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DESIGN AND SCALEUP OF MIXER-SETTLERS  
FOR THE DAPEX SOLVENT EXTRACTION PROCESS

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ABSTRACT

The basis for design and scaleup of mixer-settlers for the Dapex solvent extraction process was determined by a unit operations study in scale model units. Rate constants for uranium extraction involving stage efficiency, phase ratio, and residence time are directly proportional to the cube root of power input in the mixer. Typical mixing requirements for 90% stage efficiency are 30 hp/1000 gal and 2 min residence for extraction, and 0.7 hp/1000 gal and 1.5 min for stripping. The scaleup of geometrically similar mixers is based on constant specific power input. The flow capacity of gravity settlers is limited by a dispersion band, which increases exponentially with flow rate. The scaleup of settlers is based on constant flow rate of dispersed phase per unit settler area and constant band thickness. The nominal flow capacity (50% of flooding) of the dispersed phase is approximately 1 gpm/ft<sup>2</sup> of settler area. Emulsions and solvent entrainment are minimized by control of mixing to form solvent-continuous dispersions. Operating data from two uranium mills confirm the scaleup relations over a range of flow rates of 200-fold for mixers and 1000-fold for settlers.

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## 1.0 SUMMARY

A basis for design of mixer-settlers was obtained by a unit operations study of single mixer-settler units. The variables affecting stage efficiency of the mixer and phase separation in the settler were successfully correlated in small, 6-in.-dia, units. Scaleup was demonstrated in three sizes and was confirmed by plant data showing an over-all scaleup of throughput capacity of 200-fold for mixers and 1000-fold for settlers.

Mixer. Standard baffled-tank mixers were used because the fluid mechanics are well defined and the power input can be calculated from turbine size and speed and the liquid properties. The mixers were evaluated with both batch and continuous flow to determine the effect of mixing on the rate of uranium extraction and stripping.

Experimentally, the rate of uranium extraction is first order with respect to the uranium concentration in the aqueous phase. The rate constant,  $k_a$ , is related to the stage efficiency,  $E$ , the fraction of aqueous in the mixer,  $R/V$ , and the contact time,  $t$ , by the equations

$$\text{Batch:} \quad k_a = -\ln(1 - E) \frac{R}{V} \frac{1}{t}$$

$$\text{Continuous:} \quad k_a = \frac{E}{1 - E} \frac{R}{V} \frac{1}{t}$$

The equations were confirmed at  $A/O$  phase ratios of 4/1 and 1/1 and at contact times from 0.5 to 5.0 min (continuous flow). The values of the rate constant were then used to correlate the effects of the mixing variables and to determine scaleup factors.

Batch rate constants, although consistently greater than those for continuous flow, were useful for determining the relative effects of variables and for guiding the continuous-flow studies.

The extraction rate constants are directly proportional to the cube root of power input over a range of 2.5 to 220 hp/1000 gal. Data for two different chemical systems involving differences in aqueous composition, D2EHPA concentration, and phase ratio (4/1 and 1/1) fit on a single curve, indicating that the power dependence of the extraction rate is not affected by the usual composition variations expected in most uranium-mill feed liquors.

At constant power input the rate constant increased nearly linearly with decreased turbine-tank diameter ratio ( $D/T$ ), being nearly doubled at 1/6 over that at 2/3. A  $D/T$  ratio of 1/3 is commonly used for mixing and was selected as a standard for comparison of different sized mixers.

The extraction rate increases with increased temperature, being doubled for a 15°C rise. The data show a good fit in the Arrhenius equation, giving a value for  $\Delta H$  of 8-9 kcal/mole.

Scaleup data in 6-, 12-, and 20-in.-dia mixers show that the extraction rate constant at constant specific power input and constant D/T is virtually independent of mixer size for the different chemical systems, phase ratios, and power inputs tested. Therefore the criteria for design of large mixers can be selected from data obtained in small mixers. Direct scaleup is based on constant power input per unit volume of geometrically similar mixers. Confirmation of scaleup was obtained from the performance of commercial mixers (36 in. dia) in the Dapex circuit of a commercial uranium mill.

Scaleup based on the rate constant allows a choice of a range of combinations of power input, residence time, and stage efficiency that can be used as a basis for design. The combination best suited for a particular plant depends on the costs of power and mixing equipment. One such combination selected for plant design was a power input of 30 hp/1000 gal and 2 min residence time, giving 90% stage efficiency.

Extraction rates for vanadium(IV) and iron(III) are slower than the rate for uranium. The values of the rate constants at the same mixing power are 1.4 for uranium, 0.84 for vanadium, and 0.10 for iron.

The rate of uranium stripping by sodium carbonate is significantly greater than for extraction, and consequently it was not so thoroughly investigated. Stage efficiency was higher than 90% at a power input of 0.7 hp/1000 gal and 1.5 min residence time or at 10 hp/1000 gal and 0.4 min. Exact scaleup data were not determined, but plant data obtained in 3- and 4-ft mixers show nearly 100% stage efficiency at a power level of 5 to 30 hp/1000 gal and a residence time of 2 to 10 min.

Settlers. The rate of primary phase separation determines the flow capacity of gravity settlers. In most cases this was limited by the rate of coalescence of the dispersed phase and not by the rate of settling of drops to the interface. In both batch and continuous-flow tests the rate of coalescence was largely dependent on the settler area and nearly independent of the dispersion volume. Consequently the best correlation of phase separation is based on the flow rate of dispersed phase per unit settler area.

At steady state with continuous flow a distinct band of dispersion existed at the interface, the thickness of which increased exponentially with flow rate. Nominal flow capacity was arbitrarily defined as the flow rate causing a 5-in. band which was about half the flow rate causing a 2-ft band. The flow capacity was virtually unaffected by mixing power and time, density and pH of aqueous phase, phase ratio, or type of dispersion within the ranges tested, which were selected primarily to bracket

expected variations in the application of the Dapex process to uranium milling. The flow capacity was markedly increased by increasing temperature, being doubled in going from 15 to 45°C.

Scaleup data in 0.5-, 1.8-, and 4.0-ft-dia settlers show that dispersion thickness is independent of settler size at the same flow rate per unit area. Therefore the nominal flow capacity expressed as flow rate per unit area of small models can be used directly as a basis for design of large settlers. The liquid depth in the settler is determined mainly by the sensitivity of the interface control device. A depth of 2 ft is sufficient for simple gravity leg control. The use of nominal flow capacity allows a safety factor of 2 for flooding of a 2-ft liquid depth. The nominal flow capacity of the dispersed phase ranged from 1.0 to 1.5 gpm/ft<sup>2</sup> in different-size settlers and for aqueous-continuous dispersion at A/O ratios of 4/1 and 1/1 and for solvent-continuous dispersions at A/O of 1/1 and 1/2. Scaleup data were confirmed in plant settlers of 7 and 16 ft dia, which showed constant flow capacity per unit area over about a 1000-fold area increase.

Entrainment of solvent in the aqueous was consistently less than 0.2 gal/1000 gal for solvent-continuous dispersions at A/O phase ratios of 1/1 and 1/2. Entrainment for aqueous-continuous mixing ranged from 0.2 to 3.0 gal/1000 gal, increasing with increased ratio of aqueous to solvent. High entrainment was reduced to less than 0.2 gal/1000 gal by passing the aqueous phase through a secondary settler designed for low turbulence with an aqueous residence time of about 30 min.

Emulsions were formed with a number of plant leach liquors which contained silica ranging from 0.1 to 2.5 g of SiO<sub>2</sub> per liter. The emulsions were an oil-in-water type and could be prevented either by adding certain surfactants or by controlling the mixing for a solvent-continuous dispersion. The latter is preferred because it minimizes both emulsion formation and solvent entrainment without affecting stage efficiency.

## 2.0 INTRODUCTION

This report summarizes the engineering studies of mixer-settlers for the Dapex solvent extraction process. Most of the results have been published in monthly progress reports.<sup>1,2</sup> The variables affecting stage efficiency, phase separation, and scaleup were measured to provide a basis for design of plant equipment. The work was directed toward application of the process for recovery of uranium from sulfuric acid leach liquors in uranium mills. Design data generally are required for any new process, and in this case it was particularly true because solvent extraction techniques were novel to the uranium milling industry.

Although the mixer-settler is one of the oldest types of contactors used for liquid-liquid extraction, there are relatively few published methods of scaleup. Even more rarely has there been any attempt to



verify the methods from the operational data of full-scale plants. Results of previous studies of agitated vessels have suggested several different methods of scaleup of mixers. Miller and Mann<sup>3</sup> in their study of degree of mixing in unbaffled tanks suggested scaleup by use of constant power input per unit mixer volume; Flynn and Treybal<sup>4</sup> found that stage efficiency was independent of mixer size at constant energy input per unit volume of liquid processed; and Karr and Scheibel<sup>5</sup> demonstrated scaleup as a function of powers of agitator speed and diameter. For settlers, the suggested scaleup method has often been based on residence time.<sup>6</sup> Many patents have been granted for particular settler configurations, but information on experimental demonstration of scaleup generally has not been published.

Geography has precluded pilot plant operation with uranium ore liquors at ORNL. Consequently engineering studies have been on a unit operations basis for evaluation of the principal variables affecting the rate of extraction (and stripping) in the mixer and phase separation in the settler. The effects of power input, phase ratio, type of dispersion, and temperature on the uranium transfer rate were measured in baffled-tank turbine-agitated mixers. Both batch and continuous-flow mixing techniques were used.

Phase separation was studied in gravity settlers to determine the flow capacity for primary phase break and residual entrainment. The effects of mixing power, type of dispersion, settler geometry, phase ratio, and temperature were evaluated. In addition, emulsion prevention with certain ore leach liquors containing silica and/or ore slimes was studied.

Scaleup relations for both mixers and settlers were developed from data obtained in three sizes of geometrically similar units. A check on the scaleup relations was obtained from analysis of the equipment performance data of two operating uranium mills: Kerr-McGee Oil Industries, Shiprock, N. M., and Climax Uranium Co., Grand Junction, Colo.

Acknowledgments. This report is a summary of work carried out over several years by a number of people. The program was directed by H. M. McLeod and K. B. Brown. Experimental work was done by H. F. Bauman, J. R. Buchanan, J. F. Mannes Schmidt, R. R. Wiethaup, F. G. Kilpatrick, C. H. Tipton, and K. Ladd.

The analyses required by the experiments were performed in the Y-12 section of the Oak Ridge National Laboratory Analytical Chemistry Division under the direction of C. D. Susano.

Operational data of solvent extraction circuits in the uranium mills were obtained with the excellent cooperation of the management and technical staff of Climax Uranium Company, Grand Junction, Colo., and Kerr-McGee Oil Industries, Inc., Shiprock, N. M.

### 3.0 DAPEX PROCESS FLOWSHEET

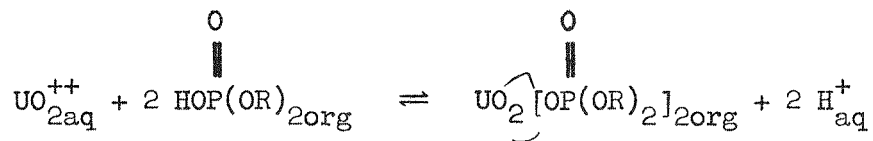
The solvent in the Dapex process<sup>7-11</sup> (Fig. 3.1) for recovery of uranium from sulfuric acid solution is a dialkylphosphoric acid, typically di(2-ethylhexyl)phosphoric acid (D2EHPA), dissolved in a kerosene-type diluent, usually modified with tributyl phosphate (TBP). Uranium is stripped from the solvent by sodium or ammonium carbonate.

The liquor fed to the system is reduced to minimize extraction of ferric ion. By appropriate adjustment of the extractant concentration and flow rates the uranium is essentially completely extracted from the liquor in 3-6 countercurrent stages. The uranium loading of the solvent depends on selection of process conditions, but with 0.1 M D2EHPA it will usually be of the order of 4.5-7 g of U<sub>3</sub>O<sub>8</sub> per liter. In the carbonate stripping cycle the uranium and any small amounts of impurities are removed in 2-3 countercurrent stages, giving a solution containing 50-60 g of U<sub>3</sub>O<sub>8</sub> per liter. The extractant, in the form of the sodium salt, is recycled to the extraction section.

Uranium is recovered from the loaded strip solution by established methods, one of which involves destruction of the carbonate with sulfuric acid followed by addition of ammonia or magnesia to precipitate the uranium. Prior to uranium recovery the strip solution can be filtered to remove precipitated metal contaminants if their removal is desired.

### 4.0 KINETICS OF URANIUM EXTRACTION

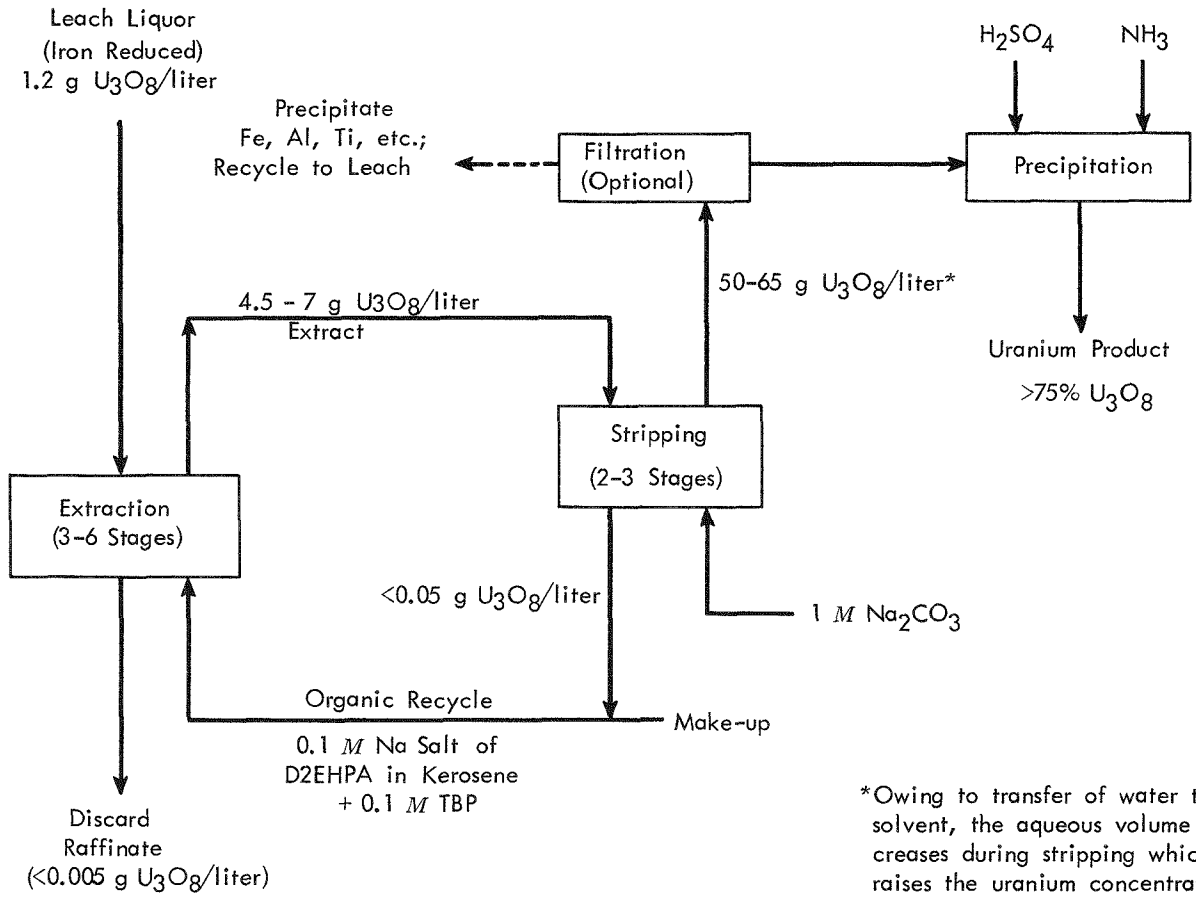
Uranium is extracted by cation exchange of uranyl ions and hydrogen ions between the aqueous phase and the di(2-ethylhexyl)phosphoric acid in the solvent phase. The stoichiometry of the exchange reaction is



This equation is an over-simplification and does not take into account polymerization<sup>12,13</sup> of the organophosphoric acid in the solvent or the competitive effects of uranium sulfate complexes in the aqueous phase.

Experimentally, the rate of uranium extraction was found to be first order with respect to the uranium concentration in the aqueous phase over a wide range of mixing conditions and uranium concentrations. If the reaction is treated as reversible and first order in both directions, the kinetic equation for batch extraction is

$$-dN/dt = -R \, dC/dt = A' [k_1 C - k_{-1} (C_0 - C)] \quad (1)$$



\*Owing to transfer of water to the solvent, the aqueous volume decreases during stripping which raises the uranium concentration above that expected from the initial flow volumes.

Fig. 3.1. Dapex process for uranium.

At equilibrium

$$k_1 C_{eq} = k_{-1} (C_0 - C_{eq}) \quad (2)$$

Substituting in eq. 1 and regrouping,

$$(k_1 + k_{-1}) \frac{A'}{R} dt = \frac{dC}{C - C_{eq}}$$

On integration, when  $t = 0$ ,  $C = C_0$ ,

$$(k_1 + k_{-1}) \frac{A'}{R} t = -\ln \left( \frac{C - C_{eq}}{C_0 - C_{eq}} \right) \quad (3)$$

Let  $k = k_1 + k_{-1}$  and substitute  $aV$  for  $A'$ ; then

$$ka = -\ln \left( \frac{C - C_{eq}}{C_0 - C_{eq}} \right) \frac{R}{V} \frac{1}{t} \quad (4)$$

If the stage efficiency,  $E$ , is defined by

$$E = \frac{C_0 - C}{C_0 - C_{eq}} \quad (5)$$

the final form of the batch kinetic equation is

$$ka = -\ln (1 - E) \frac{R}{V} \frac{1}{t} \quad (6)$$

The effective rate constant,  $ka$ , is evaluated by sampling over a period of mixing time at constant power input and phase ratio. The rate constant is directly proportional to the slope of the curve obtained by plotting  $\ln (1 - E)$  vs  $t$ .

For continuous flow the kinetic equation derived from eq. 6 and the theory of short-circuiting presented by MacMullin and Weber<sup>14</sup> is

$$ka = \left( \frac{E}{1 - E} \right) \frac{R}{V} \frac{1}{t} = \left( \frac{E}{1 - E} \right) \frac{A}{V} \quad (7)$$

Here the effective rate constant is evaluated by operating a continuous-flow mixer under constant mixing conditions and flow rates and sampling at steady state. The rate constant is calculated from the measured stage efficiency, mixer volume, and flow rates. The numerical values of  $ka$  are very nearly the same as those for the over-all mass transfer coefficients based on the aqueous phase. The latter are calculated from Murphree stage efficiencies and are not so easily calculated for batch mixing because graphical integration is required.

Two systems (Table 4.1) were investigated to cover a range of chemical compositions, solvent strengths, and phase ratios for uranium recovery from sulfuric acid leach liquors. System A is typical of the conditions

Table 4.1. Composition and Physical Properties

	System A	System B
Solvent		
Di(2-ethylhexyl)phosphoric acid, <u>M</u>	0.16	0.10
Tributyl phosphate, g/liter	35	30
Diluent	kerosene	kerosene
Density, g/cc	0.83	0.82
Viscosity, centistokes	2.4	2.3
Aqueous feed		
U, g/liter	5.2	1.2
Fe <sup>3+</sup> , g/liter	0.52	0.33
Al, g/liter	2.6	2.8
V <sub>i</sub> (total), g/liter	4.8	0.94
V <sup>4+</sup> , g/liter	1.4	0.94
SO <sub>4</sub> <sup>2-</sup> , g/liter	89	51
pH	0.3	1.1
Density, g/cc	1.08	1.05
Viscosity, centistokes	1.14	1.06
Interfacial tension, dynes/cm	18.3	19.0
Phase ratio (A/O)	1/1	4/1

in mills processing uranium-vanadium ores and system B is typical of conditions for processing of most uranium ores. The equilibrium distribution of uranium for both systems is shown in Fig. 4.1. The mixer was a baffled tank agitated with a flat-bladed turbine in the center of the liquid. The liquid depth was equal to the tank diameter. Three geometrically similar mixers, 6-, 12-, and 20-in. dia, were used (Fig. 4.2). The total power input to the mixer was calculated from correlations of turbine size and speed and liquid properties as reported by Rushton<sup>15</sup> and Laity:<sup>16</sup>

$$P = K\rho N^3 D^5 / g$$

The specific power input was calculated in horsepower per 1000 gal of mixer contents from the total power and total liquid volume of the mixer.

The mixers were operated with both batch and continuous flow to study the effects of power input, phase ratio, temperature, and type of dispersion on the rate of uranium extraction. Scaleup relations were determined batchwise in 6-, 12-, and 20-in.-dia mixers and with continuous flow in 6- and 12-in.-dia mixers.

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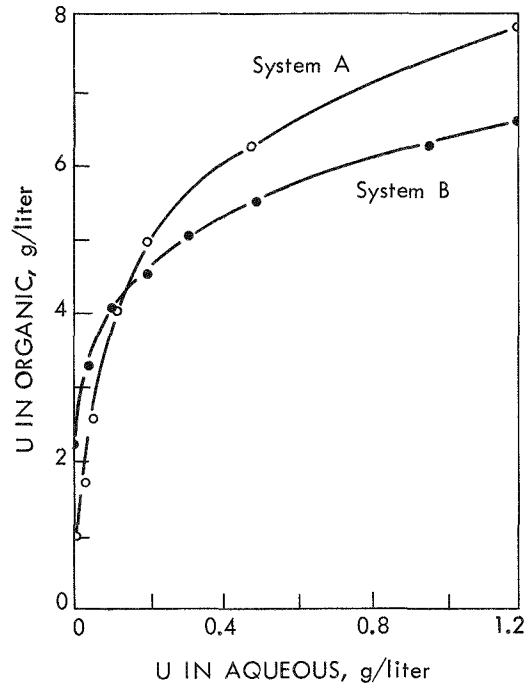
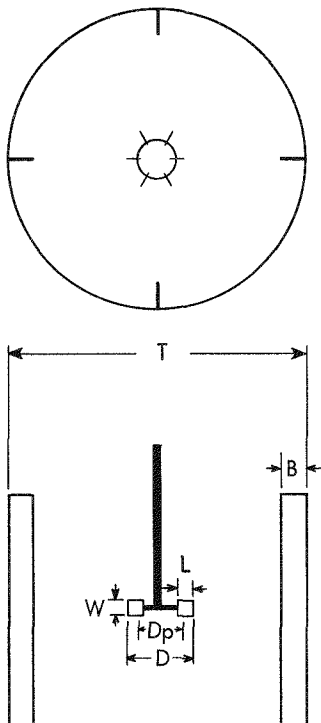


Fig. 4.1. Equilibrium distribution of uranium between aqueous and organic phases.



T	D	B	V
Inches		Gallon	
6	2	0.6	0.74
12	4	1.2	5.9
20	6	2.0	27.0

DIMENSION RATIOS OF TURBINE

$$D:D_p:L:W = 24:16:6:5$$

Fig. 4.2. Baffled tank mixers.

4.1 Batch Mixing (See Table 10.1, Appendix)

The batch technique for determining the rate constant consisted of mixing measured volumes of aqueous feed and solvent under constant mixing conditions and sampling the mixture over a time sequence sufficient to obtain equilibrium. The samples were dipped from the mixer without interruption of mixing, and the separated phases were analyzed. The effective rate constant was evaluated by plotting  $\ln(1 - E)$  vs  $t$  according to eq. 6. Typical curves (Fig. 4.3) are straight lines to about 95% approach to equilibrium. The intercept at zero time is not  $\ln 1.0$  because extraction continues during the time required for the phases to separate after the sample is withdrawn from the mixer; therefore the actual contact time is greater than the indicated sample time by  $\Delta t$ . The observed time for phase separation of the samples is approximately  $2\Delta t$ . However, the evaluation of the rate constant is not affected by  $\Delta t$  because it is calculated from the slope of the curve  $\ln(1 - E)$  vs  $t$ .

Effect of Type of Dispersion. With proper startup procedures either an oil-in-water (aqueous continuous) or a water-in-oil (organic continuous) type dispersion can be formed and maintained at phase ratios ranging from 10% to 80% dispersed phase without phase inversion. For example, an oil-in-water type dispersion is obtained by starting the agitator with only aqueous phase in the mixer and then adding the solvent. Conversely, water-in-oil type dispersions are obtained by starting the agitator with only solvent in the mixer. The type of dispersion is easily determined by measuring the electrical resistance of the mixture. Since the resistance of the solvent is much higher than that of the aqueous feed, when the organic phase is continuous the resistance of the dispersion is high, >10,000 ohms, and when the aqueous phase is continuous the resistance is low, 1-100 ohms.

The type of dispersion is important in practical solvent extraction applications because of its effect on phase separation rates and entrainment. Consequently, the rate of uranium extraction was compared for both aqueous continuous and organic continuous dispersions over a range of power inputs. The results show no significant effect of dispersion type on the rate of uranium extraction:

Power, hp/1000 gal	$k_a, \text{min}^{-1} D/T = 1/2$	
	<u>Aq. Cont.</u>	<u>Org. Cont.</u>
5.9	0.88	0.79
20	1.35	1.29
67	2.18	2.18
220	2.68	2.89

Effect of Temperature. The effect of temperature on rate of extraction was investigated because of its practical importance in the plant and also to aid in determining whether the rate-limiting step was

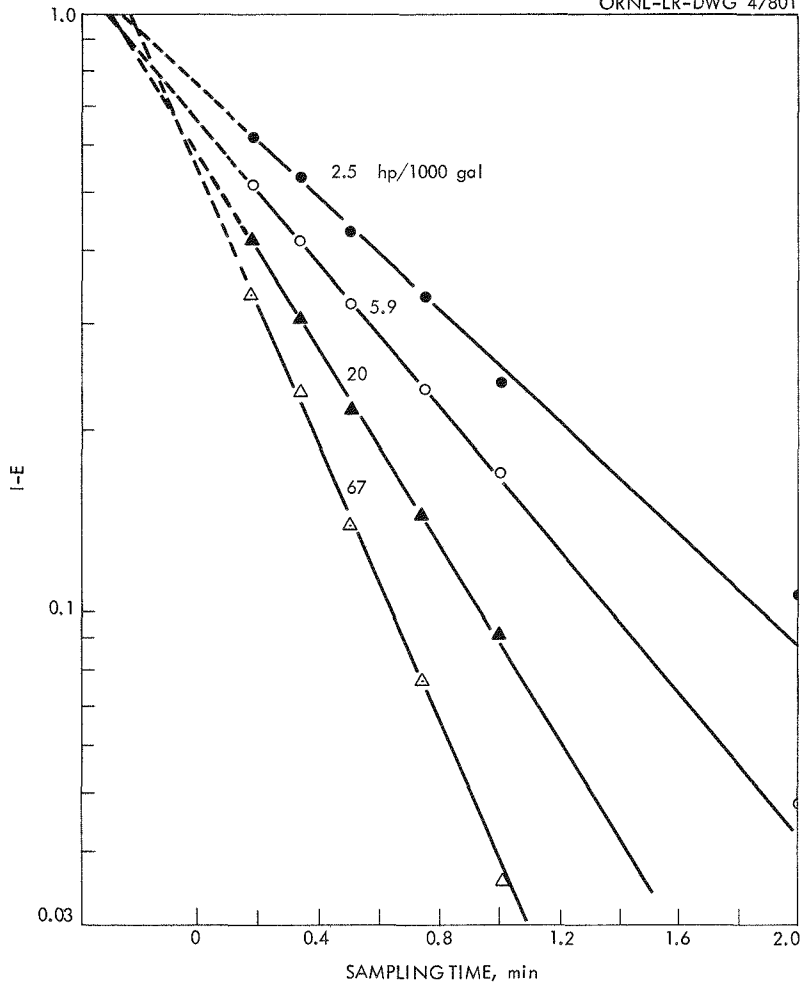


Fig. 4.3. Typical rate curves for batch mixing.

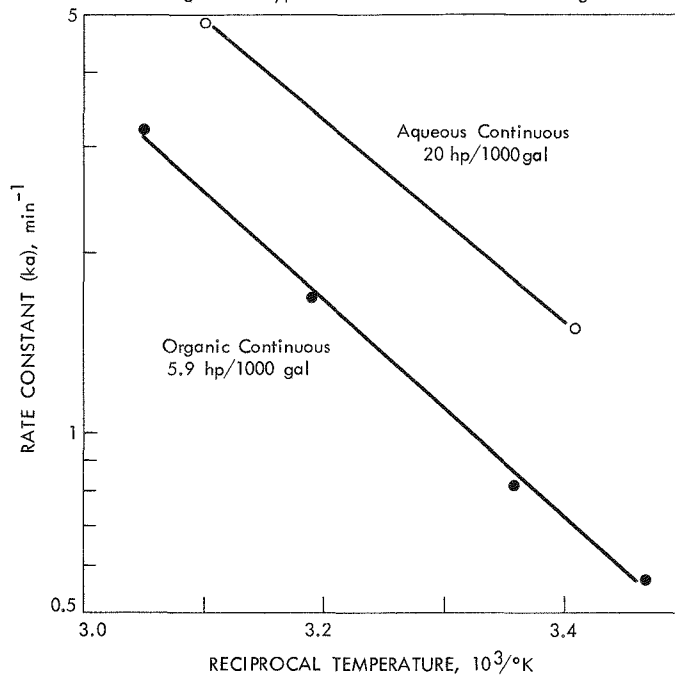


Fig. 4.4. Temperature dependence of uranium extraction rate.



diffusion or ion exchange. In a series of organic-continuous tests in a 6-in.-dia mixer, with a constant power input of 5.9 hp/1000 gal and a turbine speed of 400 rpm,  $k_a$  varied from 0.57  $\text{min}^{-1}$  at 15°C to 3.23 at 55°C. The interfacial area per unit volume in two aqueous-continuous tests, at 20 hp/1000 gal and 600 rpm, was nearly independent of temperature as calculated from Rodger's<sup>17</sup> correlation: 46.9  $\text{cm}^{-1}$  at 20°C and 48.2  $\text{cm}^{-1}$  at 49°C. Therefore the observed change in rate of extraction with temperature was not caused by the change in interfacial area but by the change in the specific rate of transfer.

The plot (Fig. 4.4) of  $\log k_a$  vs reciprocal temperature is a straight line for the organic-continuous tests at 400 rpm, and the two points for aqueous-continuous mixing at 600 rpm lie parallel to this line. The heat of activation  $\Delta H_a$ , was calculated from the data by means of the integrated form of Arrhenius' equation,

$$\log \frac{k_{a2}}{k_{a1}} = \frac{\Delta H_a}{2.303R} \left( \frac{T_2 - T_1}{T_2 T_1} \right)$$

The  $\Delta H_a$  was 8950 cal/mole for the organic-continuous and 7920 for the aqueous-continuous tests. For diffusion processes  $\Delta H_a$  is generally of the order of 5000 cal/mole, while for most chemical reactions it is greater than 10,000 cal/mole;<sup>18</sup> hence the temperature effect is inconclusive as to whether the rate-controlling step is an exchange reaction or diffusion.

Effect of Ratio of Turbine Diameter to Tank Diameter (D/T). In general, at constant power input, the efficiency of mixing of immiscible liquids increases with decreasing ratio of turbine to tank diameter (D/T) because of increasing turbulence.<sup>19</sup> The effect of D/T on uranium extraction was measured in a 12-in.-dia tank with 2-, 3-, 4-, 6-, and 8-in. turbines at a constant power input of 20 hp/1000 gal. The resulting rate constants (Fig. 4.5) increased nearly linearly with decreasing turbine diameter. The rate constant at D/T = 1/6 was about double that at D/T = 2/3. The lower limit of D/T will be determined by the pumping rate of the turbine, which is proportional to  $ND^3$ . If the pumping rate in a continuous-flow mixer is too low, excessive short-circuiting can occur with resultant loss of efficiency. To avoid this effect, a D/T value of 1/3 was selected as a standard for study of other mixing variables.

Effect of Power Input. The effect of power input on the rate of uranium extraction for both systems A and B (Table 4.1) was measured in 6-, 12-, and 20-in.-dia mixers with the power input varied from 2.5 to 220 hp per 1000 gal of mixer volume (Table 4.4). The rate constants,  $k_a$ , calculated at a D/T ratio of 1/3 for each test are plotted against power input in Fig. 4.6. A single curve through all points is a straight line with a slope of 1/3, showing that  $k_a$  is proportional to approximately the cube root of power input. The good agreement of data obtained from different systems confirms the expected dependence of  $k_a$  on phase ratio in eq. 7. The agreement shows that the wide differences in chemical composition of the feed liquor and extraction agent concentration have virtually no effect on the rate of uranium extraction.

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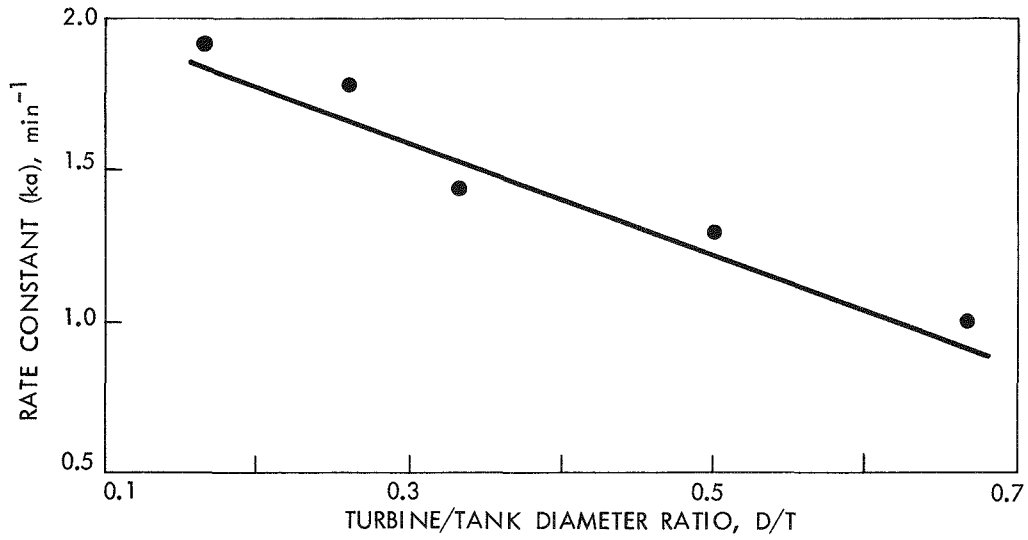


Fig. 4.5. Effect of D/T ratio on rate of extraction in batch mixer.

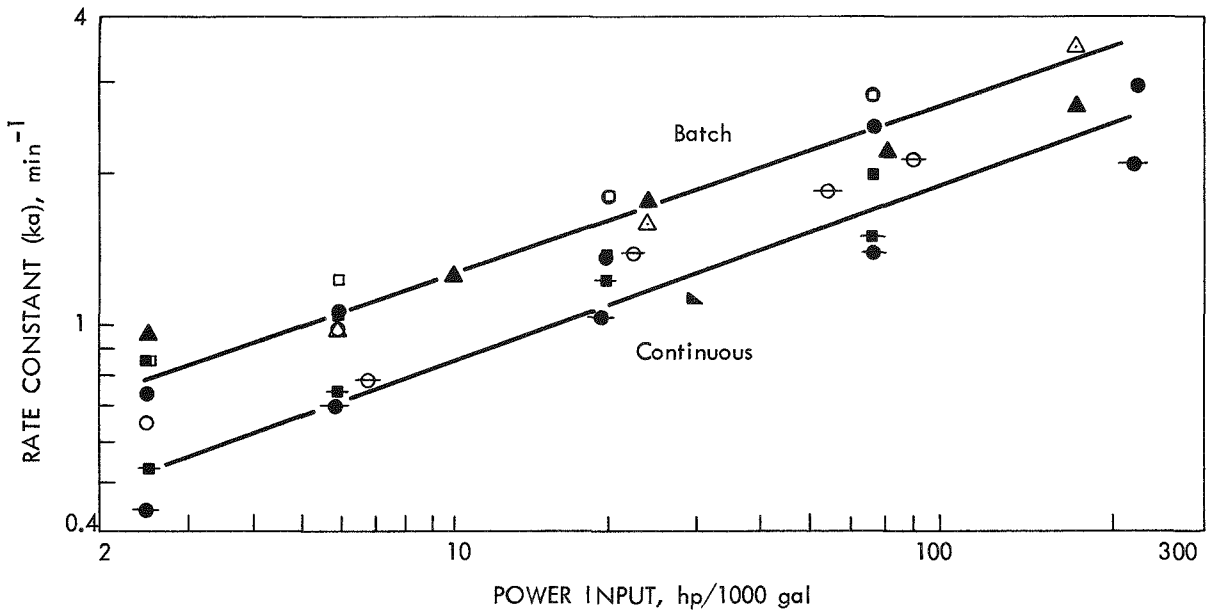


Fig. 4.6. Correlation of rate constants with power input in batch and continuous flow experiments.

As shown in Fig. 4.6, the rate constant at constant power input is nearly independent of the size of geometrically similar mixers over the entire range of power inputs tested. Thus scaleup of mixers can be based simply on constant power input per unit volume of mixer contents.

Effect of Interfacial Area. Although the term  $ka$  is all that is required for design and scaleup purposes, fundamentally it is of interest to see how  $k$  alone varies with the mixing parameters. From specific interfacial areas measured photographically by Rodger and Trice<sup>20</sup> for 50% oil-in-water type dispersions in 6-in.-dia mixing vessels geometrically similar to those used here, the values calculated for  $k$  are virtually constant and independent of turbine speed:

<u>Turbine Speed,</u> <u>rpm</u>	<u><math>ka,</math></u> <u><math>\text{min}^{-1}</math></u>	<u><math>a,</math></u> <u><math>\text{cm}^{-1}</math></u>	<u><math>k,</math></u> <u><math>\text{cm}/\text{min}</math></u>
350	0.88	162 <sup>a</sup>	$5.4 \times 10^{-3}$
524	1.35	242 <sup>a</sup>	$5.6 \times 10^{-3}$
787	2.18	365 <sup>a</sup>	$6.0 \times 10^{-3}$
1170	2.68	520 <sup>a</sup>	$5.0 \times 10^{-3}$

<sup>a</sup>Data obtained by extrapolation.

To explore further the value of  $k$ , the rate of uranium extraction was measured in a mixer where a fixed interface was maintained between the phases and each phase was agitated with separate turbines. The rate was determined at turbine speeds of 30 and 60 rpm and  $ka$  was calculated from a plot of  $\ln(1-E)$  vs sample time (eq. 6). The data fit on a straight line going through the origin, as expected. The value of  $ka$  was independent of turbine speed,  $1.95 \times 10^{-4} \text{ min}^{-1}$  at 30 rpm and  $2.07 \times 10^{-4}$  at 60 rpm. The area of the interface was  $186 \text{ cm}^2$ , the total volume was 5 liters, and the specific interfacial area was  $0.0372 \text{ cm}^{-1}$ . The calculated value of  $k$  was  $5.40 \times 10^{-3} \text{ cm}/\text{min}$ , which agrees with the value of  $k$  obtained with turbulent mixing at a 4000-fold greater specific area,  $160 \text{ cm}^{-1}$ .

#### 4.2 Continuous-flow Mixing (Table 10.2, Appendix)

The kinetics of uranium extraction were studied in 6- and 12-in.-dia mixers at flow rates ranging from 0.15 to 5 gpm in two chemical systems (Table 4.1). The stage efficiency was calculated by eq. 5 from the equilibrium curves and effluent samples. The extraction rate constant was calculated from eq. 7.

The equipment used in continuous-flow tests is shown in Fig. 4.7. The aqueous feed and the solvent were prepared in large batches and stored in head tanks. Each phase was pumped by a metering pump to the mixer. The phases entered through a concentric entrance to minimize pre-mixing. The mixed phases overflowed from the mixer to a catch tank,

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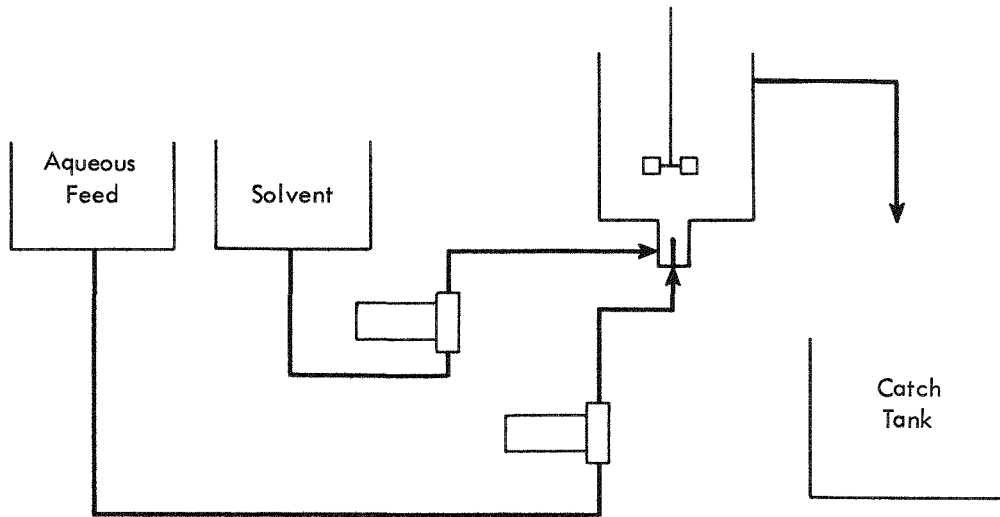


Fig. 4.7. Equipment for continuous-flow mixing.

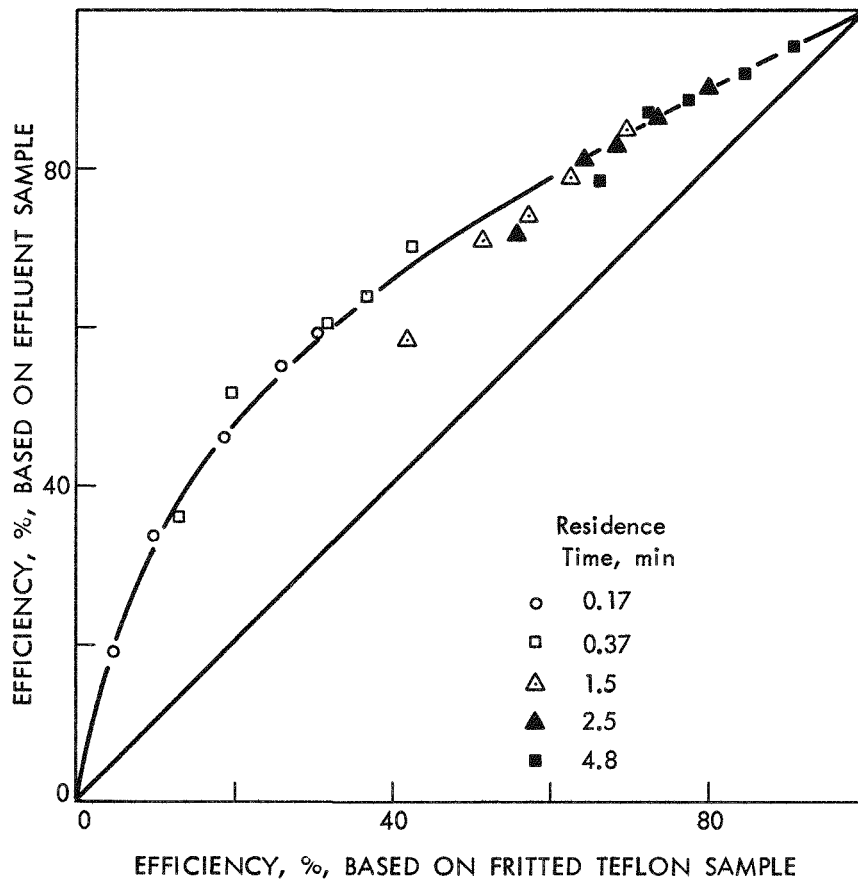


Fig. 4.8. Comparison of sampling methods (system B).

where the phases were separated and measured after each run. The solvent was stripped with sodium carbonate and was acidified with sulfuric acid before re-use.

Sampling. The mixer contents were sampled at steady state, a minimum of ten volume changes being allowed for steady state to be reached. Samples of the dispersed phase were the best measure of the mixer contents, these being taken through fritted Teflon with aqueous-continuous dispersions and through coarse fritted glass with organic-continuous dispersions. Samples of the mixed-phase effluent from the mixer do not give representative data on the extraction in the mixer because extraction continues while the phases are separating. With the preferentially wetted filters, controlling the head across the filter at 3-4 in. H<sub>2</sub>O made it possible to obtain in about 2 min a 15-ml sample of one phase containing virtually none of the other. It was difficult to obtain only one phase at high power input or at low phase ratio of dispersed phases because of the small drop size of the dispersed phase. Although material balances were poor with the filtered samples, with both types of dispersion they indicated less uranium transference to the solvent than did mixed-phase samples (Table 4.2).

Table 4.2. Comparison of Sampling Methods

Run No.	Uranium Concentration, g/liter				
	Aqueous		Organic		
	Fritted Glass	Effluent Stream	Fritted Teflon	Calc from Fritted Glass	Effluent Stream
Aqueous Continuous					
573	3.6	3.2	0.84	1.9	2.3
574	2.5	1.9	1.6	2.8	3.4
575	2.1	1.3	2.4	3.2	4.0
Organic Continuous					
576	4.7	3.2	1.7	1.2	2.7
581	3.8	1.3	3.0	1.7	4.2
583	2.9	0.9	3.4	2.5	4.5

Both Teflon and glass filters were tested with both types of dispersion. Under aqueous-continuous conditions the uranium concentration of the solvent calculated from a fritted-glass sample of the aqueous phase was consistently higher than the concentration measured in the solvent sample taken through Teflon. It is not possible for the solvent to lose uranium as it is filtered through the fritted Teflon; and the measured uranium concentration in the solvent therefore cannot be too low. The other possibility is that the calculated value is too high, which would

be the case if further extraction of uranium occurred while the aqueous sample was being taken with the fritted glass. Similarly, for solvent-continuous dispersion the value calculated on the solvent from the aqueous fritted glass sample is less than the value measured in the fritted Teflon solvent sample. The extraction based on the effluent sample, which approximates the total efficiency of a single mixer-settler stage, differs most from the efficiency based on the fritted Teflon sample when the residence time in the mixer is small (Fig. 4.8). This is because the contact time during phase separation is constant and therefore the proportion of the extraction that occurs during phase separation is greatest at low residence time in the mixer.

Effect of Residence Time on Rate Constants. Equation 7 predicts that the rate constant,  $k_a$ , will be independent of residence time. Data obtained with the 6-in.-dia mixer at total flow rates ranging from 0.15 to 5.0 gpm showed no systematic change in  $k_a$  for residence times varying from 0.17 to 4.8 min at different levels of power input.

Effect of Power Input. The rate constants were correlated with power input for both systems A and B in 6- and 12-in. mixers (Fig. 4.6, curve b). The average value for  $k_a$  at various residence times was used (Table 4.3). The D/T ratio was 1/2 in the 6-in.-dia mixer and 1/3 in the 12-in.-dia mixer. In order to compare the data at a constant D/T ratio, the rate constants for 6-in.-dia mixers were calculated at D/T = 1/3, using the curve (Fig. 4.5) showing the D/T effect for batch mixing.

The slope of curve b in Fig. 4.6 is about 1/3, showing that the rate constant is directly proportional to the cube root of power input. In the curve are included data obtained in routine operation of plant-size mixers in the Dapex extraction circuit at Climax Uranium Co., Grand Junction, Colo. (detailed data in Table 8.1). Good agreement of data from 6-, 12-, and 36-in.-dia mixers shows the independence of rate with respect to mixer size.

Effect of Phase Ratio. Rate constants are slightly greater for System A than for system B. The two systems differ in uranium concentration, pH, D2EHPA concentration, and phase ratio. In order to define the effect of only phase ratio on rate of extraction, one series of runs was made with system B simulating solvent recycle so that the phase ratio in the mixer was 1/1 instead of the normal ratio (A/O) of 4/1. The mixing was controlled to maintain the solvent phase continuous. The rate constants agreed very closely at low power input, but at high power input were greater at a phase ratio of 1/1 than at 4/1 (Table 4.4). Most of the difference is probably caused by a change in the specific interfacial area. Extrapolation of interfacial area data reported by Vermeulen<sup>21</sup> shows that the value of "a" should be about 40% greater at a phase ratio of 1/1 than at 4/1.

The stage efficiencies were nearly independent of phase ratio at constant aqueous flow rate but not at constant residence time, which is predicted by eq. 7. This is of practical importance to operating plants; for example, if the mixers were designed for a phase ratio of 4/1

Table 4.3. Effect of Residence Time on Extraction Rate Constant

Residence Time, min	Continuous Phase	Extraction Rate Constant ( $k_a$ ), min <sup>-1</sup>							
		2.5 hp/ 1000 gal	5.9 hp/ 1000 gal	6.0 hp/ 1000 gal	20 hp/ 1000 gal	55 hp/ 1000 gal	67 hp/ 1000 gal	80 hp/ 1000 gal	220 hp/ 1000 gal
<u>System A</u>									
0.37	Aqueous	0.31		0.78	1.50	-	3.09	-	
0.37	Organic	0.23		0.28	0.70	-	1.50	-	
0.82	Organic	-		0.86	1.40	2.02	-	2.25	
1.5	Organic	-		0.65	1.10	1.51	-	1.51	
1.5	Organic <sup>a</sup>	0.49		0.66	1.13	-	1.55	-	
1.5	Aqueous <sup>a</sup>	-		0.83	1.40	-	1.60	-	
avg.		0.34		0.68	1.21	1.77	1.93	1.88	
<u>System B</u>									
0.17	Aqueous	0.30	0.64		1.36		2.09		2.69
0.37	Aqueous	0.41	0.69		1.25		1.58		2.06
1.5	Aqueous	0.54	0.81		0.98		1.33		1.74
2.5	Aqueous	0.53	0.75		0.89		1.15		1.75
4.8	Aqueous	0.43	0.59		0.74		1.15		2.15
avg.	Aqueous	0.44	0.70		1.04		1.46		2.08
Standard deviation, % of avg.		21	12		24		26		18

<sup>a</sup>Uranium concentration in aqueous feed was 1.7 g/liter.

Table 4.4. Effect of Phase Ratio on Extraction at Various Aqueous Flow Rates and Residence Times,

System B						
	E, * %			ka, * min <sup>-1</sup>		
	A/O = 4/1		A/O = 1/1	A/O = 4/1		A/O = 1/1
Power Input, hp/1000 gal	0.40 gpm, 1.5 min	0.24 gpm, 2.5 min	0.24 gpm, 1.5 min	0.40 gpm, 1.5 min	0.24 gpm, 2.5 min	0.24 gpm, 1.5 min
2.5	43.2	56.3	53.9	0.43	0.42	0.39
5.9	53.7	65.2	66.0	0.65	0.60	0.64
20	58.3	68.6	76.2	0.78	0.71	1.06
67	65.1	74.1	84.5	1.06	0.92	1.81
220	71.2	81.3	86.4	1.39	1.40	2.11

\* Based on samples of dispersed phase taken with fritted samplers.

and subsequently it was necessary to operate solvent-continuous with recycle so that the phase ratio in the mixer was 1/1 to prevent emulsions (see Sec. 7.1, p.30), the stage efficiency would not be significantly different at the same aqueous throughput.

#### 4.3 Correlation of Batch and Continuous Data

The agreement between the rate constants for batch and continuous flow is close enough to warrant the use of the batch technique to scout a new system because of its relative simplicity. The rate of uranium extraction is described by the rate constants calculated from eq. 6 for batch mixing and eq. 7 for continuous flow. Assuming that a homogeneous dispersion is formed in the mixer and the interfacial area is the same for batch and continuous mixing, the rate constants should be equal at the same power input. The slope of the average batch rate curve is virtually the same as that for continuous flow, showing the same dependence on power input and independence of mixer size and chemical system. The batch rate constants are consistently greater than those for continuous flow, and the reason for the discrepancy is not known. The most probable causes are the method of sampling and differences in interfacial area between batch and continuous mixing. As pointed out in the sampling section, several different methods were used for the continuous flow tests and the method selected was to sample the dispersed phase because it was found to be farthest from equilibrium. If the sample of the continuous phase is used, the agreement between batch and continuous mixing is good. However, this may be only fortuitous. There are no published data for comparison of interfacial area for continuous flow with batch mixing; consequently it is not possible to check this as an explanation for the lower rate constants with continuous-flow mixing.



#### 4.4 Scaleup of Mixers

Both batch and continuous-flow mixing tests show that the rate constant for uranium extraction is independent of mixer diameter at constant specific power input and D/T ratio. This is the basis for direct scaleup of geometrically similar mixers.

The first step of scaleup is to select the combination of power input and residence time required to give the desired stage efficiency according to eq. 7 in the scale model mixer. Next, the volume of the large scale mixer is calculated from the plant flow rates to obtain the required residence time. The total power input to the mixer is calculated from the volume and the required specific power input. The mixer diameter and turbine diameter are fixed by geometric similitude. Finally, the turbine speed is calculated from the total power and turbine diameter from the power equation,  $P = K_p N^3 D^5 / g$ . The parameters and scaleup method are summarized in Table 4.5.

Table 4.5. Scaleup of Mixers

Parameter	Small Model	Large Size
Flow rate	$Q_1$	$Q_2$
Specific power input	$P_1/V_1$	$P_2/V_2 = P_1/V_1$
Residence time	$t_1$	$t_2 = t_1$
Mixer volume	$V_1 = t_1 Q_1$	$V_2 = t_1 Q_2$
Total power	$P_1 = (P_1/V_1) \times V_1$	$P_2 = (P_1/V_1) \times V_2$
Mixer diameter	$T_1 = (4/\pi V_1)^{1/3}$	$T_2 = (4/\pi V_2)^{1/3}$
Turbine-mixer dia ratio	$D_1/T_1$	$D_2/T_2 = D_1/T_1$
Turbine speed	$N_1 \sim (P_1/D_1^5)^{1/3}$	$N \sim (P_2/D_2^5)^{1/3}$

#### 5.0 KINETICS OF VANADIUM AND IRON EXTRACTION

The rates of extraction of vanadium(IV) and iron(III) were surveyed in batch tests with 6- and 12-in.-dia mixers. The conditions were similar to those encountered in application of the Dapex process for recovery of vanadium. The solvent was 0.24 M di(2-ethylhexyl)phosphoric acid in kerosene containing 50 g of tributylphosphate per liter and the composition of the aqueous feed in grams per liter, was 0.9 V(IV), 0.2 Fe(III), 3.0 Al, 50 SO<sub>4</sub>, and 1.9 PO<sub>4</sub> at pH 1.8. The rates of vanadium and iron

extraction were measured simultaneously as a function of power input at A/O phase ratios of 4/1 and 1/1. The rate constants,  $k_a$ , were evaluated in the same way as described for uranium in Sec. 4.0. The test data are shown in Table 10.3 (Appendix) and the rate constants are correlated with power input in Fig. 5.1.

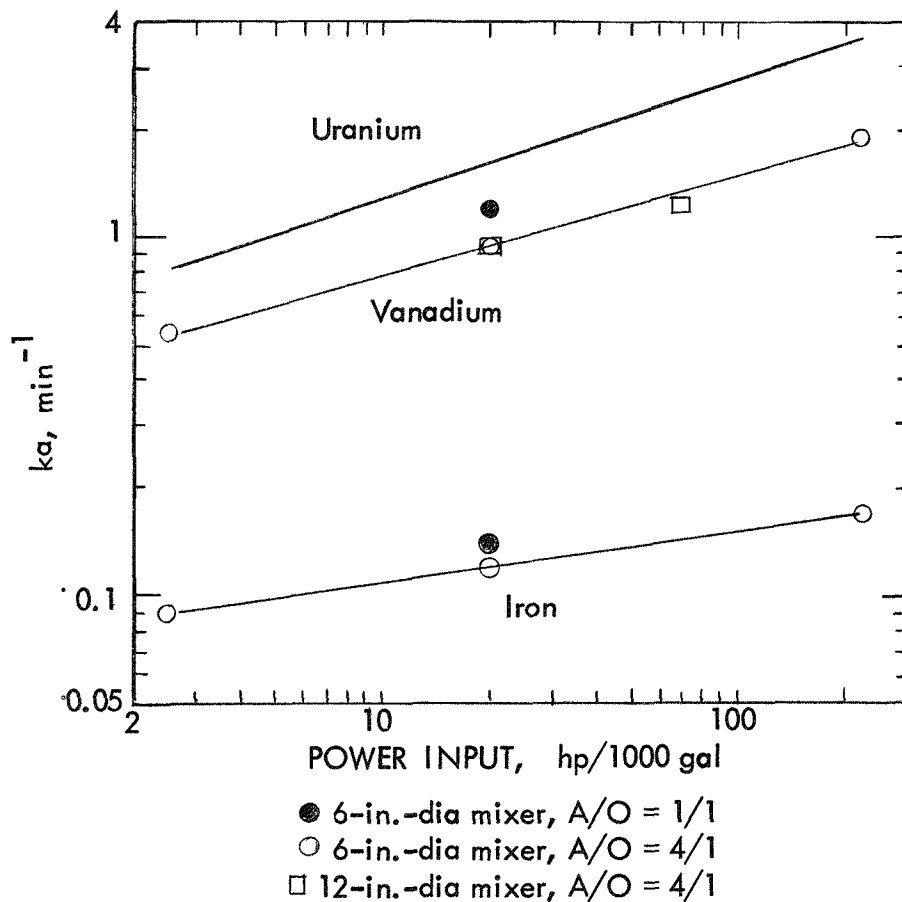


Fig. 5.1. Batch extraction rate constants for uranium, vanadium, and iron.

The slopes of the curves (Fig. 5.1) are very similar for uranium, vanadium, and iron, showing that the rate constant for each is dependent on the cube root of power input for both 6- and 12-in.-dia mixers. The relative magnitude of the rate constants at constant power shows that the rate of vanadium extraction is lower than that of uranium and that the iron rate is about one order of magnitude lower. For example, at a power input of 20 hp/1000 gal, the values of the rate constants are 1.4 for uranium, 0.84 for vanadium, and 0.10 for iron.

Direct comparison of the rate constants at an A/O phase ratio of 1/1 vs 4/1 shows that the rates of vanadium and iron extraction are about 25% greater at 1/1. The rates were not affected by the type of dispersion, being the same for both oil-in-water and water-in-oil type dispersions at a phase ratio of 1/1. These results are consistent with those found for uranium extraction.

The batch values of the rate constants can be used to calculate the stage efficiency of the mixer operated with continuous flow by using eq. 6. For example, with mixing conditions for good uranium extraction, 30 hp/1000 gal and 2 min residence time at a phase ratio of 1/1, the calculated stage efficiency is 87% for uranium, 79% for vanadium, and 31% for iron.

## 6.0 KINETICS OF URANIUM STRIPPING WITH SODIUM CARBONATE

The rate of uranium stripping from the solvent with solutions of sodium carbonate was determined in a 6-in.-dia mixer by both the batch technique and continuous flow. The rate of stripping is significantly greater than the rate of extraction; therefore only a survey study was made. Batch data showed that the rate of stripping increases with increased excess sodium carbonate and increased power input. Continuous-flow tests showed that greater than 90% stage efficiency was attained over a range of uranium concentrations simulating two countercurrent stripping stages.

The solvent was 0.16 M di(2-ethylhexyl)phosphoric acid and 35 g of TBP per liter in kerosene. It was loaded with uranium by contacting with a synthetic leach solution containing uranium, iron, aluminum, tetravalent vanadium, phosphate, and sulfate. The stripping agent was aqueous sodium carbonate.\* The O/A phase ratio was constant at 5/1, and the concentration of sodium carbonate was adjusted to give the desired excess. The mixing was controlled to produce solvent-continuous dispersions and the temperature was 24 to 30°C.

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\*The stoichiometric requirements were assumed to be 3 moles of  $\text{Na}_2\text{CO}_3$  per mole of uranium plus 0.5 mole per mole of free D2EHPA.

## 6.1 Batch Stripping

The batch technique described for uranium extraction (Sec. 4.1, p. 13) was used to study the range of variables affecting stripping. The first-order kinetic equation

$$k_a = -\ln(1 - E) \frac{R}{V} \frac{1}{t}$$

was used where stage efficiency,  $E$ , was calculated from the uranium concentrations in the solvent and  $R/V$  was the fraction of solvent in the mixer. The rate constant was evaluated from the slope of the curve obtained by plotting  $\ln(1 - E)$  vs  $t$ . In several cases rate curves were not well-defined because the rate was so high that equilibrium was reached in less than 20 sec.

Effect of Excess Sodium Carbonate. The effect of excess sodium carbonate on the stripping rate was measured at constant mixing power (420 rpm, 6 hp/1000 gal) and constant O/A phase ratio, 5/1. The amount of excess carbonate was varied by adjusting the concentration of the aqueous solution. The value of  $k_a$  for uranium stripping increased from 3.5 to 9.3  $\text{min}^{-1}$  as the excess sodium carbonate was increased from 19 to 55% (Table 6.1, Fig. 6.1).

For practical application the amount of excess sodium carbonate should be kept low to minimize chemical cost. Therefore the rate of stripping at 19% excess probably is typical for actual plant operation. Comparison of stripping rate with extraction shows that stripping is about three times faster at the same power input.

Effect of Power Input. The effect of power input on the rate of stripping was measured at constant O/A phase ratio, 5/1, with 55% excess sodium carbonate. The rate constant increased about threefold (9.3 to 24  $\text{min}^{-1}$ ) when the power was tripled (6 to 19 hp/1000 gal) (Table 6.1). The rate was too large to be measured at the next higher power level tested (63 hp/1000 gal).

## 6.2 Continuous Flow Mixing

Greater than 90% stripping stage efficiency was readily attained in both stages of a two-stage circuit with the 6-in.-dia mixer. At a residence time of 1.5 min the efficiency was greater than 90% at the lowest power input tested, 0.7 hp/1000 gal; at a residence time of 0.38 min the power required for 90% efficiency was about 10 hp/1000 gal (Table 6.2).

A two-stage stripping circuit was simulated by first contacting the loaded solvent with sodium carbonate and subsequently contacting the partially stripped solvent with fresh sodium carbonate. The solvent was the same as that used for the batch tests. The concentration of the stripping solution was 1 M  $\text{Na}_2\text{CO}_3$  and the phase ratio was adjusted so that 45% excess carbonate was used. The mixer was the 6-in.-dia baffled tank shown

Table 6.1. Effect of Carbonate Excess and Power Input on Stripping Rate

Mixer: 6 in. dia with 3-in. turbine  
 Solvent: 0.16 M DEHPA, 35 g of TBP per liter  
 Phase ratio, O/A: 5/1

Run No.	Power Input, gal/1000	Na <sub>2</sub> CO <sub>3</sub> Excess, %	Na <sub>2</sub> CO <sub>3</sub> Conc., M	Uranium Concentration in Solvent, g/liter								k <sub>a</sub> , min <sup>-1</sup>
				0 sec	10 sec	20 sec	30 sec	60 sec	120 sec	240 sec	480 sec	
11	6	19	0.73	5.2	2.9	1.5	0.68	0.37	0.37	0.38	0.36	3.5
9	6	32	0.81	5.2	2.3	0.56	0.31	0.36	0.33	0.33	0.33	4.7
7	6	41	0.87	5.2	1.6	0.33	0.30	0.33	0.32	0.32	0.32	6.8
1	6	55	0.95	5.2	1.1	0.32	0.29	0.30	0.31	0.31	0.31	9.3
3	19	55	0.95	5.2	0.34	0.30	0.31	0.30	0.30	0.30	0.30	24
5	63	55	0.95	5.2	0.31	0.29	0.28	0.30	0.29	0.30	0.30	>24

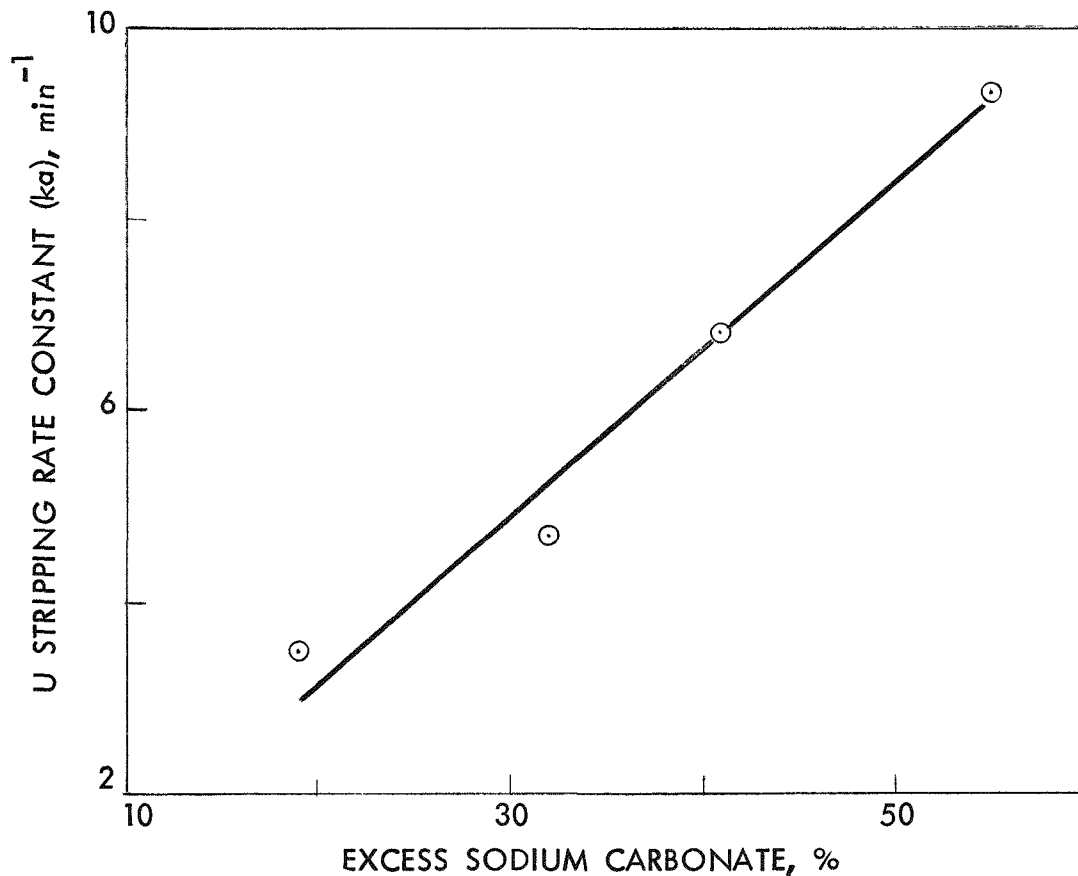


Fig. 6.1. Effect of excess sodium carbonate on uranium stripping rate.

Table 6.2. Stage Efficiency for Uranium Stripping With Sodium Carbonate

Turbine Speed, rpm	Power Input, hp/1000 gal	Stage Efficiency, %			
		0.38 min Residence		1.5 min Residence	
		1st Stage	2nd Stage	1st Stage	2nd Stage
200	0.7	-	-	93	94
300	2.2	73	96	98	99
400	5.3	82	98	99	99
600	18	95	98	99	99
900	60	98	98	100	-

in Fig. 4.2. The stage efficiency was calculated from the analysis of the organic effluent stream. The operating data are given in Table 10.4, Appendix.

### 6.3 Scaleup

Scaleup of mixers for uranium stripping has been assumed to follow the same relations developed for extraction, i.e. geometric similitude with constant power input and residence time. Limited confirmation of scaleup is shown by plant data (Table 6.3), although the plant mixers were the same size as those in the extraction circuit for simplicity of operation and maintenance. Consequently, the plant mixers are over-designed with respect to requirements for uranium stripping. However, the plant data do show that nearly 100% stage efficiency was obtained at power inputs and residence times of the same order of magnitude as that required in the small scale mixer.

Table 6.3. Comparison of Different Size Mixers for Uranium Stripping

Mixer		Total	Residence	Power	Stage
Dia,	Vol,	Flow Rate,	Time,	Input,	Efficiency,
ft	gal	gpm	min	hp/1000 gal	%
0.5	0.74	0.50	1.5	5.3	99
3.0	150	76	2.0	30	99
4.0	300	30	10	5	99

## 7.0 PHASE SEPARATION

Phase separation is generally considered a two-step process consisting of a primary break in which the bulk of the dispersion separates by sedimentation and coalescence into its component phases, followed by a secondary break in which drops of one phase entrained in the other settle out, leaving the phases clear. The rate of primary break is the controlling factor for design and operation of settlers. In both batch and continuous-flow tests, the flow capacity of settlers was determined as a function of the more important variables—type of dispersion, phase ratio, mixing power, and temperature. The use of a secondary settler for recovering solvent entrained in the raffinate was evaluated, and scaleup data were determined from three sizes of settlers. The amount of entrainment of aqueous in the extract and stripped solvent and of solvent in the loaded carbonate was measured, but no special effort was made to decrease it. Most of the phase separation studies were directed toward extraction rather than stripping problems because higher flow rates and larger equipment are involved. The basic principles of scaleup developed for settlers in the extraction section are assumed to apply in stripping.

The amount of entrainment after the primary break is usually not sufficient to cause loss of stage efficiency due to back mixing. As much as 10% backflow of either phase can be tolerated in either the extraction or stripping circuit.<sup>22,23</sup> However, entrainment in the streams leaving the end stages of the extraction and stripping circuits is important. Loss of solvent in the aqueous raffinate is economically significant because the uranium concentration in most feed liquors is relatively low. For example, for average uranium mill leach liquors, a loss of 1 gal of solvent per 1000 gal of raffinate is equivalent to 5¢/lb of U<sub>3</sub>O<sub>8</sub> product.

For both batch and continuous-flow tests the solvent was 0.1 M di(2-ethylhexyl)phosphoric acid in kerosene containing 30 g of TBP per liter. It was scrubbed with 10 vol of 1 M sodium carbonate solution and 50 vol of 0.5 M sulfuric acid to remove readily soluble components, thereby making it similar to plant solvent. The aqueous phase for most tests was a uranium-barren sulfate solution containing sodium sulfate and sulfuric acid, although actual plant liquors were used in some tests (Table 7.1).

#### 7.1 Batch Primary Break Tests

For the batch phase separation studies baffled tank mixers (Fig. 4.2) of 3, 6, and 12 in. dia were used. A dispersion was formed by agitation at a power input sufficient to yield a uniform dispersion, and the break time was the time required after the agitator was stopped until a sharp interface was formed between the bulk phases.

The general appearance of a dispersion during the primary break resembles a three-phase system in which a rather sharply defined dispersion band is sandwiched between relatively clear layers of organic and aqueous.<sup>6,17</sup> At one boundary the rate of travel toward the final interface is dependent on sedimentation of the dispersed drops and shows little activity. The other boundary is characterized by active coalescence of drops with neighboring drops and with the bulk phase which they contact. The coalescing interface for an aqueous-continuous dispersion is at the organic dispersion boundary, while for an organic-continuous dispersion the coalescing interface is at the aqueous dispersion boundary (Fig. 7.1).

As shown by settling profiles (Fig. 7.2), the rate of movement of both boundaries of several different dispersions in 12-in.-dia batch settlers is linear over most of the total break time. This shows that the rate of coalescence is virtually independent of dispersion volume and indicates that most of the coalescence occurs at the active interface rather than within the dispersion. Comparison of settling profiles for aqueous-continuous dispersions at A/O phase ratios of 4/1 and 1/1 shows a faster sedimentation rate for the dilute, 4/1, dispersion. This causes packing of the drops toward the coalescing interface until a close-packed dispersion is formed, after which the sedimentation rate falls off to about the same rate as that over the entire break time for the 1/1 dispersion.



Table 7.1. Physical Properties of Liquids Used

Liquid	Concentration, g/liter					pH	Density, g/ml		Viscosity, cp		Interfacial Tension with Solvent, dynes/cm	
	U	V	SO <sub>4</sub>	SiO <sub>2</sub>	Na <sub>2</sub> CO <sub>3</sub>		25°C	50°C	25°C	50°C	25°C	50°C
Solvent <sup>a</sup>							0.82	0.80	2.3	1.6		
Sulfate feed			70			1.0	1.07		1.0	0.70	20	22
Sodium Carbonate					100		1.09	1.08	1.3	0.83	1.5	1.7
<u>Kerr-McGee</u>												
Section 10	0.9	0.1	20	0.70		1.4	1.02		0.94		18	
Holly	1.3	0.1	31	2.0		1.6	1.05		1.07		19	
St. Anthony	1.1	0.1	30	2.2		0.8	1.03		0.97		17	
Recycle	0.8	0.1	48	0.94		0.6	1.04		1.03		19	
Shiprock	0.7	0.8	23	0.34		1.4	1.02		1.02		19	
KM 11	1.1	4.1	110	<0.10		0.5	-		-			
<u>Climax Uranium</u>												
Sand liquor No. 19	1.8	2.3	109	1.0		0.7	1.1		1.25		20	
Slime liquor, 5/8/58	5.1	3.8	78	<0.10		0.5	1.08		1.14		18	
<u>U. C. Nuclear</u>												
Old Rifle	2.1	2.9	31	1.6		1.4	1.05		1.08		19	
New Rifle	4.0	3.1	55	2.5		0.9	1.06		1.10		20	

<sup>a</sup>0.1 M di(2-ethylhexyl)phosphoric acid in kerosene containing tributyl phosphate, 30 g per liter.

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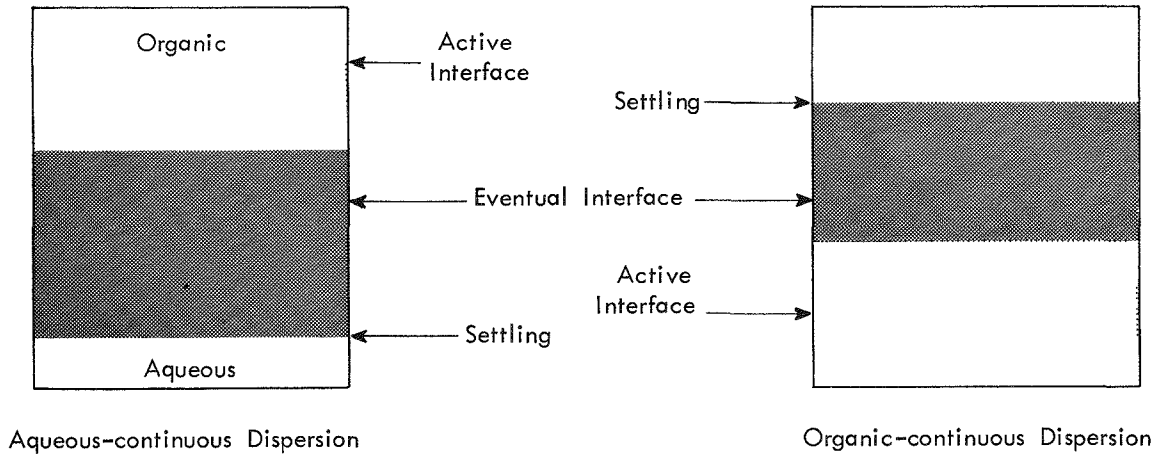


Fig. 7.1. Illustrations of types of coalescence.

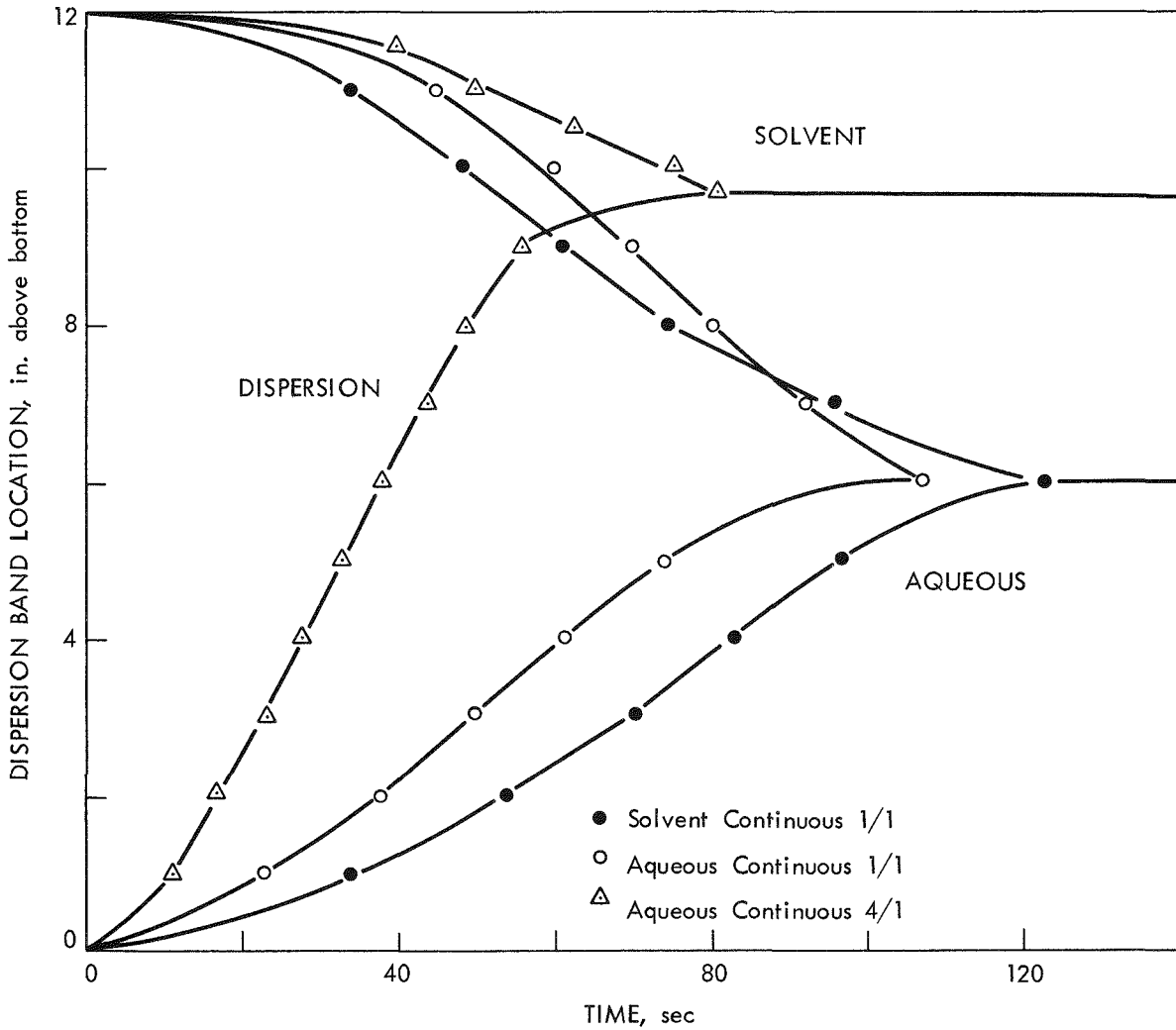


Fig. 7.2. Typical batch settling profiles in 12-in.-dia settler.

Effect of Mixing Power and Time on Primary Break. Neither mixing power from 4 to 160 hp/1000 gal nor mixing time from 1 to 30 min had any effect on primary break time for either aqueous- or organic-continuous dispersions (Table 7.2). This simplifies the design of mixer-settler units because the mixing can be selected to optimize stage efficiency without changing settler capacity.

Table 7.2. Effect of Mixing Power and Time on Primary Break

Settler: 6 in. dia  
Phase ratio, A/O: 1/1  
Temperature: 25°C

Mixer Power, hp/1000 gal	Mixing Time, min	Primary Break Time, sec	
		Aqueous Continuous	Organic Continuous
4	10	52	65
20		50	64
50		49	65
160		50	61
20	1	46	-
	5	45	-
	10	50	-
	30	49	-

Effect of Temperature on Primary Break. For both types of dispersion, the break time decreased with increasing temperature, from 150 sec at 15°C to 70 sec at 55°C in a 12-in.-dia settler (Fig. 7.3). Probably most of the temperature effect is due to a change in the viscosity of both phases, which decreased in about the same proportion as the break time with increasing temperature, while density and interfacial tension were relatively independent of temperature.

Effects of pH and Density of Aqueous Phase on Primary Break. Variations in the aqueous phase of pH from 0.5 to 1.5 and of density from 1.02 to 1.08 g/ml had no significant effect on primary break time (Table 7.3). These pH and density limits include the variations of most plant leach liquors.

Plant Leach Liquors. Primary break times for 10 different leach liquors received from uranium mills on the Colorado plateau were markedly affected by the type of dispersion (Table 7.4). With most of the liquors the break time was greater than 300 sec for aqueous-continuous dispersions at A/O phase ratios of 4/1 and 1/1. However, for solvent-continuous

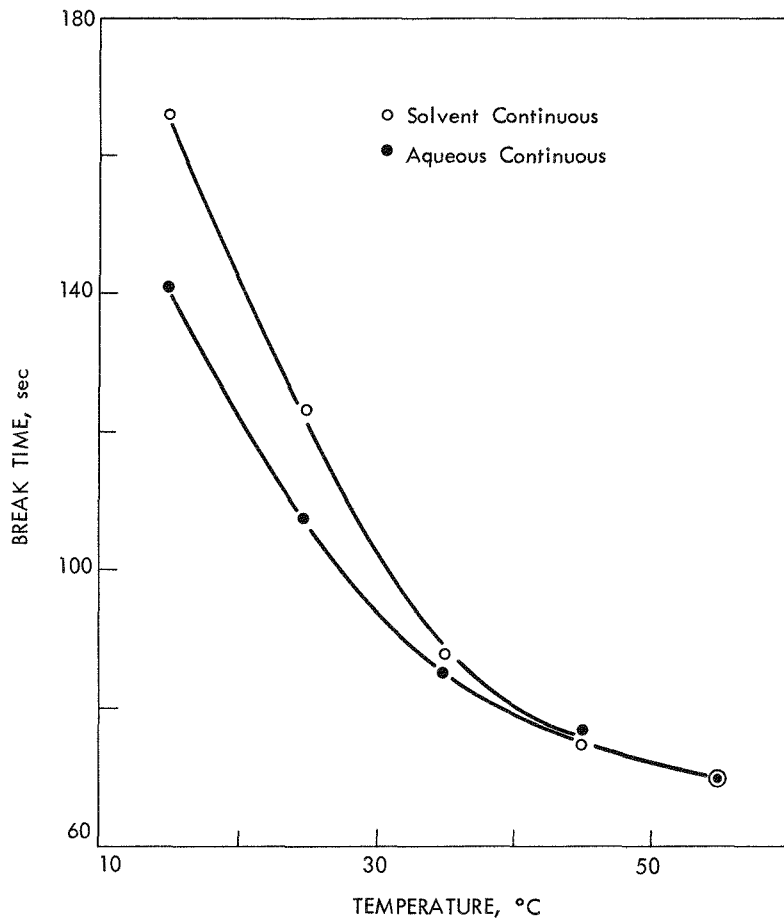


Fig. 7.3. Effect of temperature on batch break time in 12-in.-dia settler at 1/1 phase ratio.

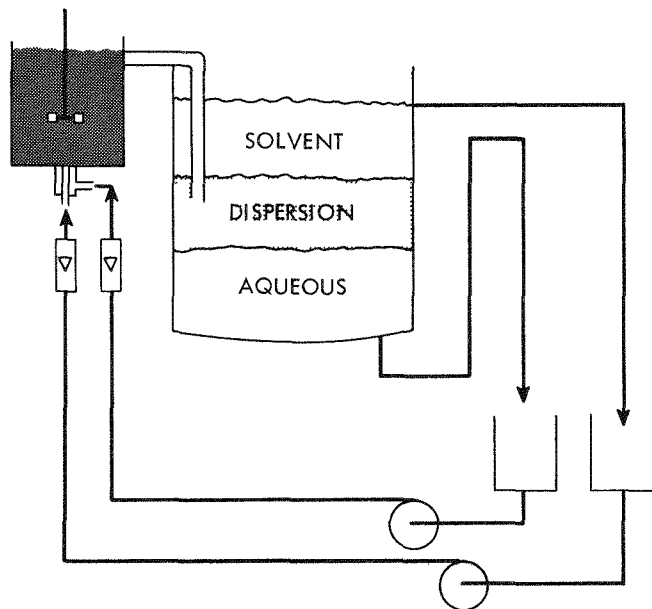


Fig. 7.4. Single mixer-settler unit for phase separation tests.

Table 7.3. Effects of pH and Density on Primary Break Time

Settler: 6 in. dia  
 Phase ratio, A/O: 1/1  
 Temperature: 25°C

pH	Density, g/ml	Primary Break Time, sec	
		Aqueous Continuous	Solvent Continuous
0.5	1.07	50	63
1.0	1.07	50	63
1.5	1.07	57	61
1.0	1.02	47	65
1.0	1.05	47	65
1.0	1.06	45	63
1.0	1.07	50	63
1.0	1.08	-	68

dispersions at phase ratios of 1/1 and 1/2, the break times ranged from 50 to 100 sec. In many of the aqueous-continuous tests an emulsion formed which was stable overnight. All the liquors contained silica, from <0.1 to 2.5 g/liter, which could not be removed by centrifugation or filtration. The break time with liquors containing less than 0.1 g of SiO<sub>2</sub> per liter was about the same as that for synthetic sulfate solutions. At silica concentrations greater than 0.7 g/liter the break time was greater than 300 sec and a varying amount of stable emulsion was formed, indicating that silica is the most likely cause of slow phase separation although it may not be the only emulsifier in the plant liquors. That silica alone can cause emulsion was confirmed by adding silicic acid to sodium sulfate solutions, which formed an emulsion with solvent when the aqueous phase was continuous and none when the organic was continuous.

Emulsion Prevention. The majority of the liquors tested contain emulsifying agents which tend to stabilize an aqueous-continuous dispersion. Control of mixing to produce an organic-continuous dispersion is a simple method of preventing emulsions. Usually organic-continuous mixing can be maintained by starting the mixer with only solvent present and then adding the aqueous phase and keeping at least 50% solvent in the mixer. If the concentration of emulsifier is very high it may be necessary to maintain greater than 50% solvent to prevent localized formation of aqueous-continuous dispersion. All except one of the solvent extraction circuits operate with organic-continuous mixing in commercial uranium mills to prevent emulsions.

Table 7.4. Batch Phase Separation Tests with Actual Plant Liquors

Settler: 3 in. dia x 3-5/8 in. depth

Temperature: 25°C

Aqueous Phase	SiO <sub>2</sub> , g/liter	Primary Break Time, sec			
		Aqueous Cont.		Organic Cont.	
		4/1	1/1	1/1	1/2
Kerr-McGee					
Section 10	0.7	>300	120	80	54
Holly	1.0	>300	>300	62	53
St. Anthony	2.2	>300	>300	62	60
Recycle	0.9	>300	>300	82	73
Shiprock	0.3	120	110	68	52
KM-11	<0.1	49	64	79	72
Climax Uranium					
Sand liquor No. 19	1.0	>300	>300	87	106
Slime liquor 5/8/58	<0.1	72	81	53	48
Union Carbide Nuclear Co.					
Old Rifle Plant, treated with Lomar PW <sup>a</sup>	1.6 1.6	>300 46	>300 61	54 54	51 51
New Rifle Plant, treated with gelatin <sup>b</sup>	2.5 -	>300 -	>300 60	75 75	85 -
Sodium sulfate					
solution, pH 1.0	<0.1	37	51	53	48
silicic acid added	4.0	-	>300	65	-

<sup>a</sup>40 ppm of Lomar PW added to aqueous phase.

<sup>b</sup>Silica coagulated by addition of 300 ppm of gelatin to aqueous and removed by centrifugation.

Another method of preventing emulsions that has been successful in laboratory tests is the addition of certain surfactants to the aqueous feed before contact with the solvent. Cationic types, e.g. Lomar PW,\*

\*Lomar PW is the sodium salt of condensed mononaphthalene sulfonic acid, available from Jacques Wolf and Co.

have been most effective. Addition of about 40 ppm to the aqueous liquor will permit mixing with either phase continuous without emulsion formation<sup>8</sup> (Table 7.4).

In cases where silica alone is the cause of emulsion the silica can be flocculated with gelatin.<sup>24</sup> After filtration or settling out of the silica, the aqueous phase can be contacted with solvent without difficulty (Table 7.4). Flocculating agents that are normally used to aid in settling ore slimes are not very effective for flocculation of silica.

## 7.2 Continuous-flow Settler Tests

For the continuous-flow settler tests a single mixer-settler unit (Fig. 7.4) was used in which both the solvent and aqueous phases of constant composition were recycled. The mixer was a baffled tank agitated with a flat bladed turbine and the settler was a cylindrical tank in which the total liquid depth was approximately 30 in. The interface was controlled near the midpoint of the settler by a gravity leg on the aqueous discharge. Three sizes, 0.5, 1.8, and 4 ft dia, were used to obtain scaleup data. The temperature was maintained at 25°C except where noted otherwise.

Correlation of Settler Capacity with Dispersion Band Thickness. At steady state there was at the interface a distinct dispersion band which was of uniform thickness across the entire area of the settler, very similar to previously reported observations with other systems.<sup>25</sup> Steady state was usually attained within 20 min of operation and the dispersion thickness was measured over a period of 1 hr. The band was stable with respect to operation time, 7 hr being the longest time tested. The dispersion thickness increased exponentially with increased flow rate without showing a sharp flooding point. The best correlation was obtained by plotting dispersion thickness against flow rate of dispersed phase per unit settler area (Figs. 7.5 and 7.6). The result is a family of nearly parallel lines showing relatively small effects of phase ratio and settler diameter for both aqueous- and solvent-continuous dispersions.

The data were summarized by arbitrarily selecting a flooding point at a dispersion thickness of 2 ft. This is somewhat justified because usually a total liquid depth of 2 ft is sufficient for simple gravity leg control of the interface position with most systems. The nominal flow capacity was chosen to be the flow at 5 in. dispersion band, which was about half the flow rate that produced flooding (2-ft band), thereby allowing a safety factor of 2 for design purposes.

Effect of Phase Ratio and Type of Dispersion. The nominal flow capacity for organic dispersed in aqueous increased from 0.9 to 1.4 gpm/ft<sup>2</sup> as the O/A ratio was increased from 1/4 to 1/1 (Table 7.5). Similarly, the nominal flow of aqueous dispersed in organic increased from 1.0 to 1.4 gpm/ft<sup>2</sup> as the A/O ratio was increased from 1/2 to 3/1.

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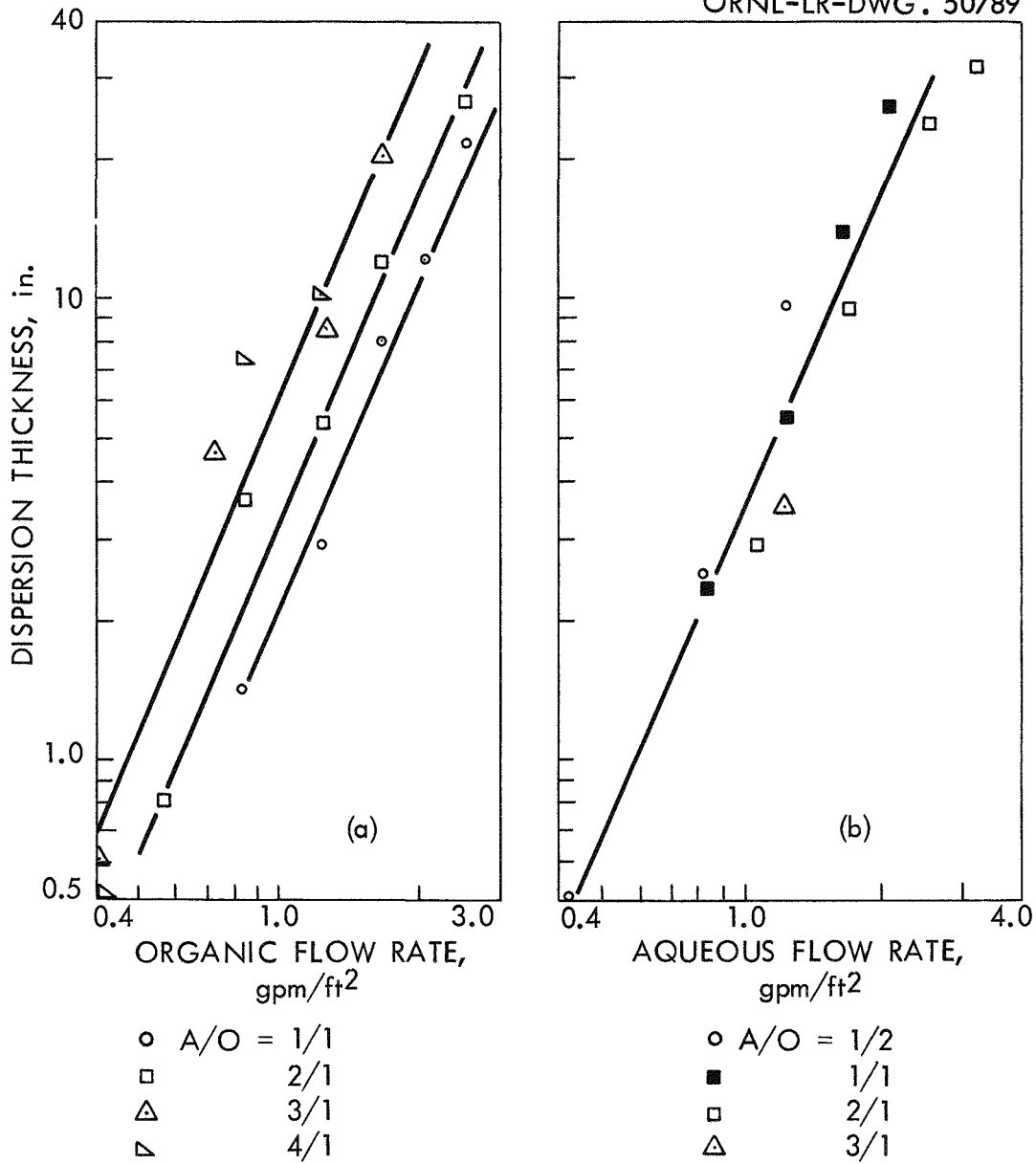
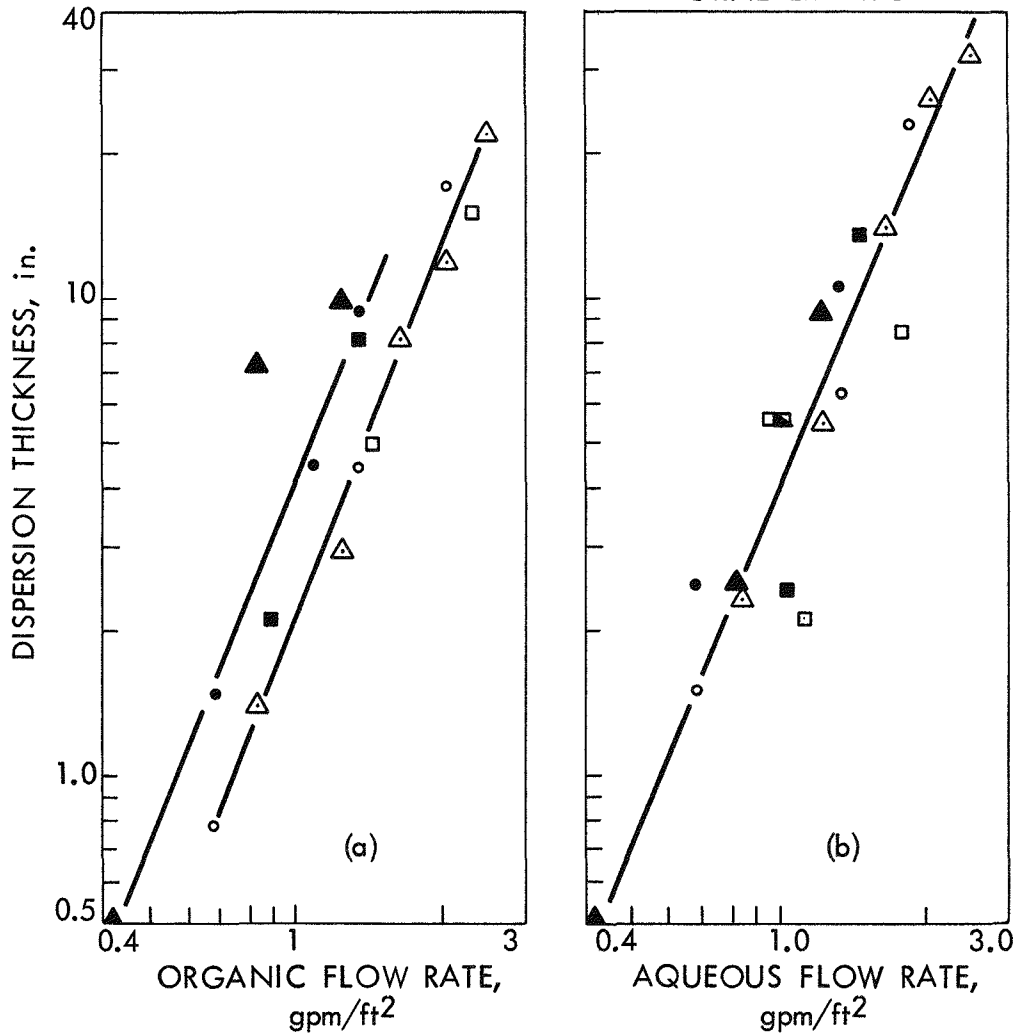


Fig. 7.5. Effect of phase ratio on phase separation in 4-ft-dia settler. (a) Aqueous continuous. (b) Organic continuous.



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A/O Phase Ratio		Settler Dia, ft	A/O Phase Ratio		Settler Dia, ft
1/1	4/1		1/1	1/2	
○	●	0.5	○	●	0.5
□	■	1.8	□	■	1.8
△	▲	4.0	△	▲	4.0
-	◄	7.0	-	◄	7.0
-	-	16.0	◻	-	16.0

Fig. 7.6. Comparison of settler size. (a) Aqueous continuous.  
(b) Organic continuous.

Table 7.5. Effect of Phase Ratio on Nominal Flow Capacity<sup>a</sup>

Ratio of Dispersed to Continuous Phase	Flow Rate of Dispersed Phase, <i>gpm/ft<sup>2</sup></i>	
	Aqueous Continuous	Organic Continuous
1/4	0.9	-
1/3	0.9	-
1/2	1.2	1.0
1/1	1.4	1.2
2/1	-	1.4
3/1	-	1.4

<sup>a</sup>Table derived from Fig. 7.5.

Organic-continuous mixing was maintained at A/O ratios as high as 3/1, i.e. the major phase was dispersed. This was accomplished by starting the mixer with only solvent present and then starting the aqueous flow. The dispersion was much more viscous than when the minor phase was dispersed, but there was no marked effect on the rate of phase separation. When the ratio of dispersed phase to continuous was increased to 4/1 or greater, inversion occurred to form a dispersion with the major phase continuous, and in the presence of silica inversion occurred at lower ratios. Therefore for plant practice it is desirable to maintain the ratio of dispersed to continuous phase less than 1/1.

The nominal flow capacity of dispersed phase was slightly greater for aqueous continuous than for organic continuous, 1.2 vs 1.0 *gpm/ft<sup>2</sup>* at a phase ratio of 1/1. The effect of type of dispersion is greater with several amines used in the Amex process,<sup>26</sup> and indicates that nominal flow capacity decreases with increased viscosity of the continuous phase and decreased interfacial tension.

Effect of Temperature. Nominal flow capacity increased nearly linearly with increased temperature over the range 15 to 45°C, being nearly doubled for 30°C temperature rise (Table 7.6, Fig. 7.7). This agrees with the temperature effect observed in batch tests, where it was shown that the decrease of primary break time with increased temperature corresponds roughly with the change of viscosity with temperature.

The temperature in most uranium mill solvent extraction circuits depends on ambient conditions and is usually within the range 15 to 45°C. Because of the relatively large temperature effect on settler flow capacity, settler size should be designed for the minimum temperature expected or facilities for heating to design temperature should be provided.

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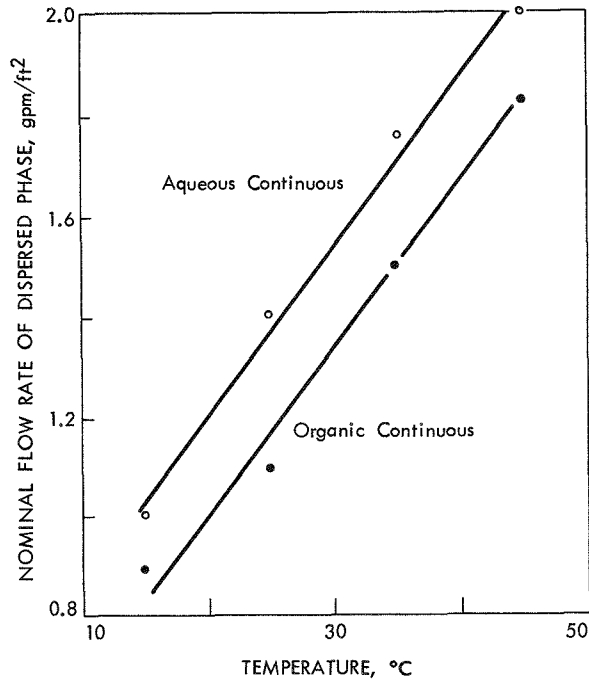


Fig. 7.7. Effect of temperature on nominal flow capacity of 6-in.-dia settler for A/O phase ratio of 1/1.

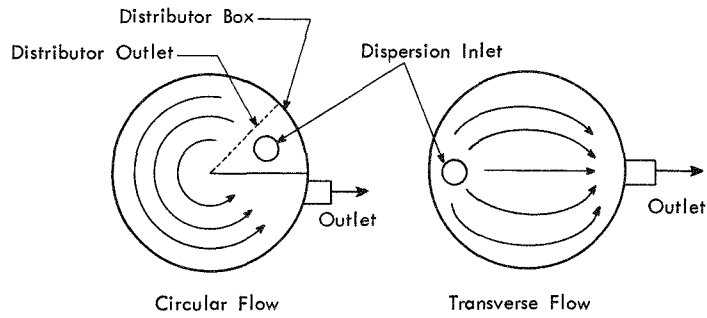


Fig. 7.8. Flow patterns (plan view) in 4-ft-dia settler.

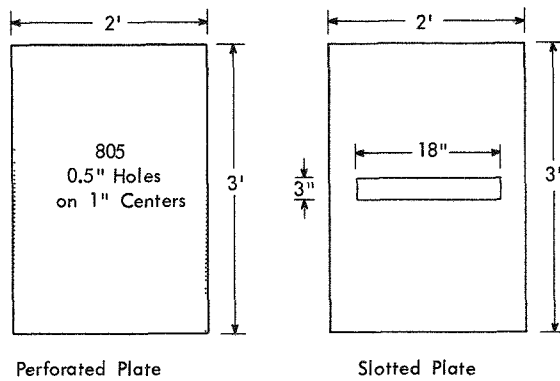


Fig. 7.9. Distributor outlets in 4-ft-dia settler.

Table 7.6. Effect of Temperature on Dispersion Thickness in 6-in.-dia Settler at Phase Ratio of 1/1

Type of Dispersion	Temp., °C	Flow Rate of Each Phase, gpm/ft <sup>2</sup>	Band Thickness, in.
Aqueous continuous	15	0.95	4.7
		1.49	19.3
	25	0.68	0.8
		1.35	4.4
		2.03	17.0
	35	1.89	6.4
		2.57	20.8
	45	1.89	4.3
2.70		18.4	
Organic continuous	15	0.68	1.8
		1.35	19.0
	25	0.68	1.5
		1.35	6.3
		1.84	23.0
	35	0.68	0.4
		1.35	3.3
		2.03	13.8
	45	2.03	5.5
		2.70	17.8

Effect of Mixing Power. Power input to the mixer was varied from zero to 90 hp/1000 gal without effect on dispersion thickness in the settler (Table 7.7), confirming the observed lack of dependence of batch break time on power input. The tests were made with a 12-in.-dia mixer and a 4-ft-dia settler, using an aqueous-continuous dispersion with equal flow of each phase (2.5 gpm/ft<sup>2</sup>). The turbulence in the mixer at this flow rate was sufficient to cause mixing with the agitator shut off. Independence of phase separation from mixing power simplifies the design of mixer-settlers because the power input can be chosen to optimize extraction efficiency without affecting settler capacity.

Flow Patterns in the Settler. Two flow patterns in the 4-ft settler were investigated (Fig. 7.8). One pattern was circular, formed when the mixed phases were introduced through a distributor box which was a 45°

Table 7.7. Effect of Mixing Power on Dispersion Band

Mixer: 12 in. dia  
 Settler: 4 ft dia  
 Flow rate, each phase: 2.5 gpm/ft<sup>2</sup>  
 Phase ratio: 1/1  
 Continuous phase: aqueous

Dia, in.	Turbine	Power Input, hp/1000 gal	Dispersion Band Thickness, in.
	Speed, rpm		
6	0	0	18
	200	4	22
	300	15	21
	400	40	23
	550	90	22
3	1340	40	22

sector of the settler with a distributor outlet located 315° from the settler outlets. An unbaffled distributor box was compared with boxes having either a perforated plate or a slot as the distributor outlet (Fig. 7.9). The other flow pattern was transverse, the result of feeding the mixed phases at the side opposite the outlets. There was no marked difference in dispersion thickness with any of the flow patterns tested (Table 7.8). Because there was a distinct dispersion band in the distributor box which was only slightly thicker than that in the rest of the settler, the transverse flow pattern was used in subsequent tests.

Vertical location of the point of entry of the mixed phases in relation to the dispersion band was important in the 0.5-ft-dia settler. Introduction of the feed either above or below the dispersion band resulted in a thicker band than that obtained when the feed entered near the midpoint. Although this was not observed in the larger settlers, the feed point was standardized at the midpoint of the dispersion band.

Wall Effects. When the settler wall was preferentially wetted by the dispersed phase, water drops on glass or solvent on plastic, coalescence was observed on the wall and the dispersion band was thinner than when the dispersed phase did not wet the wall (Table 7.9). The wall effect is greatest in small-diameter settlers where the ratio of wall area to cross sectional area is large. Consequently the wall effect should be minimized for valid comparisons of small and large settlers. For the 0.5-ft-dia settler, glass walls were used for aqueous-continuous dispersions and a plastic liner, nonaqueous wetted, was inserted for solvent-continuous dispersions. For the larger settlers (1.8 and 4 ft), which were constructed of stainless steel, no attempt was made to minimize the wall effect.

Table 7.8 Comparison of Flow Patterns in 4-ft-dia Settler

Organic-continuous dispersion, phase ratio 1/1

Flow Pattern	Dispersion Thickness, in.	
	Aqueous Flow	Aqueous Flow
	0.83 gpm/ft <sup>2</sup>	2.0 gpm/ft <sup>2</sup>
Circular		
Distributor box with perforated plate	1.3	10
Distributor box with 3 x 18 in. slot	3.0	18
Distributor box with 24 x 32 in. opening	2.4	14
Transverse flow	2.3	16

Table 7.9. Wall Effect on Dispersion Thickness in 0.5-ft-dia Settler

Continuous Phase	A/O Phase Ratio	Aqueous Flow Rate, gpm/ft <sup>2</sup>	Dispersion Thickness, in.	
			Glass Wall	Plastic Wall
Aqueous	1/1	1.35	4.4	2.4
Organic	1/1	1.35	2.8	7.0
		1.84	8.3	23
	1/2	1.35	2.8	11
		1.82	9.8	>27

### 7.3 Scaleup of Settlers

Dispersion thickness as a function of flow rate per unit cross-sectional area is virtually independent of settler diameter for aqueous-continuous dispersion at A/O ratios of 4/1 and 1/1 and for solvent continuous at 1/1 and 1/2 (Fig. 7.6). Therefore scaleup can be based on constant flow rate per square feet and constant dispersion thickness.

Summarizing the data as nominal flow capacity of the dispersed phase demonstrates this more clearly (Table 7.10). The nominal flow capacity was read from Fig. 7.6 at 5 in. dispersion thickness and is about half the flow rate causing flooding for a total liquid depth of 2 ft. The nominal flow capacity of dispersed phase ranges from 1.0 to 1.5 gpm/ft<sup>2</sup>

Table 7.10. Nominal Flow Capacity of Dispersed Phase

Settler		Flow Rate of Dispersed Phase, gpm/ft <sup>2</sup>			
Dia, ft	Area, ft <sup>2</sup>	Aq Continuous		Org Continuous	
		A/O = 4/1	A/O = 1/1	A/O = 1/1	A/O = 1/2
0.5	0.2	1.1	1.4	1.1	1.0
1.8	2.6	1.2	1.4	1.5	1.2
4.0	12	1.0	1.4	1.1	1.0
7.0	38	-	-	-	1.0
16.0	190	-	-	1.0	-

for all variations of phase ratio and for both types of dispersion in settlers 0.5, 1.8, 4.0, 7, and 16 ft dia. Over-all, this represents an area scaleup of nearly 1000-fold.

The data for 7- and 16-ft settlers were obtained from commercial Dapex process circuits in uranium mills (details in Sec. 8.0), which confirm the practical use and validity of the scaleup relations. Because of unknown factors that could affect phase separation, flow capacity tests should be made with representative samples of actual plant solutions. The scaleup data indicate that this can be done in settlers as small as 0.5 ft dia to provide a reliable basis for design of large-scale equipment.

#### 7.4 Correlation of Batch and Continuous Settling

An accurate correlation of batch primary phase separation with continuous-flow capacity has not been possible with the present data. However, in both cases the rate of coalescence appears to be dependent on the area of the coalescing interface and not on the dispersion volume. The linear rate of movement of the active interface in batch tests should represent the limiting velocity that could be attained in a continuous-flow settler. This was tested by using the linear rate for the coalescing interface from the batch profile (Fig. 7.2) to calculate the maximum flow capacity of the dispersed phase. The results indicate flow rates of 3.2 gpm/ft<sup>2</sup> at A/O of 1/1 for aqueous-continuous dispersions and 2.6 gpm/ft<sup>2</sup> for organic continuous. These are both slightly greater than actual flooding capacity measured in continuous-flow tests, being equivalent to a 40-in. dispersion thickness (Fig. 7.6). So it may be concluded that batch tests are useful as a rough method of predicting continuous-flow capacity in addition to demonstrating the relative effects of mixing power, temperature, and type of dispersion.

#### 7.5 Entrainment in Extraction Section

Entrainment in both solvent and aqueous streams was measured as a part of the continuous-flow tests of single-stage mixer-settler units to

determine the effects of phase ratio, temperature, and flow rate. A secondary settler was tested for recovery of solvent entrained in the aqueous phase.

A centrifugation method was developed measuring the amount of solvent entrained in the aqueous. It consists in centrifuging 250-ml samples in a bottle with a neck calibrated in 0.01-ml divisions. Tests with spiked samples showed an accuracy of  $\pm 20\%$  for samples containing as little as 0.04 gal of solvent/1000 gal aqueous. Aqueous entrainment in solvent was determined by centrifugation of 140-ml samples in calibrated (0.01-ml divisions) cone-bottom tubes.

Solvent Entrainment in Aqueous. The type of dispersion formed during mixing had a significant effect on solvent entrainment in the aqueous phase after primary phase separation. The entrainment was consistently lower for organic-continuous dispersions than for aqueous-continuous. Solvent entrainment was less than 0.2 gal/1000 gal in 0.5-ft (Table 7.16), 4-ft (Table 7.12), and plant settlers (Sec. 8.0) for solvent-continuous mixing at flow rates up to flooding and at A/O phase ratios ranging from 1/2 to 3/1. For typical application of the Dapex process in uranium milling plants, the cost of solvent lost by entrainment at this level is about 1¢ per pound of  $U_3O_8$  recovered, which is less than the estimated cost of reagent lost by solubility.<sup>9</sup>

Solvent entrainment for aqueous-continuous mixing increased both with increased A/O phase ratio and flow rate, with some inconsistencies between data for 0.5- and 4-ft-dia settlers. The effect of phase ratio at constant aqueous flow rate (1.35 gpm/ft<sup>2</sup>) in Table 7.11 shows that solvent entrainment is about an order of magnitude higher at 4/1 than at 1/1.

The effect of flow rate at an A/O phase ratio of 4/1 is more marked in the 0.5- than in the 4-ft-dia settler; solvent entrainment increased from 1.0 to 6.2 gal/1000 gal as the aqueous flow rate was increased from 1.35 to 5.4 gpm/ft<sup>2</sup> (Table 7.13).

Table 7.11. Effect of Phase Ratio on Solvent Entrainment

Aqueous flow rate: 1.35 gpm/ft<sup>2</sup>  
 Temperature: 25°C

Continuous Phase	A/O Phase Ratio	Solvent in Aqueous, gal/1000 gal	
		0.5-ft-dia Settler	4-ft-dia Settler
Aqueous	4/1	1.0	2.0
	1/1	<0.04	0.3
Organic	1/1	<0.04	<0.2
	1/2	<0.04	<0.2



Table 7.12. Solvent Entrainment Data for 4-ft-dia Settler

Temperature: 25°C

A/O Phase Ratio	Aqueous Flow Rate, gpm/ft <sup>2</sup>	Solvent in Aqueous, gal/1000 gal	
		Aqueous-continuous	Solvent-continuous
4/1	1.67	2.1	-
	3.33	2.0	-
	5.00	2.4	-
3/1	1.25	2.3	<0.2
	3.75	2.2	<0.2
	5.00	2.2	<0.2
2/1	1.67	1.1	<0.2
	2.50	1.0	<0.2
	3.30	1.7	<0.2
	5.00	1.6	<0.2
1/1	0.83	0.4	<0.2
	1.25	0.3	<0.2
	1.67	0.2	<0.2
	2.08	0.5	<0.2
	2.50	0.2	<0.2
1/2	0.42	0.3	<0.2

Table 7.13. Effect of Aqueous Flow Rate on Solvent Entrainment

A/O phase ratio: 4/1

Temperature: 25°C

Dispersion: aqueous-continuous

Aqueous Flow Rate, gpm/ft <sup>2</sup>	Solvent in Aqueous, gal/1000 gal	
	0.5-ft-dia Settler	4-ft-dia Settler
1.35	1.0	-
1.67	-	2.1
2.70	2.2	-
3.33	-	2.0
4.35	3.8	-
5.00	-	2.4
5.40	6.2	-

Increasing the temperature from 25 to 50°C reduced solvent entrainment 4- to 5-fold at an A/O ratio of 4/1 (Table 7.14), but it was still higher than that obtained for organic-continuous mixing.

Table 7.14. Effect of Temperature on Solvent Entrainment

Settler: 0.5 ft dia  
 A/O phase ratio: 4/1  
 Dispersion: aqueous-continuous

Temp., °C	Solvent in Aqueous, gal/1000 gal	
	Aqueous Flow Rate	Aqueous Flow Rate
	1.35 gpm/ft <sup>2</sup>	4.35 gpm/ft <sup>2</sup>
15	2.0	5.6
25	1.0	3.8
35	0.6	2.6
50	0.4	1.6

Aqueous Entrainment in Solvent. Entrainment of aqueous feed in the extract is not an important economic factor but is of interest because carryover can contaminate the stripping circuit and the uranium product. In all tests the aqueous entrainment was less than 1 vol % of the solvent. For a typical Dapex uranium installation where the ratio of feed liquor to extract is 4/1, an entrainment value of 1 vol % would result in a ratio of each contaminant to uranium in the extract of 1/400 of that in the feed liquor. For average mill liquor and present product specification, this amount of contamination would be unimportant.

Secondary Settler for Recovery of Entrained Solvent. Although it has been demonstrated that organic-continuous mixing is desirable to avoid emulsions and to minimize solvent entrainment, there are situations due to existing equipment limitations where the system is operated aqueous-continuous and it is desirable to reduce the amount of solvent lost by entrainment. A secondary settler of the type used by the petroleum industry to remove oil from waste water<sup>27</sup> decreased solvent entrainment from an average of 3 to less than 0.2 gal/1000 gal at an aqueous flow rate of 2 gpm (Table 7.15). This is equivalent to 25 min residence time in the settler. Filling the film rupture chamber with various packings such as Tellerettes, Tuffies, or oil shale which were solvent wetted did not decrease entrainment loss. A single mixer-settler unit was operated aqueous-continuous at an A/O ratio of 4/1 to supply aqueous phase containing entrained solvent to the secondary settler (Fig. 7.10), which was evaluated by measuring the entrainment in the aqueous phase leaving it. The secondary settler was a rectangular box 6 by 1 by 1.5 ft high with a flow distributor to promote low turbulence in the settler where entrained solvent drops would rise and form a layer which could be reclaimed. The liquid depth was 1 ft, set by a fixed gravity leg on the aqueous discharge.

Table 7.15. Evaluation of Secondary Settler for Reducing Solvent Entrainment in Aqueous

Solvent in feed: 3 gal/1000 gal

Packing in Rupture Chamber	Coalescer in Filter Frame	Pressure Drop, in. H <sub>2</sub> O	Aqueous Flow Rate, gpm	Solvent Entrainment, gal/1000 gal
None	-	-	2	0.16
Tellerettes <sup>a</sup>	-	-	2	0.20
Tuffies <sup>b</sup>	-	-	1	0.10
Oil shale <sup>c</sup>	-	-	1	0.10
None	Glass wool	2.5	2	0.04
None	Glass wool	2.5	4	0.04
None	1 sheet of glass cloth <sup>d</sup>	-	2	0.13
None	1 sheet of glass cloth <sup>d</sup>	-	4	0.14
None	4 sheets of glass cloth <sup>d</sup>	2.3	2	0.04
None	4 sheets of glass cloth <sup>d</sup>	3.3	4	0.04

<sup>a</sup>Polyethylene tower packing, Harshaw Chemical Co.

<sup>b</sup>Woven polyethylene scouring pad, SOS Co.

<sup>c</sup>Oil shale, 3/8 in.

<sup>d</sup>Fiberglas cloth, Code N41A40, Owens Corning Fiberglas Co.

To test various coalescing media, a filter frame (area 1 ft<sup>2</sup>) was inserted in the vertical plane at position C (Fig. 7.10). Glass wool mat and glass cloth reduced entrainment to <0.04 gal/1000 at flow rates up to 4 gpm with a pressure drop of 2-3 in. H<sub>2</sub>O. Dynel cloth caused a pressure drop greater than 6 in. H<sub>2</sub>O at a flow rate of 1 gpm, which caused the aqueous phase to flow over the top of the filter frame making evaluation impossible.

The practical usefulness of coalescing media in the secondary settler probably would depend on the amount of slime in the aqueous phase, which would blind the coalescer and cause an excessive pressure drop. The coalescer could be backwashed by reversing the direction of aqueous flow or by wash nozzles. The slime problem was not studied; therefore the final feasibility of coalescers should be determined with actual plant raffinate.

#### 7.6. Phase Separation in Stripping Circuit

Phase separation tests of the uranium stripping circuit using sodium carbonate was not investigated as thoroughly as extraction because the flow rates are relatively smaller and settler sizing is not so important

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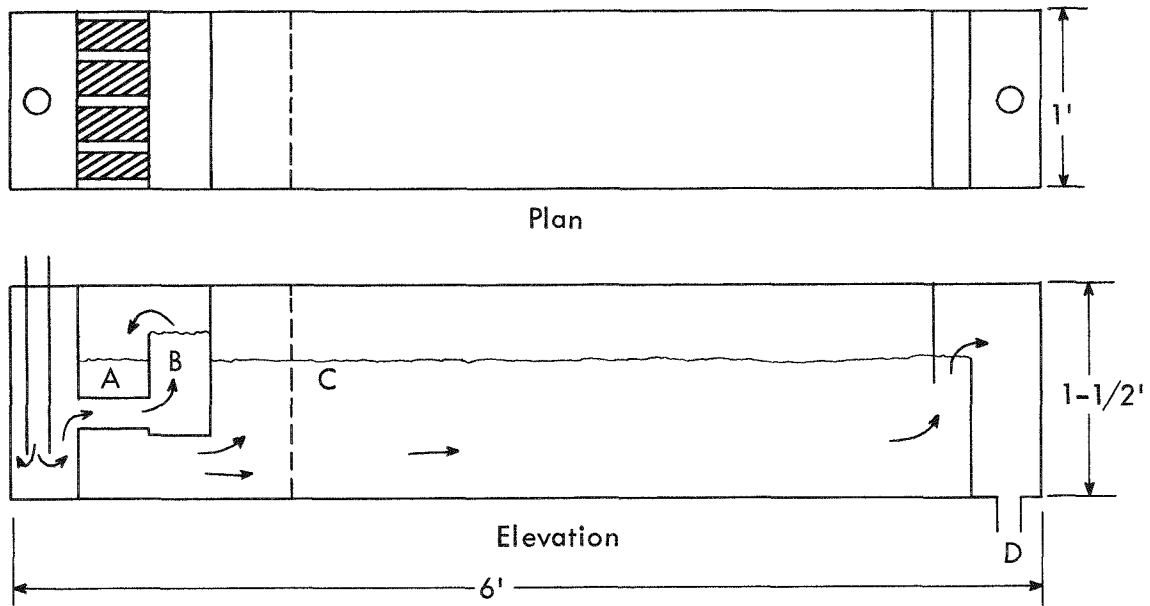


Fig. 7.10. American Petroleum Institute type secondary settler. (A) Film rupture chamber; (B) distribution box and adjustable weir; (C) filter frame; (D) sample point.

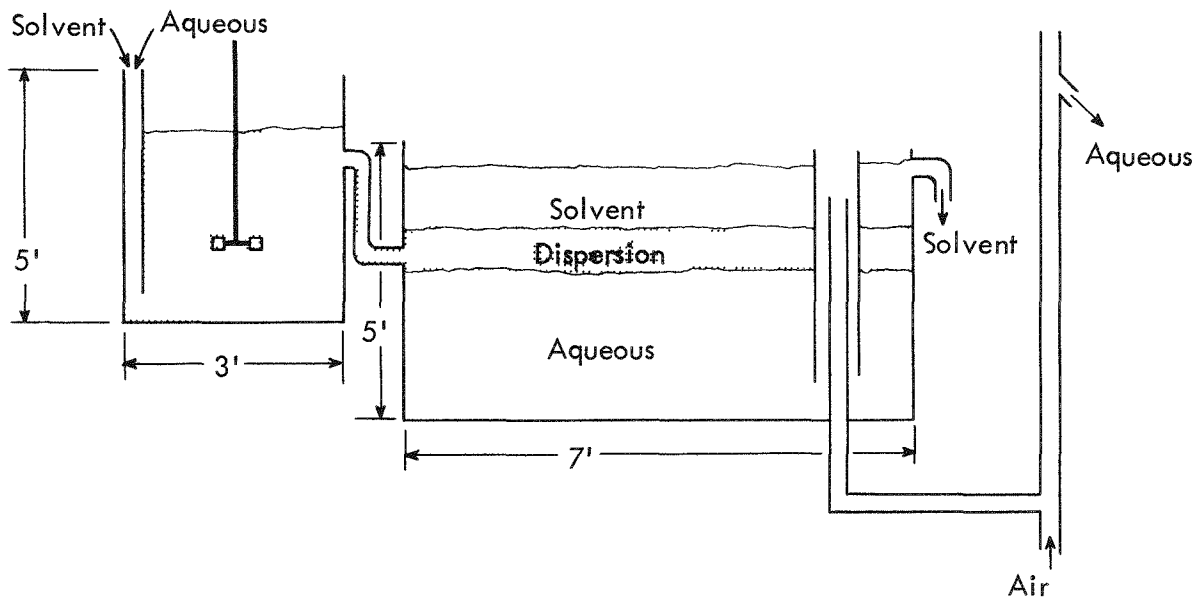


Fig. 8.1. Climax mixer-settler.

economically. Scouting tests were made in the 0.5-ft-dia settler with mixtures of solvent and 10% Na<sub>2</sub>CO<sub>3</sub> solutions (Table 7.1). The mixing was controlled to produce organic-continuous dispersion with a phase ratio (A/O) of 1/6 in most tests, which is in the range of typical stripping circuits. Only a thin dispersion band was discernible at a phase ratio of 1/6 and the thickness increased very little with increased flow (Table 7.16). However, the entrainment of aqueous in solvent increased sharply,

Table 7.16. Phase Separation for Stripping Circuit

Settler: 0.5 ft dia

Continuous Phase	A/O Phase Ratio	Flow Rate, gpm/ft <sup>2</sup>		Dispersion Thickness, in.	Entrainment, gal/1000 gal		
		Aq.	Org.		Aqueous in Solvent	Solvent in Aqueous	
Organic	1/6	0.14	0.84	<0.1	7.0	<0.04	
		0.27	1.6	0.1	7.0	<0.04	
		0.41	2.4	0.3	7.7	<0.04	
		0.54	3.2	0.4	27.3	<0.04	
	1/1	0.34	0.34	0.8	1.3	<0.04	
		0.78	0.78	14.	0.7	<0.04	
	Aqueous	1/1	0.54	0.54	1.3	2.1	<0.04

approaching the flooding point where the entrainment was equal to the phase ratio, 1/6, of the feed to the settler. The sharp increase in entrainment occurs between a solvent flow rate of 2.4 and 3.2 gpm/ft<sup>2</sup>, so a good choice for the maximum practical solvent flow capacity is 2 gpm/ft<sup>2</sup> which could be used as a basis for design. At higher flow rates the amount of entrainment is large enough to be economically important because of loss of sodium carbonate, and to lower stage efficiency as a result of back-mixing of the aqueous phase between stages.

Tests at a phase ratio of 1/1 showed that recycling of aqueous would lower entrainment, but the flow capacity would be reduced because a dispersion band formed at the interface similar to that obtained in the extraction circuit. This limited the solvent flow capacity to about 0.6 gpm/ft<sup>2</sup> at a 5-in. band, which is only about 25% of the capacity at a phase ratio of 1/6.

Entrainment of solvent in aqueous was very low in all tests (<0.04 gal/1000 gal). It should be noted, however, that this system contained no impurities or solids which might tend to increase the entrainment losses.

Tests of the settler for the stripping circuit were not scaled up. However, the data obtained in the uranium mill Dapex circuits (Sec. 8.0) showed satisfactory phase separation in 7-ft-dia settlers at a solvent flow rate of 1.8 gpm/ft<sup>2</sup>, which agrees with the flow capacity determined in the small-scale equipment. It may therefore be concluded the settlers can be scaled up at constant flow rate per unit cross-sectional area.

## 8.0 OPERATIONS IN URANIUM MILLS

The first commercial application of solvent extraction in uranium mills was by Kerr-McGee Oil Industries, Shiprock, N. M., and Climax Uranium Co., Grand Junction, Colo., in September 1956. Both mills installed the Dapex process without actual pilot plant operation. The design was based on data obtained from bench-scale laboratory counter-current tests and the basic principles of mixing and phase separation obtained from the unit operations study at ORNL. Since the extraction equipment at Climax is almost exactly the same as the models described above, operational data from these mills provide a good check of the scale-up relations developed in the small-scale tests. Through the courtesy of the Kerr-McGee and Climax managements, both mills were visited in May 1958 to obtain samples and information required in analyzing performance of the plant equipment.

### 8.1 Climax Uranium Company

The mill uses the salt-roast process for recovering uranium and vanadium from ores. The Dapex solvent extraction process is used for recovery of uranium from sulfuric acid leach of residues after most of the vanadium has been removed. The solvent is 0.08 M D2EHPA and 3% TBP dissolved in kerosene or No. 1 fuel oil. The aqueous feed is filtered through drum filters and although not sparkling, usually contains less than 0.5% insoluble solids. Typical analysis of the feed, in grams per liter: U 5.2, V 4.0, Fe 0.3, Al 3, Mo <0.1, SO<sub>4</sub> 70, Cl 2, and PO<sub>4</sub> 2; pH 0.5. The liquor is highly oxidized so that the vanadium is present in the pentavalent state.

The uranium is extracted in four stages (six installed) and stripped with 1 M Na<sub>2</sub>CO<sub>3</sub> in four stages. The flow rates of feed, solvent, and strip are 40, 70, and 6.6 gpm, producing a loaded strip solution containing about 50 g of uranium per liter. The uranium is recovered, after destroying the carbonate with sulfuric acid at pH 4, by precipitation with ammonia at pH 7. The precipitate is filtered and calcined at 900°C.

The process is controlled by regulating the flow rates of aqueous feed, solvent, and sodium carbonate. The solvent and carbonate flow rates are adjusted according to feed concentration and flow rate to maintain high uranium loading in the solvent without excessive loss of uranium in the raffinate. Each stream flows by gravity from a constant-level head tank and is metered by a recording rotameter.

The mixer-settlers are the same size in the extraction and stripping circuits (Fig. 8.1). They were designed based on extraction requirements and are slightly oversized for stripping service. The mixers are wooden tanks (3 by 5 ft), which are a direct scaleup of the small model shown in Fig. 4.2. The agitator is a flat-bladed turbine, 1 ft dia, driven by a 5-hp motor at 380 rpm, which results in a power input of 30 hp/1000 gal. The mixing is controlled to maintain the solvent phase continuous to avoid emulsions caused by silica or solids. The settlers are wooden tanks (7 by 5 ft) with a horizontal settling area of 38 ft<sup>2</sup>. The interface level is controlled by an internal aqueous gravity leg. The mixer-settlers are installed on a grade so that the solvent flows by gravity between stages and the aqueous is advanced by air-lifts.

Uranium Profile. The concentration of uranium in each phase leaving the settlers was determined; the resulting profile is shown in Fig. 8.2. In the extraction circuit the uranium concentration of the aqueous phase was decreased from 5.2 to 0.014 g/liter in four stages, giving 99.7% recovery of uranium. In the stripping circuit the uranium in the solvent was effectively removed in four stages. The profile shows back-extraction of uranium in the No. 1 stage, which can occur when the pH of the aqueous carbonate is too low. The profile also shows that the uranium content of the aqueous phase from stage No. 4 is considerably greater than that calculated by material balance from the solvent stream entering and leaving that stage. The most logical cause is back-mixing of aqueous from No. 3 stage due to entrainment of aqueous in the solvent. Based on material balance, the entrainment would be 3 vol %, which is reasonable at the operating A/O phase ratio of 1/10 and temperature of 32°C. The entrainment was not measured, but thief samples of the No. 1 and No. 4 settlers showed that the aqueous content of the solvent was about 5% within 4 in. of the top of the solvent layer.

The loaded aqueous carbonate stream leaving the strip circuit contained an insoluble yellow solid (1 wt %), analysis (dry) of which showed:

U	45.4 wt %	Na	5.8 wt %
V	9.8	CO <sub>3</sub>	1.6

The U/V/Na mole ratio was very nearly 1, indicating that the solid is probably synthetic carnotite. The vanadium in the solid represents about 65% of the total vanadium present in the loaded strip. Separation of this solid may offer a simple method of decreasing the vanadium content of the final uranium product.

Stage Efficiency. The stage efficiencies (Table 8.1) of the mixer-settlers were calculated from samples taken from the settler effluent streams. The efficiency of the mixers alone was determined from samples taken through fritted glass filters immersed in the mixers (Sec. 4.0). Equilibrium data were obtained by taking samples of dispersion from the mixer and shaking for 10 min in separatory funnels.

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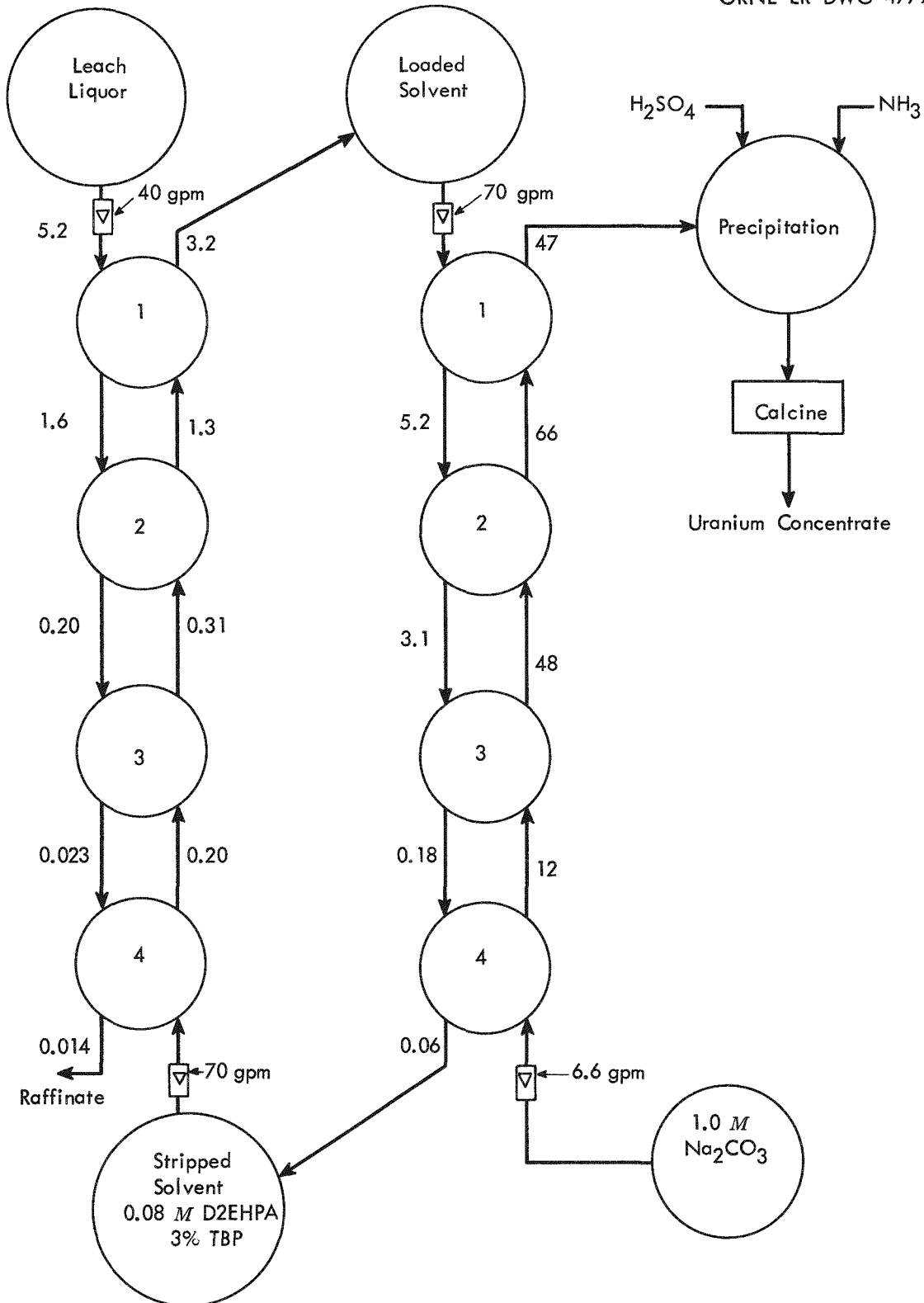


Fig. 8.2. Climax extraction circuit. Figures beside arrows, unless otherwise identified, denote uranium concentration in grams per liter.



Table 8.1. Stage Efficiencies of Climax Mixer-Settlers

Stage No.	Uranium Concentration, <sup>a</sup> g/liter				Efficiency, %	
	Feed	Equil.	Mixer <sup>b</sup>	Settler Effluent	Mixer	Mixer-Settler
<u>Extraction Circuit</u>						
1	5.2	1.35	2.10	1.62	80.5	93.0
2	1.62	0.072	0.40	0.20	78.7	91.6
3	0.20	0.010	0.053	0.023	77.4	93.2
<u>Stripping Circuit</u>						
1	3.2	5.2	-	5.2	-	100
2	5.2	2.7	-	3.1	-	84
3	3.1	0.08	-	0.18	-	97

<sup>a</sup>Uranium concentration in aqueous in extraction circuit and in solvent in stripping circuit.

<sup>b</sup>Aqueous sample taken through fritted glass filter immersed in mixer.

In the extraction circuit the efficiency of three adjacent stages was nearly constant over a wide range of uranium concentrations. The over-all efficiency of each mixer-settler was significantly greater than that of only the mixer because some extraction occurred during phase separation. The residence time of the dispersion in the mixer was 1.2 min and in the settler was 1.3 min. The rate constants (eq. 7) based on average stage efficiencies were 1.1 in the mixer and 1.7 in the mixer-settler. The close agreement of these data with those obtained in small-scale mixers (Fig. 4.6) confirms the scaleup relations based on constant power input per unit mixer volume.

In the stripping section most of the uranium was stripped in one stage (No. 3) where the stage efficiency was 97%. Stages 1 and 2 were complicated by the pH being too low for effective stripping.

Phase Separation. The dispersion band thickness in each extraction settler and the end strip settlers was located and measured by repeated sampling at measured depths with a thief sampler. Its thickness was nearly the same in all four extraction settlers, ranging from 5 to 6 in. (Table 8.2). The batch primary break time (measured in 400-ml beakers) of the dispersion dipped out of the mixers was 57 sec, which is only slightly more than the break time for synthetic feed used in scale-model tests at 25°C (Sec. 7.0). Therefore the band thickness in the Climax settlers and scale models can be compared directly without temperature correction. The plant data fit very well on the curve for solvent-continuous dispersion in Fig. 7.6, demonstrating valid scaleup of settlers based on constant flow per unit area at constant dispersion thickness.

Table 8.2. Profile of Climax Settlers

Stage	Layer Thickness, in.		
	Solvent	Aqueous	Dispersion
Extraction 1	12	31	5.5
2	9	35	6.0
3	10	33	5.5
4	9	35	5.0
Stripping 1	4	27	16
4	4	27	16

The entrainment of solvent in the aqueous raffinate was measured at two points in the plant. The raffinate from stage 4 was passed through a spare settler before it was discharged. The average solvent entrainment (based on three grab samples) in raffinate entering and leaving the spare settler was 0.37 and 0.20 gal/1000 gal, respectively, showing that an additional hold time of 25 min reduced the entrainment by half. The average total inventory loss for nearly two years of operation is equivalent to 1.6 gal per 1000 gal of aqueous. This includes loss due to entrainment and solubility in the raffinate and loaded carbonate and loss due to spillage and evaporation. Although the measured entrainment was a small part of the total loss, it may not be realistic because of the short run time represented by the entrainment samples.

Entrainment of aqueous in the loaded solvent was 4.2 gal per 1000 gal of solvent. This is not sufficient to seriously affect the operation of the stripping circuit or the product purity.

In the strip settlers no sharp dispersion band was found. A dilute dispersion of the same aqueous content as measured in the mixer, ~10% aqueous, was at the aqueous-solvent interface. The aqueous content of solvent gradually decreased to less than 5% near the top of the 20-in. solvent layer. This is very similar to the results obtained in scale-model settler tests with dilute solvent-continuous dispersions (Sec. 7.0), where the flow capacity is limited by entrainment and not by a distinct dispersion band.

## 8.2 Kerr-McGee Oil Industries, Shiprock, New Mexico<sup>29</sup>

Uranium ore is leached with sulfuric acid and the solids are removed by classifiers and thickeners arranged for countercurrent decantation. The leach liquor from the thickeners is treated with scrap iron to reduce the ferric ion content before uranium extraction.

The solvent is 0.11 M D2EHPA and 3% TBP dissolved in a high-flash-point kerosene (Napoleum 470). The aqueous feed is not filtered and

contains variable amounts of insoluble solids, usually less than 0.5%. Typical chemical analysis of the feed, in grams per liter, is: U 0.7, V 1.1, and Fe 1.8; pH 1.4. The emf of the liquor is usually less than 300 mv, so that the iron and vanadium are in reduced valence states.

Each mixer-settler in the extraction section is composed of one 16-ft-dia by 6-ft-deep wood settler with a 4- by 4-ft stainless steel mixing tank supported within the settler. The agitator is an 18-in.-dia turbine driven by a 3-hp motor. The settler area is 190 ft<sup>2</sup>. The mixer-settlers are installed on a grade so that the aqueous flows by gravity and the solvent is advanced by air-lifts. The interface level is set by a variable weir on the aqueous discharge leg. The mixing is aqueous-continuous in all stages except No. 4 where solvent is recycled to maintain a phase ratio of 1/1 in the mixer. The temperature is 40°C.

In the stripping section each mixer-settler is comprised of a 4- by 4-ft mixer followed by an 8-ft-dia cone-bottom settler. The solvent flows by gravity and the aqueous strip solution by pump. The mixing is solvent-continuous in both stages. Steam-heating coils are installed in the mixers and the temperature is maintained at 45°C.

Uranium Profile. The profile of uranium concentrations in both phases from each stage is shown in Fig. 8.3. In the extraction section the uranium concentration in the aqueous phase was reduced from 0.68 to less than 0.001 g/liter in four stages, giving 99.9% uranium recovery. The extraction was essentially complete in three stages, the fourth stage serving as reserve capacity. In the stripping circuit the profile shows that virtually all the uranium is stripped in the second stage. Stripping does not occur in the first stage because the flow of sodium carbonate to the circuit is controlled to maintain the pH of the loaded strip at less than 7.5, which is too low for effective stripping.

Stage Efficiency. The stage efficiencies of the mixer-settlers were calculated from samples taken from the settler effluent streams and equilibrium values obtained by shakeout of samples dipped from the mixers. The efficiency of the mixer-settlers in the extraction circuit ranged from 89 to 99%. The measurements were complicated by fluctuating recycle within each mixer-settler, which caused rapid changes of phase ratio within the mixer. Therefore, although the stage efficiency is apparently greater than 90% most of the time, the data are not useful for checking scaleup factors. Attempts to measure the efficiency of the mixers alone by sampling the dispersed phase with fritted filters were unsuccessful because the dispersion was so fine that the filters did not give good separation of the phases.

Phase Separation. The dispersion band thickness in each settler in the extraction circuit is shown in Table 8.3. The first three stages were operated with aqueous-continuous dispersion at an A/O phase ratio of 9/1 and the fourth stage was solvent-continuous with solvent recycled by a centrifugal pump to maintain a phase ratio of 1/1 in the mixer. Phase

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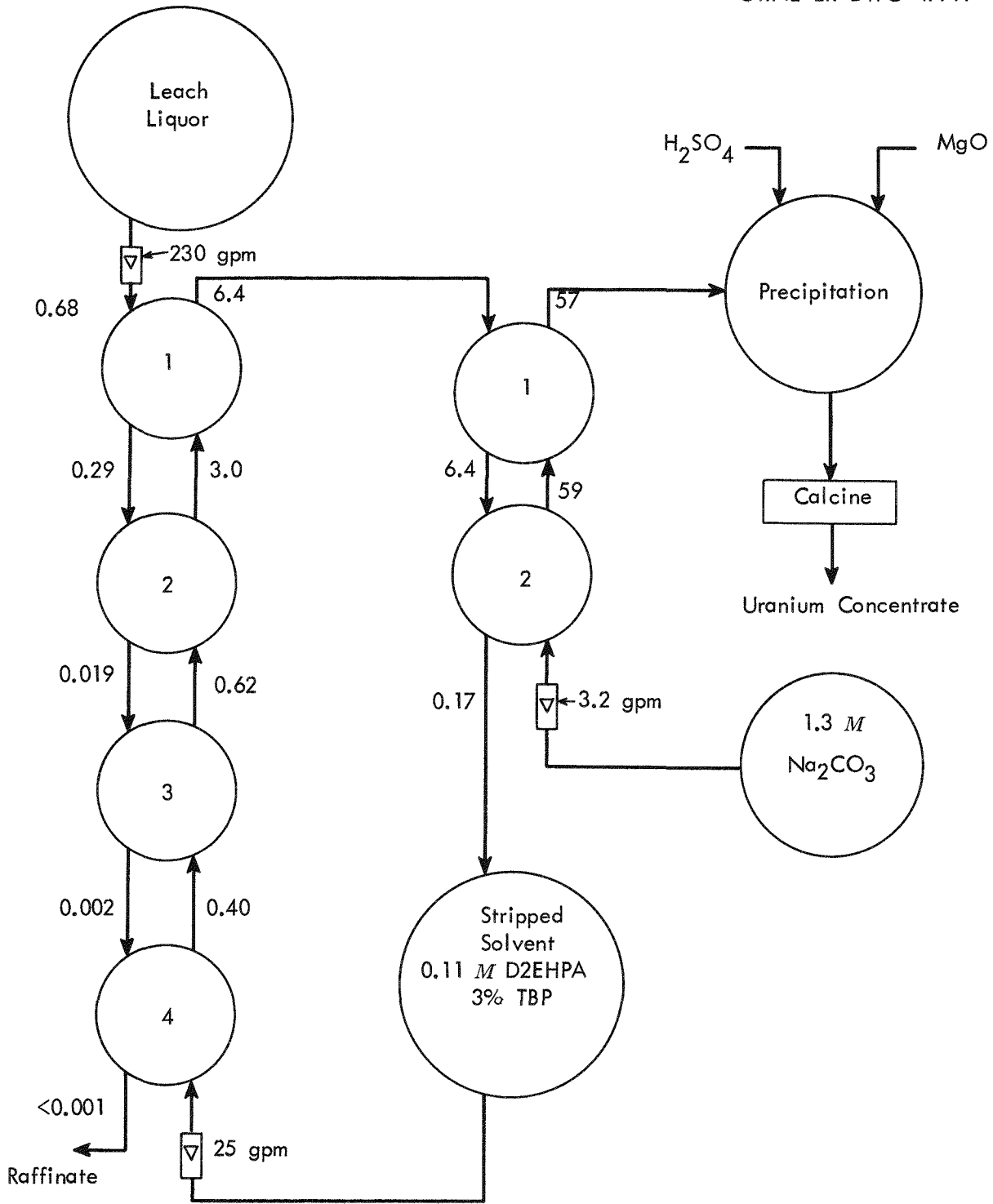


Fig. 8.3 Kerr-McGee shiprock extraction circuit. Figures beside arrows, unless otherwise identified, denote uranium concentration in grams per liter.

Table 8.3. Profile of Shiprock Extraction Settlers

Stage No.	Continuous Phase	A/O Phase Ratio	Layer Thickness, in.		
			Aqueous	Solvent	Dispersion
1	Aqueous	9/1	39	3	18
2	Aqueous	9/1	39	5	16
3	Aqueous	9/1	41	8	11
4	Organic	1/1	45	9	6

separation was markedly affected by the type of dispersion, as shown by a band thickness of 6 in. for solvent-continuous compared to 11 to 18 in. for aqueous-continuous. The dispersion band depth was greatest in the first stage settler; in fact, the dispersion periodically overflowed with the loaded solvent into the stripping circuit. Most of the dispersion in the settler was a coarse emulsion consisting of a relatively large volume of solvent dispersed in aqueous. The emulsion was easily broken by pouring it into a mixer containing solvent and thereby inverting it to a solvent-continuous dispersion. The cause of the emulsion is believed to be decomposition products of certain vegetable gums used for flocculation of ore slimes in the thickener circuit. The difficulty is avoided by using synthetic flocculating agents or by operating with solvent phase continuous as was done in the fourth stage.

Because of the emulsion difficulty, only the data for solvent-continuous dispersion in the fourth stage can be used for checking the scaleup relations. The batch break time of 400-ml samples dipped from the mixer was 40 sec, which is only slightly less than that for the system used in the scale-model settlers; consequently the band thickness can be compared directly with that measured in the scale models. The good agreement of the plant data with the curves shown in Fig. 7.6 confirms the scaleup relations based on constant dispersion thickness at constant flow rate per unit settler area.

The entrainment of solvent in the aqueous raffinate was measured in grab samples taken from the No. 4 settler discharge. The entrainment in eight samples was less than 0.1 gal/1000 gal. Plant records for April 1958 of daily composites of the raffinate showed an average entrainment of 0.2 gal/1000 gal except for two days when emulsions were encountered and the entrainment was as high as 1.5 gal/1000 gal. The total inventory loss of solvent since plant startup has been equivalent to 0.8 gal/1000 gal. This includes loss due to entrainment, solubility, spillage, and evaporation. When no spillage was recorded the inventory loss was less than 0.5 gal/1000 gal.

In the strip circuit no sharp dispersion band was observed in the settlers. Primary break time of dispersion dipped from the mixers was less than 1 min. The entrainment of aqueous in the stripped solvent was

4.0 gal/1000 gal which was equivalent to 0.2 g of uranium per liter of solvent entering the raffinate stage of the extraction circuit. However, because of the high uranium extraction coefficient this did not cause high loss of uranium in the raffinate.

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## 10.0 APPENDIX

### 10.1 Nomenclature

- A - Aqueous flow rate, gal/min
- A' - Interfacial area,  $\text{cm}^2$
- a - Interfacial surface area, per unit volume of dispersion,  $\text{cm}^2/\text{cm}^3$
- C - Uranium concentration in aqueous after contact time, t, g/liter
- C<sub>0</sub> - Initial uranium concentration in aqueous, g/liter
- C<sub>eq</sub> - Equilibrium uranium concentration in aqueous, g/liter
- D - Turbine diameter, in.
- E - Stage efficiency, %
- g - Gravity conversion factor
- K - Turbine constant
- k - Rate constant, cm/min
- ka - Effective rate constant,  $\text{min}^{-1}$
- N - Turbine speed, rpm
- O - Solvent flow rate, gal/min
- P - Power input to mixer, hp
- R - Aqueous volume in mixer, gal
- T - Mixer diameter, in.
- t - Contact time in mixer, min
- V - Mixer volume, gal
- $\rho$  - Mean density of dispersion, g/ml

### 10.2 Detailed Data Tables

Detailed data from the batch and continuous mixing tests are given in Tables 10.1 and 10.2



Table 10.1. Batch Uranium Extraction Tests

Mixer Dia, in.	Turbine Dia, in.	Speed, rpm	Power Input, hp/ 1000 gal	Uranium Concentration, g/liter												ka, min <sup>-1</sup>	
				0 sec	5 sec	10 sec	20 sec	30 sec	45 sec	60 sec	75 sec	120 sec	240 sec	480 sec	At D/T shown	At D/T = 1/3	
<u>System A</u>																	
6	3	262	2.5	5:20	3.60	3.35	2.80	2.40	1.88	1.56	1.25	0.74	0.32	0.24	0.51	0.64	
		350	5.9	5.20	3.20	2.70	2.05	1.62	1.16	0.89	0.69	0.38	0.25	0.24	0.83	1.04	
		524	20	5.20	2.25	1.83	1.20	0.89	0.53	0.36	0.26	0.27	0.23	0.24	1.48	1.85	
		787	67	5.20	1.80	1.31	0.73	0.47	0.29	0.26	0.24	0.25	0.25	0.28	2.36	2.95	
		1170	220	5.20	1.55	0.85	0.47	0.29	0.25	0.24	0.25	0.25	0.25	0.26	0.27	3.70	4.63
12	4	323	2.5	5.20	-	3.25	2.45	1.89	1.52	1.07	-	0.47	0.26	0.25	0.86	0.86	
		430	5.9	5.20	-	1.71	1.27	0.87	0.64	0.46	-	0.26	0.23	0.25	1.22	1.22	
		647	20	5.20	-	1.55	0.90	0.61	0.41	0.29	-	0.24	0.24	0.25	1.82	1.83	
		970	67	5.20	-	0.92	0.50	0.35	0.27	0.24	-	0.23	0.24	0.27	2.92	2.92	
20	6	370	5.9	5.40	-	3.15	2.20	1.52	1.02	0.70	0.52	0.31	0.25	0.25	1.10	1.07	
		4	1140	24	5.40	2.70	-	1.16	0.73	0.44	0.32	0.27	0.24	0.24	0.26	1.82	1.57
		6	1140	170	5.40	1.50	0.93	0.45	0.29	0.24	0.22	0.24	0.26	0.27	0.30	3.60	3.50
<u>System B</u>																	
6	3	262	2.5	1.16		0.89	0.83	0.75	0.63	0.57		0.38	0.23	0.16	0.59	0.74	
		350	5.9	1.16		0.78	0.67	0.61	0.49	0.43		0.29	0.19	0.16	0.86	1.07	
		524	20	1.16		0.71	0.62	0.50	0.41	0.34		0.22	0.17	0.16	1.15	1.44	
		787	67	1.16		0.52	0.41	0.32	0.26	0.23		0.17	0.16	0.17	1.97	2.46	
		1170	220	1.16		0.52	0.38	0.30	0.24	0.20		0.16	0.18	0.19	2.35	2.94	

Table 10.1 (continued)

Mixer Dia, in.	Turbine Dia, in.	Speed, rpm	Power Input, hp/ 1000 gal	Uranium Concentration, g/liter											$k_a, \text{min}^{-1}$	
				0 sec	5 sec	10 sec	20 sec	30 sec	45 sec	60 sec	75 sec	120 sec	240 sec	480 sec	At D/T shown	At D/T = 1/3
System B (continued)																
12	4	323	2.5	1.16		0.79	0.70	0.60	0.51	0.42		0.29	0.20	0.17	0.88	0.88
		430	5.9	1.16		0.68	0.58	0.49	0.40	0.34		0.22	0.17	0.17	1.11	1.11
		647	20	1.16		0.59	0.49	0.40	0.33	0.28		0.19	0.19	0.19	1.43	1.43
		970	67	1.16		0.51	0.41	0.32	0.26	0.22		0.18	0.18	0.20	1.97	1.97
20	6	290	2.5	1.25	-	0.93	0.82	0.58	0.48			0.19	0.12	0.12	1.00	0.95
	3	1140	5.8	1.16	-	-	-	0.42	0.34			0.14	0.11	0.12	1.40	1.15
	6	1140	10	1.27		0.84	0.65	0.52	0.39	0.30		0.16	0.11	0.12	1.29	1.25
	4	1140	24	1.16		0.58	0.42	0.31	0.20	0.15		0.10	0.10	0.12	2.06	1.77
	5	1140	73	1.16		0.56	0.40	0.26	0.18	0.14		0.09	0.09	0.13	2.32	2.10
	6	1140	170	1.16		0.50	0.35	0.23	0.16	0.12		-	-	0.13	2.79	2.71
12	2	2050	20	1.16		0.52	0.41	0.31	0.26	0.23		0.17	0.18	0.18	1.92	-
	3	1040	20	1.16		0.52	0.39	0.34	0.28	0.24		0.18	0.17	0.18	1.77	-
	4	647	20	1.16		0.59	0.49	0.40	0.33	0.28		0.19	0.19	0.19	1.43	-
	6	329	20	1.16		0.69	0.55	0.45	0.37	0.31		0.22	0.17	0.18	1.28	-
	8	204	20	1.16		0.76	0.63	0.53	0.45	0.39		0.26	0.21	0.20	0.97	-

Table 10.2. Continuous Flow Uranium Extraction TestsSystem A

Mixer: 6-in. with 3-in. turbine (4 blade)

A/O phase ratio: 1/1

Aqueous feed: 5.3 g U/liter; at equilibrium, 0.20 g U/liter

Run No.	Turbine Speed, rpm	Power Input, hp/1000 gal	U in Raffinate, g/liter		Efficiency, %		ka, min <sup>-1</sup>		
			Effluent Sample	Calc. from Fritted Teflon	Effluent	Fritted Teflon	Fritted Teflon	Calc. at D/T = 1/3	
Aqueous-continuous dispersion									
Residence time 0.37 min; aqueous 1.0 gpm, solvent 1.0 gpm									
573	300	2.5	3.2	4.5	41.2	15.7	0.25	0.31	
574	400	6.0	1.9	3.7	66.7	31.4	0.62	0.78	
575	600	20	1.3	2.9	78.4	47.1	1.20	1.50	
579	900	68	1.3	1.8	78.4	64.7	2.47	3.09	
Residence time 0.74 min; aqueous 0.5 gpm, solvent 0.5 gpm									
557	420	6.9	1.1	-	82.4	-	-	-	
558	630	23	0.86	-	87.1	-	-	-	
559	840	55	0.66	-	91.0	-	-	-	
560	950	80	0.62	-	92.8	-	-	-	
Residence time 1.5 min; aqueous 0.25 gpm, solvent 0.25 gpm									
543	420	6.9	0.72	-	89.8	-	-	-	
544	630	23	0.45	-	95.1	-	-	-	
545	840	55	0.40	-	96.1	-	-	-	
546	950	80	0.38	-	96.5	-	-	-	

Table 10.2 (continued)

Run No.	Turbine Speed, rpm	Power Input, hp/1000 gal	U Conc. in Raffinate, g/liter		Efficiency, %		ka, min <sup>-1</sup>	
			Effluent Sample	Fritted Glass	Effluent	Fritted Glass	Fritted Glass	Calc. at D/T = 1/3
Solvent-continuous dispersion								
Residence time 0.37 min; aqueous 1.0 gpm, solvent 1.0 gpm								
576	300	2.5	3.2	4.7	41.2	11.8	0.18	0.23
580	400	6.0	2.3	4.6	58.8	13.8	0.22	0.28
581	600	20	1.3	3.8	78.4	29.4	0.56	0.70
583	900	68	0.93	2.9	85.7	47.1	1.20	1.50
Residence time 0.82 min; aqueous 0.45 gpm, solvent 0.45 gpm								
564	420	6.9	1.1	2.6	82.4	53.0	0.68	0.86
565	630	23	0.72	2.0	89.8	64.7	1.12	1.40
566	840	55	0.59	1.6	92.4	72.6	1.61	2.02
567	950	80	0.60	1.5	92.2	74.5	1.80	2.25
Residence time 1.5 min; aqueous 0.25 gpm, solvent 0.25 gpm								
551	420	6.9	0.66	2.2	91.0	60.8	0.52	0.65
552	630	23	0.53	1.6	93.5	72.6	0.88	1.10
553	840	55	0.42	1.3	95.7	78.4	1.21	1.51
554	950	80	0.40	1.3	96.1	78.4	1.21	1.51

Table 10.2 (continued)

## Second Pass

Aqueous feed: 1.7 g/liter; at equilibrium, 0.021 g/liter

Residence time: 1.5 min

Flow rate, each phase: 0.25 gpm

Run No.	Turbine Speed, rpm	Power Input, hp/1000 gal	U Conc. in Raffinate, g/liter		Efficiency, %		ka, min <sup>-1</sup>		
			Effluent Sample	Calc. from Fritted Sample	Effluent Sample	Fritted Sample	Fritted Sample	Calc. at D/T = 1/3	
Aqueous-continuous, Teflon sampler									
590	400	5.9	0.18	0.59	90.6	66.5	0.66	0.83	
591	600	20	0.11	0.41	94.7	77.1	1.12	1.40	
593	900	67	0.087	0.37	96.1	79.4	1.28	1.60	
Solvent-continuous, glass sampler									
588	300	2.5	0.42	0.80	76.5	54.1	0.39	0.49	
584	400	5.9	0.21	0.68	88.8	61.2	0.53	0.66	
585	600	20	0.14	0.48	92.9	73.0	0.90	1.13	
587	900	67	0.078	0.38	96.6	78.8	1.24	1.55	

Table 10.2 (continued)

System B

Mixer: 6-in. with 3-in. turbine (6 blade)

A/O phase ratio: 4/1

Aqueous-continuous dispersion

Run No.	Turbine Speed, rpm	Power Input, hp/1000 gal	U Conc. in Raffinate, g/liter		Efficiency, %		ka, min <sup>-1</sup>		
			Effluent Sample	Calc. from Fritted Teflon	Effluent	Fritted Teflon	Fritted Teflon	Calc. at D/T = 1/3	
Residence time 0.17 min; aqueous 4.0 gpm, solvent 1.0 gpm									
646	0	0	1.10	1.15	-	-	-	-	-
647	262	2.5	0.96	1.11	19.3	4.8	0.24	0.30	-
648	350	5.9	0.81	1.06	34.3	9.8	0.51	0.64	-
649	524	20	0.68	0.97	46.4	18.8	1.09	1.36	-
650	787	67	0.59	0.89	55.1	26.2	1.67	2.09	-
651	1170	220	0.55	0.84	58.9	31.4	2.15	2.69	-
Equil.	-	-	0.13	0.13	-	-	-	-	-
Residence time 0.37 min; aqueous 1.6 gpm, solvent 0.40 gpm									
634	0	0	1.06	1.16	-	-	-	-	-
635	262	2.5	0.79	1.03	36.3	13.1	0.33	0.41	-
636	350	5.9	0.63	0.95	51.8	20.2	0.55	0.69	-
637	524	20	0.55	0.81	59.5	31.7	1.00	1.25	-
638	787	67	0.50	0.78	63.9	36.8	1.26	1.58	-
639	1170	220	0.44	0.71	70.2	43.2	1.65	2.06	-
Equil.	-	-	0.13	0.13	-	-	-	-	-
Residence time 1.5 min; aqueous 0.40 gpm, solvent 0.10 gpm									
652	0	0	1.14	1.15	-	-	-	-	-
653	262	2.5	0.58	0.72	56.6	43.2	0.43	0.54	-
654	350	5.9	0.46	0.61	68.3	53.7	0.65	0.81	-
655	524	20	0.42	0.56	72.2	58.3	0.78	0.98	-
656	787	67	0.36	0.49	78.4	65.1	1.06	1.33	-
657	1170	220	0.31	0.43	83.6	71.2	1.39	1.74	-
Equil.	-	-	0.14	0.14	-	-	-	-	-

Table 10.2 (continued)

Run No.	Turbine Speed, rpm	Power Input, hp/1000 gal	U Conc. in Raffinate, g/liter		Efficiency, %		ka, min <sup>-1</sup>	
			Effluent Sample	Calc. from Fritted Teflon	Effluent	Fritted Teflon	Fritted Teflon	Calc. at D/T = 1/3
Residence time 2.5 min; aqueous 0.24 gpm, solvent 0.06 gpm								
640	0	0	1.07	1.15	-	-	-	-
641	262	2.5	0.42	0.50	72.3	56.3	0.42	0.53
642	350	5.9	0.32	0.49	81.6	65.2	0.60	0.75
643	524	20	0.31	0.45	83.0	68.6	0.71	0.89
644	787	67	0.28	0.40	85.9	74.1	0.92	1.15
645	1170	220	0.23	0.32	90.3	81.3	1.40	1.75
Equil.	-	-	0.13	0.13	-	-	-	-
Residence time 4.8 min; aqueous 0.12 gpm, solvent 0.03 gpm								
658	0	0	-	1.26	-	-	-	-
659	262	2.5	0.41	0.53	79.1	66.9	0.34	0.43
660	350	5.9	0.33	0.46	86.5	73.6	0.47	0.59
661	524	20	0.31	0.42	88.8	77.9	0.59	0.74
662	787	67	0.27	0.34	92.1	84.5	0.92	1.15
663	1170	220	0.23	0.27	95.8	91.1	1.72	2.15
Residence time 1.5 min; aqueous 0.24 gpm, solvent 0.24 gpm*								
671	0	0	-	1.21	-	-	-	-
672	262	2.5	0.57	0.66	61.1	53.9	0.39	0.49
673	350	5.9	0.45	0.53	72.0	66.0	0.64	0.80
674	524	20	0.34	0.43	82.9	76.2	1.06	1.33
675	787	67	0.28	0.34	88.2	84.5	1.81	2.26
676	1170	220	0.27	0.32	92.5	86.4	2.11	2.64
Equil.	-	-	0.18	0.18	-	-	-	-

\* A/O phase ratio = 1/1, organic continuous dispersion; fritted glass sampler used.

Table 10.2 (continued)

Mixer: 12 in. with 4-in. turbine (6 blade)

Run No.	Turbine Speed, rpm	Power Input, hp/1000 gal	U Conc. in Raffinate, g/liter		Efficiency, %		ka, min <sup>-1</sup> Fritted Teflon
			Effluent Sample	Calc. from Fritted Teflon	Effluent	Fritted Teflon	
Residence time 2.2 min; aqueous 2.1 gpm, solvent 0.52 gpm							
663	0	0	1.25	-	-	-	-
664	323	2.5	0.42	0.60	74.8	58.8	0.53
665	430	5.9	0.37	0.52	79.3	65.8	0.71
666	647	20	0.32	0.38	83.8	78.5	1.35
667	970	67	0.29	0.36	86.5	80.8	1.56
Equil.	-	-	0.14	0.14	-	-	-
Residence time 5.0 min; aqueous 0.92 gpm, solvent 0.23 gpm							
667	0	0	1.21	-	-	-	-
668	323	2.5	0.28	0.39	86.5	76.0	0.51
669	430	5.9	0.27	0.32	87.4	83.0	0.78
670	647	20	0.24	0.27	90.2	87.6	1.13
671	970	67	0.21	0.26	93.0	88.8	1.27
Equil.	-	-	0.14	0.14	-	-	-



Table 10.3. Iron and Vanadium Batch Extraction Rate Data

Mixer Dia, in.	Turbine		Power Input, hp/1000 gal	A/O Phase Ratio	Concentration in Aqueous, g/liter									ka, min <sup>-1</sup>	
	Dia, in.	Speed, rpm			0 sec	10 sec	20 sec	30 sec	60 sec	120 sec	240 sec	480 sec	960 sec	At D/T shown	Calc. at D/T = 1/3
Iron(III) Extraction															
6	3	262	2.5	4/1	0.22	0.22	0.21	0.21	0.20	0.19	0.17	0.15	0.13	0.070	0.088
		524	20	4/1	0.22	0.21	0.20	0.20	0.19	0.18	0.16	0.13	0.11	0.092	0.12
		1170	220	4/1	0.22	0.20	0.19	0.19	0.17	0.16	0.13	0.11	0.07	0.14	0.17
		524	20	4/1	0.22	0.20	0.19	0.18	0.16	0.12	0.08	0.04	0.01	0.11	0.14
		524	20	1/1*	0.22	0.19	0.18	0.18	0.15	0.12	0.08	0.04	0.01	0.11	0.14
Vanadium(IV) Extraction															
6	3	262	2.5	4/1	0.94	0.89	0.85	0.81	0.71	0.60	0.49	0.43	-	0.43	0.54
		524	20	4/1	0.94	0.78	0.74	0.70	0.60	0.48	0.43	0.44	-	0.76	0.95
		1170	220	4/1	0.94	0.72	0.66	0.60	0.51	0.46	0.46	0.50	-	1.51	1.89
		524	20	1/1	0.99	0.62	0.49	0.40	0.26	0.18	0.17	0.20	-	0.97	1.20
		524	20	1/1*	1.00	0.55	0.46	0.39	0.26	0.19	0.18	0.20	-	0.97	1.20
12	4	647	20	4/1	0.94	0.76	0.70	0.66	0.55	0.46	0.42	0.44	-	0.89	0.89
12	4	970	67	4/1	0.94	0.71	0.65	0.59	0.50	0.43	0.42	0.45	-	1.20	1.20

\* Solvent-continuous dispersion.

Table 10.4. Continuous-flow Uranium Stripping Data

Mixer: 6-in. dia, 3-in. turbine  
 Solvent: 0.16 M DEHPA, 35 g TBP/liter  
 Stripping agent: 1.0 M Na<sub>2</sub>CO<sub>3</sub>, 45% excess  
 O/A phase ratio: 5/1

Run No.	Flow Rates, gpm		Solvent Conc., g/liter		Turbine Speed, rpm	Efficiency, %
	Solvent	Aqueous	In	Out		
<u>First Stage</u>						
603	0.43	0.083	5.4	0.62	200	93
604	0.43	0.083	5.4	0.36	300	98
605	0.43	0.083	5.4	0.35	400	99
606	0.43	0.083	5.4	0.34	600	99
611	1.8	0.33	5.4	1.71	300	73
612	1.8	0.33	5.4	1.25	400	82
613	1.8	0.33	5.4	0.60	600	95
614	1.8	0.33	5.4	0.42	900	98
<u>Second Stage</u>						
607	0.43	0.079	0.43	0.042	200	94
608	0.43	0.079	0.43	0.022	300	99
609	0.43	0.079	0.43	0.021	400	99
610	0.43	0.079	0.43	0.021	600	99
615	1.7	0.34	1.0	0.086	300	96
616	1.7	0.34	1.0	0.066	400	98
617	1.7	0.34	1.0	0.058	600	98
618	1.7	0.34	1.0	0.058	900	98

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