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A POLYETHYLENE BOX-TYPE MIXER-SETTLER EXTRACTOR

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and Morton Smutz

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A Polyethylene Box-Type Mixer-Settler Extractor

by

Lester Knapp, Roman Schoenherr, John Barghusen
and Morton Smutz

ABSTRACT

The design features of a small multistage mixer-settler extractor are described. The polyethylene construction permits the use of corrosive systems such as those containing hydrofluoric acid. The design permits precise interface control in each settler. Complete cost data are given.

INTRODUCTION

The unit operation of liquid-liquid extraction has developed greatly within the last two decades and is now regarded as an important technique for separating and refining materials. The basic information needed for efficient design of extraction equipment, however, has not progressed as rapidly as the need for the application of the unit operation. Consequently, the design of extraction equipment has developed through experience rather than through application of theory. This is evidenced by the great variety of extraction equipment in use today.

Several articles have been published recently which review extraction equipment. Morello and Poffenberger (4) describe and classify a number of extractors, especially the column extractor. A complete survey of extractors was made by Pratt (5, 6) in which he evaluated and classified each type of extractor according to its physical characteristics and performance. Davis et al. (2) present a comprehensive review of mixer-settler contactors.

It will be noted that the function of extraction equipment is to provide the interfacial area necessary for the transfer of solute from one liquid phase to the other. The mixer-settler type of extractor is a stagewise contactor in which contact is provided in discrete stages by the action of an impeller.

The arrangement of stages in most patented designs of mixer-settlers is either vertical or horizontal. The vertical units usually possess a common shaft on which the agitators are mounted, while separate shafts and agitators are provided for each stage in the horizontal units.

Mixer-settlers have been in use since 1904 (4), but only recently has there been much interest in the design of a compact box-type horizontal mixer-settler. The need for compact mixer-settlers developed when atomic energy programs required that a number of mixtures of inorganic materials be separated in apparatus requiring a minimum of shielding.

Previous Designs of Mixer-Settlers

For the past few years workers at the Ames Laboratory have developed several separation processes which incorporate multi-stage liquid-liquid extraction techniques. Concurrent with the research work on process development was the design of an efficient multistage mixer-settler. The mixer-settler had to be able to handle liquids of high viscosity and also systems of liquids in which the density difference between phases was often quite small. Also, the extractor had to supply a constant flow of liquids from one stage to another even though there might exist a sharp liquid density gradient between adjacent stages.

In 1952, M. E. Whatley (7, 8) designed a multistage mixer-settler which consisted of a system of interconnected tubes (see Figure 1). The mixers and settlers were separate tubes connected by lengths of pipe or tubing. A third tube, which acted as an antechamber to the mixer, was included in the design. This tube (called a Tee from its appearance) served effectively as an interface controller since the height at which the tube was placed automatically positioned the level of the interface in the adjacent settling chamber. The light phase from stage n-1 and heavy phase from stage n+1 entered the side arms of the Tee. From there the phases were drawn into the mixing chamber by the pumping action of the stirrer. After agitation the mixture was pumped radially into the settler in which the phases separated.

A more compact mixer-settler, which employed the same principle of interface control as the Whatley extractor, was developed by Bochinski (1). Each stage in this extractor (see Figure 2) consisted of a mixing chamber, an antechamber, and a settling chamber. A unit stage was essentially a system of three concentric cylinders; with the mixing chamber and antechamber of stage n located within the settling chamber of stage n+1. The light phase from stage n-1 and the heavy phase from stage n+1 flowed into the antechamber of stage n from which they flowed simultaneously through an inlet weir into the mixing chamber of stage n. In the mixing chamber, by the action of an impeller, the phases were mixed and then pumped into the settling chamber of stage n.

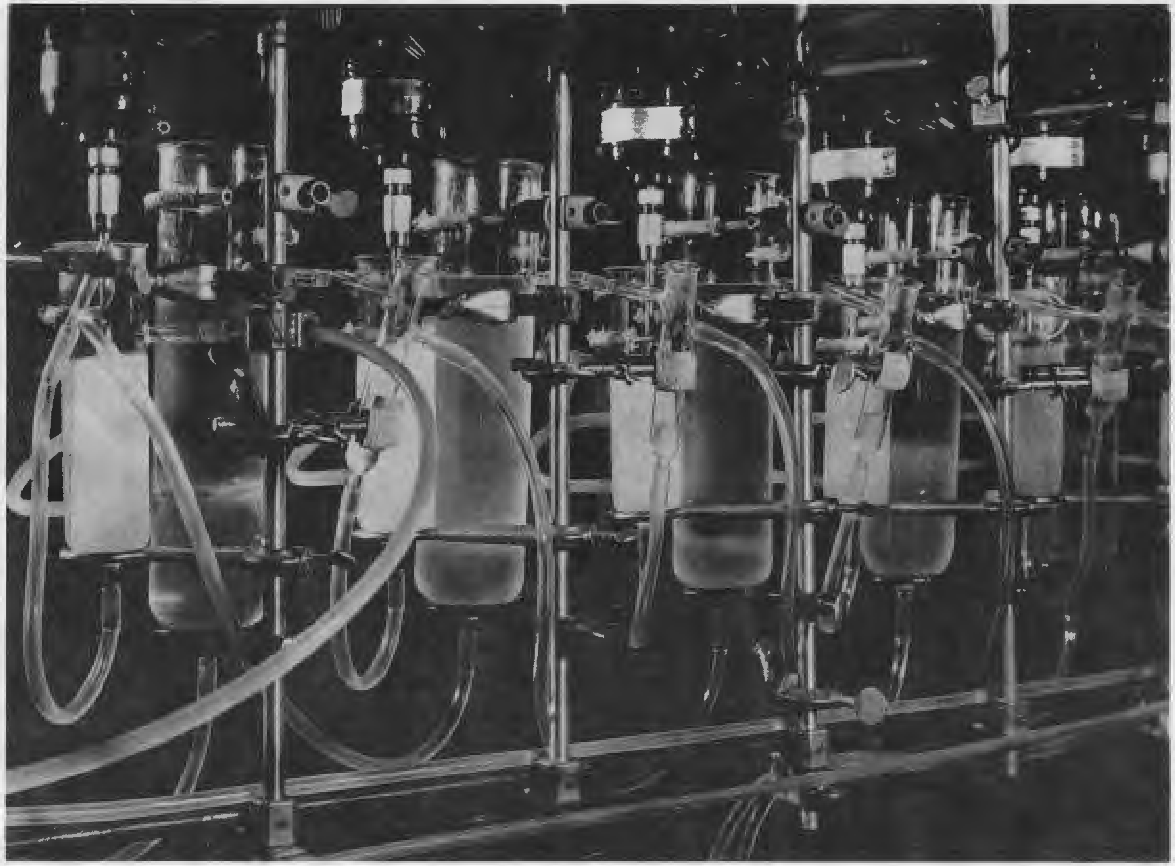
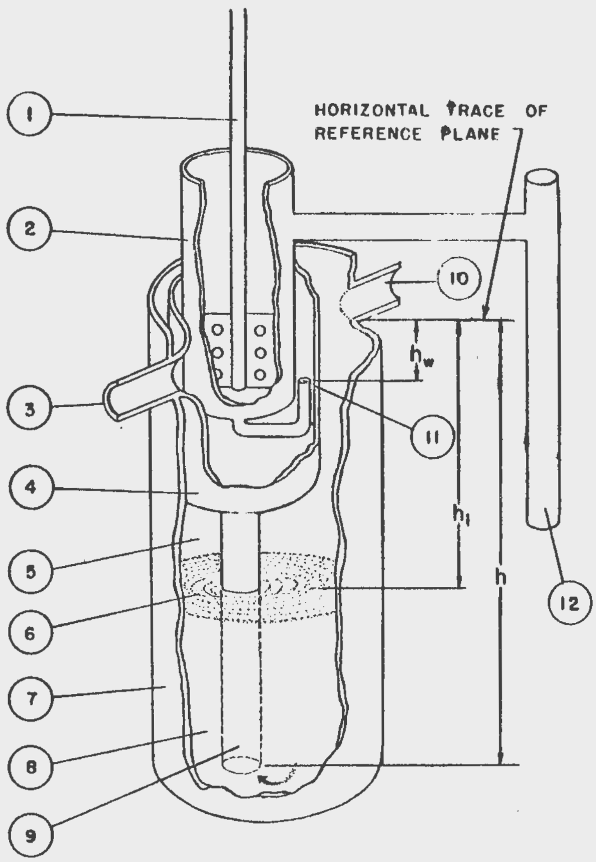


Figure 1. Mixer-settler extractor designed by M. E. Whatley.



LEGEND

1. MIXER PADDLE, 1" X 1.5"
2. MIXER TUBE, 5.5" DEEP, 1.2" D.
3. ORGANIC OVERFLOW FROM PRECEDING SETTLER
4. INNER RESERVOIR
5. ORGANIC LAYER
6. INTERFACE BETWEEN PHASES
7. SETTLING CHAMBER, 9" DEEP, 3" D.
8. AQUEOUS LAYER
9. EXIT TUBE FOR AQUEOUS LAYER
10. OVERFLOW FROM SETTLER TO FOLLOWING STAGE
11. INTERFACE CONTROL WEIR
12. OVERFLOW DELIVERY ARM TO ADJOINING SETTLER

Figure 2. Design features of the Bochinski extractor.

The mixer-settler designs developed by Bochinski and Whatley were satisfactory, but leaks frequently developed at some of the connections, and also there was an appreciable pressure loss as fluid flowed through the tubing. A box-type mixer-settler minimizes pressure losses and also the number of interstage connections.

This paper describes a new box-type mixer-settler which was recently developed at the Ames Laboratory. The design of this unit incorporates the basic feature of the Whatley extractor - interface control by adjustment of the height of the heavy phase in the antechamber.

Three salient features of the box-type extractor are: 1. horizontal arrangement of stages, whereby any desired number of stages can be made immediately available for any process; 2. interface control in each stage; 3. compactness of the unit, whereby no interstage piping is necessary to transport the fluids from one stage to another.

Design of the Mixer-Settler

The extractor, as shown in Figure 3, is a horizontal contactor constructed in the form of a rectangular box. The box is partitioned into discrete stages which, in turn, are divided into two chambers - an antechamber and a settling chamber. A mixing chamber is suspended in the antechamber from an overhead clamp. Each

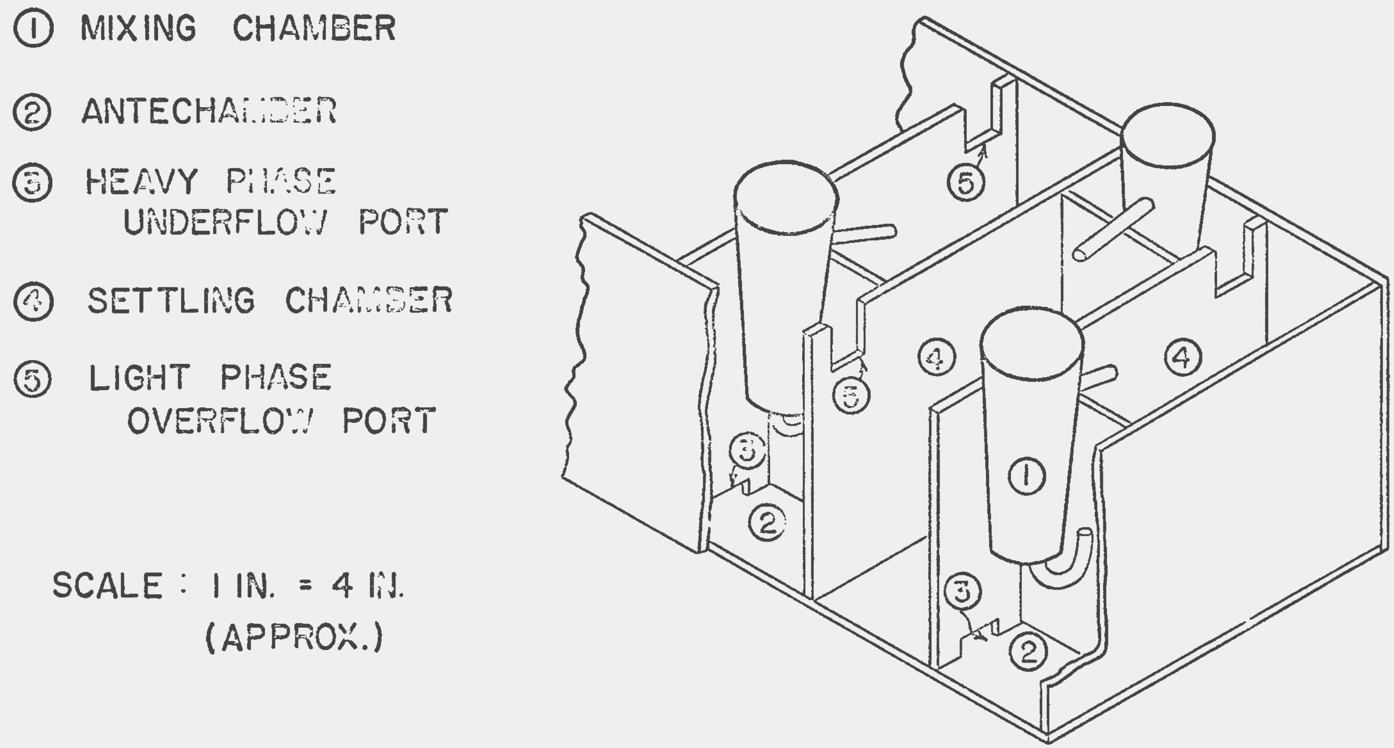


Figure 3. Design of the box-type mixer-settler extractor.

mixing chamber is provided with an impeller which enters through the top of the chamber. Each impeller is connected by means of a belt and pulleys to a shaft which drives all the stages simultaneously. The drive shaft is driven by a belt from an electric motor.

As illustrated in Figure 4, the flow of liquids through the extractor is counter-current. The heavy phase from stage n+1 and the light phase from stage n-1 flow through respective underflow and underflow ports into the antechamber of stage n. In the antechamber the heavy phase rises to the level of the inlet weir to the mixing chamber. The light phase accumulates as a film on the surface of the heavy phase. The two phases flow simultaneously through the inlet weir into the mixing chamber of stage n. In the mixing chamber, by the action of the impeller, the two phases are mixed and then pumped through an overflow arm into the settling chamber of stage n. Here the phases separate by gravity and then flow through the appropriate ports into the adjoining stages.

Thus the mixing section provides both mixing of the phases and pumping of the dispersed phases in the same operation. This is an essential feature of this mixer-settler. The need for an external means to provide the flow of the heavy phase from one stage to another is obviated with this mechanism.

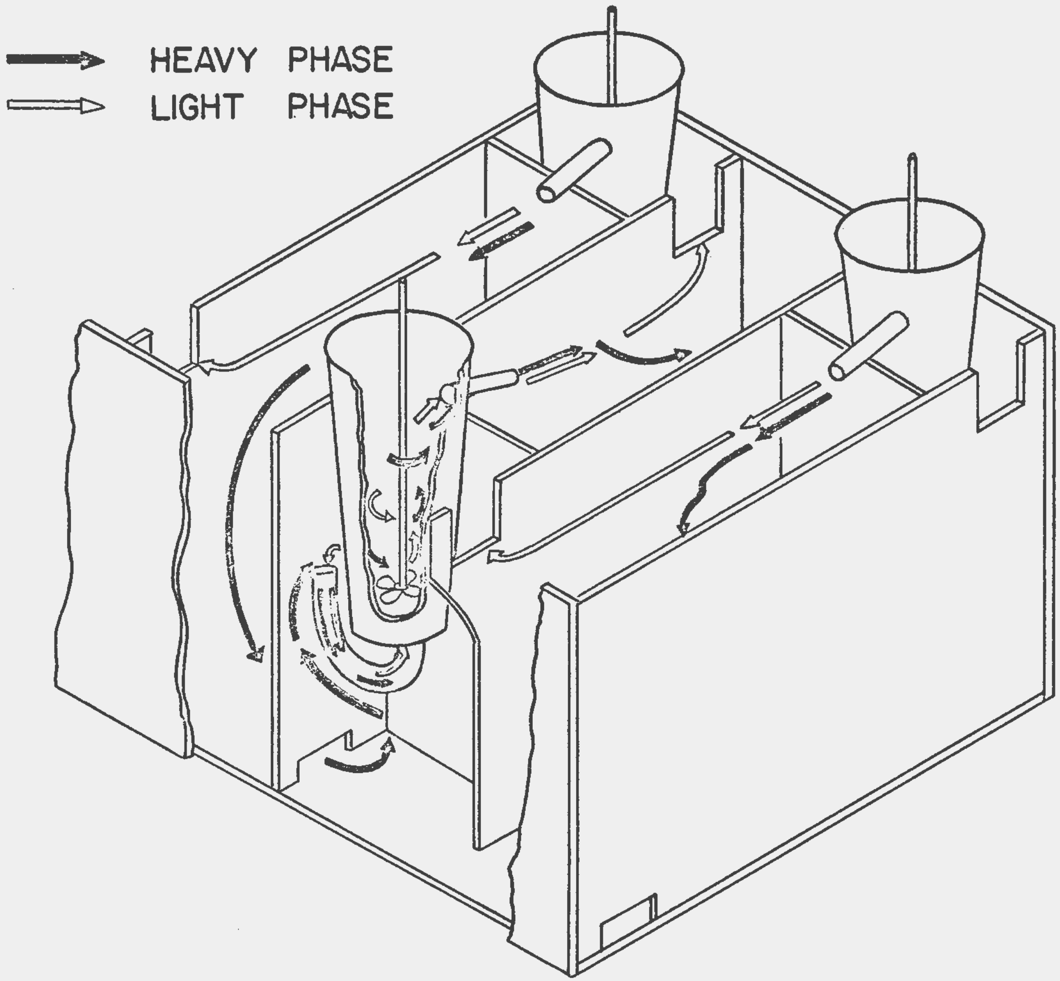


Figure 4. Flow pattern through the box-type mixer-settler extractor.

Construction of the Mixer-Settler

Several separation processes recently developed at the Ames Laboratory involved multistage liquid-liquid extraction. Therefore, one 16-stage and one 54-stage mixer-settler extractors were constructed to carry out these operations. Because the processes involved the use of corrosive chemicals, both of these units were constructed from polyethylene and stainless steel.

A section of one of the polyethylene boxes is shown in Figure 5. The boxes were constructed from polyethylene sheets. The sides and bottoms were cut in one piece which extended the full length of the box. The partitions were cut to size and the underflow and overflow ports cut into them. Slots were milled at all joints to provide a semi-force fit. After the boxes were assembled, the joints were reinforced and made water-tight by welding them with molten polyethylene.

Figure 6 shows the components of the mixer assembly. The mixing chamber was made from a polyethylene drinking cup. The weir and overflow arm were made from polyethylene tubing which was welded to the cup.

The impeller assembly consisted of a saran plate for mounting it onto the tube holder, a stainless steel bearing, a polyethylene-coated stainless steel shaft, a cover, a polyethylene impeller, and a pulley (not shown in Figure 6). The shaft was force fitted into

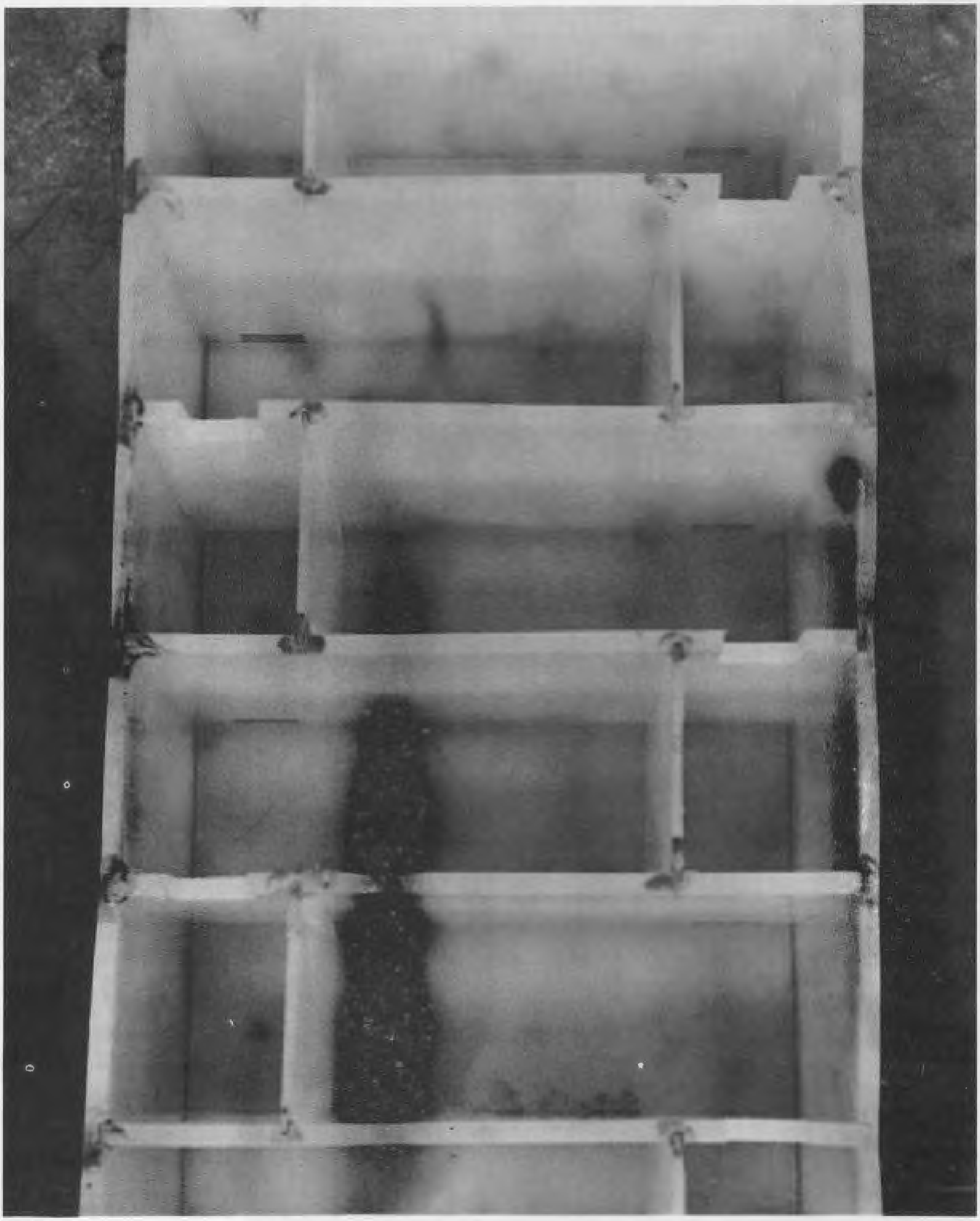


Figure 5. Section of assembled polyethylene box.

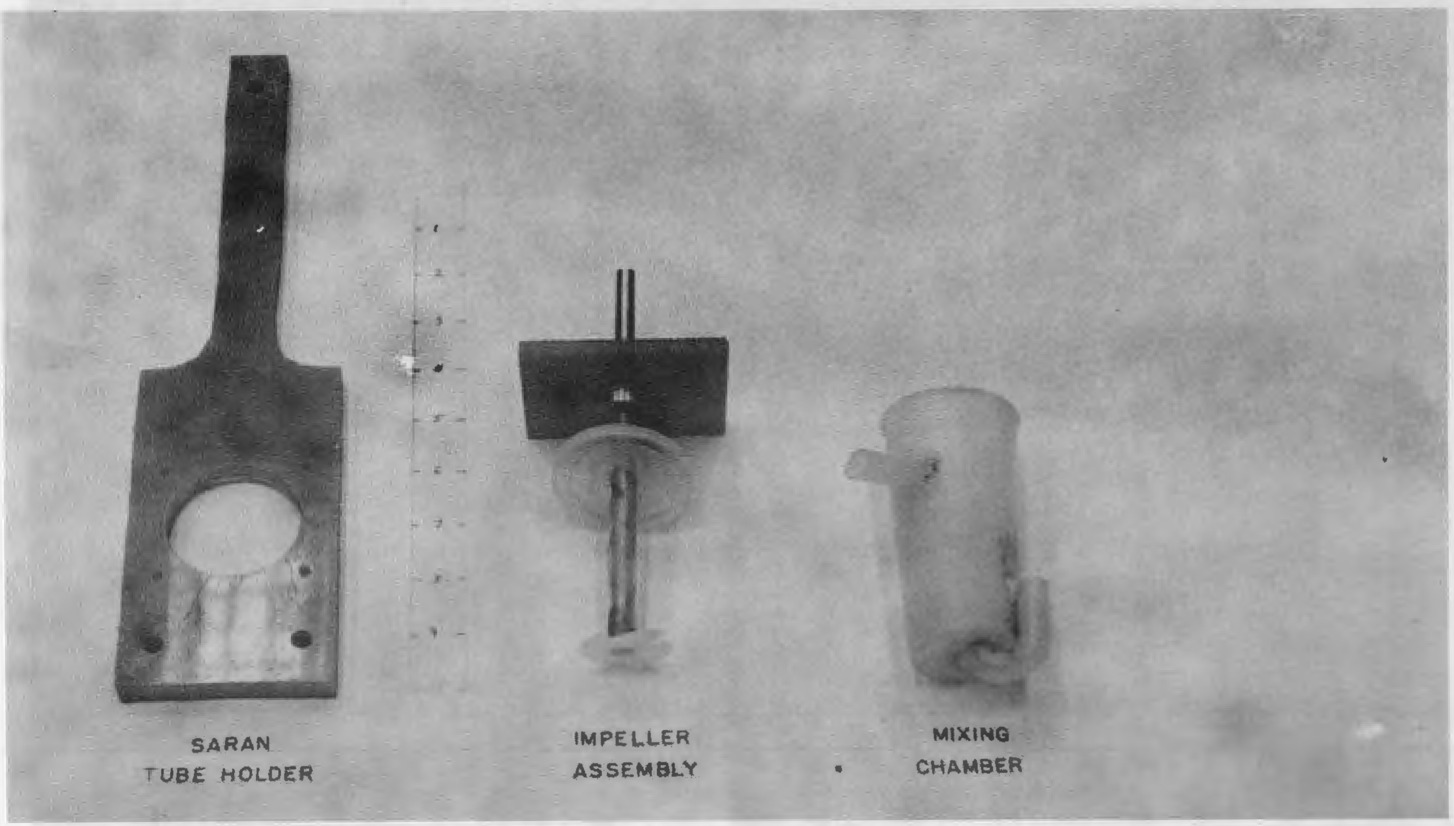


Figure 6. Components of the impeller assembly.

the bearing and then coated by shrinking polyethylene tubing over it. After adding the cover for the cup, the impeller was welded to the polyethylene coating on the shaft. The bearing was then force fitted into the saran plate.

The mixer was assembled in the following manner: The mixing chamber was tightly fitted into the hole in the saran tube holder, the impeller assembly was mounted to the tube holder with bolts and spacers, the cup lid was snapped on, and the pulley was attached.

The assembled holder was attached to a stainless steel frame which extended the length of the extractor. The holder can be positioned to any desired height by adjusting the bolts which affix the holder to the frame. Therefore, the height of the inlet weir can be positioned and the interface controlled by adjusting these bolts.

The drive mechanism for the impellers consisted of a system of belts and pulleys. Each impeller was driven by a belt from a single drive shaft. The drive shaft was powered by a V-belt from an electric motor.

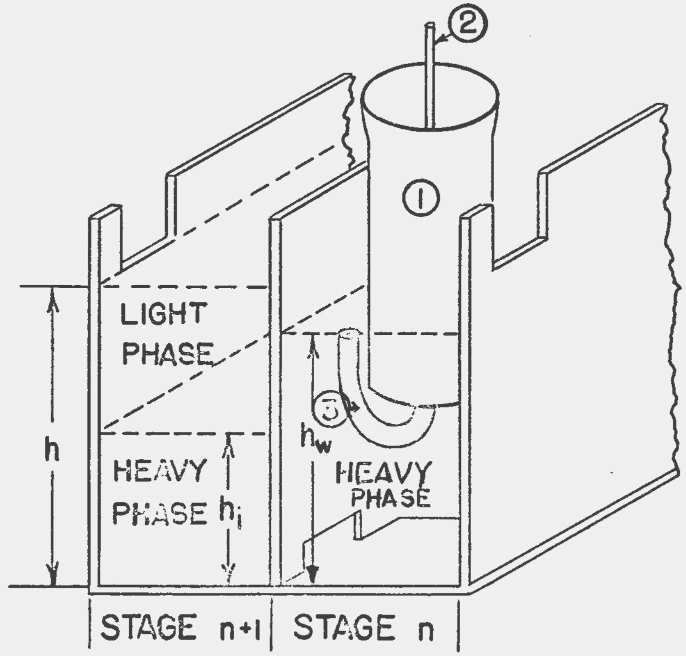
The assembled extractor is shown in Figure 7. This photograph is of the 54-stage extractor which was actually used to separate a mixture of rare earth nitrates by liquid-liquid extraction. Since an extractor of this size could not be fabricated in the form

Figure 7. 54-Stage mixer-settler extractor.

of one box and still retain some degree of portability, three boxes, each containing 18 stages, were used. No piping or tubing was used to transport the liquids from one box to the next. Instead, the end stages opposite each other were used only to pump the liquids from one box to the next. One mixer pumped the heavy phase into the settling chamber of the end stage in the next box, while the mixer of that stage pumped the light phase into the settling chamber contiguous to the first mixer.

Interface Control

The interface between the two liquid phases in the settling chamber of stage n+1 is maintained at any desired height by adjusting the position of the inlet weir to the mixing chamber of stage n. The hydraulic analysis of the interface control is shown in Figure 8. The position of the interface depends not only upon the densities of the individual phases but also upon the difference in the densities. It is apparent that the more nearly identical the densities of the two phases are, the more critical the position of the inlet weir becomes. A system with a density difference of 0.03 g/ml could only be handled if the position of the inlet weir could be set accurately to 1/32 of an inch. A deviation of 1/32 of an inch would produce a one-inch change in the level of the interface.



h = TOTAL HEIGHT OF LIQUID IN SETTLING CHAMBER OF STAGE n+1

h_i = HEIGHT OF INTERFACE IN STAGE n+1

h_w = HEIGHT OF INLET WEIR TO MIXING CHAMBER OF STAGE n

ρ_h = DENSITY OF HEAVY PHASE

ρ_L = DENSITY OF LIGHT PHASE

$$(h - h_i)\rho_L + h_i \rho_h = h_w \rho_h$$

$$h_i = \frac{h_w \rho_h - h \rho_L}{\rho_h - \rho_L}$$

① MIXING CHAMBER

② STIRRER

③ INLET WEIR TO MIXING CHAMBER

Figure 8. Hydraulic analysis of interface control in the box-type mixer-settler extractor.

In Figure 6 the mechanism for holding the mixing chamber is shown. The mechanism was designed so that, by a simple adjustment, the height of the inlet weir could be positioned accurately in the antechamber. Consequently, every stage has its own interface control. By knowing only the densities of the two phases and the depth of the settling chamber, the height of the interface in each stage can be pre-set by adjusting the position of the mixing chamber in the adjacent stage.

When the holders for the mixing chambers were assembled, great care was taken to insure that the top of the inlet weirs were in all cases a certain distance below the bottom of the saran plate. The bottom face of the plate was taken as a datum for positioning the height of the inlet weir in the antechamber. For each stage in the extractor, the distance between the top of the inlet weir and the saran plate was the same. Any changes which were required, due to density gradients in the extraction system, were made by adjusting the bolts which affixed the holder to the steel frame which extended the length of the extractor.

Cost of Constructing the Extractor

In connection with the construction of the 54-stage mixer-settler, an estimate of the cost for materials and labor involved was made. Table I itemizes the specifications and costs for the materials which were used in constructing this unit. Although this

Table I. Cost for 54-stage mixer-settler

Item	Material cost	Labor cost	Total cost
Mixer-settler box and table			
Polyethylene	$ 84.24	$114.76	$ 199.00
Angle iron table	31.60	92.25	123.85
Steel frame	74.62	132.75	207.37
	$190.46	$339.76	$ 530.22
Mixing holder assembly			
Bearings, shafts and spacers	$214.93	$174.28	$ 389.21
Mixing cups	5.50		5.50
Saran tube holders	190.00		190.00
Saran bearing plates	21.00		21.00
Bolts and nuts	159.41		159.41
	$590.84	$174.28	$ 765.12
Drive Assembly			
Main drive shaft	$ 16.75		$ 16.75
Universal joints	26.10		26.10
Drive pulleys from motor	2.00		2.00
Neoprene "O" ring belts	21.81		21.81
V-drive belt	1.27		1.27
Aluminum pulleys	57.70	$419.63	477.33
Supports	36.00		36.00
	$161.63	$419.63	$ 581.26
Total cost	$942.93	$933.67	$1,876.60

cost analysis is rather detailed, the resulting total cost is only an order of magnitude; and any extrapolation to other units could not be made with great accuracy. A considerable labor cost has been omitted from the table. This was the labor used in the construction of the boxes and in the final assembly of the mixer-settler. If this labor cost were included the final total cost of the mixer-settler might be approximately $2,500.

Summary of Characteristics of the Extractor

The extractor has been used successfully in two different extraction processes. Koerner et al. (3) used a 16-stage box-type mixer-settler of this design in separating niobium and tantalum by liquid-liquid extraction. The feed to the extractor was a solution of tantalum, niobium and hydrofluoric acid in methyliso-butyl ketone. No difficulty was experienced in handling the hydro-fluoric acid system. The niobium was scrubbed from the tantalum with 0.1M sulfuric acid.

The 54-stage extractor was used in the separation of mixed rare earth nitrates with tributyl phosphate as a solvent. The feed, solvent and scrub solutions were all 5N in nitric acid. No corrosion difficulties were experienced during a run lasting 300 hours.

There are several characteristics of this box-type mixer-settler which distinguish it from other extractor types. The box-type mixer-settler has a built-in interface controller which can be adjusted even when the extractor is in operation. The interface controller can be positioned with a fine degree of accuracy.

It is known that for optimum mixing of liquids in a continuous process, the liquids must enter the mixing chamber along the axis of the impeller. This is adequately provided for in this design. Both phases enter the mixing chamber along the axis of the impeller.

No auxiliary means are necessary to transport the heavy phase from one end of the extractor to the other. In some box-type units, the extractor is tilted to move the heavy phase from stage to stage. In this unit, however, the necessary head is provided by the impeller which pumps the phases through the extractor.

This box-type design is compact. There is no need for any interstage piping connections. The liquids flow easily from one stage to the next through the ports in the partitions.

The number of stages which can be added to the extractor is almost unlimited. It can be envisioned that an extractor containing 150 to 200 stages is not at all beyond the realm of possibility. The stages can be easily added without being concerned about interstage connections.

Unlike many mixer-settlers, there is no back-mixing of liquids in this design. The unit was designed so that it was impossible for back-mixing to occur.

A wide latitude in impeller speed is allowed in this design. The impeller may rotate within a wide range of speed above a minimum without decreasing mixing efficiency. Therefore, by having the impeller rotate at a speed above the minimum, the mixer-settler can easily handle surges in liquid flow and still provide maximum mixing efficiency.

The box-type unit is simple in design. Therefore, construction of metal or plastic units is easily accomplished.

Feed lines can be introduced at any stage without revising the extractor design or construction. This is usually true with most mixer-settler designs, but is not necessarily the case with column extractors.

During the continuous run made with the rare earth nitrate-tributyl phosphate system, the extractor performed quite well even though the liquids were about ten times as viscous as water. The impeller was able to displace all of the liquids entering the mixing chamber. Also, there was no build-up of liquid in the ante-chamber.

BIBLIOGRAPHY

1. Bochinski, J. H. Separation of individual rare earths by liquid-liquid extraction from multicomponent monazite rare earth nitrates. Unpublished Ph.D. thesis. Iowa State College, Ames, Iowa. 1954.

2. Davis, M. W., Hicks, T. E. and Vermeulen, T. Mixer-settler extraction equipment. Chem. Eng. Prog. 50: 188-197. April 1954.

3. Koerner, Ernest L., Smutz, Morton and Wilhelm, H. A. Separation of niobium and tantalum by liquid extraction. U. S. Atomic Energy Commission Report ISC-802. December 1956.

4. Morello, V. S. and Poffenberger, N. Commercial extraction equipment. Ind. Eng. Chem. 42: 1021-1035. June 1950.

5. Pratt, H. R. C. Liquid-liquid extraction in theory and practice. Ind. Chem. 30: 475-482. October 1954.

6. Pratt, H. R. C. Liquid-liquid extraction in theory and practice. Ind. Chem. 30: 597-601. December 1954.

7. Whatley, M. E. Mixer-settler. U. S. Patent 2,754,179. July 10, 1956.

8. Whatley, M. E. Purification of thorium by solvent extraction. Unpublished Ph.D. thesis. Iowa State College, Ames, Iowa. July 22, 1953.