

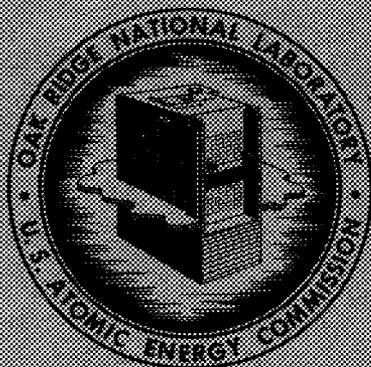
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SOLVENT EXTRACTION OF URANIUM
FROM REDUCTION SLAG SLURRY

A. D. Ryan
F. L. Daley



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CHEMICAL TECHNOLOGY DIVISION
Chemical Development Section C

SOLVENT EXTRACTION OF URANIUM
FROM REDUCTION SLAG SLURRY

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ABSTRACT

The chemical feasibility of the Dapex and Amex solvent extraction processes for recovery of uranium from sulfuric acid leach slurry (20 wt % solid) of magnesium reduction slag was demonstrated. Extraction isotherms show that only 3 to 4 theoretical stages are needed for good uranium recovery. Phase separation is rapid with virtually no emulsion if the mixing is controlled to disperse slurry in continuous solvent phase.

In continuous countercurrent tests with the Dapex process using 5 extraction stages and 3 strip stages uranium recovery was 99.8%, and the product contained <500 ppm of any one impurity. The solvent loss was less than 0.5 gal/1000 gal of slurry. The cost of chemicals for extraction, stripping, precipitation, and solvent loss was 13¢ per pound of uranium recovered.

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1.0 INTRODUCTION

Uranium was recovered from the slag resulting from magnesium reduction of UF_4 to metal, by modified Amex and Dapex processes. The slag consists of finely divided uranium metal and unreacted UF_4 dispersed in magnesium fluoride. In order to demonstrate the applicability of these processes to the recovery of uranium from slag slurry, it was necessary to test the effects of several factors which differed from the established technology developed for uranium mills. In addition to uranium extraction isotherms, the important factors were phase separation, reagent adsorption on slurry solids, and solvent loss. Counter-current tests were used to demonstrate one of several possible flowsheets.

One method for the recovery of uranium from this slag, in use at the Y-12 plant at Oak Ridge, Tennessee, uses sulfuric acid leach, followed by pH adjustment with sodium carbonate and filtration to prepare feed for the Higgins ion exchange column process.^{1,2}

As an alternative to ion exchange, solvent extraction was tested for recovery of uranium from the leach slurry without prior pH adjustment or filtration. The solvents considered were those used in the Amex and Dapex solvent extraction processes, both of which are used commercially for recovery of uranium from ore leach solutions in uranium mills. The Amex process^{3,4} uses certain long-chain amines dissolved in kerosene, which extract uranium by anion exchange analogous to resins. Uranium can be stripped from the solvent by bases such as sodium carbonate or magnesia, and acid chloride or nitrate salt solutions. The Dapex process^{4,5} uses di(2-ethylhexyl)phosphoric acid (D2EHPA) in kerosene, usually modified with tributyl phosphate (TBP) to prevent third phase formation and to enhance the uranium extraction coefficients. Uranium is usually stripped with solutions of sodium or ammonium carbonate.

2.0 EXTRACTION REAGENTS

Several batches of slag leach slurry (Table 1), obtained from the Y-12 plant, were used in the tests. The slurry contained 20 wt % insoluble solids and about 5% free sulfuric acid. The insoluble solids were nearly an equimolar mixture of calcium sulfate and magnesium fluoride. The major constituents in solution were uranium, magnesium, and sulfate.

Table 1. Slag Slurry Feed
~20 wt % solids; pH 0.4 to 0.7

Filtrate Analysis,		Solids Analysis,	
g/liter		%	
U	8.5	CaSO ₄	51
SO ₄	200	MgF ₂	42
Mg	50		
F	4		
Mn	0.7		
Fe	0.3		

2.1 Extraction Isotherms

Batch equilibration tests were made to study the effect of slurry pH in the range 0.4 to 0.7 and reagent molarity on the extraction coefficients. The organic reagents studied were a secondary amine (Rohm and Haas, LA-1), a tertiary amine (tri-iso-octyl) and di(2-ethylhexyl)phosphoric acid (D2EHPA). The extraction coefficients for each amine at a slurry pH of 0.4 were nearly the same while that for D2EHPA at a slurry pH of 0.4 was higher by a factor of 2 (Table 2). Raising the pH of the slurry from 0.4 to 0.7 increased the extraction coefficient 2- to 3-fold for both the 0.1 M Rohm and Haas LA-1 and the 0.1 M D2EHPA (Table 2).

Isotherms for the 0.1 M solutions of D2EHPA (3% TBP) and the amines at a slurry pH of 0.4 and 0.7 are plotted in Fig. 1. The uranium concentrations in the slurry at equilibrium with solvent uranium loadings of 0.1, 2.0, and 4.0 g/liter were used to calculate the extraction coefficient (E_{2}^{0}) for each extractant.

Table 2. Uranium Extraction Coefficients for 0.1 M Reagents

Solvent	Slurry pH	Extraction Coefficient (E_{2}^{0})		
		0.1 g U per liter of solvent	2.0 g U per liter of solvent	4.0 g U per liter of solvent
Tri-iso-octylamine, 5% tridecyl alcohol	0.4	10	6.8	1.8
	0.7	23	13	2.7
Rohm and Haas LA-1, 3% tridecyl alcohol	0.4	10	6.8	1.8
	0.7	23	13	2.7
D2EHPA, 3% TBP	0.4	18	11	2.7
	0.7	53	24	8.0

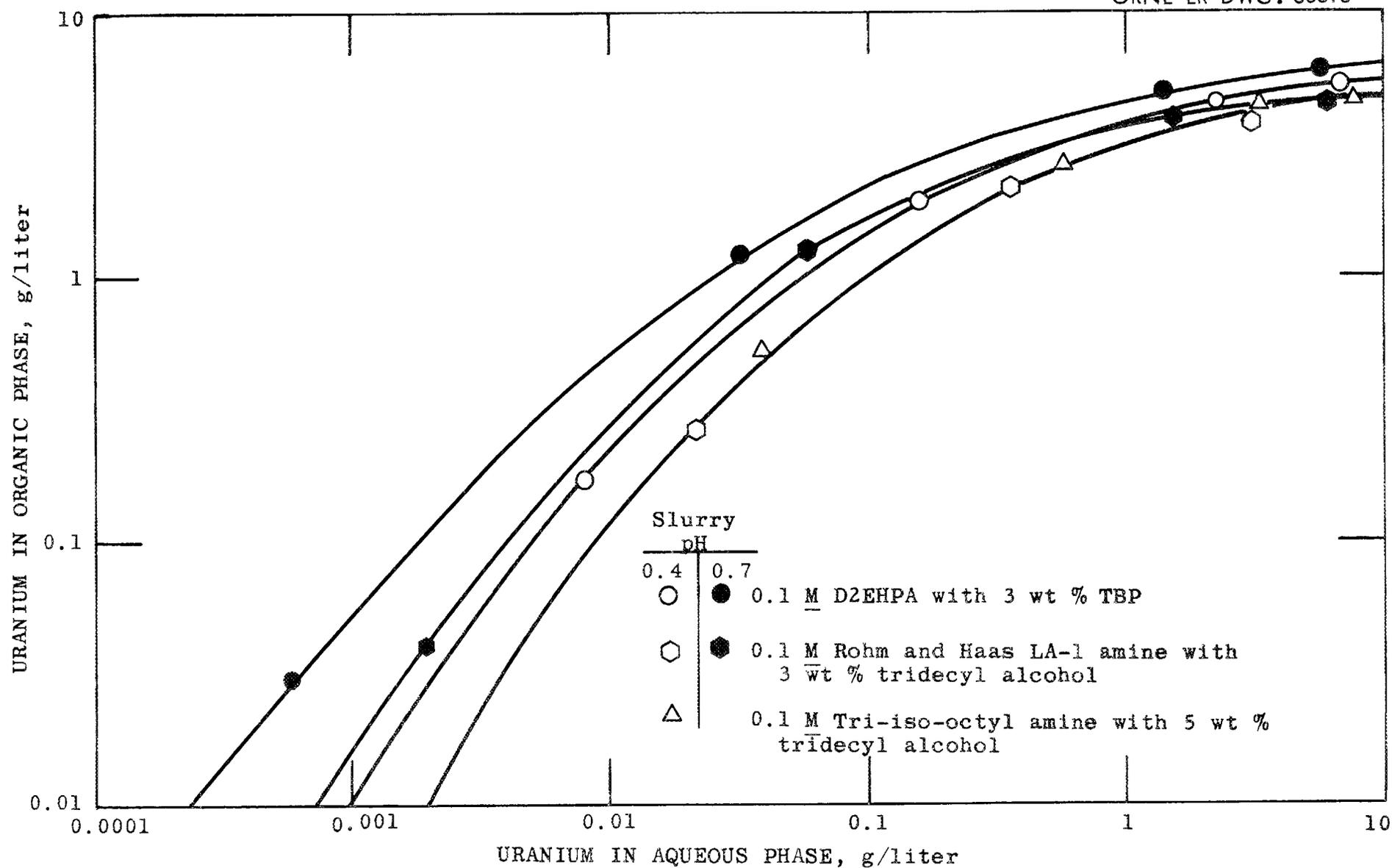


Fig. 1. Uranium extraction isotherms.

The isotherm for 0.24 M D2EHPA (5% TBP) is plotted in Fig. 2. Extraction coefficients were calculated at the same mole ratio of uranium to extractant as was used for 0.1 M D2EHPA to show the relative effect of reagent concentration on extraction. The increase in the extraction coefficient for the 0.24 M D2EHPA over that found for the 0.1 M D2EHPA approximates the increase expected, that is, by the square of the concentration of the uncomplexed reagent⁶ (Table 3).

Table 3. Effect of Extractant Molarity on Uranium Extraction
Slurry pH 0.7

0.1 M D2EHPA, 3% TBP		0.24 M D2EHPA, 5% TBP	
Solvent Uranium Loading, g/liter	E_a^0	Solvent Uranium Loading, g/liter	E_a^0
0.1	53	0.24	390
2.0	24	4.8	77
4.0	8.0	9.6	17

The number of theoretical stages required for 99.9% extraction of uranium with each solvent at a uranium loading 80% of maximum is shown in Table 4. An example of the graphical determination of stages is shown in Fig. 2. Although the uranium loading varied from 3.8 to 5.0 g/liter for 0.1 M solvents, the number of stages for all solvents tested was in the range 2.7 to 3.6. The pH of the slurry had more effect than either type or concentration of extractant.

Table 4. Theoretical Stage Requirements

Solvent	Uranium Conc.* in Solvent, g/liter	Slurry pH	No. of Stages
0.1 M Rohm and Haas, LA-1, 3% tridecyl alcohol	3.8	0.4	3.6
	3.8	0.7	2.9
0.1 M D2EHPA, 3% TBP	4.3	0.4	3.0
	5.0	0.7	2.8
0.24 M D2EHPA, 5% TBP	12.0	0.7	2.7

*Loading of 80% of maximum in equilibrium with slurry.

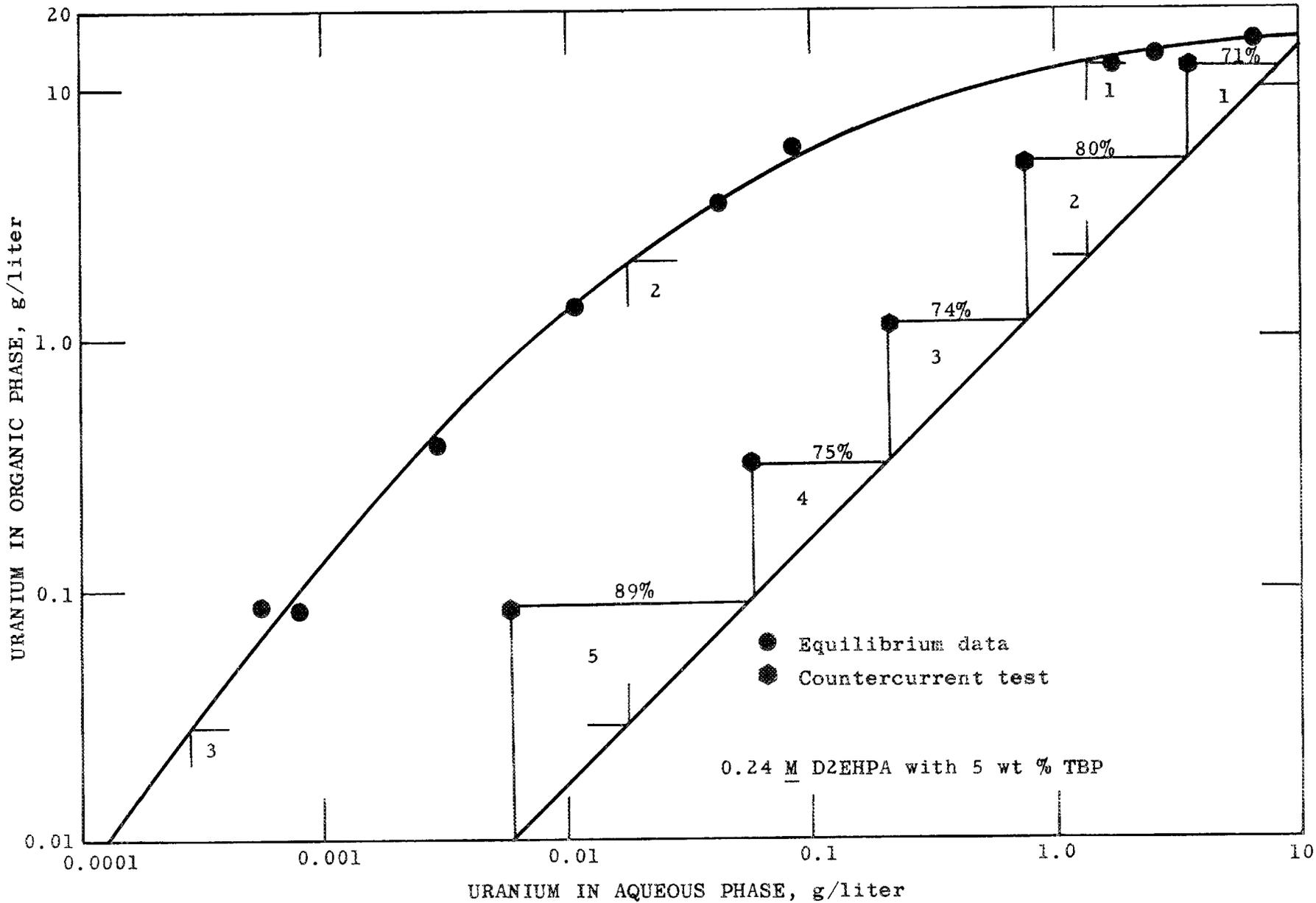


Fig. 2. McCabe-Thiele diagram.

2.2 Reagent Adsorption

The adsorption of amine and D2EHPA on slurry solids was negligible (Table 5). Past experience with TBP extraction in other slurry systems has shown no serious loss. The amine and D2EHPA adsorption was tested by contacting 1 vol each of the ~0.1 M organic solutions with 900 vol of slurry for 45 min. The loss of reagent from the solvent phase was determined from analyses made before and after contact.

Table 5. Adsorption of Extractants by Slurry

Organic Extractant	Molarity	
	Before Contact	After Contact
Tri-iso-octylamine	0.090	0.091
D2EHPA	0.104	0.105

2.3 Extraction Rate

Extraction rate constants (Table 6) show that the rate of uranium extraction with amines is about one order of magnitude greater than that with D2EHPA. Comparison with the rates obtained with clarified western leach liquors⁸ shows that the rates with both solvents are significantly lower with slurry, probably because less interfacial area is produced with slurry at the same power input. However, the lower rate is not a serious problem. The extraction rates were determined batchwise in a 6-in. baffled tank mixer.⁷ The slurry was dispersed in continuous solvent phase at an a/o ratio of 1 to 5.

The residence time required for 80% stage efficiency of a continuous flow mixer, calculated from the batch rate constants,⁹ is 2.5 min for Dapex at a power input of 67 hp/1000 gal and 0.4 min for Amex at 20 hp/1000 gal.

Table 6. Uranium Extraction Rate

Solvent	Mixer Speed, rpm	Power Input, hp/1000 gal	Rate
			Constant, min ⁻¹
0.1 M D2EHPA, 3% TBP	350	6	0.128
	520	20	0.169
	790	67	0.258
0.1 M Rohm and Haas LA-1, 3% tridecyl alcohol	520	20	1.92

2.4 Comparison of Reagents

The choice between the Dapex and Amex processes for application to slag slurry is not clearcut. The main advantages of Dapex are: (1) uranium extraction is by cation exchange which permits good decontamination from sulfate, while in the Amex process extraction is by sulfate anion complex similar to anion exchange resins; (2) the extraction coefficients are higher than those for the amines tested. The main advantages of Amex are: (1) amines are more selective for uranium and permit better separation from other metals, particularly iron(III); (2) the rate of extraction with amines is rapid, which would allow more freedom in choice of type of contactor best suited for slurry extraction.

3.0 PHASE SEPARATION

3.1 Batch Tests

Because of the importance of primary phase separation on continuous operation, batch tests were made on samples of 12 different batches of slurry to study the effect of phase ratio and continuous phase mixing on primary phase break time. In batch tests there was virtually no permanent emulsion formation. For both Dapex and Amex type solvents with aqueous-continuous mixing break times were approximately 5 min while with solvent-continuous mixing in each case break times were 0.5 to 1 min. A break time of 5 min is too long for practical use in a countercurrent mixer-settler system; therefore solvent-continuous mixing should be used. Both aqueous-continuous and solvent-continuous mixing at a/o phase ratios of 1/1 and 1/5 were tested in a 3-in. baffled tank mixer. Solvent-continuous mixing could not be maintained with either solvent at the 1/1 phase ratio, and the ratio had to be decreased to 1/5.

The characteristics of the slurry feed, observed in the phase break tests, indicated that a contactor with a low aqueous/solvent ratio was needed to ensure proper mixing conditions (solvent continuous). The operation of a conventional mixer-settler under these conditions necessitates a forced external solvent recycle for each unit, and two types of contactors, a mixer column and a Turbo mixer-settler, designed for a low aqueous/solvent ratio, were tested for the extraction of uranium in a countercurrent system. The Turbo mixer-settler was the most successful.

3.2 Mixer Column

A 4-in. 10-compartment mixer column¹⁰ was tested to find the effect of impeller speed, slurry throughput, horizontal baffle hold size, and impeller location on the slurry retention time in the column. The slurry was introduced into the top of the column countercurrently to the solvent at a set impeller speed and the column was allowed to come to steady state. The flows were then shut down and the slurry holdup was measured. The slurry retention time in the column remained virtually constant (2.4 to 2.8 min) over the range of impeller speeds, 500 to 700 rpm, and slurry throughput of 64 to 440 ml/min (Table 7). The location of the impeller in the compartments or size of the hole in the horizontal baffle (1 or 2 in.) seemed to have little effect on the slurry retention time. Increasing the impeller speed above 700 rpm or the slurry throughput over 440 ml/min caused the mixing to invert to aqueous continuous, which flooded the column. The maximum slurry throughput was equivalent to 1.4 gal/min per square foot of column area.

Table 7. Mixer Column Holdup Tests

Aqueous feed: 20 wt % slurry
 Solvent feed: D2EHPA, 3% TBP
 Flow ratio, a/o: 1/1.5
 Compartments (10): 2 x 4 in.
 Impellers (10): 2 in., 4-bladed turbine

Impeller Speed, rpm	Aqueous Flow, ml/min	Aqueous Holdup, ml	Aqueous Retention Time, min
1-in. holes in horizontal baffles			
500	64	176	2.8
700	100	264	2.6
2-in. holes in horizontal baffles			
700	200	500	2.5
700	440	1060	2.4
700	500	Phase inversion	
1200	300	Phase inversion	

The stage efficiency of the mixer column was measured under the operating conditions (Table 7) which gave the greatest slurry residence time (2.8 min). At steady state the uranium content of the raffinate was 2.5 g/liter, which showed that the entire column was equivalent to one theoretical stage or that the average efficiency of each compartment was about 10%. The low efficiency is due to

inadequate residence time and backmixing of solvent in the column.

3.3 Turbo-Mixer

A commercial Turbo mixer-settler,* experimental model, was tested as a single-stage extractor to determine the effect of impeller speed and interface level on slurry retention time. The residence time of slurry was virtually independent of the impeller speed in the range 650 to 950 rpm and interface level ranging from 12 to 18 in. below the impeller (Table 8). Phase inversion to aqueous-continuous occurred if the interface level was less than 10 in. below the impeller. Although the maximum flow capacity was not determined, the flow rate of 1.1 gal/min per square foot of settler area was established, which provided sufficient residence time for good extraction efficiency.

Table 8. Turbo-Mixer Tests

Impeller Speed, rpm	Interface Level below Impeller, in.	Slurry Holdup, liters	Slurry Residence Time, min
650	12	7.5	2.3
	14.5	7.0	2.2
	16	7.4	2.3
	18	7.5	2.3
750	14.5	7.5	2.3
850	15	8.0	2.5
950	16	9.0	2.8

The Turbo mixer-settler consisted of a 4-in. aerator impeller hood ring assembly suspended in a 12-in.-dia by 27-in.-deep tank. The tank was filled with solvent so that the impeller was submerged about 6 in. Slurry feed was metered directly into the mixer chamber at a constant flow rate (3.2 liters/min).

4.0 CONTINUOUS COUNTERCURRENT TESTS

The Dapex process (Fig. 3, Table 9) was used to demonstrate recovery of uranium from leach slurry (Table 1). The

*Manufactured by Turbo Mixer, a division of General American Transportation Corporation.

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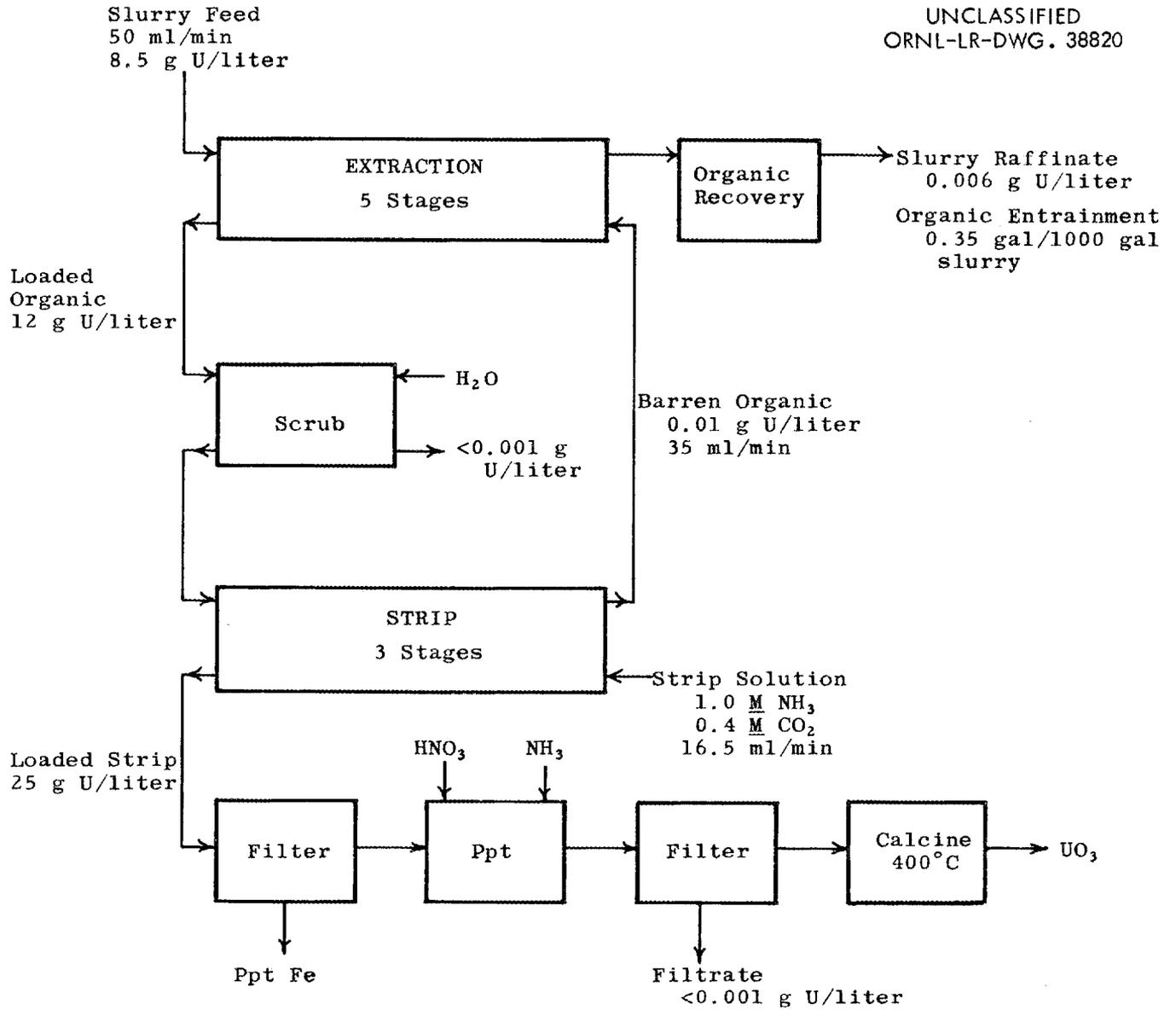


Fig. 3. Flowsheet for uranium recovery from slag slurry by Dapex process.

Table 9. Operating Conditions for Continuous
Countercurrent Tests

Solvent: 0.24 M D2EHPA in kerosene with 5% TBP
Aqueous: 20 wt % slurry, 8.5 g of uranium per liter, pH 0.7
Strip solution: ammonium carbonate, 1.0 M NH_3 , 0.4 M CO_2
Scrub solution: water

Extraction Stages (5)

Feed ratio, a/o	1.4
Residence time in mixer	2-3 min
Stirrer speed	700-800 rpm

Stripping Stages (3)

Feed ratio, a/o	0.5
Residence time in mixer	7 min
Stirrer speed	700-800 rpm

Scrub Stage (1)

Feed ratio, a/o	0.5 - with aqueous recycle
Residence time in mixer	7 min
Stirrer speed	700-800 rpm

uranium was stripped from the solvent with ammonium carbonate¹¹ to obtain a sodium-free product. The extractant was 0.24 M D2EHPA in kerosene containing 5% TBP. The extraction section consisted of five Turbo-mixer stages (details in Fig. 4) and the strip, three mixer-settler stages. An optional water scrub stage was installed to remove entrainment from the loaded solvent before stripping. The interstage solvent flow was by gravity and the aqueous flow was pumped. Interface levels were maintained by adjusting the pumping rates of the interstage aqueous pumps. Mixing was controlled to maintain organic-continuous dispersions throughout the system.

The flow rates of slurry feed, solvent, water scrub, and ammonium carbonate strip were 50, 35, 2, and 16.5 ml/min, respectively. The flow ratio in the extraction section was selected so that the uranium loading in the solvent was about 80% of the equilibrium value possible with the uranium concentration in the slurry feed. Uranium was stripped from the solvent with an ammonium carbonate solution (1.0 M NH_3 , 0.4 M CO_2). The flow rate of the stripping solution was adjusted to provide 10% excess of the stoichiometric requirement for NH_3 .

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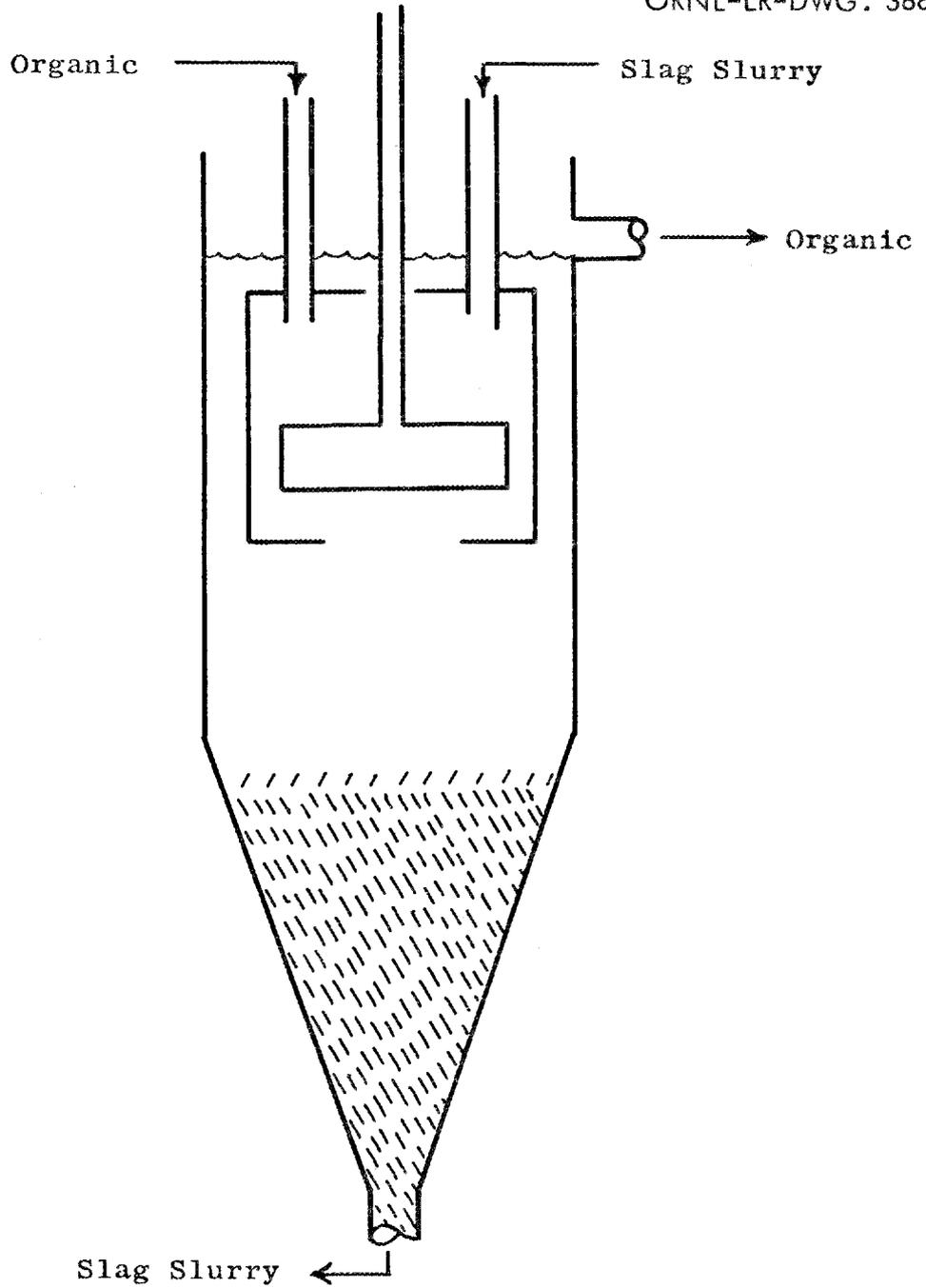


Fig. 4. Turbo mixer-settler for countercurrent tests.

4.1 Uranium Recovery

Analyses of composite samples taken during 14 hr of operation (Table 10) show that steady state was attained in less than 4 hr. The average uranium concentration in the slurry raffinate was 0.016 g/liter, representing 99.8% uranium recovery. The stage efficiency of each Turbo-mixer in the extraction section was calculated from the uranium profile data shown in Table 11 and Fig. 2. Stage efficiency, expressed as Murphree efficiency based on the uranium concentration in the aqueous phase, ranged from 71 to 89%. The average was about 80%, which is in good agreement with the predicted efficiency from the batch rate measurements.

Table 10. Analysis of Composite Samples

Time, hr	Uranium, g/liter		Solvent Entrainment, gal/1000 gal slurry
	Strip Solution	Slurry Raffinate	
2	22.5	0.016	-
4	24.8	0.029	0.39
6	25.5	0.017	0.38
8	25.4	0.013	0.34
10	24.2	0.017	0.43
12	24.6	0.014	0.26
14	25.8	0.006	0.30
Avg.	24.7	0.016	0.35

Table 11. Uranium Profile

Stage No.	U Conc., g/liter	
	Aqueous	Organic
Extraction Section, a/o = 1.43		
1	3.60	12.3
2	0.78	5.0
3	0.216	1.14
4	0.057	0.31
5	0.006	0.082
Water Scrub	<0.001	11.8
Strip Section, a/o = 0.47		
1	24.7	10.0
2	18.6	0.182
3	0.46	0.009

4.2 Uranium Purity

The loaded solvent was scrubbed with water during part of the run to determine the effect of aqueous entrainment removal on product purity. The product analyses (Table 12) show that the water scrub decreased the amount of most impurities to <500 ppm. The major impurities were sulfur, silicon, phosphorus, iron, and calcium. Decontamination factors for the major impurities in the slurry feed, magnesium and sulfate, were greater than 10,000. The loss of uranium to the water scrub was negligible (<0.001 g/liter).

Table 12. Product Analyses

Element	Amount in Product, ppm	
	No Water Scrub	Water Scrub
S	650	<160
F	-	<500
Be	<0.1	<0.1
Ni	6	1.0
Si	450	200
P	500	250
Li	<2	<2
Na	25	20
Mn	6	<1
Mg	230	30
Fe	650	100
Cu	6	5
Cr	17	<2
Ca	1000	90
B	12	1.2
Al	7	4
Cd	0.2	0.1
Co	<1	<1
V	5	<1

The uranium was effectively stripped (99.9%) from the loaded solvent in three stages with 10% excess ammonium carbonate. The loaded strip solution contained 25 g of uranium per liter and a small amount of precipitate, which was mainly ferric hydroxide. The precipitate was filtered off, the carbonate destroyed with nitric acid, and the uranium precipitated with ammonia. The product was calcined at 400°C to UO₃.

4.3 Solvent Loss

Phase separation throughout the system was satisfactory. The entrainment of solvent in the slurry raffinate from the last extraction stage was equivalent to 4 gal/1000 gal of slurry. Most of the solvent was recovered in the solvent recovery tank, which was agitated with a low speed rate and provided a liquid residence time of 1 hr. The average entrainment loss in the discarded raffinate (Table 10) was 0.35 gal/1000 gal. About 1/3 of the solvent collected in the recovery tank was in the form of an emulsion, which separated on standing overnight or upon filtration. The emulsion was apparently stabilized by black solids which were mainly manganese, magnesium, and silicon. Batch phase separation tests demonstrated that both MnO₂ and SiO₂ stabilize oil-in-water type emulsions. The difficulty caused by MnO₂ could be eliminated by using some other oxidant in the leaching step.

4.4 Chemical Consumption

The cost of chemicals consumed for extraction, stripping, and precipitation of uranium was 13¢ per pound of uranium recovered (Table 13). The amounts of each chemical used in determining the cost were those actually used in the countercurrent tests. The cost of nitric acid for destruction of carbonate is the largest single item; use of sulfuric acid would reduce costs with an increase in sulfur content of the uranium product, which would necessitate a higher calcination temperature. The cost of solvent is based on the final loss in the discarded raffinate (0.35 gal/1000 gal). If the solvent recovery tank is not used the solvent cost would be about 7¢ per pound of uranium recovered.

Table 13. Chemical Consumption Cost

	Reagent	Cost/lb	Lb/lb U	Cost/lb U
Strip	NH ₃	4¢	0.74	3.0¢
	CO ₂	4	0.76	3.0
Precipitation	HNO ₃	3	1.8	5.4
	NH ₃	4	0.22	0.9
Organic Loss (0.35 gal/1000 gal slurry)		15	0.04	<u>0.6</u>
				12.9¢

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