both of which contribute to leakage on thermal cycling.

Gasket slip for an oval and an octagonal ring were measured and are shown in Fig. 4-7. The comparative strength of the oval and octagonal gaskets are easily seen from this chart. The octagonal ring has a lower and more uniform slip, a major portion of which should be retrieved through spring-back in thermal cycling. The oval ring exhibits a similar slip up to a bolt load of some 15,000 lb; however, beyond this value the slip increases sharply (denoted by dotted line) with little increase in bolt loading.

This sharp increase in the slip of the oval gasket is the reason for obtaining the remarkably uniform hot bolt loadings of 15,000 lb mentioned in the earlier tests. Similarly, this sharp break shows the magnitude and importance of the flange-groove deformation encountered in thermal cycling. This sharp upward break also illustrates another point, namely, that hot bolt loadings for the oval gasketed joint must be kept very low (i.e., less than $\sim 12,000$ lb), or raised very high (i.e., greater than $\sim 18,000$ lb). Since a minimum 18,000 lb hot bolt load represents an excessive stress (60,000 psi) this cannot be recommended unless the oval gasket is coined with one set of bolts and these replaced with bolts at safer loadings. On the other hand, it is not desirable to design for a maximum hot load of 12,000 lb, if a known precipice exists above this value. Through this reasoning, again, the oval gasket is judged inferior to the octagonal gasket.

Table 4-9

UNCLASSIFIED
ORNL-LR-DWG. 14482HIGH PRESSURE FLANGE STUDIES
Bolt Stress-Strain Data

TORQUE FOOT POUNDS	TEST #1			TEST #2			TEST #3		
	LOAD (lb)	STRESS (psi)	$\frac{dE}{dT}$	LOAD (lb)	STRESS (psi)	$\frac{dE}{dT}$	LOAD (lb)	STRESS (psi)	$\frac{dE}{dT}$
50	5,027	16,373	3.8	7,144	23,268	5.4	4,763	15,513	3.6
100	10,319	33,608	4.0	13,493	43,947	4.8	9,789	31,883	3.8
150	15,346	49,981	3.8	19,500	63,512	4.6	14,552	47,396	3.6
200	20,100	65,466	3.6	26,723	85,571	5.4	19,048	61,942	3.4
250	24,605	80,138	3.4	32,808	106,855	4.6	23,018	74,970	3.0
300	30,691	120,215	4.6	43,126	140,461	7.8	29,103	94,788	4.6
340	----	----	----	BOLT	BROKE		----	----	----
350	84,000	273,588	40.0	----	----	----	39,687	129,260	8.0
360	88,000	286,616	----	----	----	----	----	----	----

BOLT DESCRIPTION: CADMIUM PLATED, 3/4 in. x 3.5 in. (ENGAGED LENGTH) ASTM A193
GRADE B7.

0000000000

FIGURE 4-7
HIGH PRESSURE FLANGE STUDIES
slipping characteristics of
two standard ring type gaskets.

4.4 HRT-CP Hydroclone Development Studies P. A. Haas

Enlarged photographs of the three hydroclones most recently cross-sectioned for examination show the low corrosion rates obtained (Fig. 4-8, 4-9, 4-10). These and other corrosion tests (summarized in CF-56-5-197) have shown corrosion rates of 10 mil/year or less for titanium hydroclones on underflow pots. While stainless steel hydroclone 25-SS-5 (Fig. 4-9) showed a low corrosion rate for the relatively low flow rate used, other stainless steel hydroclones have shown corrosion rates of over 100 mil/year. Therefore all hydroclones used in HRP chemical processing plants will be titanium units.

4.5 HRP-Plutonium Producer Blanket Processing Development Loops P. A. Haas, S. D. Clinton

4.5.1 Component Tests

The H₂O-cooled sample valve used with the loop P-1 hydroclone showed no corrosive attack after twenty sample cycles. Each cycle included taking a 100 ml sample of 400 g U/kg H₂O solution from the loop at 240-260°C. The valve surfaces including the stainless steel series 400 stem remained clean and scale-free. The 400 stems were badly corroded by 10 g U/kg H₂O soup in other loops. The lack of attack on the valve tested might also be due to the cooling coil on the hydroclone sample line.

Titanium components of the T-1 loop including the pump impeller, pump scroll case, and flow restrictors show no apparent attack after over 1000 hours of operation with 350-400 g U/kg H₂O solution. Room temperature checks of the loop P-1 hydroclone and of an all titanium hydroclone after 160 and 850 hours, respectively at blanket conditions are partially complete.

4.5.2 Loop T-1 Pump

The loop T-1 ORNL canned rotor pump showed continued rapid corrosion of the stainless steel flange which is the back of the pump scroll. The maximum attack has continued at the 1000 mil/year rate reported for the first 350 hours (CF-56-4-210). The attack has been limited to the 3/8-in. wide annular ring behind the impeller and outside the lock-in washer. The washer itself shows only minor amounts of attack. Shavings from the washer and the flange are being analyzed to determine the difference in compositions; both should be 347 stainless steel. The thermal barrier and the pump can flange are being altered to use a 0.094-in. thick titanium washer as the surface behind the impeller. The 0.094 inches machined from the flange to provide a seat for this titanium washer did not quite equal the depth of maximum corrosion. The total time of pump operation is now about 1080 hours.

Fig. 4-8. Corrosion of a 0.40 in. D. Titanium Lined Hydroclone.

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PHOTO 17722

0 2 3 4

INCHES

VORTEX FINDER

UNDERFLOW PORT

INDUCED
UNDERFLOW
POT

DIAMETER: 0.25 IN.
LENGTH: 1.50 IN.
MATERIAL: 347 STAINLESS STEEL
CONDITIONS:
10 g U/kg H₂O
320°C
TEST PERIOD: 1200 HOURS
TEST LOOP: UNOP LOOP A
FEED FLOW: 0.13 TO 0.20 GPM

25-SS-5

Fig. 4-9. Corrosion of a 0.25 in. D. Stainless Steel Hydroclone.

INCHES

DIAMETER: 0.25 IN.
LENGTH: 1.50 IN.
MATERIAL: TITANIUM 55-A
CONDITIONS:
10 g U/kg H₂O
300°C
TEST PERIOD: 3975 HOURS
TEST LOOP: REED LOOP K
FEED FLOW: 0.23 GPM

25-Ti-4

Fig. 4-10. Corrosion of a 0.25 in. D. Titanium Lined Hydroclone.

4.5.3 Traced Zr Addition

Of the Zr-95 and Nb-95 added to loop T-1 to trace zirconium added as $\text{Zr}(\text{SO}_4)_2$; only about eight percent of the γ activity was recovered from the loop. (See Tables 4-10 and 4-11). The activity was evenly distributed around the loop as shown by readings averaging 5 mr/hr at the insulation surface. There were no points of high activity although the pump discharge line showed on the order of 150% of the average loop readings.

The experimental procedure used three cycles with addition of 146 mg Zr as $\text{Zr}(\text{SO}_4)_2$ at the start of each cycle. The zirconium in 100 ml of UO_2SO_4 solution was flushed in by 300 ml of additional UO_2SO_4 solution. Hydroclone pot samples were taken at 10, 30, and 60 minutes after the start of zirconium addition. The hydroclone pot samples were 100 ml and were intended to be complete removal of the 55 ml of pot solution. Loop samples were taken at the end of each cycle. After the third cycle, the loop was shut down and cooled over a weekend. After a half hour of low temperature circulation, the loop was drained.

The loop uranium concentration decreased throughout the run with high uranium concentrations in the initial samples (See Table 4-10). This could be caused by formation of a heavy phase although the loop temperatures of 247 to 265°C (See Table 4-12) should not result in heavy phase formation. The uranium recovery was 98% (See Table 4-13). The decrease in nickel concentration could be explained by assuming the surge tank contained 300 ml of stagnant, nickel free solution. Probably some nickel was removed by the zirconium which was precipitated.

The radiochemical γ activity analyses indicated that the γ activity of the tracer was 35% Zr-95, 65% Nb-95. The γ activities of loop and hydroclone samples (corrected for the uranium activity) ranged from the feed Zr-Nb ratio to 15% Zr, 85% Nb. The disappearance of more of the Zr activity might be explained by the presence of inert Zr "carrier" while no inert Nb was added.

4.5.4 Loop P-1

The tests for $\text{H}_2\text{-O}_2$ detonations in the Loop P-1 pressurizer will be carried out at X-10 ² probably in a UNOP cell -- since the REED Division does not have facilities for these tests. Results of a Syracuse University research program will probably supply some of the information needed. Their research program, under an AEC contract initiated by the REED Division, was specifically to study $\text{H}_2\text{-O}_2$ detonations.

Fabrication of loop P-1 components is continuing in the ORNL shops. The elements of the Westinghouse A-150 pump were photographed while the pump was disassembled for inspection (See Figure 4-11).

Table 4-10

SAMPLE ANALYSIS - LOOP T-1 RUN NO. 15

BASIS: Concentrations and Activities Based on Room Temperature Densities

Sample Time	Sample Source	U Conc. (g/l)	Activity 10^5 c/min cc	Ni Conc. (mg/l)	Radiochemically Separated γ	
					Zr-95 (10^4 c/min cc)	Nb-95 (10^4 c/min cc)
--	Zr Stock	0	653	0	1800	3300
5-14-1340	Fresh Feed	368	0.21, 0.29	<5	0	0.02
5-29-0005	Fresh Feed	356	0.24	2	---	---
5-29-0910	Zr-UO ₂ SO ₄ Feed	304	101	---	290	540
5-29-0031	Hydroclone Pot	584	3.52	1290	5.9	16
-0051	Hydroclone Pot	---	1.64	---	---	---
-0121	Hydroclone Pot	---	1.28	---	---	---
5-29-0130	Loop Pre-Sample Flush	---	1.64	1010	---	---
-0131	Loop Sample	551	1.35	1200	---	---
5-29-0215	Hydroclone Pot	473	1.84	858	1.7	9.6
-0235	Hydroclone Pot	---	1.27	---	---	---
-0305	Hydroclone Pot	---	1.09	---	---	---
5-29-0314	Loop Pre-Sample Flush	---	2.85	866	---	---
-0315	Loop Sample	492	1.15	1050	1.3	4.5
5-29-0335	Hydroclone Pot	421	1.77	---	---	---
-0355	Hydroclone Pot	---	0.98	---	---	---
-0425	Hydroclone Pot	424	0.92	---	---	---

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Table 4-10 (cont.)

Sample Time	Sample Source	U Conc. (g/l)	Activity 10^5 c/min cc	Ni Conc. (mg/l)	Radiochemically Separated γ	
					Zr-95 (10^4 c/min cc)	(10^4 c/min cc)
5-29-0434	Loop Pre-Sample	---	3.23	---		
-0435	Flush					
	Loop Sample	---	0.99, 0.99	632	0.92	4.3
5-29-1005	Loop Sample	350	0.36	647		
5-29-1205	Loop Sample	355	0.45	716		
5-29-1430	Loop Sample	320	0.31	560		
5-29-1515	Hydroclone Pot	253	0.93	480		
6-4-0925	Hydroclone Pot	201	2.48	390, 419		
-0930	Loop Sample	207	0.24	---	0.23	0.39
6-5-1800	Loop Drain	175, 175	0.28, 0.14, 0.12	362		
6-5-2330	1st H ₂ O Wash	15.6, 15.7	0.18, 0.24	---		
6-6-2000	2nd H ₂ O Wash	1.8, 1.8	0.02, 0.02	---		

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Table 4-11

γ ACTIVITY BALANCE - LOOP T-1 RUN NO. 15

BASIS: γ Scintillation Count Through Aluminum at Room Temperature Corrected for 75 c/min gm U; Zr-95 and Nb-95

RUN CONDITIONS: ~400 g U/kg H₂O, 247 to 265° C

Stream	Volume (cc)	Corrected γ Activity	
		10 ⁵ c/min cc	10 ⁵ c/min
Zr Stock Solution	46.9	653	30,600
Diluted Zr Stock	300	101	30,300
INPUT γ ACTIVITY		(Average of Above)	30,500
1st Cycle Clone Samples	300	1/3(3.12 + 1.24 + 0.88)	524
1st Cycle Loop Flush and Sample	100	1.2	120
2nd Cycle Clone Samples	300	1/3(1.5 + 0.9 + 0.75)	315
2nd Cycle Loop Flush and Sample	100	2.2	220
3rd Cycle Clone Samples	300	1/3(1.45 + 0.7 + 0.65)	280
3rd Cycle Loop Flush and Sample	100	2.4	240
Post Run Loop Samples	400	1/4(0.12 + 0.21 + 0.07)	40
Post Run Clone Samples	200	1/2(0.7 + 2.2)	290
Loop Solution When Drained	1150	0.06	69
1st H ₂ O Wash Solution	1800	0.20	360
2nd H ₂ O Wash Solution	1800	0.02	36
TOTAL γ ACTIVITY RECOVERED			2,494
% γ ACTIVITY RECOVERED			8.2%

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Table 4-12

LOOP T-1 CONDITIONS DURING RUN NO. 15

DATE: May 29, 1956

RUN CONDITIONS: 955 to 1115 Psig, 74 to 76 Cycles A.C.,
to Pump, Hydroclone $P_F - P_0$ of 40 to 60 ft. of Liquid

Time	Pressure (psig)	Temperatures ($^{\circ}\text{C}$)				Loop Heater Input (kw)
		Pressurizer	Pump Suction	Pump Discharge	Hydroclone Discharge	
0016	970	257	255	254	247	2.44
0041	1115	265	264	261	255	1.30
0111	1045	263	262	260	253	1.91
0201	955	257	255	255	249	3.17
0225	1080	261	261	259	249	3.32
0255	1035	262	261	260	254	2.65
0321	955	264	263	262	252	3.17
0345	1115	259	259	256	250	2.20
0415	1060	258	257	255	251	2.30
0620	955	251	250	248	243	2.64

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Table 4-13

URANIUM AND NICKEL BALANCES - LOOP T-1 RUN NO. 15

RUN CONDITIONS: Loop Temperatures of 247 to 265°C, 350 Psi O₂ Over Pressure

BASIS: Analyses for Concentrations at Room Temperature

Material	Volume (cc)	Uranium		Nickel	
		Conc. (g/l)	Amount (gm)	Conc. (mg/l)	Amount (mg)
Diluted Zr Feed	300	304	91	0	0
Zr Flush Solution	900	356	310	0	0
Loop Solution	1700	356	605	1300	2210
H ₂ O Washes	<u>3600</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>
TOTAL INPUT	6500		1006		2210
1st Cycle Clone	300	584	175	1290	387
1st Cycle Loop	100	551	55	1105	111
2nd Cycle Clone	300	473	142	858	257
2nd Cycle Loop	100	492	49	958	96
3rd Cycle Clone	300	423	127	632	190
3rd Cycle Loop	100	423	42	632	63
Post Run Loop	400	308	123	600	240
Post Run Clone	200	227	45	405	81
Loop Drain	1150	175	201	362	416
1st H ₂ O Wash	1790	15.7	28	--	--
2nd H ₂ O Wash	<u>1800</u>	<u>1.8</u>	<u>3</u>	<u>--</u>	<u>--</u>
TOTAL RECOVERED	6540		990		1841
PERCENT RECOVERY	101		98		83

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Fig. 4-11. Loop P-1 Westinghouse 150A Pump.

5.0 METALLEX

J. C. Suddath

The development of the Dry Salt flowsheet was continued with emphasis on the installation of the 1 1/2 gal. Dispersall and 4-in. D wash column as continuous contactors with a batch feed system and product filtration.

The installation of the Interim Facility for the production of thorium-mercury "quasi" was completed, but immediate plans call for only a few development runs to be made without any large production.

6.0 POWER REACTOR FUEL REPROCESSING STUDIES

C. D. Watson

Introduction:

Experimental programs active during this period were:

- (1) Discharging Solids from a Dissolver
- (2) Hydrochlorination of Zr-U Alloys
- (3) Aqua Regia Dissolution of Stainless Steel-U Fuel Elements
- (4) HCl Distillation
- (5) Materials Evaluation
- (6) Experimental Mechanical Handling Facility
- (7) Universal Slug Loader
- (8) Radiation Damage Studies
- (9) Continuous Ion Exchange Purification

Programs in progress but inactive during this period were:

- (1) Recovery of Spent Solvent with a Hydroclone
- (2) Shearing and Shredding Studies
- (3) Slug Dejacketing Studies

6.1 Hydrochlorination of Zirconium-Uranium Alloys

R. J. McNamee, J. J. Perona, F. L. Rogers

It was shown (May monthly) that only 12% of the heat of the zirconium hydrochlorination reaction could be removed by the stoichiometric reaction gases, provided the maximum temperature allowable was 500°C. At this temperature and a total reaction time of four hours, only 7% of the heat of reaction would be removed by radiation. Hence, either the balance of the heat will

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cause the temperature to rise, or the reaction time can be increased so that the radiation rate will be able to handle the rate of heat evolution. The temperature rise will be investigated in a small hydrochlorinator, currently being fabricated. Calculations were made to estimate reaction times necessary for adequate heat dissipation.

Since there is some question about the emissivity of a zirconium fuel element (could range from literature value of 0.18 to 1--a "black body", for it is oxidized and black in appearance), this could greatly affect the final temperature to which the element would rise. For example, calculations have been made which show that this temperature could vary from 583 to 904°C for a four hour reaction time. In order to evaluate what temperature can be expected, as a function of reaction rate, a small unit for hydrochlorinating about a three inch length of an STR sub-assembly has been designed and is being fabricated (see Figure 6-1). The thermowell coming from the bottom into the middle of the reactor will be inserted between two plates of the element, thereby measuring its temperature. This unit will make possible a thorough investigation of the heat transfer problems associated with such a reactor. It will also show the feasibility of a jacketed pipe $ZrCl_4$ condenser, make possible a determination of the maximum operating temperature (such that U will not volatilize), and evaluate entrainment of UCl_3 in the gas stream.

Calculation of times necessary to process a STR sub-assembly, as a function of maximum temperature allowable and assumed emissivity, gave the following:

Max. Temp.	Theoretical ϵ	"Black Body"
500°C	47.4 hr.	7.6 hr.
700	11.3	2.0

This was obtained by dividing the net heat which would have to be removed by radiation (heat of reaction for producing $ZrCl_4$ gas minus sensible heat picked up by reaction gases) by the rate of heat loss by radiation. Since the former is a function of the amount of metal reacted and the latter a function of radiating surface area, and since these are proportional to each other, it can readily be shown that this time is independent of the amount of element reacted in a batch (provided they are stacked singly, one on top of another). Thus, there will be a maximum allowable reaction rate, and this can be determined experimentally as a function of limiting maximum temperature and uranium loss in the small hydrochlorinator, which will soon be ready for operation. This information will be valuable for scale up design, since it is theoretically independent of batch capacity.

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SMALL-SCALE EXPERIMENTAL HYDROCHLORINATOR
 FIGURE 6-1

6.2 Aqua Regia Dissolution of Stainless Steel-Uranium Fuel Elements

R. J. McNamee, J. J. Perona

A flowsheet for the Unit Operations aqua regia continuous dissolver for stainless steel (APPR) fuel elements has been drawn (see Figure 6-2). This unit will not only provide continuous dissolution data, but will also provide feed for evaluation of the other components of the overall aqua regia flowsheet--HCl removal by distillation, feed clarification using a magnetic filter, and finally uranium removal from the stainless steel-nitric acid solution by solvent extraction.

The dissolver has been designed. It consists of a vessel 8 in. in diameter and 2 1/2 ft. tall, water jacketed for heat removal. The liquid level will be controlled at different depths to produce variable submergence of the element, making possible the determination of the maximum depth allowable, due to its effect on the time necessary for dissolution (and hence, rate of heat evolution compared to the maximum possible rate of heat removal).

6.3 HCl Distillation

H. F. Johnson, W. J. Lee, F. G. Kitts

If a feasible process for the aqua regia dissolution of S. S.-U fuel elements is to be developed, a method for completely removing the chloride from the dissolution product prior to solvent extraction must be devised. If the chloride were not removed, corrosion rates in the S. S. solvent extraction equipment would be prohibitive and in addition, uranium extraction would not proceed normally in the presence of chloride. The present concept of such a system includes a dissolver, and columns to remove aqua regia from dissolution product, to remove HCl from aqua regia, and to concentrate acids to usable strengths for recycle.

To design a column for the separation of hydrochloric acid from nitric acid, equilibrium data for aqua regia are necessary. A correlation of vapor-liquid equilibrium data for the $\text{HCl-HNO}_3\text{-H}_2\text{O}$ system has been made, the basis being experimental work performed with a Gillespie Still by MIT Practice School students. The ultimate purpose of this work is to devise a distillation system which will selectively remove HCl from an aqua regia dissolver product solution and provide HCl and HNO_3 solutions of suitable strength for use in making up fresh aqua regia dissolver solution.

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FLOW SHEET FOR AQUA REGIA DISSOLVER

FIGURE 6-2

This final correlation shows that the volatility of HCl relative to that of HNO_3 increases as total acid concentration increases and as HCl concentration decreases. The y/x for HCl does not become greater than one until total acid concentration is greater than about 12 mol %.

The fundamental consideration in the work was the correlation of activity coefficients, where the activity coefficient is defined as:

$$\gamma_1 = \frac{Py_1}{\bar{P}_1 x_1}$$

where:

γ_1 = activity coefficient of component 1 in the liquid phase

P = total pressure of the system

x_1 = mole fraction of 1 in the liquid phase

$\bar{P}_1$ = vapor pressure of pure 1 at the temperature of the system

y_1 = mole fraction of 1 in the vapor phase

P, x_1 , y_1 , temperature and specific gravity for the system at atmospheric pressure were reported directly by the MIT students, and $\bar{P}_1$ was determined from data reported in the literature.

The calculated activity coefficients, γ_1 , were then plotted vs. x_1 on semi-log paper for each component and values of parameters were tabulated for each point on the plot. Parameters employed were as follows:

$$\gamma_A \text{ vs. } x_A, \text{ parameter} = \frac{x_B}{x_B + x_C}$$

$$\gamma_B \text{ vs. } x_B, \text{ parameter} = \frac{x_A}{x_A + x_C}$$

$$\gamma_C \text{ vs. } x_C, \text{ parameter} = \frac{x_A}{x_A + x_B}$$

where the subscript "A" refers to HCl, "B", to HNO_3 , and "C", to H_2O . Reference curves with parameter = 0 were then plotted for each activity coefficient plot; e.g., for the γ_A plot, the reference curve consisted of γ_A 's in the binary system HCl- H_2O , for which the parameter = 0.

From the line $\gamma_i = 1$, the perpendicular distances to the plotted points and to the reference curve were measured. The ratios of the former to the latter were then plotted vs. parameter. From a smooth curve through these points, for given parameters, ratios could be found, and smooth lines, each of constant parameter, were drawn, these being the γ_i vs. x_i plots.

Using the facts that $y_A + y_B + y_C = 1$, $y_i = \frac{x_i \gamma_i \bar{P}_i}{P}$, and

$P = \gamma_A \bar{P}_A x_A + \gamma_B \bar{P}_B x_B + \gamma_C \bar{P}_C x_C$, $\frac{y_i}{x_i}$'s were computed for various liquid phase compositions.

Values for x_A and x_B were arbitrarily taken and temperatures were computed by trial and error use of the above equation for P . Having established temperatures, y_i/x_i 's were computed from $y_i/x_i = \gamma_i \bar{P}_i/P$.

The plots of y_A/x_A vs. $x_A + x_B$ and y_B/x_B vs. $x_A + x_B$, using $x_A/(x_A + x_B)$ as parameter, are presented here on a single plot for comparison purposes. See Figure 6-3.

A work order was submitted for the installation of a 4 in. Pyrex distillation column. This "laboratory column" will be used to gather data necessary for the design of the final columns. The following are conservative preliminary calculations based on a maximum allowable vapor flow rate up the columns.

Determination of vapor velocity up the column:

$$u = K \sqrt{\frac{\rho_L}{\rho_V}} \quad (\text{Chem. Engrs. Handbook, Perry})$$

where:

u = vapor velocity ft/sec

ρ = density at column conditions

K = .04 constant for column with 6" plate spacing and 1/2" liquid seal

Assuming 25 lb/lb mole and operating temp = 240°F

$$\rho_V = \frac{25}{359} \times \frac{492}{700} = .05 \text{ lb/cu ft}$$

$$\rho_L \text{ ranges } 1.17 \text{ to } 1.32$$

$$\text{Assume } 1.25 (62.4) = 78 \text{ lb/cu ft}$$

$$u = .04 \frac{78}{.05} = .04(39.5)$$

$$u = 1.58 \text{ ft/sec}$$

Figure 6-3

Vapor-Liquid Equilibrium in HCl-HNO₃-H₂O System (Based on experimental data from MIT EPS-X-258).

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This corresponds to a volumetric flow rate of:

$$1.58(60) \frac{(4\pi)}{144} = 8.3 \text{ cu.ft./min.}$$

And a mass flow rate of:

$$\frac{8.3}{359} \times \frac{492}{700} \times 25 = .41 \text{ lb/min} = \frac{.41 \times 454}{1.25} = 145 \text{ cc/min condensate}$$

Heat to be removed by condenser:

$$\text{Estimate } \Delta H_v = 900 \text{ BTU/lb.}$$

$$.41 \times 900 = 370 \text{ BTU/min.}$$

Capacity of condenser: (conservative)

$$\text{Assume } U = 60 \text{ BTU/hr.sq.ft. } ^\circ\text{F}, \Delta T_c = 240 - 120 = 120$$

$$q = UA \Delta T_c = \frac{(60)(13)(120)}{60} = 1560 \text{ BTU/min.}$$

Water required thru condenser:

$$W = \text{lb./min. required}$$

$$\Delta T_w = \text{change in bulk water temp, estimate } 40^\circ\text{F}$$

$$q = W \Delta T_w$$

$$370 = W 40$$

$$W = 9.25 \text{ lb./min.} = 1.11 \text{ gal./min.}$$

Heat to be supplied by reboiler = 370 BTU/min.

Assume condensing 100 lb. steam yields 325 F in reboiler.

Assume U through reboiler wall = 60 BTU/hr.sq.ft. $^\circ\text{F}$.

$$q = UA \Delta T \quad \Delta T = 325 - 240 = 85^\circ\text{F}$$

$$370 = \frac{(60)(A)(85)}{60}$$

$$A = 4.35 \text{ sq.ft.}$$

To cover this area in the 20 in. diameter 20 gal. reboiler only requires approximately 7 gallons. Estimating the hold-up in the column at about one gallon, the hold-up of the entire apparatus will be only 8 gallons and a complete column change can be brought about in 3.7 hours.

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Using a two-plate section of the column and air and water as the fluids, visual inspection of flow rates up to this velocity indicated that vapor-liquid contact was satisfactory throughout the range.

6.4 Material Evaluation (Corrosion Tests)

F. L. Rogers, G. A. West, G. B. Dinsmore

A corrosion testing program is in progress to evaluate materials of construction for the components of the proposed aqua regia and hydrochlorination dissolving systems.

Several power reactor fuels will be clad, or alloyed, with stainless steel or zirconium. The reprocessing of these materials involves a corrosive media that has very little user history on materials of construction. Therefore, corrosion tests are being made with boiling ($\sim 110^{\circ}\text{C}$) simulated dissolver solutions of (1) aqua regia, 2N HCl-5N HNO_3 and 4N HCl-6N HNO_3 , (2) aqua regia (4 and 6N) with ~ 100 gms/liter of 304 stainless steel, (3) aqua regia (4 and 6N) with stainless steel and ~ 15 gms/l of U and (4) anhydrous HCl gas at 500°C . The materials being evaluated for the aqua regia system are: titanium, tantalum and Pfaudler #53 glass-lined steel. Durichlor and Hastalloy "F" have been eliminated due to excessive corrosion. The materials being tested for use in the HCl system are: Inconel, monel, "A" nickel and 304-L stainless steel.

Tests with the boiling aqua regia indicates tantalum to have superior resistance. Titanium is satisfactory except in the liquid phase of aqua regia (2N HCl-5N HNO_3) where the corrosion is slightly greater than 2 mils per month when the specimen is defilmed. When not defilmed the corrosion is less than 2 mils per month (~ 1.6). The titanium in the vapor phase was satisfactory in that the average corrosion rate was approximately 0.35 mils per month. Pfaudler #53 glass had less than 0.1 mil/month over a 900 hour test period. The aqua regia, 4N HCl-6N HNO_3 , in which ~ 100 gms/liter of 304 S/S was dissolved was reduced to 1.5N acid. The corrosion in this solution over a 240 hour period was nil for tantalum and 0.01 mil per month for titanium. The aqua regia-stainless steel solution was made 1.5M in sulfate to simulate the HCl removal step in which sulfate might be added to break up the Fe-Cl complex. This does not appreciably increase the corrosion rate in that only 0.013 and 0.014 mils per month was had on tantalum and titanium, respectively over a 240 hour test period.

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The hydrochlorination corrosion test was conducted with anhydrous HCl gas in a furnace at 500°C. The corrosion over a 138 hour test period was as follows:

Inconel: 0.69 mils per month
"A" Nickel: 0.74 mils per month
Monel: Failure - Complete deterioration

6.5 Experimental Mechanical Handling Facility

F. G. Kitts

Before the chemical reprocessing of spent power reactor fuel elements can begin, certain mechanical operations such as auxiliary removal, dejacketing and shearing must be performed. A facility with simulated radiation shielding has been designed in which various machines will be evaluated and the remote operation of such machines demonstrated. Preliminary prints of the enclosure and manipulator carriers have been received and a work order is being written for their construction.

6.6 Universal Slug Loader

F. G. Kitts

A continuous dissolution process requires the charging of spent fuel to a dissolver at a specified rate. To accomplish this, slugs must be conveyed from beneath the surface of a storage reservoir to the dissolver at a constant rate with a high degree of reliability. A working model of a device to perform such functions is still being modified by Engineering and Mechanical Division. The hopper was recently lengthened and presently a new set of cams is being machined. These cams time the cycling of the apparatus by activating micro-switches in a fixed sequence.

6.7 Radiation Damage Studies

G. A. West, F. L. Rogers

The program to irradiate protective coating with gamma and neutrons and gamma alone in the Homogeneous Reactor shield tank is active in that 12 of 20 paint manufacturers participating have submitted specimens which have been placed on stringers for suspension around the shield tank.

6.8 Continuous Product Purification

The 3" ion exchange contactor for the isolation of plutonium from uranium has been completed and leak tested by pulsing resin and flowing water counter-current through the contactor. Some minor changes or modifications of component parts (primarily valves) are being made to prevent contactor leakage while plutonium is being processed. A chemical shake-down test using neptunium is planned as soon as the changes are made.

6.9 Discharging Solids from a Dissolver

F. G. Kitts

The continuous dissolution of spent power reactor fuels clad with materials insoluble in nitric acid results in a constant build-up of inert materials in the dissolution vessel. If this material is not removed, the dissolver is soon clogged and no further dissolution can take place. Methods of removing these solids are being studied at the present time.

A system utilizing a vertically mounted spiral vibratory conveyor with a central reservoir as the dissolution vessel is still under consideration. One design, probably the most promising one to date, incorporates a stationary outer housing with the spiral revolving inside. The spiral is aligned by bearings mounted on a stationary hollow cylinder extending down inside the spiral. The dissolution chamber is located within the stationary hollow cylinder (see figure 6-4). This arrangement allows the fuel and acid feed, acid drain and solids discharge lines to be attached directly to the dissolver and connected to the other components of the system with rigid pipe. It also provides accessibility to the area surrounding the reaction chamber and allows the alignment bearing to function in an acid free atmosphere.

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VIBRATORY DISSOLVER WITH SOLIDS DISCHARGE

FIGURE 6-4

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