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Chemical Separation Processes
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**EVALUATION OF A CONTINUOUS SOLID BOWL CENTRIFUGE FOR
PROCESSING SIMULATED ROVER DISSOLVER EFFLUENT**

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ABSTRACT

Pilot plant evaluation of the applicability of a continuous solid bowl centrifuge to solids-liquid separation during Rover fuels headend processing is described.

SUMMARY

Pilot-plant studies using a plant-scale continuous Bird centrifuge were made to determine the solids-liquid separation and subsequent solids washing efficiencies for processing simulated Rover dissolver effluent. The studies were under-taken to provide a basis for acceptance of the continuous solid bowl centrifuge as a method of solids-liquid separation and solids washing in the Rover process.

Test results showed the solid bowl centrifuge reduced the undissolved solids content of the liquid effluent to a level compatible with the TBP extraction system in the range of flow rates studied. Although wash injection nozzles were located within the centrifuge bowl, little or no washing of occluded uranium from the solids was possible. The most effective removal of occluded uranium, and thus the most efficient uranium recovery, resulted from repulping undissolved solids in water in two or more stages and recycling the repulped slurry through the centrifuge. Discharged solids were in the form of a fluid mass after the initial centrifugation and all subsequent repulp stages.

Pilot-plant experience indicates the plant centrifuge should operate satisfactorily under remote conditions. Troublefree operation should result if there is no significant deviation from process conditions specified in the Rover flow sheet and operating procedures outlined in this report.

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EVALUATION OF A CONTINUOUS SOLID BOWL CENTRIFUGE FOR
PROCESSING SIMULATED ROVER DISSOLVER EFFLUENT

I. INTRODUCTION

The reprocessing of spent Rover fuels requires combustion of the graphite fuel matrix in a continuous primary burner followed by burning of the primary ash in a secondary burner. The resulting secondary burner ash is dissolved in HNO_3 -HF yielding a solution of UO_2F_2 and $\text{UO}_2(\text{NO}_3)_2$. Undissolved solids which must be separated from the solution and washed of uranium are Nb_2O_5 , Al_2O_3 , and trace quantities of unburned carbon.

The continuous solid bowl centrifuge is attractive for use in the Rover process because it requires less space than alternate settler/washer systems, can be operated continuously, and is critically safe. (Refer to ICP-1016⁽¹⁾ for a description of a batch centrifuge/settler-washer system.) In continuous operation, slurry from the dissolver can be fed directly into the centrifuge with continuous discharge of solid and liquid effluents.

II. SOLID BOWL CENTRIFUGE SYSTEM DESCRIPTION

The continuous solid bowl centrifuge test system consisted of the following primary components (Figure 1): 1) a centrifuge complete with slurry feed inlet, in-bowl wash nozzles, in-bowl decontamination wash nozzles, and solid and liquid effluent outlets, 2) a slurry feed tank equipped with provisions for air sparging to promote solids suspension, 3) a feed line for slurry transport by gravity from the slurry feed tank to the centrifuge feed inlet.

The centrifuge used for the experimental testing was a 6-inch diameter continuous solid bowl centrifuge manufactured by Bird Machine Company. The bowl contour was a combination 6-inch diameter cylinder 8 inches long connected to a 10° truncated cone (6 inches in diameter at the largest end) 4 inches long. The conveyor (auger), mounted inside the bowl was coated with chromium oxide for maximum corrosion and erosion resistance. The conveyor trunnion contained compartments for introducing feed and wash solution. Wash nozzles distributed water on the inside surface of the bowl. Feed ports were used to distribute feed at the approximate junction of the cylinder and the 10° cone.

Figure 1. Solid Bowl Centrifuge Test System

Five decontamination wash nozzles were provided at the top of the bowl cover for rinsing the outside of the bowl. Solids were discharged vertically downward. All materials not coated with chromium oxide that were in contact with the dissolver effluent were constructed of 304 ELC, stainless steel. The centrifuge was driven by a 5 HP, 3460 RPM motor having a V-belt pulley drive.

The slurry feed tank was a 12-inch diameter cylinder having a 60° conical bottom and 25-liter capacity. Sparge air at 100 psig was metered through the apex of the cone to maintain adequate solids suspension. A 3/4-inch ball valve located 2½ inches below the air sparge line regulated feed addition to the centrifuge. A 90°, 3/4-inch elbow located below the ball valve resulted in no solids plugging problems during slurry transport. A short length of 3/4-inch, 304 SS flexible metal hose served as the slurry feed line to the centrifuge. The apex of the slurry feed tank cone was approximately 15 inches above the centrifuge bowl centerline.

III. SIMULATED ROVER FEED SLURRY

Simulated Rover dissolver effluent for experimental use as centrifuge feed slurry consisted of the material compositions shown in Table I. Particle size data for suspended solids in the simulated feed slurry are given in Table II. Although fluoride exists in the actual dissolver effluent, it is complexed by aluminum to reduce the corrosion potential downstream of the dissolver. The complexed fluoride should have a negligible effect on solids-liquid separation and thus was not added to the simulated feed slurry. Unburned graphite, because of the small amount present, was also not included in the slurry.

IV. EXPERIMENTAL PROCEDURES AND SAMPLE ANALYSIS

All experimental runs using the continuous centrifuge were batchwise. For preliminary solids-liquid separation tests, 11 grams/liter of Nb_2O_5 in water served as feed slurry. Tests of solids washing and repulping were made with a slurry having the composition shown in Table I. The specific feed was mixed in the slurry feed tank at an air sparge rate of 2.7 scfm/ft² of cross-sectional area to maintain solids suspension.

TABLE I

COMPOSITION OF SIMULATED ROVER DISSOLVER EFFLUENT

<u>Dissolved Component</u>	<u>Solid Component</u>	<u>Concentration</u>
Uranium	-	38.4 g/l
HNO ₃	-	3.88 <u>M</u>
-	Al ₂ O ₃	2.7 g/l
-	Nb ₂ O ₅	7.8 g/l
-	Total Solids	10.5 g/l

TABLE II

PARTICLE SIZE DISTRIBUTION OF SUSPENDED SOLIDS

<u>Solid Component</u>	<u>Particle Size</u>	<u>WT%</u>
Al ₂ O ₃	60 Mesh (250μ)	25.7
Nb ₂ O ₅	+ 100 (+ 149μ)	4.1
Nb ₂ O ₅	100 - 140 (105 - 149μ)	10.0
Nb ₂ O ₅	140 - 200 (74 - 105μ)	11.3
Nb ₂ O ₅	200 - 325 (44 - 74μ)	23.3
Nb ₂ O ₅	- 325 (-44μ)	25.6

The centrifuge rotation rate was fixed by selection of the motor drive sheave diameter. The slurry feed rate was controlled by adjusting the ball valve opening at the base of the feed tank. The liquid supernate was usually "cloudy" during the initial slurry feeding, but cleared after approximately 2 minutes of operation. At this point, a supernate sample was taken for analysis of undissolved solids content. The same procedure was followed after each flow rate readjustment.

Flowrate measurements were taken with a stopwatch and a graduated cylinder. Discharged solids were collected in a polyethylene bag attached to the solids discharge flange of the centrifuge. After an entire batch of slurry had been fed, the centrifuge was shut down. The hold-up volume of the bowl was 750 ml. and consisted of a solids-rich liquor which would discharge from the liquid effluent outlet when rotation ceased. The undissolved solids concentration of this effluent was approximately twice that of the feed slurry. Discharge of this hold-up volume should present no problem in plant operation provided the downstream diverter valve is positioned to direct the liquor to the repulp tank.

For testing of solids washing effectiveness using the in-bowl wash system, the desired wash-water flow rate was adjusted using the flowmeter prior to slurry feeding. Only a combined wash-water rate for the two nozzles was possible, since the total flow was regulated to both nozzle locations. The solids repulping study involved collection of the wet separated solids from a 25-liter batch and re-slurring the solids in 5 liters of water. The resulting slurry was then fed to the centrifuge. This procedure was repeated for each repulp cycle for a maximum of five cycles. A sample of wet solids from each cycle was dried for analysis of occluded uranium.

The undissolved solids content of the centrifuge-effluent samples was determined by collection of the solids on a 0.45 μ millipore filter. Analysis of occluded uranium in the discharged solids was made by x-ray fluorescence.

V. RESULTS AND DISCUSSION

A. Solids-Liquid Separation

Results of solids-liquid separation tests using Nb_2O_5 in water are shown in Figure 2. The centrifuge was run at four rotation rates; at each rotation rate, the slurry feed rate was changed to determine its effect on separation efficiency. The separation efficiency, SE, is defined as:

$$\text{SE} = 1 - \frac{C_e}{C_f}$$

Where C_e = Concentration of suspended solids in the liquid effluent, grams/liter.

C_f = Concentration of suspended solids in the feed slurry, grams/liter.

The percent solids recovery is $100 \times \text{SE}$.

The separation efficiency decreased with increasing slurry feed rate as also shown in Figure 2. This result was anticipated, since slurry residence time and settling time within the centrifuge bowl was shortened as throughput was increased. Operation of the centrifuge at the two highest centrifugal forces tested, 1300 and 2175 g's (corresponding to 3900 and 5050 rpm, respectively), resulted in decreased separation efficiencies for a given feed rate. Increased turbulence at the higher rotation rates may have caused some of the fines to be carried over in the effluent.

Maximum solids-liquid separation efficiencies were attained at 500 and 800 g's (corresponding to 2400 and 3100 rpm). Solids recoveries at these rotation rates ranged from 99.44 to 99.86% at slurry feed rates ranging from 0.3 to 2.2 l/min. Suspended solids content in the centrifuge liquid effluent ranged from 0.0014 to .031% by weight (0.014 - 0.310 g/l). Although no precise data are available, extraction columns at ICPP have tolerated suspended solids concentrations of 0.5 g/l. Thus, solids in the liquid effluent stream are not likely to present problems in the TBP extraction system. The separation efficiencies obtained at 2400 and 3100 rpm, represented by the two uppermost lines in Figure 2, are essentially the same. Therefore, the lower rotation rate which is more advantageous from the standpoint of reduced wear and maintenance should be used.

Figure 2. Centrifuge Separation Efficiencies - Nb₂O₅ In Water

B. In-Bowl and Repulp Washing of Solids

Tests using simulated Rover dissolver effluent (Table I) as centrifuge feed yielded solids of a paste-like rather than a granular consistency. Occluded uranium and nitric acid within the discharged solids obviously produced the cohesive effect. Bridging of solids in the discharge chute can be minimized provided: 1) the solids fall vertically into either a repulp tank or after the last wash cycle, a combination drier and washed solids collection vessel, and 2) a spray system is located in the discharge chute to aid the transport of wet solids to down-stream vessels. A schematic representation of the proposed centrifuge/repulp wash system is shown in Figure 3.

The in-bowl wash nozzle system was not able to meet process requirements for removal of occluded uranium from the Rover solids. This result was expected, since the solid bowl centrifuge functions as a continuous decanter. Wash liquor simply flows over the cake of solids which is compressed against the bowl wall. Thus, the liquor does not pass through the cake to remove occluded solubles as in porous membrane filtration. Bird Machine Company's Engineering Bulletin 30-110 states that if a substantial quantity of solids are smaller than 200 mesh, it is unlikely that a thorough wash will be obtained, and repulp washing will be required. Data from in-bowl wash testing appear in Table III. The uranium content of solids discharged from the centrifuge without in-bowl washing ranged from 0.98 to 1.5%. Those solids which had been washed within the bowl contained 0.71 to 1.2% uranium.

Since in-bowl solids washing could not affect a sufficient uranium recovery in the supernate, repulp washing was required. Two repulp test runs were made to determine the number of repulp cycles required for adequate uranium recovery. Results of the two tests are summarized in Table IV.

TABLE III

SUMMARY OF RESULTS OF IN-BOWL SOLIDS WASHING

<u>Run</u>	<u>Slurry Feed Rate, (ℓ/min)</u>	<u>Wash-Water Rates Per Nozzle</u> <u>Location (cc/min)^a</u>		<u>Occluded Uranium</u> <u>in Solids, wt%</u>
		<u>Location 1</u>	<u>Location 2</u>	
1 ^b	1	6	0	1.2
2 ^b	1	0	6	0.71
3 ^b	3	18	0	1.5
4 ^b	3	0	18	0.96
5 ^c	1	6	6	1.6
6 ^c	1	17.5	17.5	0.78

a - Wash nozzle locations are:

Location 1 : 2-7/8 inches from solids discharge weir directed upward

Location 2 : 7/8 inches from solids discharge weir directed downward

b - Wash water rate based on 0.5 g. wash water /g. solids

c - Wash water rate based on 1.0 g. wash water /g. solids

Figure 3. Proposed Rover Centrifuge/Repulp Wash System

TABLE IV

SUMMARY OF RESULTS OF SOLIDS REPULP WASHING

<u>Run A</u>	<u>Cycle No.</u>	<u>Repulp Liquor</u>	<u>Occluded U-238 In Solids, wt%</u>	<u>Cumulative U-238 Recovery In Supernate, %</u>
	1(1st pass)	-	1.5	99.63
	2(1st repulp)	15% HNO ₃	0.63	99.85
	3(2nd ")	15% HNO ₃	0.49	99.885
	4(3rd ")	Water	0.38	99.914
<u>Run B</u>	1(1st pass)	-	0.98	99.74
	2(1st repulp)	Water	0.85	99.78
	3(2nd ")	"	<0.2	>99.95
	4(3rd ")	"	<0.2	>99.95
	5(4th ")	"	<0.2	>99.95
	6(5th ")	"	<0.2	>99.95

Uranium recoveries in the supernate were calculated from the equation:

$$\% \text{ URANIUM RECOVERY} = 100 \left(1 - \frac{(\text{SE})^n M_s X_n}{M_u} \right)$$

Where:

SE = Separation efficiency, 0.97

n = Centrifuge cycle number (n = 1, 2, 3)
assuming 97% solids recovery each cycle.

M_s = Mass of solids initially in batch, g.

M_u = Mass of U-238 in batch (960g).

X_n = wt% U-238 in solids in the nth cycle.

The volume of each repulp slurry was 5 liters, 20% of a feed slurry volume. Water as repulp wash solution was more effective than a 15% nitric acid solution. Results show that greater than 99.5% uranium recoveries are possible after the initial separation. Subsequent repulping and centrifugation provided further uranium recoveries in excess of 99.9% after two or more repulp cycles. Initial cold Rover plant operation will determine the exact requirements of wash water volume and number of repulp cycles. However these requirements are not expected to exceed the requirements determined by this study.

The Rover flowsheet provides for 781 liters of complexed dissolver effluent to be processed per day. At feed and repulp slurry flows of 3 l/min, the centrifuge, which would be plant scale, will operate for approximately 8 hours per day, or 2.7 hours per 8-hour shift, assuming four repulp cycles at 20% of a feed slurry volume per cycle.

C. Results of Centrifuge Bowl Disassembly

After completion of the study using simulated Rover dissolver effluent, the bowl was washed repetitively by introducing water through the feed line during rotation and while at rest. The decontamination wash system (Figure 3) was used to rinse the outside of the bowl. A thin layer of solids remained on the inner bowl surface after rinsing. Essentially complete removal of solids should be possible, however, by feeding water or nitric acid into the bowl during reverse rotation, the remaining solids will exit with the liquid via the liquid effluent outlet.

The centrifuge bowl was disassembled after rinsing was completed. All equipment parts were cleaned, and a thorough inspection for corrosion and erosion of the equipment components was made. Comparison with photographs taken prior to the study indicated no visual damage to the bowl, conveyor, or bowl housing. Visual inspection, however, indicated that some abrasion of the chromium oxide coating may have occurred on the outer edges of the blades. Actual measurement of erosion was not possible because of the conveyor blade configuration.

D. Adaptability to Remote Operation

The continuous solid bowl centrifuge should conform satisfactorily to remote operation, since direct manual operation of any form would not be required. All controls and instrumentation for streams entering and leaving the centrifuge will be located outside Cell 2 of the Rover Fuels Reprocessing Facility. Variations in process conditions, however, may cause centrifuge operational problems. For example, plugging of solids in the feed line may be encountered if solids loading in the slurry is significantly greater than provided by the Standard Rover flowsheet.

VI. CONCLUSIONS AND RECOMMENDATIONS

The following conclusions were drawn from the continuous solid bowl centrifuge studies:

- 1) The solid bowl centrifuge is effective in separating greater than 99% of the undissolved solids from the simulated Rover dissolver effluent.
- 2) The most effective solids-liquid separation was attained at centrifuge rotation rates of 2400 and 3100 rpm (500 and 800 g's respectively).
- 3) The undissolved solids content of the centrifuge liquid effluent, under the conditions studied, should present no problems in the TBP extraction system.
- 4) The in-bowl solids wash system removes essentially no occluded uranium from the solids.
- 5) Greater than 99.9% of the uranium can be recovered by repulp washing with water in two or more cycles.
- 6) At feed and repulp slurry flows of 3 l/min, the centrifuge will operate approximately 8 hours per day or 2.7 hours per 8-hour shift, assuming four repulp cycles at 20% of a feed slurry volume per cycle.
- 7) Discharge of the solids-rich hold-up volume should present no problems in plant operation provided the downstream diverter valve is positioned to direct the liquor to the repulp tank.
- 8) A volume of wash water equivalent to 20% of the feed slurry volume was adequate for repulp washing. Initial cold Rover plant operation will determine the exact wash volume requirements; however, the volume ratio should not exceed the ratio tested in this study.
- 9) Essentially complete removal of solids from the bowl is possible by feeding water or nitric acid into the bowl during reverse rotation of the centrifuge bowl. The remaining solids will exit with the liquid via the liquid effluent outlet.
- 10) The continuous solid bowl centrifuge should operate under remote conditions satisfactorily.

Recommendations for the proposed centrifuge/repulp wash system are as follows:

- 1) For maximum solids-liquid separation efficiency and minimum required maintenance, the solid bowl centrifuge should be operated at a rotation rate of 2400 - 3100 rpm (500 - 800 g's).
- 2) The slurry feed rate should be maintained at 2 l/min or less to achieve separation efficiencies above 99%. Higher feed rates can be used if lower separation efficiencies are permissible.
- 3) A maximum of 4 solids repulp washing cycles should follow each separation from the rich liquor.
- 4) Discharged solids should fall vertically from the centrifuge to either a repulp tank, or after the last wash cycle, to a drier/collection vessel.
- 5) A water-spray system should be located in the solids discharge chute to aid the transport of wet solids to downstream vessels, if necessary.

VII. REFERENCES

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