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TRIP REPORT - SPECIAL REDOX RUNS AT
OAK RIDGE NATIONAL LABORATORIES

AUTHOR

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and

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HANFORD LABORATORIES OPERATION

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TRIP REPORT - SPECIAL REDOX RUNS AT
OAK RIDGE NATIONAL LABORATORIES

I. INTRODUCTION

The authors visited Oak Ridge National Laboratory during the period October 13, 1958 to November 26, 1958. The purpose of the trip was to supply technical assistance and to help coordinate pilot plant solvent extraction runs demonstrating the use of modified Redox flowsheets for reprocessing solutions of the type resulting from Darex or Niflex dissolution of stainless steel-clad fuels. The runs were made to obtain data on column operability, column flooding capacity, chemical compatibility and solution stability, decontamination factors, waste losses, effectiveness of feed oxidation, and flowsheet variables.

The work was performed at ORNL rather than HAPO because suitable (electrically explosion-proof) facilities and operating man power were available at ORNL. Extensive modifications to existing development facilities would have been required if the work had been done at HAPO. All costs of the program, with the exception of HAPO technical assistance and a few of the chemical requirements, were absorbed by the Chemical Technology Division at ORNL.

The program was divided into two parts. The first part consisted of determining flooding capacities and general stability characteristics of the flowsheets with non-radioactive solutions. This portion of the work was done by the Unit Operations Section of the Chemical Technology Division. The second part of the program consisted of testing the flowsheets with radioactive solutions. This work was done by the Pilot Plant Section of the Chemical Technology Division. All of the work was done in the 3503 Building at the X-10 Area.

Some details of the test program are not included in this report, but will probably be included in a summary document to be issued by ORNL personnel at a later date. Until the summary report is issued, details of the work should be solicited from the writers of this report.

It was planned that the Unit Operations Section investigation of flowsheets with cold solutions would precede the tests with radioactive solutions. This work will be discussed in Section III and the "hot" pilot plant work will be discussed in Section IV.

II. CONCLUSIONS

A number of modified Redox first cycle flowsheet conditions were demonstrated in pilot-plant-scale packed columns for the processing of Darex and Niflex type of low-acid feed solutions. On the basis of this work, several conclusions may be drawn pertaining to both (molybdenum-free) systems.

a. Column operability with feed solutions reasonably near the instability point is good.

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b. Dichromate oxidation of plutonium in feed solutions is good.

c. The HAW stream is stable for periods of minutes to several hours after leaving the column. Acidification of the HAW stream by 0.1 to 0.2 M acid before precipitation occurs will render the stream stable for several days or more.

d. High HA Column scrub flow rates are ineffective in removing fission products from the organic stream in the scrub section.

e. For the systems tested, reasonable flows of acid-deficient scrub may be used in the HA Column without reducing the acidity of the HAP stream below that in the typical Redox flowsheet. (Other effects of acid-deficient versus acidic scrub were not determined, however.)

f. Stainless steel nitrates are effective salting agents for uranium and plutonium in the Redox flowsheet.

g. No difficulties in stripping the products from the hexone stream are to be expected.

h. An analytical method for determination of free acid in solutions containing stainless steel⁽⁴⁾ must be adapted to routine use.

Conclusions concerning the processing of (molybdenum-free) Darex feeds specifically may be summarized as follows:

a. The maximum over-all gamma decontamination factors expected from the first cycle are in the range of 300 to 500.

b. The optimum HA Column flowsheets for maximum decontamination performance combined with reasonable waste losses are probably bracketed by the following conditions:

- (1) Salt concentration in waste stream 1 to 1.10 M equivalent $\text{Al}(\text{NO}_3)_3$.
- (2) Extraction section L/V 0.5 to 0.65.
- (3) Scrub section L/V about 0.1 or less.

c. The presence of appreciable quantities of silicon in the feed solution lowers significantly the flooding capacity of the HA Column. In the absence of relatively high concentrations of silicon the flooding rates are at least as high as with standard Redox flowsheets.

d. Nearly exhaustive scrubbing of the HAP stream with acid-deficient $\text{Al}(\text{NO}_3)_3$ scrub is relatively ineffective in improving decontamination.

Conclusions concerning the processing of (molybdenum-free) Niflex feeds specifically may be summarized as follows:

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a. The maximum over-all gamma decontamination factors expected from the first cycle are in the range of 100 to 200.

b. The optimum HA Column flowsheets for maximum decontamination performance combined with reasonable waste losses are probably bracketed by the following conditions:

- (1) Salt concentration in the waste stream 1 to 1.05 M equivalent $\text{Al}(\text{NO}_3)_3$.
- (2) Extraction section L/V 0.5 to 0.65.
- (3) Scrub section L/V about 0.1 or less.

c. The presence of appreciable quantities of silicon in feed solutions has no appreciable effect on the flooding capacity of the HA Column with stainless steel concentrations as high as 0.15 M in the feed solutions. (High silicon concentrations in solutions containing more than 0.15 M stainless steel were not tested.) It should be noted, however, that with existing limitations on Niflex feed solutions, the maximum amount of stainless steel in solution is about 0.4 M, compared to greater than 2.0 M for Darex solutions.

III. "COLD" FLOODING AND STEADY-STATE RUNS - UNIT OPERATIONS SECTION

A. Description of Equipment

The "cold" runs were made in two-inch-diameter glass columns by R. J. McNamee of the Unit Operations Section. The HA Column had a 22-foot extraction section and a four-foot scrub section packed with 1/4-inch diameter by 3/8-inch long (24 gage) split stainless steel Raschig rings. The top and bottom disengaging sections were made of four-inch glass pipe and were 45 and 12 inches long, respectively. The feed was introduced flush at the wall of the column with no special provision for distribution into the packing. The extractant was introduced through an inverted conical distributor buried in the packed section of the enlarged lower disengaging section, 5-1/2 inches above the packing support. Interface control was by means of dip tube differential pressure which was used to control the HAW flow through an automatic control valve.

The HC Column was a concatenated pulsed column with two 12-foot sections containing stainless steel sieve plates at two-inch spacing. The HC Columns served only to strip uranium from the hexone which was then washed with water in a six-inch pulse column prior to being recycled.

All streams were metered with positive displacement metering pumps. The actual pumping rate was checked by measuring the rate of liquid fall in calibrated titre tubes connected to each tank.

B. Description of Experimental Method

Flooding volume velocities, chemical stability (tendency to precipitate), and

uranium waste losses were investigated in the flowsheets studied by the Unit Operations Section. Flooding was determined both visually and instrumentally. The instrumental method used is one that R. J. McNamee has been investigating for some time with pulsed columns, but this was the first time it had been applied to packed columns. The method depends upon the linear increase of the volume of total organic phase in a column with increasing volume velocity. When flooding begins, the organic hold-up exceeds the linear relationship, thereby providing a sensitive method of determining the flooding capacity. In practice, the column was started on a given flow sheet and operated at a low flow rate until steady state was attained (two to three hours) as determined by HAP density measurements. At that time, all flows to and from the HA Column were shut off and all the organic in the column allowed to accumulate in the upper disengaging section. The downward displacement of the interface then afforded a measurement of the organic hold-up in the column. The column was pulsed after the column was shut down to hasten accumulation of the organic phase (which usually required about 10 minutes). After the measurement was made, the column was restarted at a higher flow rate, operated 30 minutes, and shut down for another organic hold-up measurement. This procedure was repeated until a non-linear increase in organic hold-up indicated the flooding point had been exceeded. As a further check, the column was operated one hour at the previously tested rate just below the indicated flooding rate, since incipient flooding might not be detected in the shorter run.

After the flooding volume velocity was determined, a steady-state run of about eight hours' duration was usually made at 80 percent of flooding to determine uranium waste losses and general operability of the flowsheet.

C. Solution Sources and Make-Up

Solutions were prepared from commercial salts with the exception of aluminum nitrate used in the upper scrub stream, uranium, and stainless steel. Uranium was obtained from the ORNL Metal Recovery Plant as an approximately 1.5 M $\text{UO}_2(\text{NO}_3)_2$ solution (usually containing about 0.5 M HNO_3). The stainless steel solution was obtained from pilot plant Darex dissolver runs in the 321 Building at Hanford as a nitrate solution of 3.0 M stainless steel, 0.11 M Cl^- , and about 0.3 M HNO_3 . The $\text{Al}(\text{NO}_3)_3$ used in the upper scrub stream for most of the runs was obtained from Hanford Plant solution.

No facilities were available for concentrating or refluxing solutions. Consequently, no attempt was made to simulate head-end treatment of the feed in these runs.

D. Data and Results

The results of the flooding and steady-state runs are summarized in Table I. The first run made was with a typical Redox flowsheet⁽²⁾ which was used as a comparison basis for subsequent runs. Actually, the true flooding points were known only to lie between two values which differed by about 10 percent. In all cases the reported flooding rate was chosen as the mean of the last non-flooding and the first flooding flow rate tested, so that the reported

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flooding rate is within five percent of the true flooding rate. All values are shown in the table with the exception of Run 3a where testing was discontinued before the flooding volume velocity was reached.

The first two Darex (Runs 3b and 3a) and the single Niflex (Run 8a) flowsheets tested flooded at volume velocities comparable to, or slightly higher than, the typical Redox flow sheet. Feeds for these runs contained relatively low concentrations of silicon. One Darex run (Run 3c) was made with 0.013 M Si in the feed which corresponds to about 0.7 weight percent of the stainless steel present. This run was made when it was reported⁽¹⁾ that essentially all of the silicon present in stainless steel will remain in solution during Darex feed solution preparation. The increased silicon concentration (added as sodium silicate) decreased the flooding volume velocity by about 40 percent.

Solution stability did not prove to be a serious problem when proper precautions were taken. Solids were formed in the HAW streams from both Darex and Niflex runs usually in a matter of hours (or less) after leaving the Column. In all cases, however, addition of concentrated HNO₃ to increase the acidity by 0.1 M delayed precipitation from several hours to several days and an increase in acidity of 0.2 M prevented precipitation for at least several days. It should be noted that after the precipitates once formed, much more acid was required to re-dissolve them than was originally required to prevent precipitation.

On several occasions the columns were shut down overnight without stripping, draining, or flushing. Precipitates invariably formed in the bottom portion of the HA Column. Attempts to dissolve the precipitate with dilute acid were ineffective. The removal technique usually used was to drain about two feet of liquid from the column and refill with water through a tee in the HAW line. Repeating this procedure three to four times removed the solids completely. On this basis, simple velocity-type water flushes should provide adequate removal of these precipitates.

It became standard practice to fill the HA Column with scrub acidified to 0.2 M acid (even when acid-deficient scrub was used during the run) prior to start-up of a Darex or Niflex flowsheet. This technique prevented the precipitation which occurred when the feed first entered the column filled with acid-deficient scrub.

Stability of Darex and Niflex feed solutions is primarily a function of acidity (for the solutions used in these runs), and the acidity was not known precisely. A reliable and time-proven method for analysis of free acid in these solutions was not available at the time the runs were performed. The nominal acidities given in Table I are based upon pH correlation, acidity contributions of individual components, and to some extent upon actual analyses. In eight-hour steady-state runs with the typical Redox flowsheet and with Darex Flowsheets 3b and 3c (the only three steady-state runs made), very low uranium waste losses to the HAW stream were obtained. In both Darex flowsheets the losses were slightly less than in the standardizing (typical Redox) run, which indicates that the Darex flowsheets were conservative on the basis of waste loss considerations.

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E. Future Work

The unit Operations Section plans to continue several more flooding and operability tests in the two-inch glass columns during the next few weeks. The planning of these runs will be done in close cooperation with HAPO personnel.

IV. "HOT" PILOT PLANT STUDIES - PILOT PLANT SECTION

A. Description of Equipment and Facilities

The radioactively "hot" solvent extraction runs were performed by the Metal Recovery Pilot Plant group headed by R. E. Brooksbank and assisted technically by C. D. Hylton. The work was done in the "Interim-23" Pilot Plant by a three-man/shift operating crew on an around-the-clock schedule.

The "hot" equipment consists basically of a dissolver vessel, a feed adjustment tank, a feed tank, a compound extraction-scrub column, a simple strip column, waste handling tanks, a batch-type organic still, and necessary auxiliaries. The "cold" auxiliary equipment consists of several chemical make-up and chemical addition tanks. A schematic flow diagram of the equipment used (some available equipment was not needed) is shown in Fig. I. All equipment is constructed of 347 or 309 SCb stainless steel. The "hot" equipment is located in three cells, each with two feet of concrete shielding. (An additional four inches of lead shielding is placed around tanks containing feed and "hot" waste solution.)

Both solvent extraction columns are constructed of two-inch stainless steel pipe and packed with the same stainless steel Raschig rings used in the "cold" work of the Unit Operations Section. The packed heights of the HA Column scrub section, HA Column extraction section and HC Column are 21, 17, and 22 feet, respectively. Both columns have upper disengaging sections of six-inch pipe and lower disengaging sections of four-inch pipe. The interface locations were measured by dip tubes in the upper disengaging sections. The interface controllers controlled air-operated valves in the aqueous effluent stream lines of each column.

The vessels within the cells are 20 to 100 gallons in size. All feed make-up, adjustments, sampling, and concentration was performed in the feed make-up tank. The organic still (which was operated batch-wise) consists of a simple vessel equipped with a steam sparger.

Aside from the extraction column interface control instrumentation described above, the only additional "hot" instrumentation was various combinations of liquid level, specific gravity, and temperature measurements on most of the vessels. All "hot" stream flow rates were determined by build-up or depletion rates in the respective vessels. All non-radioactive stream flow rates were determined by depletion rates from calibrated titre tubes. The HAF stream was moved by a remote-head diaphragm pump; all cold streams were pumped by piston-type metering pumps.

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B. Solution Sources and Make-Up

Radioactivity in the runs was provided by aluminum-jacketed natural uranium slugs irradiated in the Oak Ridge graphite reactor to exposures of 100 to 300 MWD/ton at rates of 0.07 to 0.15 MW/ton and cooled four to eight months. The jacketed slugs were charged to the pilot plant dissolver, de-jacketed with NaOH-NaNO₃ solution, and dissolved by the semi-continuous technique used in the Redox Plant. With the exception of the standardizing run, the last Darex run and the last Niflex run, all runs were made with no dilution of the radioactivity by non-radioactive uranium solution. The small amount of "cold" uranium solution used was product solution from the ORNL Metal Recovery Pilot Plant.

All upper scrub streams (except those in the last few runs) were made up from Hanford stock 2.5 M Al(NO₃)₃ solution. All hexone (except a small amount in the later runs) was Hanford unused hexone. All hexone was pre-treated by steam distillation before use and was retreated by steam distillation after use and before being recycled to the solvent extraction system. Stainless steel was made up synthetically; all other constituents in the feed and scrub streams were supplied by bulk chemicals available at ORNL.

To provide for plutonium oxidation, all Darex and Niflex feed batches were made 0.2 M in Na₂Cr₂O₇ and boiled three to four hours. Similarly, feeds for standard runs were made 0.2 M in Na₂Cr₂O₇ and boiled one hour under acidic conditions and three hours under acid-deficient conditions.

Feed acidity measurement of the Darex and Niflex solutions was primarily by calculation of the summation of acidities of the individual constituents. These acidities were checked by the pH correlation data of the Development and Corrosion Chemistry Operation at HAPO. The feed solutions were generally about 0.1 to 0.2 M more acidic than required for borderline stability control.

Acidity control and measurement of the "cold" streams, the dissolver solutions, the HAP and HCU streams, and the feed for the standardizing runs were by actual analyses.

C. General Run Description

Eight Darex, seven Niflex, and one typical Redox⁽²⁾ flowsheets containing radioactivity in the feed solutions were tested. The length of each run was generally between 12 and 24 hours after the feed was up to nominal rate. The initial runs were performed at 75 percent of flooding rates indicated by the "cold" work of the Unit Operations Section. Later runs were performed generally at rates of 60 to 70 percent of flooding to permit more time for leveling out the equipment operation. The rates used resulted in about one aqueous throughput per hour in the HA Column extraction section and about one aqueous throughput per two to five hours in the scrub section. Because of the limiting size of the feed make-up tank, no more than one run for each feed batch could be satisfactorily performed and evaluated. The runs were performed in four series of three to five runs in each series. A several-day period between each series was required for flushing and preparation for subsequent runs.

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All feed solutions were transferred to the unheated and unagitated feed tank at temperatures of 60 to 80 C. The amount of temperature lowering due to heat losses from the feed tank during the progress of each run is unknown.

D. Data and Results

1. General

Prior to the performance of the radioactively "hot" runs, the equipment was operated with radioactivity-free feed on a typical Redox flowsheet⁽²⁾ for several days. This run served as a shake-down run for the equipment and an orientation run for the operators. Operating data on this run will not be reported here. Another standardizing flowsheet run was made which contained radioactivity. The results of this run were used to supply a comparison basis for the "hot" performance of the pilot plant equipment.

Generally speaking, the operation of the equipment was quite good. Sharp changes in performance with changes in run conditions were observed in most cases. Steady-state conditions were achieved within the first eight hours or less in all runs, as indicated by sample data. Consequently, it is believed that the data should be representative.

The data presented here are based on laboratory analyses of the samples from four flowing streams taken every two to four hours after steady-state conditions were reached. Early sample data during each run were used to determine when steady-state conditions had been obtained. Because of the large sample load and the small laboratory crew available during most of the test runs, the laboratory analyses were generally delayed considerably. These delays prevented making major changes in scheduled run plans for several days.

The stability data of the various solutions are very rough. The stability data consist of periodic observations of the samples (by the writers) in which the samples were inverted and inspected for the presence of solids in the bottom of the sample bottle.

At the time of this writing, the only radio-chemical analyses of the major sources of gamma activity in the dissolver solutions are the following:

<u>Dissolver Sample</u>	<u>Gross Gamma</u> <u>c/m/ml</u>	<u>Ru Gamma</u> <u>c/m/ml</u>
MD-3	3.17×10^8	9.05×10^7
MD-7	4.60×10^8	9.84×10^7

Considerably more radiochemical analytical data have been requested.

2. Darex Runs

The early Darex runs were originally established to approximate standard

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Redox HA and HC Column flowsheets. The scrub flow was increased (to greater than that used in the typical Redox HA Column flowsheet) in an attempt to improve decontamination. The organic flow rate was reduced slightly and the salting strength in the extraction section was kept essentially unchanged (assuming stainless steel nitrate is 80 to 90 percent as effective a salt as is aluminum nitrate). As the test runs progressed, the HA Column extraction section salting strength was reduced; the extraction L/V was increased, then reduced; acid-deficient scrub was used; and the salting strength of the scrub was reduced. No testing of molybdenum-containing Darex feeds was performed. A summary of the nominal run conditions and the more important results is presented in Table II. For comparison, plant data of November 1956(3) are also presented.

A detailed discussion of the data will not be presented in this report. However, some of the more important points to be observed are:

- a. Decontamination factors as affected by extraction section salt concentration.
- b. Decontamination factors as affected by scrub section L/V.
- c. HA Column waste losses as affected by extraction section salt concentration and L/V.
- d. HA Column plutonium waste losses as an indication of oxidation effectiveness.
- e. Stability of the HAW stream.
- f. Acidity of the HAP stream.
- g. Comparison of pilot plant run to plant run.

3. Niflex Runs

The early Niflex runs were established using the same general bases as those used for the early Darex runs, with the exception that the aluminum equivalent of the fluoride present was assumed to be ineffective as a salting agent. On the basis of the apparently low losses encountered during some of the preceding Darex runs, the extractant flow rate was reduced considerably. Also, on the basis of the relatively short stability time of the Darex HAW stream, the HAX stream was acidified slightly in an attempt to improve HAW stability. As the runs progressed, the salting strength in the HA Column extraction section was reduced significantly, the extraction section L/V was reduced, and the extractant was not acidified.

The compositions of the feed solutions were based on the following premises:

- a. Five moles of fluoride ion per mole of stainless steel are required during a Niflex dissolution.

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b. One mole of aluminum per mole of fluoride ion is required for satisfactory inhibition of corrosion.

c. Ammonium fluoride and nitric acid are the reagents used in a Niflex dissolution.

Only one Niflex feed solution contained molybdenum. This feed contained 0.009 M molybdenum, which was the maximum solubility of molybdenum in this solution as determined by the Unit Operations Section.

A summary of the nominal flowsheets tested and the more important results are presented in Table III. As in the case of the Darex runs, a detailed discussion of the results will not be presented here.

4. Additional Information

To determine the effect of scrubbing the HAP stream with acid-deficient $\text{Al}(\text{NO}_3)_3$ solutions, seven HAP samples from Darex Runs 3a, 3b-1, 3b-2 and 3c were each contacted batch-wise in the laboratory with three successive equal-volume portions of scrub of the following composition:

1.19 M $\text{Al}(\text{NO}_3)_3$, -0.16 M HNO_3 , 0.07 M $\text{UO}_2(\text{NO}_3)_2$, 0.01 M $\text{Na}_2\text{Cr}_2\text{O}_7$.

The gamma decontamination factor due to the batch scrubbing ranged from 1 to 12 and averaged about 5.

An attempt to determine the decontamination expected from a second cycle operated on a standard type of Redox flowsheet was performed in the laboratory. Special HCU samples (from some of the later Niflex and Darex runs) were boiled down with the addition of $\text{Al}(\text{NO}_3)_3$, NaOH , $\text{Na}_2\text{Cr}_2\text{O}_7$ to a final composition approximating that of the present Redox Plant IAFS solution. This newly formed IAFS solution was then contacted with one equal volume of hexone; the contacted organic was decanted, then contacted with an equal volume of scrub of the following composition:

1.3 M $\text{Al}(\text{NO}_3)_3$, 0 M HNO_3 , 0.2 M $\text{UO}_2(\text{NO}_3)_2$, 0.01 M $\text{Na}_2\text{Cr}_2\text{O}_7$.

The results of this work are not yet available.

J. E. Mendel
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- (1) Personal communication, J. J. Shefcik and K. J. Schneider, November 10, 1958.
 - (2) Letter, M. K. Harmon to G. L. O'Neill, "Standard Redox Flowsheets for ORNL Test Runs on Non-Production Fuels," August 21, 1958.
 - (3) HW-47907, "Dichromate Oxidation of Redox Feed Solution: Current Status of Technology," by K. L. Adler, January 21, 1957.
 - (4) HW-58004, "Determination of Free Acid in the Presence of Stainless Steel," by K. Koyama, October 29, 1958.
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TABLE I

UNIT OPERATIONS FLOODING AND STEADY-STATE TESTS

Feed Composition M	Run Numbers				
	1a(1)	3b	3a	8a	3e
UO ₂ (NO ₃) ₂	1.24	0.6	0.6	0.5	0.45
SS(NO ₃) ₂ .9	-	1.2	1.2	0.25	0.90
HNO ₃	-0.2	-0.2	-0.2	+0.4	-0.3
Al(NO ₃) ₃	1.11	-	-	0.84	-
NaNO ₃	0.33	1.5	1.5	0.25	0.9
AlF ₃	-	-	-	0.42	-
NH ₄ NO ₃	-	-	-	1.25	-
Na ₂ SiO ₃	-	0.0008	0.0008	0.0013	0.013
Flow Ratio(5)					
HAS(2)	12.5	84	42	40	67
HAFS	81.5	167	167	200	222
HAX(3)	188	420	330	370	457
Run Conditions					
Scrub L/V(7)	0.07	0.20	0.13	0.11	0.15
Extraction L/V(7)	0.50	0.60	0.63	0.65	0.63
M Ann equivalent in HAW(4)	1.36	1.43	1.46	1.59	1.15
Run Results					
Volume	No flood	545	555	640	665
Velocity (6)(gal/hr-ft ²)	Flood	615	635	-	725
	Assumed Flood pt.	585	600	640	690
		0.0035	0.0018	-	0.0028
Percent U loss HA Column					

Notes:

- (1) HAFS combined with HAFS in feed tank.
- (2) HAS composition was 1.3 M ANN, 0.01 M Na₂Cr₂O₇ in all cases; HNO₃ was 0.2 M in Runs 1a, 3b, and 3a; HNO₃ was -0.15 M in Runs 8a and 3e.
- (3) Neutral hexone in all runs.
- (4) Equivalent Al(NO₃)₃ salt strength is calculated by Al M + SS M + 1/6(NaNO₃ M + NH₄NO₃ M).
- (5) Flow ratios are relative to HAFS flow. HAFS flow ratio is 100/U M in HAFS.
- (6) Extraction section volume velocity is based on rates of influent streams.
- (7) L/V numbers are based on influent flow rates.

TABLE II. "HOT" DAREX RUN SUMMARY
Run Numbers

Stream	Components, M, etc.	Typical Redox		3a	3b-1	3b-2	3c	3e	3f	3g-1 (5)	3g-2 (6)
		Plant (8)	1a (9)								
HAS	Al(NO ₃) ₃	1.3	1.3	1.3	1.3	1.3	1.3	1.3	1.3	1.0	1.0
	HNO ₃	-0.2	+0.2	+0.2	+0.2	+0.2	+0.2	-0.15	-0.15	-0.12	-0.12
	Na ₂ Cr ₂ O ₇	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.008	0.008
	Flow ratio (1)	12.5	12.5	42	84	84	84	67	400	20	20
HAFS	UO ₂ (NO ₃) ₂	2.0	1.87	0.6	0.6	0.6	0.6	0.45	0.2	0.8	0.8
	SS(NO ₃) _{2.9}	-	-	1.2	1.2	1.2	1.2	0.9	0.4	0.4	0.4
	HNO ₃	-0.3	-0.2	-0.3	-0.17	-0.3	+0.5	-0.3	+0.05	0.0	0.0
	Al(NO ₃) ₃	-	0.34	-	-	-	-	-	0.45	0.34	0.34
	NaNO ₃	~0.3	0.56	1.2	1.2	1.2	1.2	0.9	0.4	1.27	1.27
	Na ₂ Cr ₂ O ₇	0.15	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
	Na ₂ SiO ₃	-	-	0.001	0.001	0.001	0.001	0.0007	0.0003	0.0047	0.0047
	Flow ratio (1)	50	53.5	167	167	167	167	222	500	125	125
"Neutral" Hexone											
HAX	HNO ₃	188	188	330	420	420	420	361	1030	193	230
Demineralized Water											
HGX	HNO ₃	65	65	115	147	147	147	127	355	66.7	66.7
Run Condition Summary											
HA Scrub L/V (2)		0.067	0.067	0.13	0.20	0.20	0.20	0.19	0.39	0.10	0.087
HA Extraction L/V (2)		0.57	0.50	0.63	0.60	0.60	0.60	0.80 (7)	0.87	0.75	0.63
HA Extraction eq. Al M (3)		1.33	1.36	1.46	1.44	1.44	1.44	1.16	1.10	1.05	1.05
V.V. (4) gal/hr x ft ²		-	360	450	450	450	450	350	400	294	235
Results											
HAW Loss, U%		~0.02	0.04	<0.002	0.001	0.0005	0.003	0.25	0.02	11.4	0.33
HAW Loss, Pu%		~0.10	0.05	0.01	0.03	0.006	<0.008	1.60	<0.02	9.3	0.14
Gross ⌵ DF-HAP		-	1800	70 (10)	40 (10)	40	30	220	220	~400	-
Gross ⌵ DF-HCU		2700	2300	85	60 (est)	60	55	280	230	~450	-
HAP HNO ₃ M		~0	0.05	0.16	0.15	0.19	0.39	0.36	0.03	0.13	-
HAW Stability, hr.		-	-	~12	~24	>16	2-30	<3	>72	>3	<23
HAW pH 5:1 dilution		-	-	2.0	2.15	2.02	2.06	1.76	1.93	1.9	-
HCW Loss, U%		-	0.014	0.005	0.005	0.003	0.003	0.02	0.003	0.004	-
HCW Loss, Pu%		-	<0.006	0.007	<0.008	<0.015	<0.01	<0.007	<0.011	<0.003	-

NOTES:

- (1) Flow ratios are relative to HAFS flow. HAFS flow ratio is 100/U M in HAFS.
- (2) L/V numbers are based on influent flow rates.
- (3) Equivalent Al(NO₃)₃ salting strength was calculated by: Al M + SS M + 1/6(NaNC₃ M).
- (4) Extraction section volume velocity based on rates of influent streams.
- (5) This run was flooding a good share of the time.
- (6) This run was only 2 hours' long, and the data are indicative only.
- (7) The feed rate during this run was about 10 percent high; the actual L/V should be about 0.85.
- (8) Not shown is the HAFS stream which is 44.5 flows of 2.4 M Al(NO₃)₃, -0.10 M HNO₃.
- (9) Not shown is the HAFS stream which is 28 flows of 2.5 M Al(NO₃)₃, -0.16 M HNO₃, 0.045 M UO₂(NO₃)₂, 0.33 M NaNO₃ and 0.022 M Na₂Cr₂O₇.
- (10) Radiochemical analyses on one sample indicate the activity is predominately Ru activity.

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TABLE III. "HOT" NIFLEX RUN SUMMARY
Run Numbers.

Stream	Components, M etc.	Typical Redox		Niflex						
		Plant (5)	1a (6)	8a	8b	8e-1	8e-2	8f	8g	8h-2
HAS	Al(NO ₃) ₃	1.3	1.3	1.3	1.3	1.3	1.3	1.3	1.3	1.0
	HNO ₃	-0.2	+0.2	-0.15	-0.15	-0.15	-0.15	-0.15	-0.15	-0.12
	Na ₂ Cr ₂ O ₇	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.008
	Flow ratio (1)	12.5	12.5	40	80	80	160	60	300	55
HAFS	UO ₂ (NO ₃) ₂	2.0	1.87	0.5	0.5	0.25	0.25	0.5	0.16	0.3
	SS(NO ₃) _{2.9}	-	-	0.25	0.25	0.25	0.25	0.25	0.16	0.15
	HNO ₃	-0.3	-0.2	+0.3	+0.3	+0.4	+0.4	+0.3	+0.2	+0.2
	Al(NO ₃) ₃	-	0.34	0.84	0.84	0.84	0.84	0.84	0.8	0.75
	NaNO ₃	~0.3	0.56	0.38	0.25	0.29	0.25	0.25	0.16	0.15
	NH ₄ NO ₃	-	-	1.12	1.25	1.21	1.25	1.25	0.8	0.75
	AlF ₃	-	-	0.42	0.42	0.42	0.42	0.42	0.8(7)	0.75(7)
	Na ₂ Cr ₂ O ₇	0.15	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
	Na ₂ SiO ₃	-	-	0.001	0.001	0.001	0.001	0.001	0.0005	0.0018
	Mo(8)	-	-	-	-	-	-	-	-	0.009(8)
	Flow ratio (1)	50	53.5	200	200	400	400	200	625	333
HAX	HNO ₃	-	-	0.2	0.1-0.2	0.2	0.1	-	-	-
	Flow ratio (1)	188	188	300	269	537	537	300	1050	555
HGX	HNO ₃	-	-	Demineralized Water						
	Flow ratio (1)	65	65	86	86	172	172	86	365	188
Run Condition Summary										
HA Scrub L/V(2)		0.067	0.067	0.13	0.30	0.15	0.30	0.20	0.29	0.10
HA Extractor L/V(2)		0.57	0.50	0.80	1.04	0.89	1.04	0.87	0.88	0.70
HA Extraction eq. Al M(3)		1.33	1.36	1.62	1.58	1.62	1.58	1.60	1.20	1.08
V.V.(4) gal/hr x ft ²		-	360	406	412	383	412	420	362	413
Results										
HAW Loss, U%		~0.02	0.04	0.009	0.002	0.01	0.004	0.001	0.12	0.44
HAW Loss, Pu%		~0.10	0.05	0.01	0.02	0.03	<0.01	<0.01	0.71	0.17
Gross % DF-HAP		-	1800	20	45	30	33	33	60	160
Gross % DF-HCU		2700	2300	35	50	40	40	40	70	180
HAP HNO ₃ M		~0	0.05	0.28	0.18	0.40	0.43	0.23	0.09	0.08
HAW Stability, hr.		-	-	<7	<9	<8	<4	<1	<1	<8
HAW pH 5:1 dilution		-	-	1.4	1.45	1.75	1.8	1.95	2.04	2.05
HCU Loss U%		-	0.014	0.015	0.006	0.025	0.007	0.006	0.014	0.0026
HCU Loss Pu %		-	<0.006	0.017	0.013	0.028	<0.036	0.035	<0.019	<0.01

- Notes: (1) Flow ratios are relative to HAFS flow. HAFS flow ratio is 100/U M in HAFS.
 (2) L/V numbers are based on influent flow rates.
 (3) Equivalent Al(NO₃)₃ salting strength was calculated by: Al M + SS M + 1/6(NaNO₃ M + NH₄NO₃ M).
 (4) Extraction section volume velocity based on rates of influent streams.
 (5) Not shown is the HAFS stream which is 44.5 flows of 2.4 M Al(NO₃)₃, -0.10 M HNO₃.
 (6) Not shown is the HAFS stream which is 28 flows of 2.5 M Al(NO₃)₃, -0.16 M HNO₃, 0.45 M UO₂(NO₃)₂, 0.33 M NaNO₃ and 0.022 M Na₂Cr₂O₇.
 (7) In these two runs the fluoride ion was added as HF + NaOH and all aluminum was added as Al(NO₃)₃.
 (8) Molybdenum was added as ammonium molybdate solution. The amount of molybdenum added was based on the single solubility test of the Unit Operations Section.

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FIGURE I
SCHEMATIC FLOW DIAGRAM
"HOT" FACILITY