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ABSTRACT

A method was developed for recovering uranium from amines which yields a liquid (uranyl nitrate) concentrate rather than a solid concentrate for shipment to the refinery. This procedure offers potential cost savings by simplifying the overall uranium mill—refinery flowsheet. The process involves treatment of the amine extract with calcium nitrate solution to convert the uranium in the solvent to the nitrate complex, stripping the uranium with water or dilute nitric acid, and recovering the nitrate from the solvent for recycle by contact with a lime slurry. Reagent costs for the stripping process were estimated at 4.9¢ per pound of U_3O_8.

CONTENTS

1.0 INTRODUCTION

This report describes development of a stripping method for the amine extraction (Amex) process,[1-3] which produces a concentrated uranyl nitrate solution instead of the conventional solid concentrate. Shipment of a solution in tank cars to the refinery offers potential cost savings since it would eliminate the product calcining and packaging operations at the mill and the nitric acid dissolution step at the refinery. Dust problems associated with producing and handling a solid concentrate would also be eliminated, and sampling and analytical problems, both at mill and refinery, would be simplified. In addition, the relatively pure uranyl nitrate solution would be a convenient starting material for production of uranium dioxide at the mill site, either directly or after further purification in a TBP cycle.

After preliminary investigation of the process in batch tests, continuous runs were made in a bench-scale mixer-settler test array with synthetic ore leach liquors. Reagent costs were estimated on the basis of the continuous tests.

Analyses were performed by the Y-12 section of the ORNL Analytical Chemistry Division under the direction of C. D. Susano.

2.0 PROPOSED PROCESS

The proposed stripping process (Fig. 2.1) takes advantage of the fact that tertiary amines extract uranium effectively from highly salted nitrate solutions.[4] The uranium-loaded amine from the sulfate extraction system is contacted with a calcium nitrate solution which strips sulfate from the solvent, simultaneously precipitating it as calcium sulfate. The calcium nitrate solution, containing some uranium, is recycled after removal of the calcium sulfate by decantation and filtration. Uranium, now held in the solvent as an amine nitrate—uranyl nitrate complex, is readily stripped with water or dilute nitric acid. Following uranium stripping, nitrate is recovered as calcium nitrate for recycle to the sulfate stripping step and free amine is regenerated by contacting the amine nitrate with a lime slurry.

3.0 STRIPPING OF SULFATE

Contacting the solvent containing the amine sulfate—uranyl sulfate complex (and excess amine sulfate) with calcium nitrate (2-4 $\underline{M}$) solution strips sulfate from the solvent, simultaneously precipitating it as calcium sulfate, while most

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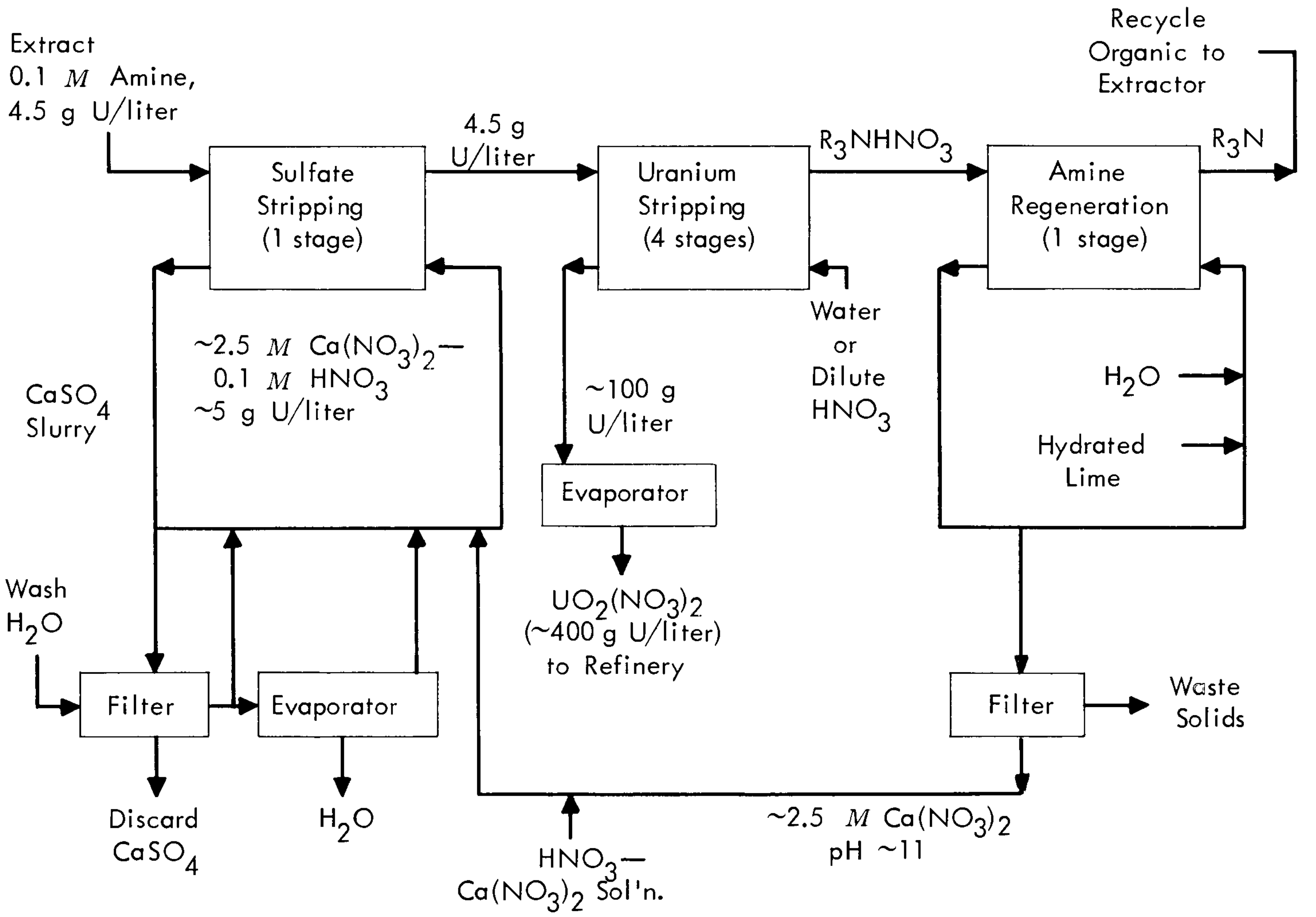

Fig. 2.1. Stripping uranium from tertiary amines by the high nitrate--water stripping method.

of the uranium remains in the solvent phase as an amine nitrate—uranyl nitrate complex:

$$\underset{\cdots\cdots\cdots\cdots\cdots\cdots}{(R_3NH)_2UO_2(SO_4)_2^*} + 2Ca(NO_3)_2 \longrightarrow \underset{\cdots\cdots\cdots\cdots\cdots\cdots}{(R_3NH)_2UO_2(NO_3)_4} + 2\underline{CaSO_4} \qquad (1)$$

and

$$\underset{\cdots\cdots\cdots\cdots}{(R_3NH)_2SO_4} + Ca(NO_3)_2 \longrightarrow \underset{\cdots\cdots\cdots}{2R_3NHNO_3} + \underline{CaSO_4} \qquad (2)$$

(the dotted underlines mark species in the organic phase). The distribution of uranium between the phases for a given amine depends primarily on the nitrate concentration (Sec. 3.2). In the process flowsheet (Fig. 2.1) the calcium nitrate solution is recycled in a closed circuit so that the net transfer of uranium between phases in the sulfate stripping step is small.

3.1 Sulfate Stripping Rate

In batch mixing tests sulfate was completely stripped from an Alamine 336 extract by contact with one-fourth its volume of 2.5 M $Ca(NO_3)_2$—0.1 M HNO_3 solution containing 5 g of uranium per liter for 1 min:

Contact Time, min	Analysis of Organic, g/liter	
	U	SO_4
1	4.0	<0.02
5	4.0	<0.02
10	4.0	<0.02
20	4.1	<0.02

Mixing, which was organic-continuous at the start of the test, reverted to aqueous-continuous within the first minute. However, the phases separated cleanly in <2 min, with the precipitated calcium sulfate settling fairly rapidly in the aqueous phase. The above test was with 0.1 M Alamine 336 in Amsco 125-82** + 8 vol % tridecanol, loaded to 4.3 g of uranium per liter from a sulfate solution. In a second test with the same solutions and a phase ratio of 1/1, results were essentially the same.

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*The amine salt-uranyl salt complexes shown here and in subsequent (Sec. 4.0) equations are not meant to represent the actual uranium complex species in the organic phase, but are used only for simplicity of presentation. The choice is not important to the discussion since any other formulation leads to an equation equivalent to the combination of eqs. 1 and 2, so that the stoichiometric quantities of stripping reagent calculated would be identical.

**Refined aliphatic petroleum fraction.

Another series of batch mixing tests with Alamine 336 showed rapid and clean separation in the sulfate stripping step regardless of whether mixing was organic-continuous or aqueous-continuous.

3.2 Effect of Nitrate Concentration on Uranium Distribution

Isotherms (Fig. 3.1) for extraction of uranium from 1.5-4 M calcium nitrate solutions with 0.1 M Alamine 336 in kerosene-tridecanol diluent show the uranium distribution between the aqueous and organic phases that may be expected during the sulfate stripping step as a function of the aqueous nitrate concentration. Assuming a solvent uranium loading of 4.5 g/liter during extraction, the uranium concentration in the calcium nitrate recycle solution at steady-state, i.e., the same concentration of uranium in the solvent entering and leaving the sulfate stripping system, would be approximately >8, 7.5, 5, 1.3, 0.6, and 0.2 g/liter for 1.5, 2.0, 2.5, 3.0 3.5, and 4.0 M $Ca(NO_3)_2$ solutions, respectively. Tridecanol requirements for avoiding separation of a third liquid phase (owing to limited miscibility of the amine nitrate—uranyl nitrate complex in kerosene) increased with increasing calcium nitrate concentration and increased uranium loading of the solvent. In contact with 1.5-2.5 M $Ca(NO_3)_2$ solutions, 5 vol % was sufficient to avoid third-phase formation in all tests. With 3-4 M $Ca(NO_3)_2$, a third liquid phase appeared at high uranium loadings when the solvent contained 5 vol % tridecanol but disappeared when the tridecanol was increased to 8 vol %. Comparison of extraction isotherms from 2.5 M $Ca(NO_3)_2$ showed slightly more favorable uranium distribution when the solvent contained 5 vol % (curve 3) than with 8 vol % tridecanol (curve 4). Extraction results from 2.5 M $Ca(NO_3)_2$ solution with another tertiary amine, XE-204 (didodecenyl-n-butylamine), were approximately the same as for Alamine 336. In contrast, a secondary amine, di(tridecyl), gave poor extraction results even from 3-4 M $Ca(NO_3)_2$. Uranium loadings of the 0.1 M Alamine 336 solutions from 3-4 M $Ca(NO_3)_2$ were higher* than is possible from sulfate solutions. For example, from 4 M $Ca(NO_3)_2$ solution, the solvent became loaded to ~10.5 g of uranium per liter as compared to 5-6 g/liter in a sulfate system. A loading of 10.5 g of uranium per liter is equivalent to approximately 1 mole of uranium per 2.3 moles of amine.

In general, the above extraction data show that, when using tertiary amines, the choice of calcium nitrate concentration for the sulfate stripping step is not critical in the range 2-4 M, since the concentration of uranium in the recycle solution would be of reasonable magnitude, i.e., <10 g/liter.

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*Extractions with kerosene + 8 vol % tridecanol (no amine) showed that a small fraction of this increased uranium capacity can be attributed to extraction of uranium by tridecanol.

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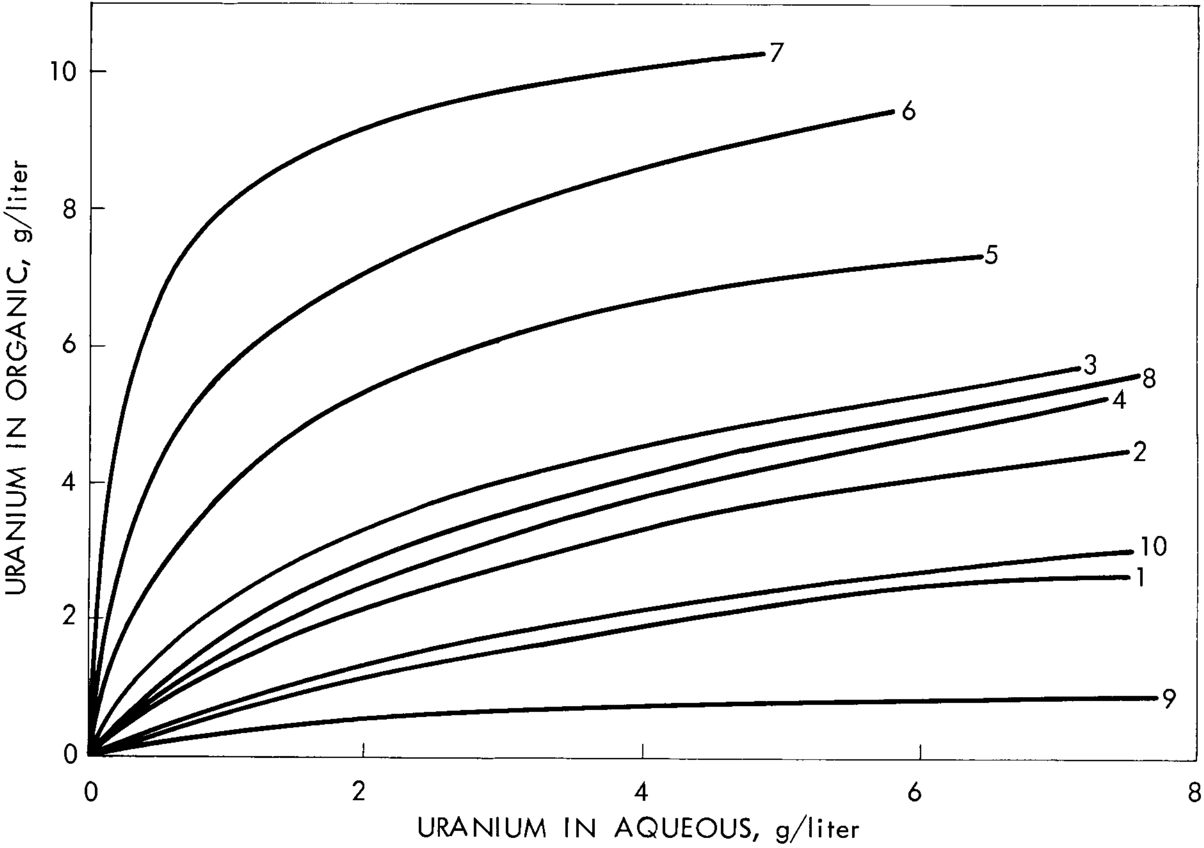

Fig. 3.1. Extraction of uranium from calcium nitrate solutions with 0.1 *M* solutions of amine nitrate in kerosene-tridecanol diluent. Head aqueous: 0.1 *M* HNO_3, 10 g U/liter as $UO_2(NO_3)_2$, $Ca(NO_3)_2$ as indicated.

Curve	Amine	Tridecanol, vol %	$Ca(NO_3)_2$, *M*
1	Alamine 336	5	1.5
2		5	2.0
3		5	2.5
4		8	2.5
5		8	3.0
6		8	3.5
7		8	4.0
8	XE-204	6	2.5
9	Di(tridecyl)	3	3.0
10		3	4.0

From the standpoint of physical operation of the sulfate stripping and amine regeneration systems, it is an advantage to operate in the lower portion of this range since the recycle solution is less viscous and the slurry solids settle more rapidly.

Effect of pH. At constant nitrate concentration (5.2 M) the uranium extraction coefficient from calcium nitrate solution increased with increase in pH from 0.0 to 2.8 but then fell off as the pH was increased to 3.4 (Table 3.1). A decrease in extraction efficiency at higher pH's would be expected since the tertiary amines are weak bases.[5,6] These data show that control of pH in the sulfate stripping step is not critical in the range 0.0-2.5, since the amount of uranium in the recycle solution, even at the lowest pH, would be tolerably low, provided the nitrate concentration is near 5 M or higher.

Table 3.1. Effect of pH on Uranium Distribution

Organic: 0.1 M XE-204 amine nitrate in kerosene + 6 vol % tridecanol

Aqueous: $Ca(NO_3)_2$-$UO_2(NO_3)_2$-HNO_3 solutions containing 9.2 g U/liter and 5.2 M total nitrate

Phase ratio, a/o: 1/1

Contact time: 10 min

Final pH	Uranium Analysis, g/liter Organic	Uranium Analysis, g/liter Aqueous	Uranium Extraction Coefficient (E_a^o)
0.0	4.4	4.7	0.9
0.5	4.9	4.0	1.2
0.7	5.2	4.2	1.2
1.1	5.6	3.7	1.5
2.8	6.0	3.4	1.8
3.4	4.6	4.1	1.1

4.0 STRIPPING OF URANIUM

Since extraction of uranium with amines from nitrate solutions is effective only with relatively high nitrate concentrations (4-8 M) in the aqueous phase (Sec. 3.2), uranyl nitrate is readily stripped from the solvent by contact with water or dilute nitric acid,

$$\underset{\cdots\cdots\cdots\cdots\cdots}{(R_3NH)_2UO_2(NO_3)_4} + H_2O \rightleftharpoons \underset{\cdots\cdots}{2R_3NHNO_3} + UO_2(NO_3)_2 + H_2O \qquad (3)$$

An isotherm (Fig. 4.1) for stripping uranium with 0.1 M HNO_3 from 0.1 M Alamine 336 nitrate showed that strip solution loadings of 100 g of uranium per liter or better could be expected.

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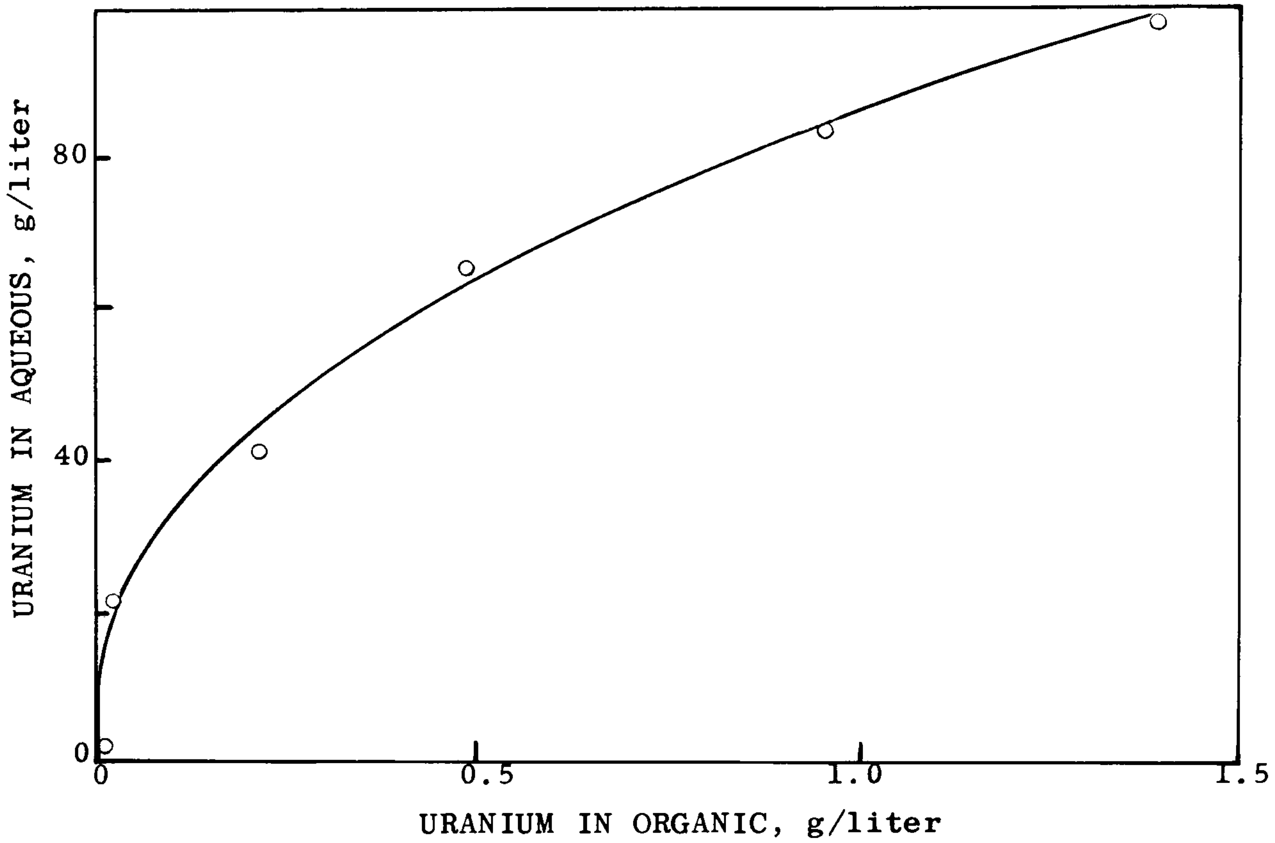

Fig. 4.1. Uranium stripping isotherm. Organic: 0.1 $\underline{M}$ Alamine 336 in Amsco 125-82+8 vol % tridecanol loaded to 4.3 g U/liter from a calcium nitrate—uranyl nitrate solution. Aqueous: 0.1 $\underline{M}$ HNO_3.

5.0 AMINE REGENERATION

The amine leaving the uranium stripping system is in the form of its nitrate salt. To recover the nitrate for reuse and to prevent interference by nitrate with uranium extraction, the amine is regenerated to the free base form with lime before it is recycled to the extraction system:

$$\underset{\cdots\cdots}{2R_3NHNO_3} + Ca(OH)_2 \longrightarrow \underset{\cdots}{2R_3N} + Ca(NO_3)_2 + 2H_2O \qquad (4)$$

Unlike the sulfate stripping system (Sec. 3.1), where control of mixing conditions was not important, phase separation in the amine regeneration system was poor unless mixing was organic-continuous. With aqueous-continuous mixing, a large fraction of the lime slurry solids collected in the solvent phase and at the aqueous-organic interface.

Continuous Tests. The feasibility of recovering nitrate from the amine as calcium nitrate was investigated in continuous equipment (Fig. 5.1) having a single mixer-settler stage for contacting the amine nitrate with a lime slurry. The settler had a conical bottom to ensure proper discharge of the slurry solids and was equipped with a slowly turning stirrer to facilitate disengagement of organic from the slurry. Organic feed for the regeneration system was provided by a continuous two-stage extractor in which 0.1 M Alamine 336 was converted to the nitrate form by countercurrent contact with dilute nitric acid.

In preliminary shake-down runs with 0.1 M Alamine 336 in kerosene + 8 vol % tridecanol diluent, the regeneration efficiency was poor and the operation was plagued by accumulation of solids and an aqueous-continuous emulsion at the settler interface. It was found that the hydrated lime being fed to the system contained ~33% carbonate, which probably accounted for the poor efficiency. A number of samples of commercial hydrated lime were obtained which contained >95% $Ca(OH)_2$ and <2% CO_3 and had high reactivity. With one of these lime samples, regeneration of the amine was consistently >95% in several continuous runs but phase separation problems were still serious. The organic in the extraction system turned deep yellow, particularly on standing during shutdown. A major portion of the yellow color was transferred to the aqueous phase during regeneration. Brief batch studies of the system showed that (1) no degradation of amine was occurring, (2) the yellow color was due to nitration of some of the kerosene constituents, and (3) the nitrated solvent had a strong tendency to form semipermanent emulsions. It was concluded that the emulsion difficulties previously encountered probably had been intensified by the degradation of the kerosene diluent, and subsequent runs were made with Amsco 125-82 (refined kerosene) diluent which showed no apparent degradation on contact with nitric acid.

A 12-hr run was then made with the operating conditions shown in Table 5.1. The calcium hydroxide content of the recycle slurry was ~20 g/liter at startup and was maintained in the range 10-20 g/liter by intermittent addition of hydrated lime. The water feed rate and calcium nitrate solution takeoff were set at ~1 ml/min, which resulted in production of a product solution approximately 2 M in $Ca(NO_3)_2$. Regeneration was >95% throughout the run and physical operation of the system was satisfactory. A small amount of solids accumulated at the settler interface but the rate of accumulation was very slow and this did not seem to represent a significant problem.

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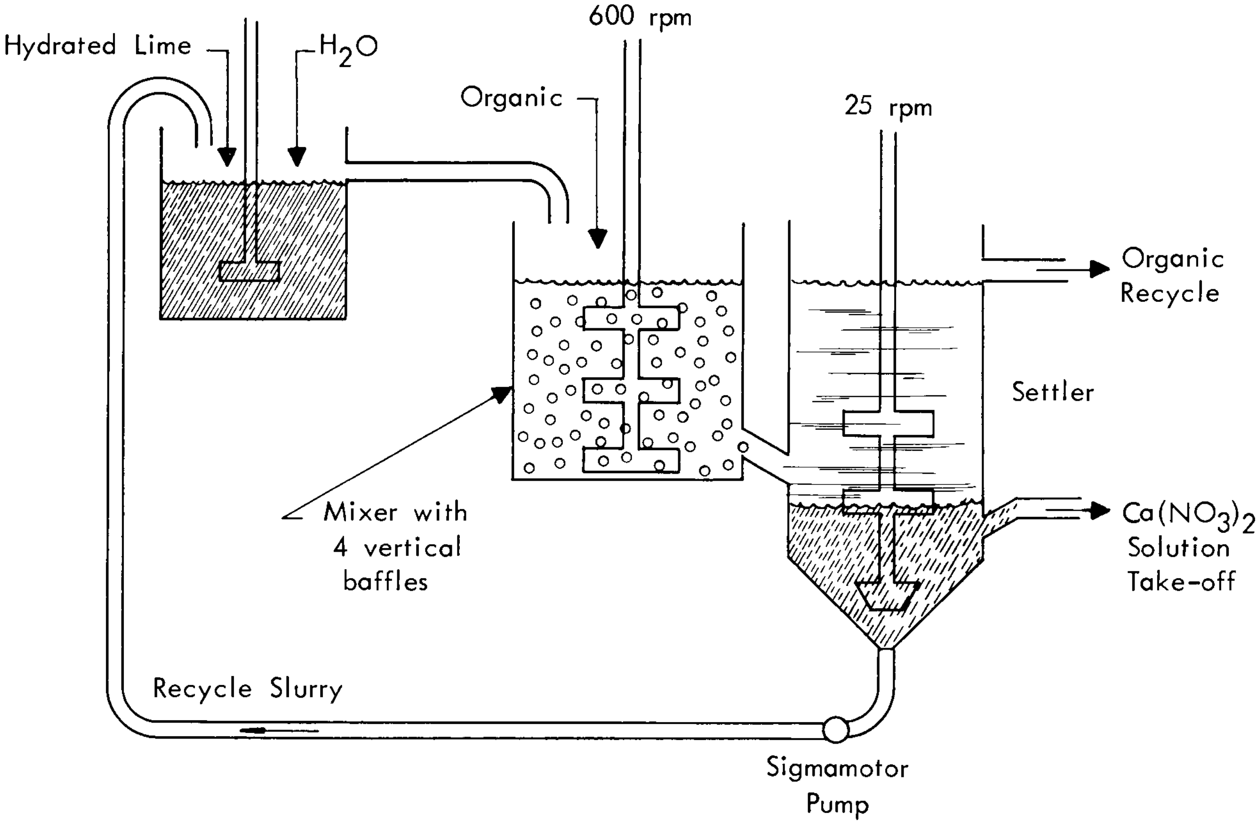


Fig. 5.1. Regeneration of amine with a lime slurry. Head organic: 0.1 *M* Alamine 336 nitrate in Amsco 125-82 + 8 vol % tridecanol.

Table 5.1. Operating Conditions for Amine Regeneration Circuit

Organic:	0.1 M Alamine 336 nitrate in Amsco 125-82 + 8 vol % tridecanol; 40 ml/min
Recycle slurry:	~2 M $Ca(NO_3)_2$ + 10-20 g of $Ca(OH)_2$ per liter; ~20 ml/min
Hydrated lime:	added intermittently at the rate of ~10 g/hr
Water:	~1 ml/min
Calcium nitrate take-off:	~1 ml/min
Residence times:	Mixer, ~8 min Settler, slurry 10 min, organic 30 min

With the same operating conditions (Table 5.1), a 14-hr run was then made to determine the effect of molybdenum in the system. Molybdenum was introduced by dissolving ammonium molybdate in the nitric acid fed to the extractor, resulting in an organic extract containing ~0.04 g of molybdenum per liter. Throughout the run, >90% of the molybdenum and >95% of the nitrate were removed from the solvent in the regeneration system. At the termination of the run the calcium nitrate recycle solution contained only 0.06 g of molybdenum per liter. Most of the molybdenum was found in the slurry solids. Since in plant practice a portion of the slurry solids would be discarded to eliminate extraneous material (silica, iron, aluminum) introduced in the lime, this represents a convenient method for eliminating molybdenum from the system.

Regeneration with Dissolved Lime. The aqueous solubility of calcium hydroxide is sufficiently high that regeneration of the amine with a calcium nitrate solution saturated with calcium hydroxide can be considered. This possibility was studied briefly in batch tests with 2.5 M $Ca(NO_3)_2$ solution that had been saturated with calcium hydroxide by contacting it with excess hydrated lime and filtering. Titration of the filtrate with nitric acid showed ~0.09 N dissolved lime. A 0.1 M amine nitrate solution was 85% regenerated by contact with this solution at an a/o phase ratio of 1/1 and 100% regenerated at ratios of 2/1 and higher (Table 5.2).

Table 5.2. Amine Regeneration with Soluble Lime

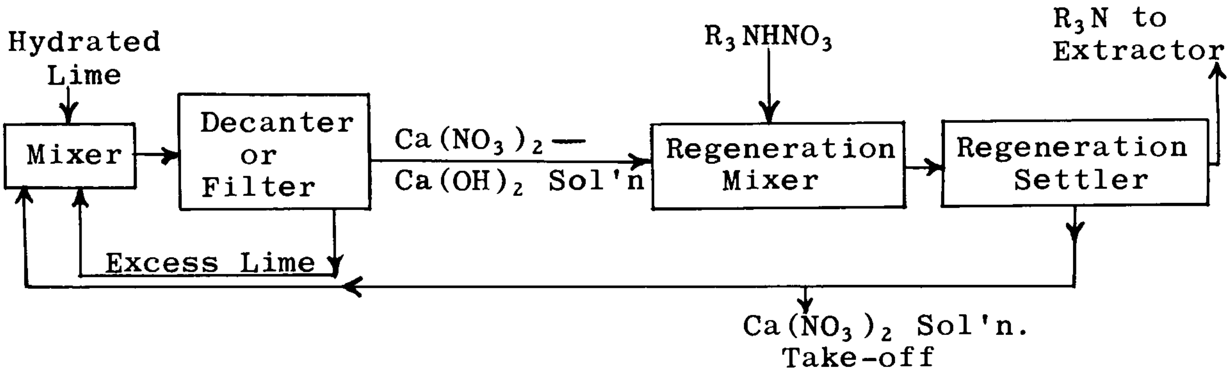
Organic: 0.1 M XE-204 amine nitrate in kerosene + 6 vol % tridecanol; analyzed 6.5 g of NO_3/liter

Aqueous: 2.5 M $Ca(NO_3)_2$ solution saturated (R.T.) with $Ca(OH)_2$, pH 11.2

Contact time: 10 min

Phase Ratio, a/o	Final pH	Nitrate in Organic, g/liter	Regeneration, %
1/2	4.9	3.9	40
1/1	5.5	1.0	85
2/1	10.8	<0.01	100
4/1	11.0	<0.01	100

In process use, the calcium nitrate solution leaving the regeneration settler would be contacted with excess lime and decanted (or filtered) before being recycled to the regeneration mixer:



More study would be needed to determine if this regeneration procedure is better than the lime slurry method. In view of the favorable results obtained with the latter (see above), it is questionable that the advantage gained in the former method of avoiding a slurry contacting step would outweigh the disadvantage of the added processing steps.

6.0 CONTINUOUS TESTS

A series of continuous runs was made with 0.105 M Amberlite XE-204 amine in Amsco 123-15, modified with 6 vol % tridecanol, and synthetic ore leach liquors to demonstrate operational feasibility of the system. Amsco 123-15 (refined kerosene, ~20¢ per gallon) rather than kerosene was used as diluent since, in studies with the latter (Sec. 5.0), emulsion difficulties resulted from nitration of the kerosene.

The total system (Fig. 6.1) included three extraction stages, one water scrub stage, one sulfate stripping stage, four uranium stripping stages, and one amine regeneration stage. Mixer-settlers were used for contacting the phases, and the settlers in the sulfate stripping and amine regeneration sections had cone-bottoms to facilitate discharge of solids. Design of the sulfate stripping and amine regeneration circuits is shown in Figs. 6.2 and 5.1, respectively. Information on the size of equipment used is listed in Sec. 9.2. Since a continuous solids feeder of sufficiently low capacity was not available for feeding lime to the amine regeneration system, lime, in ~6-g increments, was added at 30-min intervals. The excess lime in the aqueous slurry therefore varied widely at different times, tending to give somewhat erratic results in operation of this system.

Run 1. This was a 16.5-hr shakedown run with "pure" uranyl sulfate solution (pH 1), containing 5 g of uranium per liter, as feed. Recovery of uranium in the extraction section was

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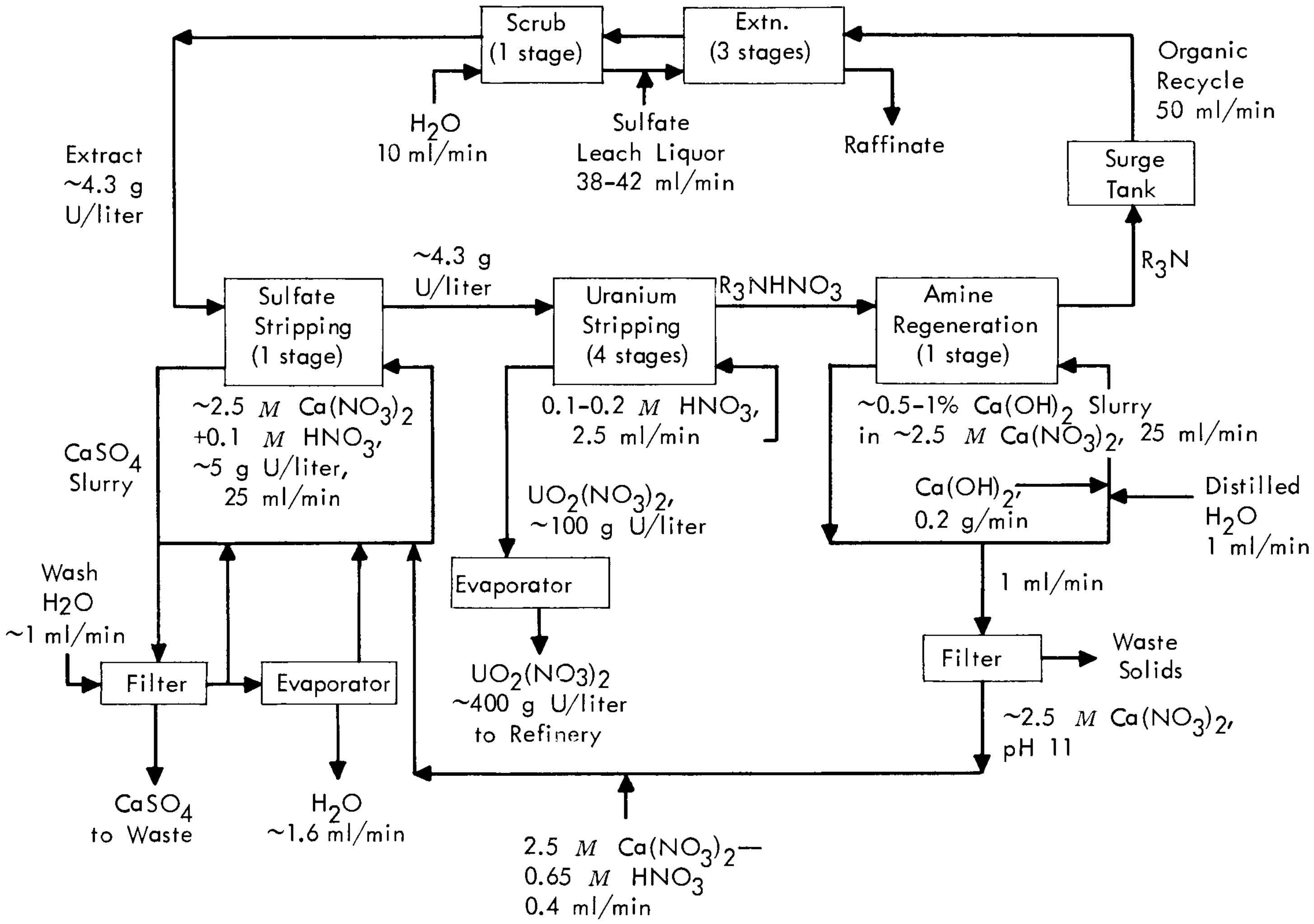

Fig. 6.1. High nitrate—water stripping method. Organic: 0.105 *M* XE-204 amine in Amsco 123-15 + 6 vol % tridecanol.

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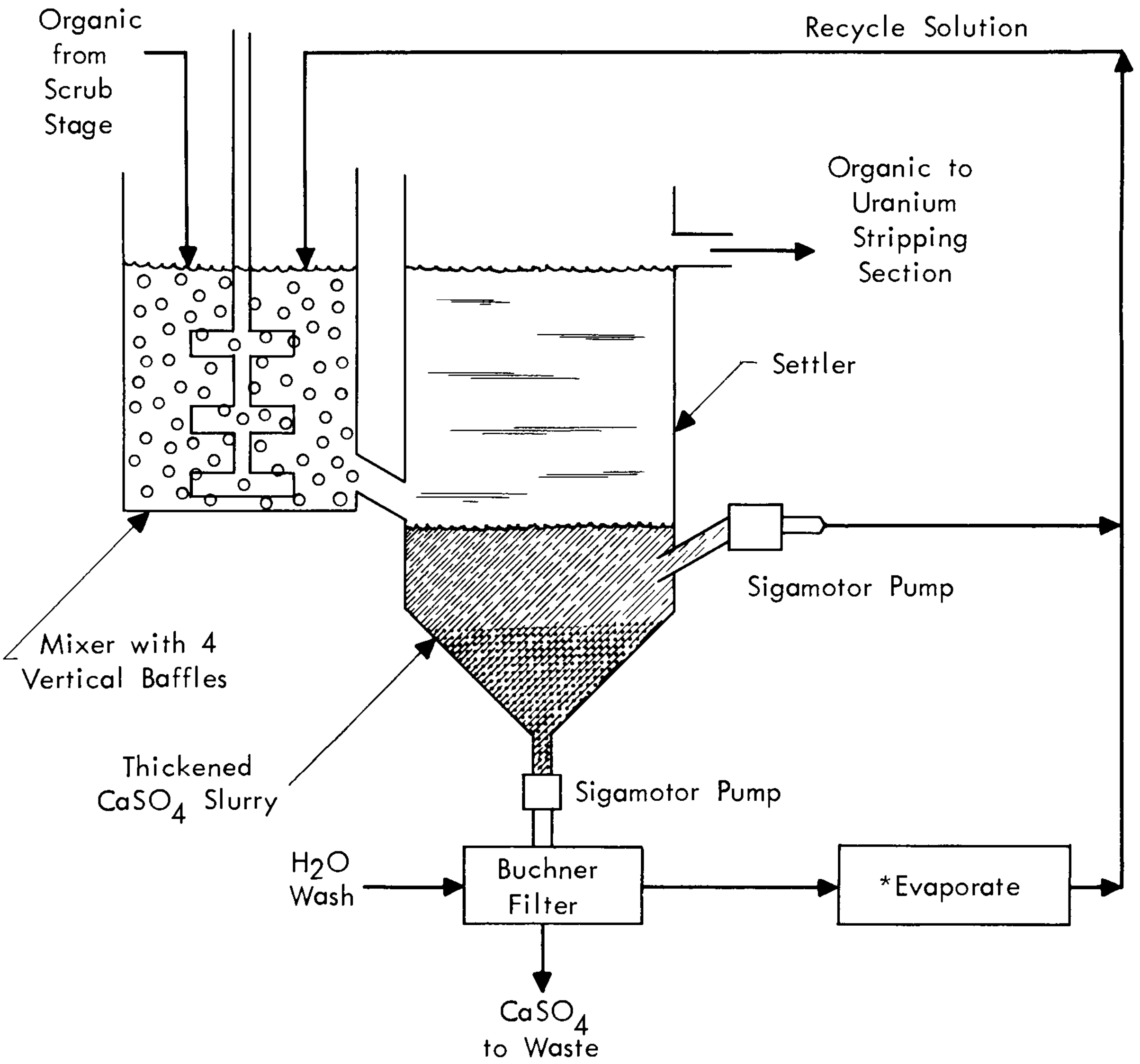

*Some evaporation needed to prevent buildup of aqueous volume.

Fig. 6.2. Sulfate stripping circuit.

>99.9% with liquor/organic/scrub ratios of 0.84/1/0.25, respectively (Table 6.1).

In the sulfate strip section, >98% of the sulfate was removed from the solvent. The nitrate concentration in the recycle solution ranged from 4.4 to 4.8 M and the uranium concentration from 6.5 to 11.4 g/liter. Physical operation of the sulfate stripping circuit was highly satisfactory, the precipitated calcium sulfate settling rapidly in the aqueous phase beneath a sparkling clear solvent phase. The calcium sulfate was filtered and washed extremely rapidly, and losses of uranium and nitrate to several batches of waste calcium solids collected during the run were negligibly low:

Weight of Dried Calcium Sulfate, g	Analysis, %	
	U	NO_3
25	0.0046	0.34
71	0.0024	0.21
70	0.0039	0.29
64	0.0021	0.20
200	0.0037	0.19

Uranium stripping with 0.1 M HNO_3 was >99.8% complete, yielding a strip liquor containing 72-83 g of uranium and 2-3 g of sulfate per liter.

Amine regeneration (based on nitrate content of the solvent) was >98% complete in the first sample (5.5 hr) but fell off to ~92% at 11 and 16.5 hr. The calcium nitrate solution withdrawn from the regeneration section for recycle to the sulfate stripping section contained 4.6-5.1 M nitrate. Physical operation in the amine regeneration system was satisfactory although phase separation was not so clean as in the sulfate stripping section. The solvent leaving the regeneration stage was slightly cloudy due to aqueous entrainment and some solids collected at the settler interface, although the rate of accumulation was slow.

Run 2 (16.5 hr duration). The principal objective of this run was to determine how molybdenum would be distributed in the system. The synthetic leach liquor (pH 1) treated contained, in grams per liter: 5.8 U, 1.40 V(IV), 2.1 Fe(III), 1.3 Fe(II), 3 Al, 0.28 Mo, 1.4 PO_4, and 49 SO_4.

Data (Table 6.2) for the run showed ~95% extraction of molybdenum and almost negligible removal of molybdenum from the solvent in the water scrub and sulfate stripping steps. Approximately 5% of the extracted molybdenum went into the uranium product liquor. Of the remainder, 80-85% was stripped from the solvent in the amine regeneration step, most of this molybdenum precipitating as calcium molybdate and being removed with the waste solids withdrawn from the regeneration system.

Table 6.1 Data for Continuous Run 1

Aqueous feed: uranyl sulfate—sodium sulfate solution adjusted to pH 1 with sulfuric acid; 5.5 g of U and 43 g of SO_4 per liter
Organic: 0.105 M XE-204 amine in Amsco 123-15 + 6 vol % tridecanol
Strip solution: 0.1 M HNO_3
Flow rates: aqueous feed, 42 ml/min; others as shown in Fig. 6.1

Run Time, hr	Stage	Analyses,[a] g/liter					
		Organic			Aqueous		
		U	NO_3	SO_4	U	NO_3	SO_4
		Extraction Section					
5.5	1	5.3	-	-	-	-	-
	3	-	-	-	0.001	-	-
11.0	1	4.3	-	-	-	-	-
	3	-	-	-	0.0006	-	-
16.5	1	5.0	-	-	2.0	-	-
	2	2.0	-	-	0.05	-	-
	3	0.07	-	-	0.0007	-	-
		H_2O Scrub Section					
5.5	-	5.0	-	-	-	-	-
11.0	-	4.6	-	-	-	-	-
16.5	-	4.8	-	-	0.52	-	-
		Sulfate Strip Section					
5.5	-	4.8	9.1	0.15	11.4	275	-
11.0	-	4.8	8.9	0.11	7.8	284	-
16.5	-	4.9	8.6	0.16	6.5	294	-
		Uranium Strip Section					
5.5	1	-	-	-	78.9	-	3.1
	4	0.0015	-	<0.05	-	-	-
11.0	1	-	-	-	82.9	-	2.4
	4	0.008	-	-	-	-	-
16.5	1	0.83	-	-	71.7	-	2.2
	2	0.31	-	-	42.2	-	-
	3	0.07	-	-	9.6	-	-
	4	0.01	-	-	0.8	-	-
		Amine Regeneration Section					
5.5	-	0.0008	0.095	-	0.003	288	-
11.0	-	0.0008	0.53	-	0.004	262	-
16.5	-	0.002	0.51	-	0.004	315	-

[a] All samples filtered before being submitted for analysis.

Table 6.2 Data for Continuous Run 2

Aqueous feed: synthetic leach liquor (pH 1) containing, in grams per liter, 5.8 U, 1.4 V(IV), 2.1 Fe(III), 1.3 Fe(II), 3.0 Al, 0.28 Mo, 1.4 PO_4, and 49 SO_4
Organic: 0.105 M XE-204 amine in Amsco 123-15 + 6 vol % tridecanol
Strip solution: 0.1 M HNO_3
Flow rates: aqueous feed, 42 ml/min; others as shown in Fig. 6.1

Run Time, hr	Stage	Analyses,[a] g/liter							
		Organic				Aqueous			
		U	Mo	NO_3	SO_4	U	Mo	NO_3	SO_4
2.75	1	4.6	0.24	0.56	-	-	-	-	-
5.5	1	4.9	0.26	-	-	2.9	0.055	-	-
	2	3.0	0.14	-	-	0.15	0.017	-	-
	3	2.9	0.07	-	-	0.016	0.005	-	-
8.25	1	4.4	0.25	-	-	-	-	-	-
	3	-	-	-	-	0.056	0.012	-	-
11.0	1	4.1	0.28	-	-	-	-	-	-
	3	-	-	-	-	0.20	0.015	-	-
16.5	1	4.0	0.33	0.98	-	3.9	0.10	-	-
	2	3.1	0.21	1.64	-	3.1	0.06	-	-
	3	2.1	0.14	2.0	-	1.4	0.04	-	-
		H_2O Scrub Section							
5.5	-	4.7	0.26	-	-	0.78	0.001	-	-
11.0	-	3.7	0.26	-	-	-	-	-	-
16.5	-	3.7	0.29	1.2	-	1.1	0.004	-	-
		Sulfate Strip Section							
5.5	-	4.2	0.25	-	0.09	7.8	0.032	275	-
8.25	-	3.6	0.27	-	0.15	8.2	0.042	268	-
11.0	-	3.5	0.27	-	0.09	8.6	0.063	274	-
16.5	-	4.0	0.29	-	0.12	6.0	0.102	325	-

Table 6.2 (Cont'd.)

Run Time, hr	Stage	Analyses, g/liter							
		Organic				Aqueous			
		U	Mo	NO_3	SO_4	U	Mo	NO_3	SO_4
		Uranium Strip Section							
5.5	1	0.77	0.26	-	-	65.0	0.091	-	2.6
	2	0.14	0.25	-	-	23.9	0.015	-	-
	3	0.05	0.24	-	-	6.6	0.003	-	-
	4	0.02	0.25	-	-	1.2	0.002	-	-
8.25	1	-	-	-	-	104	0.36	-	3.5
	4	0.013	0.23	-	-	-	-	-	-
11.0	1	-	-	-	-	110	0.25	-	4.8
	4	0.026	0.24	-	-	-	-	-	-
16.5	1	0.34	-	-	-	136	0.32	-	10.1
	2	0.27	-	-	-	30.7	0.036	-	-
	3	0.06	-	-	-	4.3	0.003	-	-
	4	0.01	-	-	-	0.7	0.002	-	-
		Amine Regeneration Section							
2.75	-	0.001	0.03	0.53	-	-	-	-	-
5.5	-	0.002	0.04	0.15	-	<0.001	0.03	301	-
8.25	-	0.001	0.04	0.10	-	<0.001	0.06	333	-
11.0	-	0.001	0.04	0.30	-	0.004	0.06	333	-
16.5	-	0.002	0.05	0.13	-	0.001	0.05	314	-

[a] All samples filtered before being submitted for analysis.

Physical operation of the total circuit was satisfactory except that entrainment of aqueous in the solvent recycle stream was relatively high, resulting in appreciable carry-over of calcium nitrate to the extraction circuit. The calcium precipitated as calcium sulfate in the last stage and was carried out in the raffinate. The nitrate, however, was extracted by the amine and interfered severely with uranium extraction, uranium recoveries at the end of the run being only ~75%.

Sulfate stripping was >98% complete.

An average of 99.6% of the uranium in the solvent was stripped in the uranium stripping section. Almost all the residual uranium was precipitated from the solvent in the amine regeneration step and was removed with the waste solids withdrawn from that system. Analysis of one batch of waste solids showed 32.4% Ca, 5.5% CO_3, 10.1% Mo, and 2.5% U.

Run 3 (16.5 hr duration). Prior to this run a much larger organic surge tank (~30 min holdup) was installed between the amine regeneration and extraction systems to allow more time for entrained aqueous to separate from the recycle solvent. In addition, since uranium stripping was incomplete in run 2, the nitric acid concentration in the strip solution was increased from 0.1 to 0.2 M in an attempt to improve the stripping efficiency.

In the sulfate stripping section the nitrate concentration in the recycle solution ranged from 4.3 to 5.1 M (Table 6.3). Under these conditions the uranium concentration in this solution remained fairly constant at ~6.5 g/liter throughout the run. As in run 1, losses of uranium and nitrate to the washed calcium sulfate solids were negligibly low:

Weight of Dried Calcium Sulfate, g	Analysis, %	
	U	NO_3
25	0.0021	0.24
43	0.0024	0.21
47	0.0024	0.21
50	0.0021	0.21
47	0.0049	0.34
180	0.0045	0.17

Uranium stripping was no more effective than in run 2, averaging ~99.5%. Since batch stripping data (Sec. 4.0) indicated that complete stripping should be obtainable in less than 4 stages with the flow ratio used, this suggests that stage efficiencies in the stripping system were relatively poor, probably because of the high organic/aqueous flow ratio, 20/1.

The amine regeneration efficiency was slightly low (~93%) in the first half but high (>97%) in the last half of the run. Analyses of several batches of waste solids removed from the regeneration system showed 1-1.5% uranium and indicated that the solids were mainly lime and calcium molybdate:

Weight of Washed and Dried Solids, g	Analysis, %			
	U	Mo	Ca	NO_3
0.65	1.5	4.4	19.5	-
0.25	1.6	17.1	19.8	-
6	1.1	11.8	21.1	4.2
7	0.9	12.6	21.0	5.9
38	1.2	17.0	20.6	5.0

The total uranium in the waste solids was equivalent to ~0.25% of the total uranium processed. This uranium loss could, of course, be decreased by increasing the efficiency of the uranium stripping step. Use of the larger organic surge tank greatly decreased the amount of entrained calcium nitrate solution entering the extraction system, but the amount entrained was still sufficiently high to cause a decrease in uranium

Table 6.3 Data for Continuous Run 3

Aqueous feed: same as in run 2 (Table 6.2)
Organic: 0.105 M XE-204 amine in Amsco 123-15 + 6 vol % tridecanol
Strip solution: 0.2 M HNO_3
Flow rates: aqueous feed, 42 ml/min; others as shown in Fig. 6.1

Run Time, hr	Stage	Analyses,[a] g/liter							
		Organic				Aqueous			
		U	Mo	NO_3	SO_4	U	Mo	NO_3	SO_4
		Extraction Section							
2.75	1	4.7	0.31	0.68	-	-	-	-	-
	3	0.28	0.04	0.83	-	0.002	0.003	-	-
5.5	1	4.4	0.35	-	-	-	-	-	-
	3	-	-	-	-	0.0008	0.003	-	-
8.5	1	3.9	0.40	-	-	-	-	-	-
	3	-	-	-	-	0.039	0.008	-	-
11.0	1	4.8	0.34	-	-	4.2	0.045	-	-
	2	4.2	0.22	-	-	1.80	0.038	-	-
	3	1.9	0.16	-	-	0.063	0.010	-	-
13.75	1	4.9	0.31	-	-	-	-	-	-
	3	-	-	-	-	0.14	0.012	-	-
16.5	1	4.9	0.35	-	-	4.2	0.095	-	-
	2	4.6	0.19	-	-	2.5	0.030	-	-
	3	4.7	0.14	-	-	0.09	0.009	-	-
		H_2O Scrub Section							
11.0	-	4.6	0.34	-	-	1.07	0.003	-	-
16.5	-	4.6	0.34	-	-	1.03	0.003	-	-
		Sulfate Strip Section							
2.75	-	4.6	0.30	-	0.10	6.4	0.055	318	-
5.5	-	4.2	0.28	-	0.16	10.2	0.037	300	-
8.25	-	4.1	0.36	-	0.11	6.4	0.036	284	-
11.0	-	4.3	0.34	-	0.18	6.7	0.036	267	-
16.5	-	4.1	0.35	-	0.09	6.7	0.036	265	-

Table 6.3 (Cont'd.)

Run Time, hr	Stage	Analyses, g/liter Organic U	Organic Mo	Organic NO_3	Organic SO_4	Aqueous U	Aqueous Mo	Aqueous NO_3	Aqueous SO_4
					Uranium Strip Section				
2.75	1	-	-	-	-	130	0.25	-	5.45
	4	0.037	0.28	-	-	-	-	-	-
5.5	1	-	-	-	-	105	0.08	-	3.09
	4	0.028	0.24	-	<0.05	-	-	-	-
8.25	1	-	-	-	-	120	0.22	-	4.06
	4	0.021	0.35	-	<0.05	-	-	-	-
11.0	1	1.27	0.33	-	-	70.8	0.131	-	3.59
	2	0.18	0.32	-	-	32.0	0.022	-	-
	3	0.057	0.33	-	-	4.7	0.006	-	-
	4	0.024	0.30	-	<0.05	0.8	0.002	-	-
13.25	1	-	-	-	-	68.1	0.115	-	2.61
	4	0.026	0.34	5.7	0.06	-	-	-	-
16.5	1	0.88	0.34	-	<0.05	78.0	0.137	-	2.3
	2	0.18	0.34	-	<0.05	27.6	0.044	-	1.9
	3	0.042	0.33	-	<0.05	4.3	0.009	-	0.4
	4	0.034	0.30	-	<0.05	0.7	0.006	-	0.1
					Amine Regeneration Section				
2.75	-	0.019	0.105	0.40	-	0.004	0.082	296	-
5.5	-	0.015	0.083	0.51	-	0.002	0.026	300	-
8.25	-	0.019	0.158	0.42	-	<0.001	0.059	268	-
11.0	-	0.004	0.100	0.18	-	<0.001	0.035	266	-
13.25	-	0.002	0.085	0.31	0.09	-	-	-	-
16.5	-	0.003	0.031	0.08	-	<0.001	0.033	266	-

[a] All samples filtered before being submitted for analysis.

recovery (due to competition from nitrate) to only ~98% near the end of the run. This problem, however, was satisfactorily overcome in run 4.

Evaporation of the uranyl nitrate product solution was studied briefly in a large beaker equipped with a tantalum steam coil. A 2-liter volume of solution containing 82 g of uranium, 0.12 g of molybdenum, and 3.2 g of sulfate per liter was evaporated to ~400 ml, giving a concentrated solution containing ~400 g of uranium per liter. This is an ideal concentration for shipment to the refinery since the freezing point[7] of uranyl nitrate solution reaches a minimum (~0°C) at ~400 g of uranium per liter. There was no foaming of the solution during evaporation.

Run 4 (18 hr duration). To the synthetic leach liquor used in run 4 (Table 6.4) were added a number of elements to determine the separation of uranium from these elements that would be obtained in the overall extraction-stripping process. Since uranium extractions in previous runs were relatively inefficient owing to competition from nitrate carried into the extraction system by the recycle solvent, the aqueous/solvent flow ratio in run 4 was lowered slightly to provide more excess amine for uranium extraction.

The uranium content of raffinate samples ranged from 0.0002 to 0.009 g/liter, averaging 0.003 g/liter which was equivalent to a uranium recovery of >99.9%. The organic phase in the extraction system showed a higher concentration of nitrate than did filtered samples of solvent leaving the regeneration stage, indicating that some nitrate entered the extraction system as entrained calcium nitrate in the recycle solvent. In spite of this, uranium recoveries, as mentioned above, were high.

Stripping of sulfate was >95% complete over most of the run, but regeneration of the amine was somewhat poorer than usual, running ~92%.

Stripping of uranium was >99.7% complete throughout most of the run. The uranium content of the product liquor was somewhat erratic, ranging from 52 to 108 g/liter in several samples. This can probably be attributed to an unsteady flow of aqueous between stages of the stripping system, owing to the very low strip solution flow rate (2.5 ml/min). The total strip liquor collected analyzed 90 g of uranium, 0.38 g of molybdenum, and 7.2 g of sulfate per liter (Table 6.5). With the exception of molybdenum and sulfate, separations of uranium from all the constituents of the feed liquor were highly effective, i.e., decontamination factors >650. The product solution was of sufficient purity to be highly amenable to purification by TBP extraction. Greater purity would be required, however, if the solution is to be directly treated for UO_2 recovery at the mill site by the ADU (ammonium diuranate) precipitation method. Precipitation of UO_4 with H_2O_2 would be expected to give a purer product than the ADU precipitation method, but it is not known whether the purity would be adequate. The peroxide precipitation method has the disadvantage of introducing uranium recycle problems, since uranium recovery in precipitation under conditions (pH <1) that give highest product purity[8] is not complete. The purity of the uranyl nitrate product solution from the amine process undoubtedly could be appreciably improved by some refinement of the system used in the continuous runs. For example, sulfate contamination of the solution, if undesirable, could be essentially eliminated by adding a second stage to the sulfate stripping system and/or by scrubbing the solvent with

Table 6.4 Data for Continuous Run 4

Aqueous feed: synthetic leach liquor (pH 1) containing, in grams per liter, 5.6 U, 1.4 V(IV), 2.2 Fe(III), 0.6 Fe(II), 2.9 Al, 0.24 Mo, 0.5 Th, 0.5 Ce(III), 0.5 Cu, 0.5 Mn, 0.5 Ni, 0.5 Cr(III), 1.5 Na, 0.1 B, 0.1 Ti, 0.1 Cd, 0.1 Zn, 0.1 Sn, 0.1 Co, 0.1 Mg, 0.1 Zr, 0.1 Cs, 0.05 Si, 1.0 As, 1.3 PO_4, and 53 SO_4

Organic: 0.105 M XE-204 amine in Amsco 123-15 + 6 vol % tridecanol

Strip solution: 0.2 M HNO_3

Flow rates: aqueous feed, 39 ml/min; others as shown in Fig. 6.1

Run Time, hr	Stage	Analyses,[a] g/liter							
		Organic				Aqueous			
		U	Mo	NO_3	SO_4	U	Mo	NO_3	SO_4
		Extraction Section							
3	1	4.3	0.19	0.7	-	-	-	-	-
	3	-	-	-	-	0.0002	0.07	-	-
6	1	4.5	0.20	0.9	-	1.74	0.08	-	-
	2	1.7	0.13	1.4	-	0.043	0.02	-	-
	3	0.04	0.06	1.2	-	0.0004	0.01	-	-
9	1	4.2	0.21	1.5	-	-	-	-	-
	3	-	-	-	-	0.009	0.02	-	-
12	1	4.5	0.23	1.2	-	3.4	0.09	-	-
	2	3.0	0.13	2.0	-	0.66	0.04	-	-
	3	0.5	0.09	2.3	-	0.007	0.02	-	-
15	1	4.4	0.20	0.8	-	-	-	-	-
	3	-	-	-	-	0.0003	0.01	-	-
18	1	4.4	0.24	0.9	-	1.90	0.09	-	-
	2	1.4	0.13	1.3	-	0.046	0.03	-	-
	3	0.03	0.06	1.1	-	0.0003	0.009	-	-
		H_2O Scrub Section							
6	-	4.3	0.22	0.8	-	0.45	0.003	-	-
12	-	4.4	0.15	1.1	-	1.13	0.004	-	-
18	-	4.2	0.22	0.9	-	0.49	-	-	-

Table 6.4 (Cont'd.)

Run Time, hr	Stage	Analyses, g/liter							
		Organic				Aqueous			
		U	Mo	NO_3	SO_4	U	Mo	NO_3	SO_4
Sulfate Strip Section									
3	-	3.7	0.15	-	0.36	2.5	0.06	292	-
6	-	3.7	0.20	-	0.38	8.2	0.13	261	-
9	-	4.5	0.22	-	1.11	4.2	0.05	311	-
12	-	4.1	0.21	-	0.49	5.6	0.06	280	-
15	-	4.3	0.18	-	0.14	3.1	0.04	326	-
18	-	3.8	0.18	-	0.16	3.9	0.05	308	-
Uranium Strip Section									
3	1	-	-	-	-	99.0	0.52	-	8.5
	4	0.01	0.12	5.7	<0.05	-	-	-	-
6	1	0.76	0.18	-	-	52.0	0.27	-	4.7
	2	0.19	0.18	-	-	10.1	0.03	-	1.7
	3	0.07	0.19	-	-	3.6	0.02	-	0.8
	4	0.01	0.18	-	-	0.6	0.01	-	0.2
9	1	-	-	-	-	82.0	0.41	-	9.4
	4	0.11	0.18	-	0.41	-	-	-	-
12	1	0.64	0.18	-	0.38	58.0	0.31	-	6.1
	2	0.11	0.18	-	0.48	13.4	0.05	-	2.6
	3	0.04	0.18	-	0.29	4.3	0.03	-	1.2
	4	0.01	0.18	-	0.17	0.7	0.02	-	0.2
15	1	-	-	-	-	108	0.69	-	4.5
	4	0.02	0.17	-	-	-	-	-	-
18	1	0.60	0.17	-	0.15	66.7	0.39	-	4.0
	2	0.18	0.16	-	-	17.3	0.07	-	2.1
	3	0.03	0.14	-	0.07	3.2	0.02	-	0.9
	4	0.01	0.15	-	0.05	0.7	0.01	-	0.3
Amine Regeneration Section									
3	-	0.003	0.03	0.59	-	0.003	0.15	292	-
6	-	0.004	0.03	0.51	-	0.001	0.12	274	-
9	-	0.002	0.05	0.53	-	0.0008	0.05	277	-
12	-	0.0009	0.03	0.25	-	0.0004	0.09	314	-
15	-	0.003	0.02	0.54	-	0.00009	0.11	264	-
18	-	0.004	0.04	0.8	-	0.0002	0.10	254	-

---

[a] All samples filtered before being submitted for analysis.

Table 6.5 Analysis of Product Solution from Run 4

Constituent	Analysis of Product Solution, g/liter Chemical	Spectrographic	Decontamination Factor[a]
U	90	-	-
SO_4	7.2	-	-
V(IV)	-	0.0004	56,000
Fe	-	<0.002	>22,000
Al	-	<0.0013	>35,000
Mo	0.38	-	8
Th	-	0.0023	3,500
Ce(III)	-	0.0004	20,000
Cu	-	<0.0002	>40,000
Mn	-	0.0006	13,000
Ni	-	<0.0025	3,200
Cr(III)	-	<0.0012	>6,600
Na	-	0.0025	9,600
B	-	0.0011	1,600
Ti	-	0.0007	2,300
Cd	-	<0.00001	>160,000
Zn	-	<0.001	>1,600
Sn	-	<0.0013	>1,200
Co	-	<0.0001	>16,000
Mg	-	0.0006	2,700
Ca	0.06	-	b
Si	-	<0.001	>750
As	<0.005	-	>3,400
P	-	<0.01	>650

[a] Decontamination factor = concentration ratio of uranium to the contaminant in the product liquor divided by the ratio in the feed liquor (see Table 6.4 for feed liquor analysis).

[b] There was no calcium in the feed liquor.

a small volume of 5-6 M HNO_3* prior to uranium stripping. The scrub would also remove entrained calcium nitrate from the solvent, decreasing the calcium concentration, which was relatively high (0.06 g/liter) in the product solution. Although it was not tested, previous studies[9,10] suggest that the molybdenum could be removed effectively from the product solution by passing it through a carbon column.

---

*The makeup HNO_3 (Fig. 2.1) could be used for scrubbing before it is combined with the basic calcium nitrate solution coming from the regeneration section.

7.0 REAGENT COSTS

Based on data from run 4, the cost of stripping chemicals is estimated (Table 7.1) at 4.9¢ per pound of U_3O_8, which compares favorably with costs estimated for ammonium carbonate stripping[10] (5-7¢ per pound of U_3O_8), chloride stripping with amine regeneration[1] (7.1¢ per pound of U_3O_8), and ammonium nitrate stripping[1] (6.4¢ per pound of U_3O_8). Total reagent costs for the process, including an allowance for loss of solvent in the extraction step, are estimated at 9¢ per pound of U_3O_8.

Table 7.1 Estimated Reagent Costs

(Assumes treatment of a liquor containing 1.2 g of U_3O_8 per liter)

Chemical	Consumption	Consumption, lb/lb U_3O_8	Unit Cost, ¢/lb	Cost, ¢/lb U_3O_8
$Ca(NO_3)_2$	Stripping	0.65	4.5[a]	2.9
HNO_3	Stripping	0.19	5	1.0
$Ca(OH)_2$	Amine regeneration	0.8	1.3	1.0
XE-204 amine	Distribution to raffinate (<5 ppm)	<0.004	125	<0.5
Organic phase	Entrainment and spillage[b]	0.05 gal	71¢/gal[c]	3.6
			Total	9.0

[a] Assumes calcium nitrate preparation from lime and nitric acid.

[b] Entrainment and spillage estimated at 0.05% of raffinate volume.

[c] Based on Amsco 123-15 cost of 20¢ per gallon and tridecanol cost of 23¢ per pound.

8.0 REFERENCES

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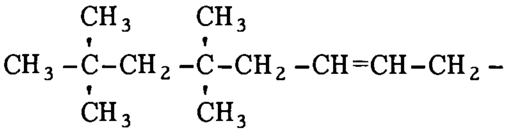
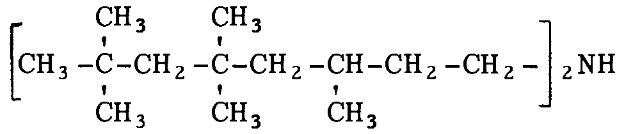
9.0 APPENDIX

9.1 Description of Amine Reagents

Information on structure, source of supply, and present availability of the amines used in this study is given in Table 9.1. Further information on compound purity, diluent compatibility, losses to aqueous liquors, etc. was reported previously in ORNL-1922, -1959, -2099, -2366, -2380, -2466, -3030).

Table 9.1 Description of Amine Reagents

Amine	Structure	Supplier	Availability
Alamine 336	Tertiary amine with mixed *n*-octyl and *n*-decyl alkyls	General Mills	a
XE-204	Didodecenyl-*n* butylamine; structure of the dodecenyl chain is  $CH_3-\overset{CH_3}{\underset{CH_3}{C}}-CH_2-\overset{CH_3}{\underset{CH_3}{C}}-CH_2-CH=CH-CH_2-$	Rohm and Haas	b
Di(tridecyl)	 $\left[CH_3-\overset{CH_3}{\underset{CH_3}{C}}-CH_2-\overset{CH_3}{\underset{CH_3}{C}}-CH_2-\underset{CH_3}{CH}-CH_2-CH_2-\right]_2NH$	Carbide	b

[a] Commercially available.

[b] Available in pilot quantities.

9.2 Description of Equipment Used in Continuous Tests

The mixer-settlers used in the extraction, scrub, and uranium stripping sections were of the type described in Sec. 11.3 of ORNL-2720. Equipment used in the sulfate stripping and amine regeneration sections is shown in Figs. 6.2 and 5.1, respectively, and information on size of the contactors used in each circuit is listed in Table 9.2.

Table 9.2 Size of Equipment Used in Continuous Runs

Uranium extraction stages and scrub stage	
Mixer volume	250 ml
Settler volume	450 ml
Settler area	5.9 $in.^2$
Sulfate stripping stage	
Mixer volume	850 ml
Settler volume	1200 ml
Settler area	11.0 $in.^2$
Uranium stripping stages	
Mixer volume	250 ml
Settler volume	450 ml
Settler area	5.9 $in.^2$

Table 9.2 (Cont'd.)

Amine regeneration stage	
Mixer volume	1100 ml
Settler volume	2200 ml
Settler area	12.6 $in.^2$
H_2O scrub stage	
Mixer volume	250 ml
Settler volume	1000 ml
Settler area	11.0 $in.^2$

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