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CHEMICAL TECHNOLOGY DIVISION
UNIT OPERATIONS SECTION
MONTHLY PROGRESS REPORT
OCTOBER 1958

Physical Chemistry

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UNIT OPERATIONS SECTION MONTHLY PROGRESS REPORT

OCTOBER 1958

CHEMICAL TECHNOLOGY DIVISION

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Date Issued

DEC 19 1958

OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee
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UNION CARBIDE CORPORATION
for the
U. S. Atomic Energy Commission

ABSTRACT

Tungsten and graphite are unsuitable materials of construction for a UF_6 inlet nozzle in a continuous DRUHM reactor. Preparation of feed was completed for an extended Fluorox test. Difficulties have been experienced in the operation of a fluidized bed ThNT denitrator. Flame denitration of UNH and ThNO has produced mixed oxides of 1 to 14 micron mean particle size. The chloride capacity of Dowex 21K was measured, and equilibria measurements of uranium sorption from sulfate solutions were continued. Siliceous deposits in the stripping column caused the termination of a Darex run with a prototype APPR fuel element. Hot runs were begun on the chemical dejacketing of irradiated FWR blanket pins. The addition of formaldehyde to neutralized "25" waste reduced the recovery of nitrate from the calciner off-gas from 76% to 23%.

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SUMMARY

1.0 DRUHM

Two continuous reaction runs were made in which a tungsten-tipped and a graphite-tipped UF_6 nozzle were used. Both tungsten and graphite proved unsuitable as nozzle materials. Cooled metallic nozzles are being developed.

2.0 FLUOROX

One hundred ninety-two lb of sized UF_4 feed material was prepared for an extended operation of the Fluorox fluidized bed reactor.

3.0 HR FUEL AND BLANKET PREPARATION AND PROCESSING

Feeding of $Th(NO_3)_4$ solution at 4 ml/min to a fluidized ThO_2 bed at $600^\circ C$ resulted in bed bogging or caking. Alterations in the gas distributor, feed nozzle, and feed solution were tested in glass equipment and will be applied to the high temperature reactor to eliminate this problem. Cylindrical hydroclones of 0.4 in. dia effectively collected ThO_2 into induced underflow receivers although maximum apparent efficiencies were only 40% compared to 60% for conical 0.4 in. dia hydroclones. Flame denitration of CH_3OH solutions of $Th(NO_3)_4-UO_2(NO_3)_3$ has been applied to produce mixed U-Th oxides of 1.1 to 13.5 micron mean diameter and 4.6 to 7.8 sq. m/g surface area with varied operating conditions. Reflectors of MgO are usable up to $1600^\circ C$; $C_3H_8-O_2$ flames are easier to handle than $C_2H_2-O_2$ flames and easily reach $1600^\circ C$. Results of toroid tests will permit selection of an optimum operating temperature.

4.0 ION EXCHANGE

The chloride capacity of Dowex 21K was determined to be 4.6 ± 0.2 meq/g of dry chloride resin, an average value obtained from five resin samples. Each sample was converted to the chloride form and then eluted with a sodium nitrate solution which was subsequently analyzed for its total chloride content. Further studies of equilibrium sorption of uranium from sulfate solutions were made, but difficulties in the analytical procedures have delayed the reporting of those results.

5.0 POWER REACTOR FUEL REPROCESSING

5.1 Darex

An 11-1/2 hr run was made with the complete integrated loop equipment being used to process a prototype APPR fuel element (~ 90% SS, ~ 7%

U and ~ 2% Si). The liquid hold-up in the stripping column (previously cleaned with hot NaOH solution) increased steadily; the upper one-third was flooded when the run was stopped. The siliceous deposits were first observed in the downcomers but were soon evident on all exposed surfaces. It is doubtful that a bubble-cap column similar to the 4-in. UNOP apparatus could be adapted to the processing of high silicon fuels. Samples of the solid material taken from the NaOH wash solution showed appreciable amounts of Si, Fe, Cr, Ni, U, and acid insoluble materials.

A 1-in. I.D. glass dissolution apparatus was completed and operated in a series of runs to demonstrate the operation of a long, vertical, cylindrical dissolver on a semi-continuous basis, and to determine the feasibility of maintaining a liquid level in such a dissolver with an airlift. In the final run, three stainless steel multiplate assemblies (0.6"x0.6"x24", weighing ~ 575 g each) were completely dissolved in 65 min, requiring 21 liters of aqua regia. From this study, it was concluded that: (1) the dissolvent should be fed into the bottom of the dissolver and the dissolution product removed through a side-arm take-off; (2) an airlift could not regulate the level in the dissolver since the reaction is quite violent, but it could be used to maintain a level in the side-arm; (3) there is a definite "dissolution zone" at the bottom of the elements; (4) there is apparently no advantage in recycling any portion of the dissolver product or the dissolution condensate.

A 6-in. I. D. glass dissolver, similar to the 1-in. model, was constructed for further testing with prototype APPR elements.

A 2-liter batch feed adjustment run was made in which detailed data on compositions, volumes and densities were obtained; Cl^- removal was to 400 ppm. A semi-continuous flowsheet was developed which has the advantages of permitting the use of a smaller feed adjustment tank, and effecting a saving in time by allowing overlapping of the dissolution and Cl^- removal steps. The two runs to date have yielded Cl^- concentrations in the solvent extraction feed of 20 ppm and 140 ppm.

5.2 PWR Decladding and Core Dissolution

Final cold runs were made on oxide coated pins; uranium loss was 0.1%. Hot runs were begun on PWR blanket pins irradiated to 250 and 500 Mwd/ton.

5.3 Solvent Extraction Studies

Expanded metal shields were installed around the extraction columns to prevent breakage and spray nozzles were installed to reduce fire hazard. The bulk chemicals were received, and the make-up of synthetic solutions for the Hanford assistance program Darex runs was begun.

5.4 Feed Clarification

A problem statement was written describing proposed experimental study of the problem of removing particulates (silica, undissolved UO_2 and UO_2 - ThO_2 fires) from Darex solutions and Sulfex decladding solutions.

6.0 WASTE PROCESSING

When neutralized "25" waste was reduced to dryness by heating, 76% of the nitrate ion was recovered in the condenser and off-gas equipment. When formaldehyde was added to the neutralized waste, only 23% of the nitrate ion was recovered. When the nitrate vapor was passed through a hot copper bed (400°C) only 27% of the nitrate ion was recovered for recycle.

When the system gas leakage in radioactive drying tests was decreased by a factor of 20, the percentage of gamma activity in the condenser and scrubbers was decreased by a factor of 1000 (from 1.0% to 0.001%).

1.0 DRUHM

C. D. Scott and W. G. Sisson

The direct reduction of uranium hexafluoride to uranium metal by use of sodium ($\text{UF}_6 + 6\text{Na} = \text{U} + 6\text{NaF}$) is being investigated. A continuous reaction study is being made with primary emphasis being placed on equipment development.

1.1 Experimental Work

Two experimental runs, D-13 and D-14, were made in which the reactants were introduced to a graphite-lined reactor by separate nozzles.

Run D-13. Run D-13 was made in the 6 in. reactor (reactor similar to that shown in Unit Operations July Monthly Report, ORNL CF No. 58-7-126) and a tungsten-tipped UF_6 nozzle was used. The run lasted for 15 min with average reactant flow rates of 96.7 g UF_6 /min and 56.6 g sodium/min. Run termination was due to reduction in the UF_6 flow rate which was caused by depletion of the UF_6 supply in the vaporizing cylinder.

Approximately 7 min of the operation was at a semi-steady state with measured reaction zone temperatures greater than the melting point of uranium.

One and one-half inches of the 2 in. tungsten tip was lost during the reduction. The loss of tungsten was due to UF_6 corrosion rather than to melting since no tungsten metal was found in the reaction products.

All of the reaction products were found in the bottom of the liner and 278 g of consolidated metal was recovered as metal flakes contained in the fused NaF slag. The maximum purity of the metal was 99.6% uranium with an average purity of 98.4%. The major contaminant was included slag and there was no tungsten found in the metal. The over-all recovery was 27.9% of the fed uranium as consolidated metal.

Run D-14. Run D-14 was made in a reactor system similar to that used in run D-13 except that no MgO sand was used between liner and reactor walls and a graphite-tipped UF_6 nozzle was used.

Average reactant flow rates were 142 g UF_6 /min and 65.7 g Na/min. The run was terminated after 9.5 min because a $3/8$ in. hole was burned in the side of the reactor. The hole was caused when the graphite tip on the UF_6 nozzle was consumed and the UF_6 stream was directed against the reactor wall.

Reaction products were deposited both inside and outside the reactor liner and a large amount of uranium metal was found alloyed with the Inconel metal in the bottom of the reactor. No attempt was made to evaluate this

run by chemical analysis.

It is felt that the loss of the graphite tip from the UF_6 nozzle was due to erosion and sodium penetration rather than to high temperatures or chemical reactions.

1.2 UF_6 Nozzle Development

The greatest problem in the development of equipment for the continuous reduction of UF_6 with sodium has been the design of a suitable UF_6 entrance nozzle. Two major factors which create this problem are the high temperature in the reaction zone and the corrosive action of UF_6 .

This design problem has been attacked in three different ways: (1) use of a corrosion and high temperature resistant material of construction for the nozzle; (2) use of an inert buffer gas around the UF_6 nozzle; and (3) use of a cooling media for the nozzle to reduce the nozzle temperature, thus reducing the high temperature and corrosion effects.

Several different resistant materials of construction have been tested. These have included Inconel, tungsten, graphite, MgO , and Lavite. It was found that in the absence of cooling, these materials would not resist UF_6 in the reactor environment.

Several attempts were made to use dry He as a buffering gas around an Inconel UF_6 nozzle. This buffering agent would both cool and protect the nozzle from corrosion. It was found that the nozzle could be protected in this way if the He velocity at the UF_6 tip was equal to the UF_6 velocity through the nozzle; however, the addition of the inert gas made it more difficult to consolidate the uranium metal into massive metal and the off-gas handling problem was made more difficult.

The use of a cooled UF_6 nozzle is also being considered. An attempt was made to cool the nozzle by increasing the UF_6 velocity through the nozzle to about 150 ft/sec; however, this proved ineffective. In another run an attempt was made to move the nozzle outside the proposed reaction zone and jet the UF_6 in through a port, thus keeping the nozzle outside of the hot reaction zone. This was also found ineffective since the sodium vapor escaped the reaction zone after high temperatures were reached and again caused reaction at the nozzle tip.

Future work will be directed towards more positive cooling of the UF_6 nozzle. Three possible cooled nozzles are shown in Figure 1.1. They include (1) a massive Inconel nozzle which will be able to remove heat rapidly from the nozzle tip by conduction back to the top flange, (2) a massive metal nozzle with internal cooling by the reactant sodium, and (3) an internally gas cooled nozzle.

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Fig. 1.1. Possible UF_6 Entrance Nozzles for the DRUHM Continuous Reactor.

2.0 FLUOROX

C. D. Scott, G. K. Ellis

One hundred ninety-two pounds of UF_4 were prepared for the Fluorox fluidized bed reactor by compacting UF_4 fines in a Chilsonator Compactor and then reducing the particles in size by use of a rolling mill to that size range used for feed in the fluidized bed reactor (greater than 100 mesh and less than 35 mesh). Intermediate and final particle size separations were made using either a Trommel screen or a Ty-Lab vibrating screen.

The procedure found most satisfactory was similar to that used in past feed preparation, but it was varied because of a new batch of feed which had different characteristics from UF_4 used in the past. This new material has a higher UF_4 content than has been used before and was lower in water content. A detailed analysis is not available at this time, but UF_4 content is expected to exceed 99%. Approximately 90% was the average UF_4 content of feed used in the past. The most difficult problem encountered using this new batch of UF_4 was the production of excessive quantities of fines, which lowered the over-all yield of feed size UF_4 . The most satisfactory procedure found for minimizing these fines gave only a 60% yield (or a 12% over-all yield) of feed size material from the compacted UF_4 . The comparable yield using other batches of UF_4 had been greater than 80% (or a 16% over-all yield).

An initial pass was made through the rolling mill at the roller setting required to break up all compacted material to smaller than 8 mesh size. Then four size separations were made: the feed size, two oversize, and a fines portion which was sent back through the Chilsonator. The two oversize portions were recycled through the rolling mill. This cycle of size separations and rolling was repeated, each time decreasing the distance of the rolls apart until finally all of the Chilsonator product was converted to feed size or fines.

The Trommel screen was used in separation of the compacted material from fines in the Chilsonator product. A 20% yield of UF_4 suitable for comminution to feed size was obtained which was about the same yield obtained in this operation with other types of UF_4 . However, the size classification was different. In the past the 20% yield was composed of material greater than 16 mesh size. In this last batch of UF_4 , only 2% was greater than 16 mesh after Chilsonating; a size separation at 35 mesh screen size was required (in the Trommel) to obtain 20% of the feed suitable for comminution to feed size. Screening of the Chilsonator product was always greater than 95% efficient in removing feed size or larger particles.

3.0 HR FUEL AND BLANKET PREPARATION AND PROCESSING

P. A. Haas

Processes and equipment are being developed for preparation and processing of aqueous homogeneous reactor solution and slurry fuels and blankets. Direct denitration of thorium nitrate and/or uranium nitrate is being applied to production of 1-10 micron oxide particles by flame denitration and 50-500 micron particles by fluidized bed denitration. Engineering assistance studies are being made as required by the HRT-CPP and the PAR loop programs and for the application of hydroclones to ThO_2 slurries.

3.1 Flame Calcining Experiments - C. C. Haws, V. L. Fowler, F. N. McLain

A new burner using propane as the fuel gas was designed and operated to give flame denitrated mixed Th-U oxides which appeared superficially equal to the oxides produced using acetylene. A series of tests directed toward evaluating product fired at different temperatures over the 800-1800°C range has been started. The first of these batches (~1600°C) has been classified to < 5 micron and is ready for evaluation.

The optimum temperatures for flame denitration have not yet been determined; higher temperatures result in more difficult operation while low temperatures may result in incomplete denitration or other unacceptable product qualities. Spheroidizing of the thorium-uranium particulate in a single stage flame denitration is unlikely. Therefore, the higher temperature offered by the $\text{O}_2\text{-C}_2\text{H}_2$ flame (3400°C theoretical) no longer appears a significant advantage when compared with the easier handling and lower cost of either natural, or liquified petroleum gases and oxygen (~3000°C theoretical).

For the present equipment and procedures (incomplete insulation and small size) the cost of reagents exclusive of TNT and UNH would be:

- a. About \$10/lb for $\text{C}_2\text{H}_2\text{-O}_2$ flames
- b. About \$5/lb for $\text{C}_3\text{H}_8\text{-O}_2$ flames
- c. About \$1/lb for $\text{C}_3\text{H}_8\text{-air}$ flames

A torch was designed and fabricated for using propane and was operated to give mixed oxides comparable with previous denitrations with $\text{O}_2\text{-C}_2\text{H}_2$ flames (Table 3.1).

The low production rate of the present equipment (40 g ThO_2 per hr) requires at least two shifts of operation to produce an adequate sample. The arbitrarily selected concentration used in the first run (79 g TNT - 6 g UNH per liter) was doubled to raise the production rate (run 38) with

Table 3.1. Flame Calcining Experiments

Flame Denitration Tests

Run No.	Fuel System	Reflector Temp., °C	Quantity Produced, g	Average Particle Size, micron	Surface Area, sq. m/g	Yield Stress, lb/sq. ft	Corrosion ^a Rate, mpy	
							Oxygen	Deuterium
38	O ₂ -C ₂ H ₂	1550	270	1.2	4.9	-	2.3	4.8
39	O ₂ -C ₂ H ₂	1550	350	0.9	5.9	0.275		
40	O ₂ -C ₂ H ₂	1560	406	14	7.8	-		
41	O ₂ -C ₃ H ₈	1550	27	2.7	6.5	-		
42	O ₂ -C ₃ H ₈	1550	190	2.7	6.3	-		

a. Toroid Coupon Rate: Slurry 300 g per kg, 347 SS, 300 hr run.

negligible effect on average particle size (Table 3.1). Doubling the diameter of the alcohol injection orifice was tested in run 40 and resulted in a 10 fold increase in the average particle size. The smaller orifice (0.016 in. dia) was re-installed for runs 41 and 42. Run 42 demonstrated an extended period of operation (24 hr) and produced a quantity of material for analytical testing.

3.2 Fluidized Bed Denitration-S. D. Clinton, R. D. Arthur

A second attempt to spray a 1.5 M $\text{Th}(\text{NO}_3)_4$ aqueous solution into a fluidized ThO_2 bed at 600°C (see Figure 3.1) failed due to caking of the ThO_2 in the bed. After spraying liquid into the bed for 40 min at a feed rate of 5 ml per min, the bed temperature distribution became distorted indicating a fixed bed. Since efforts to refluidize the ThO_2 were unsuccessful, it was necessary to remove, grind, and classify the reactor contents before starting another run. A glass mock-up of the equipment was installed to study fluidization and dispersion of the liquid feed in order to eliminate caking in the ThO_2 bed.

The previously used gas distributor plate (16 gage Inconel plate with $3/64$ in. holes drilled on $1/8$ in. centers) proved to be inadequate. When the main fluidization gas was metered to the reactor, the ThO_2 particles (classified between 50 and 150 microns) came through the distributor filling the dead space at the bottom of the reactor. This allowed the gas to channel through the ThO_2 bed with no fluidization. After trying several screen type distributors with varying degrees of success, a distributor was machined from a Micro Metallic porous stainless steel sheet, $1/8$ in. thick and with a mean pore opening of 65 microns. Using this porous gas distributor a ThO_2 bed with a length to diameter ratio of approximately 3.5 could be fluidized to 120 to 130 per cent of its original length at air velocities of 0.1 to 0.3 ft per sec.

The fluidized ThO_2 bed in the glass reactor was heated to 100°C , and an attempt was made to spray water into the bed at a rate of 4 ml per min. The air line to the spray nozzle became plugged allowing the water to be pulsed into the ThO_2 by the Lapp pump with no means of atomizing the liquid. Within seconds the ThO_2 bed became fixed and full of lumps about $1/2$ to 1 in. in diameter. These lumps could not be broken down with the fluidizing air or heat but were readily crushed by hand. When both air and water flows were maintained during a later test, 5 ml $\text{H}_2\text{O}/\text{min}$ was added without caking the bed.

3.3 Hydroclone Collection of ThO_2 Slurry - P. A. Haas, F. N. McLain

Tests of cylindrical hydroclones with induced underflow receivers gave results comparable to those for conventional conical hydroclones (Figure 3.2). Although the efficiency is slightly reduced (Figure 3.3), the cylindrical design should eliminate plugging of the underflow port during operation with slurries that tend to cake. The concentration versus time curve (Figure 3.2) applies only to the loop volume/hydroclone feed

FIGURE 3.1 PROCESS FLOWSHEET FOR THE FLUIDIZED BED DENITRATION OF THORIUM NITRATE

Fig. 3.2. Hydroclone Collection of ThO₂ into an Induced Underflow Receiver.

Fig. 3.3. Apparent Hydroclone Efficiencies For Batch Collection of ThO_2 .

rate ratio for which it was obtained. The apparent efficiency versus concentration curve (Figure 3.3) would be independent of this ratio and could be used with a graphical or numerical integration to obtain a concentration versus time curve for some other system. The apparent efficiency is calculated from the slopes of the concentration versus time curve (Figure 3.2) by using the expression:

$$R(C_f) = - \left(\frac{\Delta C_f}{\Delta t} \right) \left(\frac{V}{FC_f} \right) (100)$$

C_f = circulating concentration of ThO_2 in loop

t = time

V = volume of circulating slurry

F = hydroclone throughput rate

Thus the apparent efficiency is the percentage of the ThO_2 in the hydroclone feed stream which is collected into the induced underflow stream.

The apparent efficiency, R , will be less than the hydroclone efficiency calculated from correlations for dilute slurries (ORNL-2301). For the high ThO_2 concentrations at the start of the tests, the apparent efficiency is limited by the concentration which the induced underflow stream can attain; i. e., by the rate at which ThO_2 can be transferred into the underflow receiver. During operation with dilute slurry at the end of the tests, the apparent efficiency is limited by the recycle of ThO_2 from the induced underflow receiver back into the hydroclone. Therefore, an apparent efficiency maximum occurs at an intermediate concentration.

3.4 Hydroclone Clarification of ThO_2 Slurries - P. A. Haas, W. M. Woods

Use of a single pump with a cascade of hydroclones has been suggested for controlled transfer of ThO_2 slurries. This type of system is more complicated than the collection into an induced underflow receiver as previously developed and applied. However, achieving high concentration ratios does not depend on settling in an induced underflow receiver; therefore, the cascaded transfer system would be more dependable for radioactive slurries with thermal convection or for transfer into a well mixed loop where settling is not practical.

The performance of the cascaded transfer systems can be evaluated from performance data obtained with concentrated ThO_2 slurries. Results obtained for a variety of ThO_2 slurries and hydroclone operating conditions can be organized into performance curves of the type shown in Figure 3.4. Dispersion of the ThO_2 is apparently not complete as the curves do not vary as much with particle size as would be expected for dilute, fully dispersed

Fig. 3.4. Clarification of ThO_2 Slurries by Hydroclone Cascades.

slurries. Since the feed flow is split into an underflow stream and an overflow stream of equal volumes, either the first stage must have in parallel twice the number of hydroclones of each second stage, or a 0.40 in. dia hydroclone in the first stage may be used with a 0.25 in. dia hydroclone in each second stage.

The equal flow split approximates the most efficient hydroclone operation and thus gives the most dilute overflow streams. Smaller underflow volumes would result in more concentrated underflow streams for feed concentrations of less than 500 g ThO/kg H₂O. Values of $C_u:C_f = 500:1$ may be obtained for two stages with B/F for each stage of approximately 1/50 and C_f approximately 1 g ThO₂/kg H₂O. However, only approximately 20% of the feed ThO₂ will be in the second stage underflow and the first stage will require 50 times the number of hydroclones in the second stage.

Using the concentration ratios presented (Figure 3.4), calculations were made to determine the performance of five alternate hydroclone systems (Figure 3.5) for transferring slurry between two reservoirs using a single pump. This type of system could be used for transferring a ThO₂ slurry into and out of a loop. The single pump with 80-180 ft of head limits the number of alternative cascade systems. System "E" results in the largest concentration ratio (Table 3.2) since it is the only system which has both two stage clarification for the stream to the feed tank and two stage concentration for the stream to the loop. Since the system is symmetrical, the flow control problems are simplified.

Assuming perfect mixing in both feed tank and loop, loop volume equal to tank volume, and 50/50 split of the feed to each hydroclone the differential equations for systems A, B, C, and E (Figure 3.5) were solved. The solutions are as follows:

System A

$$t = \frac{8VR}{f(1+2R)} \ln \left[\frac{1 - 4R}{3 - (2+4R)(C_t/C_{to})} \right]$$

System B

$$t = \frac{4RV(4 - S''R)}{f(1+4R - S''R)} \ln \left[\frac{4R - S''R - 1}{(1+4R - S''R)(C_t C_{to} - 2)} \right]$$

System C

$$t = \frac{16V}{f(2S+1)} \ln \left[\frac{3 - 3S}{(4 - S) - (2S+1)(C_t/C_{to})} \right]$$

System E

$$t = \frac{4V}{f} \ln \left[\frac{SR}{(1+SR)(C_t/C_{to} - 1)} \right]$$

SYSTEM "A"

SYSTEM "B"

SYSTEM "C"

SYSTEM "D"

SYSTEM "E"

CONDITIONS:

LOOP VOLUME = FEED TANK = V

COMPLETE MIXING IN TANKS

50% - 50% VOLUME SPLIT BY
EACH HYDROCLONE

CONSTANT VOLUME INVENTORIES

STARTING LOOP CONC. = $C_L^0 \approx 0$

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Fig. 3.5. Hydroclone Transfer Systems - Flow Schematics.

Table 3.2. Calculated Equilibrium Concentrations
Hydroclone Transfer Systems

Conditions: Operation until steady state concentrations with time obtained.
Flow schematics as shown on Figure 3.5.
Equal volume split by each hydroclone; i. e., overflow volume = underflow volume.
Concentration ratio as defined on Figure 3.4.

System (See Figure 3.4)	$\frac{C_t}{C_1}$	$\frac{C_f}{C_1}$	For $C_1 = 500 \text{ g ThO}_2/\text{kg H}_2\text{O}$			
			$\frac{C_f}{C_1}$	$\frac{C_f}{\text{g/kg H}_2\text{O}}$	$\frac{C_1}{C_t}$	$\frac{C_t}{\text{g/kg H}_2\text{O}}$
A	$\frac{3}{4R - 1}$	$\frac{3}{(4 - \frac{1}{R})}$	0.76	380	44	11
B	$\frac{2}{4R - (RS'' + 1)}$	$\frac{2R}{4R - (RS'' + 1)}$	0.545	270	90	6
C	$\frac{4 - S}{3S}$	$\frac{1}{S}$	0.288	144	20	25
D	$\frac{R''(4 - S) - 1}{2R''S}$	$\frac{1}{S}$	0.288	144	35	14
E	$\frac{1}{SR}$	$\frac{1}{S}$	0.288	144	200	2.5

where:

t = time, min

V = tank and loop volume, liters

f = flow rate, liters/min

C_t = feed tank concentration, g/liter

C_{t0} = starting feed tank concentration

S , S'' , R as in Figure 3.4

For the case of $V = 8$ liters, $f = 4$ liters/min and $C_{t0} = 500$ g/liter, and using values of R , S and S'' taken from Figure 3.4, values of time vs. concentration were calculated for the various systems. Curves are shown for cases A, B, C, and E in Figures 3.6-3.9. Since R , S and S'' are functions of hydroclone feed concentration (Figure 3.4) averages over appropriate intervals were used to calculate the curves for all except system E. It is interesting to note that for system E all the concentrations around the hydroclones are independent of time, i. e., depend on C_{t0} and concentration factors only.

The times for all except system B to come to essential steady state seem quite acceptable, that is, no more than an hour (Table 3.3). Considering completeness of transfer of solids as well as time, system E seems superior to the others which were analyzed.

Table 3.3. Times to Approach Steady State

System	Approximate Time to 95% of Steady State, min
A	30
B	100
C	20
E	45

Fig.3.6. C_T and C_L vs Time for System A.

Fig. 3.7. C_T and C_L vs Time for System "B".

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Fig. 3.8. C_T and C_L vs Time for System "C".

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Fig. 3.9. C_T and C_L vs Time for System "E".

4.0 ION EXCHANGE

M. E. Whatley

4.1 Mechanism and Kinetic Studies - J. S. Watson

In order to make rational predictions of the operating characteristics of uranium anion exchange contactors, an understanding of the mechanism and kinetics of the exchange is necessary. Toward this objective an effort is being made to determine the equilibrium sorption isotherms and rates of sorption of uranium from sulfate solution onto Dowex 21K resin.

One important parameter of any ion exchange study is the total exchange capacity of the resin. Because Dowex 21K is a relatively new product, few accurate capacity measurements have been made.

Five samples of resin, weighing between 1 and 3 grams, were converted to the chloride form with 1 liter of 1 N sodium chloride solution. Each sample was placed on a paper filter funnel, and 1 liter of solution was passed "drop-wise" through the funnel. The resin was then centrifuged for 30 min to remove the solution on the outside of the beads. After being placed back on the funnel, the resin was eluted with 1 liter of 1 N sodium nitrate solution. This solution was analyzed for total chloride content to determine the capacity of the resin. Since it was desirable to weigh the resin in the dry chloride form, the samples were then converted to the chloride form, washed with 250 ml of water, and dried for 3 days at 55°C before weighing.

The results of these runs are shown in Table 4.1. The average value of 4.56 ± 0.19 meq/g of dry chloride resin agrees very well with the manufacturer's estimation of 4.5 ± 0.3 meq/g.

Table 4.1. Chloride Capacity of Dowex 21K

Run	Dry Wt of Resin, g	Total meq of Chloride Ions	Resin Capacity, meq/g
1	2.760	12.0	4.35
2	2.377	11.4	4.80
3	2.613	12.0	4.60
4	2.738	12.0	4.39
5	2.999	14.0	4.67

Average value = 4.56 meq/g.

Standard deviation = 0.19 meq/g.

The bulk of the experimental work during the month was devoted to a continuation of studies on the equilibrium uranium sorption from sulfate solutions. However, difficulties with the analyses have delayed the reporting of those results.

5.0 POWER REACTOR FUEL REPROCESSING

C. D. Watson

5.1 Darex - F. G. Kitts, B. C. Finney, F. L. Rogers, J. Beams, G. B. Dinsmore, R. L. Boles

The purpose of the Darex process is to convert stainless steel-U and stainless steel-UO₂ fuel elements into chloride-free nitrate solutions suitable for processing in existing solvent extraction equipment. Chloride must be added to effect dissolution of stainless steel in HNO₃ and then removed to avoid corrosion of feed adjustment, solvent extraction, and waste handling equipment. Continuing investigations will study the dissolution, chloride removal and feed adjustment steps when processing APPR (stainless steel-UO₂).

5.1.1 Continuous Darex

Run 76 of 11-1/2 hr¹ duration (see Figure 5.1) was made using the complete integrated loop. The fuel element used was a prototype APPR (~ 91% SS, ~ 7% U, and ~ 2% silica) and the operating conditions were the same as those for run 72 (Unit Operations Monthly Report for September, CF No. 58-9-62). The stripping column had been thoroughly cleaned with hot 2 N NaOH prior to this run. Run 76 was made in an attempt to obtain steady state operation before the column became restricted due to the deposition of siliceous material.

Approximately 3 hr after the start of the run, the amount of liquid hold-up in the column began to increase and the run was terminated when the upper one-third of the column flooded to the extent that liquid was backed up into the dissolver. It has been observed that the deposition of the siliceous material occurs first in the downcomers but ultimately there is deposition on all interior surfaces of the column.

It is apparent from the difficulties encountered during runs 72 and 76 that a bubble cap column is unsatisfactory as a chloride stripping column when the fuel being dissolved contains appreciable quantities of silica (~ 2%).

Samples of the solids in the dissolver product stream, stripped product stream, and NaOH wash water were analyzed for Si, Fe, Ni, Cr, and U. The analytical results are presented in Table 5.1. The amounts of the various constituents reported does not account for the complete sample and an appreciable portion of each sample consisted of acid insolubles. A qualitative spectrographic analysis did not provide any additional information.

Fig. 5.1. Material Balance-Run 76; Basis: 1 liter of Dissolver Product in 12.4 min.

Table 5.1. Analysis of Solids in Dissolver Product, Stripped Product, and NaOH Wash Water

	Dissolver Product, Wt %	Stripped Product, Wt %	NaOH Wash Water, Wt %
Si	17.13	9.19	13.85
Fe	9.52	.87	14.69
Ni	0.52	.09	1.83
Cr	12.91	.48	4.66
U	< 0.3	< 0.30	2.24
Acid insoluble	<u>23.</u>	<u>10.16</u>	<u>11.22</u>
Total	63.38	21.09	48.49

The material balance for run 76 is also presented in Figure 5.1. The upgrading of the HNO_3 in the stripping column overhead during the run can be attributed to the increasing liquid hold-up in the column. There was no HNO_3 added to the condenser product adjustment tank for the last 3 hr of the run and consequently a steady state operation was not achieved. The material balance is based on over-all average flow rates and compositions.

5.1.2 Semi-Continuous Darex

The purpose of the work on the development of a semi-continuous Darex process is to provide data required for the design of a Darex head-end to be incorporated in the PRFR pilot plant. The flowsheet under development should provide solvent extraction feed with < 50 ppm Cl^- while regenerating a mixed acid ($\text{HNO}_3 + \text{HCl}$) stream which can be adjusted to the original dissolvent composition without exceeding the original volume, eliminating the need for a Cl^- -containing mixed acid waste stream.

Figure 5.2 shows a tentative flowsheet for semi-continuous processing. The dissolver is essentially continuous, as demanded for criticality control. The fuel is charged batchwise but the dissolvent is fed and the dissolution product removed at approximately the same rates until the charge is dissolved. This procedure provides a continuous feed to the Cl^- removal and feed adjustment vessel where Cl^- is removed and recovered by volume reduction and refluxing, with cyclic additions of concentrated HNO_3 . The dissolved metals in the dissolution product are concentrated during the Cl^- removal treatment since only mixed acid is removed from the vessel. Final feed adjustment is accomplished batchwise by the addition of water. The use of semi-continuous rather than truly batch operation allows a reduction in the size of the feed adjustment vessel and shortens the operating cycle by allowing dissolution and Cl^- removal to proceed simultaneously.

Fig. 5.2. Semi-continuous Darex Flowsheet.

Dissolution. The installation of the 1-in. I.D. glass dissolver was completed and a number of check-out runs were made. The purposes of this dissolver were:

1. To demonstrate the operation of a long cylindrical dissolver on a semi-continuous basis.
2. To determine the feasibility of maintaining a constant liquid level in such a dissolver by using an airlift.

The fuel elements used were fabricated from 24"x0.6"x0.016" 304 stainless steel sheets with 0.016" spacers between plates; the average weight of these elements was 575 g. The last and most significant run was made with three elements charged to the dissolver. These elements were completely dissolved in 65 min requiring 21 liters of aqua regia (5 M HNO_3 -2 M HCl). The dissolution rate was 26.6 g/min with a resultant stainless steel loading of approximately 82 g/liter.

The results obtained from the operation of the dissolver indicated the following:

1. The dissolvent should be fed into the dissolver at the bottom and flow upward past the element.
2. The dissolver product should be removed from the dissolver through a side-arm take-off (the location of the side-arm with respect to the bottom of the dissolver has not been optimized).
3. The dissolution reaction is too violent to regulate a liquid level in the dissolver by use of an airlift, although the liquid level in the side-arm can be regulated by this means.
4. The dissolution takes place on the bottom 12 inches (approximately) of the fuel element (probably dependent on dissolvent flow rate).
5. There appears to be no advantage to recycling a portion of the dissolver product back through the dissolver.
6. Apparently, there is no advantage to returning the dissolution condensate to the dissolver. This dilute mixed acid stream can be sent directly to an acid make-up tank; this has the advantage of decreasing the load on the feed adjustment tank.

A 6-in. I.D. glass dissolver similar to the 1-in. model has been constructed (see Figure 5.3). This dissolver will be operated to make dissolution studies of full size prototype APPR fuel elements and also determine the operability of the dissolver.

Fig. 5.3. Schematic Diagram of 6-in-ID Glass Dissolver.

Batch Cl^- Removal and Solvent Extraction Feed Adjustment. A 2-liter batch run (similar to run APPR-7 reported in August 1958) was made to obtain more detailed data and to check the effect of batch size on Cl^- removal and recovery. Data for this run, APPR-11, are shown in Figure 5.4. The data for run APPR-11 are similar to that for APPR-7 except that in APPR-7 the Cl^- was higher by a factor of 5 after the recovery and prior to the reflux steps. This is not believed to be significant since these particular pieces of data for run APPR-7 were acknowledged to be only rough approximations.

The really significant difference between the two runs is the 10 fold higher Cl^- concentration in APPR-11 after the refluxing period. This is attributed to the fact that the same power was used for both runs while the amount of liquid present in APPR-11 was about twice that for APPR-7; this resulted in a reduced boil-up (on a percentage basis) with less relative agitation and liquid-vapor contacting.

Semi-Continuous Cl^- Removal and Feed Adjustment. In the studies on semi-continuous operation of the Cl^- removal and feed adjustment vessel, dissolution product (DP) was made batchwise and metered to the vessel to simulate the discharge of a continuous dissolver. The generalized procedure for a semi-continuous run was as follows:

1. Add a portion of the DP (about $1/3$ in these runs) and 22.5 M HNO_3 in ratio 5:1.
2. Add remainder of DP at a constant rate while boiling off mixed acid at the same rate, thus maintaining a constant liquid level.
3. Add more 22.5 M HNO_3 to increase the Cl^- volatility.
4. Boil off a volume of mixed acid equal to the amount of 22.5 M HNO_3 to be added for the reflux step.
5. Add 22.5 M HNO_3 and reflux to decompose remaining Cl^- .
6. Dilute with H_2O to original volume of DP.

The first attempt at semi-continuous operation, APPR-12, was unsuccessful; Cl^- content of the product was ~ 2500 ppm after refluxing for 2 hr. In this run the second HNO_3 addition (step no. 3 above) was made continuously at the same rate at which the DP had been added; Cl^- concentration at the beginning of reflux was 1.66 M, a factor of 2 higher than in the two following successful runs. The Cl^- concentration might have been reduced to a tolerable level with a longer reflux period.

Run APPR-13 was the most successful to date; the Cl^- content of the solvent extraction feed was 20 ppm. The procedure was as outlined for APPR-12 except that in step no. 3 the HNO_3 addition was made batchwise

Fig. 5.4. Data for Batch Darex Run APPR-II

rather than continuously. After all the DP was added, the boil-off was continued to lower the liquid level so that the addition of the HNO_3 batch returned the liquid level to that maintained throughout the addition of the DP. The reflux step was carried out in a flask with two laboratory condensers mounted vertically in series serving as an up-draft condenser. Condensation was accomplished in the upper condenser and the liquid film flowed down the wall of the lower condenser which provided area for NO_2 absorption. Mechanical agitation was supplied in the flask to improve mixing in the boiling liquid phase. Compositions, volumes and densities at various points during the run are shown in Table 5.2. The mixed acid recovered in runs 11, 13 and 14 was adjustable to original dissolvent composition without exceeding the original volume; run 12 was borderline.

Run APPR-14 was made to check the use of a down-draft condenser without agitation in the reboiler. The processing of the material prior to the reflux step was batchwise, similar to run APPR-11. With the down-draft condenser and no agitation, the worst conditions for Cl^- removal, a Cl^- content of 140 ppm was reached after 2 hr of refluxing.

5.2 PWR Decladding and Core Dissolution - G. A. West, J. C. Rose, T. A. Gens (Chemical Development)

The study of decladding and core dissolution of PWR blanket elements (UO_2 clad with Zr-2) was continued to evaluate the Zirflex flowsheet in which 6 M NH_4F -1 M NH_4NO_3 is used to declad, followed with 10 M HNO_3 for core dissolution. Final cold runs were made on oxide coated PWR blanket pins with surface conditions similar to irradiated pins. The analytical results on these cold runs indicate the uranium loss to the decladding solution was 0.1%. Hot runs were started on PWR blanket pins irradiated to 250 and 500 Mwd/ton burnup. Results of the hot runs will be presented in detail in the Chemical Development Section B monthly report after analytical results are received.

Future runs will be made on PWR blanket pins with 2,500 Mwd/ton burnup. The Sulflex flowsheet will also be demonstrated with Consolidated Edison type pins, UO_2 - ThO_2 clad with stainless steel, with burnups of 250 to 5,600 Mwd/ton.

5.3 Solvent Extraction Studies - R. J. McNamee, F. L. Rogers, D. A. McWhirter

In order to assist the Hanford Atomic Products Operation adapt the Redox process for the recovery of uranium from spent power reactor fuels, the Chemical Technology Division will investigate column capacity, efficiency, decontamination, and stability for various proposed flowsheets. Unit Operations will make the cold studies in a 2-in. dia packed, glass column. The pilot plant will make runs with spiked activity.

As a safety precaution due to the use of hexone as the solvent, expanded metal shields (to prevent breakage) and spray nozzles were installed

Table 5.2. Results of Semi-Continuous Darex Run APPR-13

	H ⁺ , <u>M</u>	Cl ⁻ , <u>M</u>	Volume, cc	Density, g/cc	Metals, g/l
Dissolution product*	2.77	1.64	3700	1.233	64
Original charge to vessel (1250 cc DP + 250 cc 22.5 <u>M</u> HNO ₃)	6.12	1.38	1480	-	54
After addition of remainder of DP	3.24	2.06	1175	1.584	202
After addition of 350 cc 22.5 <u>M</u> HNO ₃	8.26	1.71	1410	-	168
After concentration prior to reflux	2.52	1.23	830	1.755	285
After addition of 600 cc 22.5 <u>M</u> HNO ₃	11.10	0.73	1400	-	169
After 2 hr refluxing	8.46	52 ppm	1270	1.660	186
After water dilution yielding solvent extraction feed	2.90	20 ppm	3700	1.257	64

*Prototype APPR was dissolved to 65 g/liter yielding DP with about 58 g SS/liter and 6 g U/liter.

on the glass columns.

Due to the limited quantity of Darex stainless steel solution available, and the desirability of using it for the more representative pilot plant runs, bulk quantities of iron, chromium, and nickel nitrates were purchased to make synthetic feed solutions.

5.4 Feed Clarification - Solid-Liquid Separation in Fuel Reprocessing Operations - Problem Statement

Purpose. The purpose of the feed clarification program is to devise methods of removing particulates (silica, UO_2 and $\text{UO}_2\text{-ThO}_2$ fines) from process and decladding solutions.

Need. Precipitated silica must be removed from solvent extraction feed solutions resulting from the dissolution of stainless steel-alloy and stainless steel- UO_2 fuels. Aqueous reprocessing of stainless steel or zirconium-clad UO_2 or $\text{UO}_2\text{-ThO}_2$ fuels requires separation of fine particles of core material from the decladding solution.

The removal of silica allows solvent extraction columns to be operated with a reduced amount of interfacial crud formation. Recovery of fine particles of UO_2 and $\text{UO}_2\text{-ThO}_2$ is necessary for economic reprocessing. The separation equipment must be simple and reliable. It must be remotely operated and decontaminated.

Scope and Method. Studies on silica removal will include filtration on a disposable media, such as sand or filter aid and on a decontaminable media support, such as metal screening and monofilament inert filter cloth (Teflon, Kel-F, glass, etc). The rate of filtration, cake washing requirements, solution clarity, and removal and transfer of contaminated media will be studied. First studies will be on a small scale with promising procedures being scaled up to an engineering test unit to develop data required for the design and operation of pilot plant equipment.

Studies on separation of UO_2 fines from decladding solutions involve a transfer system to remove the contents from a dissolver, a unit to separate the bulk of the decladding solution from the solid UO_2 , followed by the washing of the dissolver and UO_2 free from decladding solution and the return of the UO_2 to the dissolver. First studies will be on the use of a filter for removal of UO_2 fines; these will be dissolved from the filter with nitric acid with the uranium values recycled to the dissolver. On successful development of all components, an integrated test system will be built and operated.

6.0 WASTE PROCESSING

C. W. Hancher, J. C. Taylor

The purpose of the Reduction to Solids problem of the radioactive waste processing program is to reduce radioactive waste to a solid form suitable for ultimate storage. The waste now being investigated is "25-TBP" waste. Three phases of the investigation are reported: (1) nonradioactive chemical testing, (2) radioactive reduction to dry solids, and (3) high level activity reduction to solids in hot cell Building 4507.

The process procedure for treating the "25" waste was:

- a. Evaporation (0-200°C)
- b. Drying (200°-700°C)
- c. Condensing (~ 90% of original liquid volume)
- d. Treatment of condensate to remove fission products
- e. Scrubbing of noncondensables with NaOH
- f. Recycle of trapped fission products

6.1 Nonradioactive Chemical Testing

The "25" waste has the following composition:

Al(NO ₃) ₃ ·9H ₂ O	1.6 M
Hg(NO ₃) ₂	0.01 M
NH ₄ NO ₃	0.03 M
HNO ₃	0.50 M

One of the problems encountered in reducing "25" waste to solids is the recycle and build-up of nitrate ion with the fission products from the condenser and from scrubbing system. The effect of the addition of NaOH (38 g per 200 ml - pH 9.0) to the waste before evaporation was tested. Two samples were heated to 140°C. The condensate from the acid sample was 0.59 M HNO₃ while the condensate from the neutralized sample was 0.47 HNO₃ (Table 6.1). While there was only little difference in the acidity of the condensate, the corrosion in the evaporator should be less with the neutral waste.

Table 6.1. Evaporation of "25" Waste
to 10% of Original Volume

Feed - 200 ml		"25" Waste
Maximum Temp.	NaOH, g	Condensate Molarity (HNO ₃)
140	0	0.59
140	38	0.47

It has been shown by previous workers that the nitrate ion can be chemically decomposed with formaldehyde. A possible reaction might be: $4\text{HNO}_3 + \text{H}_2\text{CO} \rightarrow \text{CO}_2 + 4\text{NO}_2 + 3\text{H}_2\text{O}$. Two experiments were performed to determine the amount of nitrate ion recovered in the condensate when neutralized "25" waste was heated to dryness. The heating time from 20°C-700°C was 4-6 hr. To one of the waste samples 4% by volume of 38% formaldehyde was added. The nitrate ion recovery for the nonformaldehyde system was 76% while for the system treated with formaldehyde the recovery was 23% (Tables 6.2 and 6.3). The HNO₃ formaldehyde reaction began at 104°C and is exothermic, but was controllable if cooled.

Table 6.2. Evaporation of "25" Waste
With NaOH Added

Feed: 200 ml "25" waste

38 g NaOH

Temp.	Volume, ml	Condensate Molarity (HNO ₃)
0°-425°	40	0.2
425°-475°	40	0.9
475°-520°	40	5.2
520°-635°	46	11.4
Nitrate Recovered - 76%		

Table 6.3. Evaporization of "25" Waste With
Formaldehyde and NaOH Added

Feed: 200 ml "25" waste

38 g NaOH

8 ml 38% formaldehyde

Temp.	Volume	Condensate Molarity (HNO ₃)
0°-105° (NO ₂ fumes)	14 ml	0.90
105°-200°	14	0.25
200°-375°	32	0.06
375°-450°	35	0.05
450°-495°	37	0.03
495°-545°	30	0.02
545°-680°	19	4.68
Nitrate recovered - 23%		

One other method was tested for the minimizing of nitrate ion recovered in the condensate. A copper bed, which was heated to 400°C was inserted between the furnace and the condenser. The hot copper reduced the nitrate by the reaction: $\text{Cu} + \text{NO}_3^- \rightarrow \text{CuO} + \text{N}_x\text{O}_x$. When 100 g of copper was used at 400°C only 27% of the nitrate ion was recovered (Table 6.4).

6.2 Reduction of Radioactive "25" Waste to Solids

Synthetic "25" waste was spiked with fission products in 6 M HNO₃. The resulting radioactive waste had the following concentration:

Al(NO ₃) ₃	1.6 M	Ce β	3.0x10 ⁷ c/min/ml
HNO ₃	0.5 M	Cs γ	6.0x10 ⁸ c/min/ml
Gross β	4.5x10 ⁷ c/min/ml	Ru γ	3.0x10 ⁸ c/min/ml
Gross γ	1.1x10 ⁷ c/min/ml	Zr-Nb γ	2.0x10 ⁸ c/min/ml
Sr γ	2.0x10 ⁸ c/min/ml	TVRE γ	5x10 ⁸ c/min/ml

This waste was neutralized with 190 g of NaOH per liter (4.7 M for pH 7-9). The neutralized waste was heated in a furnace (Figure 6.1), using a programmed heating cycle. The furnace was heated to 200°C and held there until most of the liquid was distilled over and condensed (~ 170 ml). After the distillation period, the temperature was raised as quickly as possible to 650-700°C (3-7 hr).

Table 6.4. Destruction of Nitrous Oxides By
Passing Over Hot Copper

Feed: 200 ml "25" waste

38 g NaOH

Copper: 400°C

100 g

Condensate Volume	Condensate Concentration M HNO ₃
15 ml	0.01
15	0.02
15	0.02
15	0.03
15	0.09
15	0.19
15	0.60
15	1.44
15	3.96
15	11.70
Average	1.8
Nitrate recovered - 27%

Fig. 6.1. Experimental Setup For Radioactive Waste Reduction to Solids.

The system was operated under a vacuum of 10-13 in. of water. The quantity of off-gas was measured with a wet test meter. It was very evident from the results of runs 58 and 59 that the amount of fission products evolved during heating was a direct function of total off-gas volume (see Tables 6.5 and 6.6).

The solids formed during these tests were spongy and full of small air holes. An addition of a fluxing clay might increase the heat transfer properties of these solids.

Table 6.5. Reduction of "25" Waste to Solids

Run R-59

Feed: "25" waste - 200 ml

38 g NaOH

Maximum Firing Temperature: 680°C

Total Off-Gas - Scrubbed with 10% NaOH - 0.008 cu. ft

Stream	Vol., ml	H ⁺ <u>M</u>	NO ₃ ⁻	Gross β	Gross γ	Ce-β c/min/ml	Cs-γ	Ru-γ
<u>Feed</u>	200	0.52	10.9	4.5x10 ⁷	1.1x10 ⁷	3x10 ⁷	6x10 ⁶	3x10 ⁶
<u>Condensate</u>								
1	30	0	-	1.2x10 ⁴	1.3x10 ⁴	655	7.7x10 ³	8.9x10 ³
2	25	.03	-	1.6x10 ³	1.8x10 ³	76	873	1.5x10 ³
3	25	.03	-	865	932	-	361	879
4	30	.02	-	1.2x10 ³	1.3x10 ³	-	306	1.2x10 ³
5	12	.01	-	2.1x10 ³	2.2x10 ³	54	548	1.8x10 ³
<u>Packed Bed Scrubber</u>	200	3.0 <u>M</u>	NaOH	670	549	-	84	540
<u>Caustic Bubblers</u>								
1	200	3.0 <u>M</u>	NaOH	-	10	-	-	-
2	200	-	-	-	11	-	-	-
3	200	-	-	-	31	-	-	-
4	200	-	-	-	12	-	-	-

Table 6.6. Comparison of Reduction to Solids Runs

Using "25" Waste

Feed: "25" waste - 200 ml

38 g NaOH

Off-Gas - Scrubbed with 10 NaOH

Run No.	Max. Temp., °C	Off-Gas, Cu. ft	% β Evolved	% γ Evolved	% Ru-γ Evolved
52	665	0.2	0.73	0.30	19.0
58	680	0.22	1.20	1.0	19.0
59	640	0.008	0.003	0.001	0.06

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