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LIQUID-LIQUID EXTRACTION

Milton W. Davis, Jr., Thomas E. Hicks, and Theodore Vermeulen

January, 1951

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LIQUID-LIQUID EXTRACTION

Milton W. Davis, Jr., Thomas E. Hicks, and Théodore Vermeulen
Radiation Laboratory and Department of Chemistry and Chemical Engineering
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ABSTRACT

Extraction equipment of the multistage mixer-settler type has been found useful for providing the intensive contact between phases that is required in processes involving complex compounds of the heavy metals. In this report, the factors of mixer-settler design and operation are discussed with reference to the known types of mixer-settler units. Following this review, a detailed description is given of the mixer-settler units recently constructed and installed in this laboratory.

I. SURVEY OF MIXER-SETTLER DESIGNS

The unit operation of solvent extraction is an important method for separating both organic and inorganic mixtures on an industrial scale. Many different designs of contacting equipment have been used, or proposed for use, in extraction processes. Variations in the properties of each of the two liquid phases, in the rates of interphase transfer of the solutes, and in the ease of isolating the phases after they have been contacted, are partly responsible for the diversity of equipment.

In general, extraction operations are limited by equilibria which do not allow complete separation in a single stage. For this reason, they are usually carried out countercurrently in equipment which is equivalent to several equilibrium stages. Three distinct types of continuous-flow countercurrent extraction equipment are known:

1. Equipment in which the relative vertical flow of the two phases under gravity (or the relative radial flow in a centrifugal field) accomplishes
the contact between phases. Vertical columns equipped with spray nozzles, perforated plates, or ring or saddle packing are the best known examples of this type. An annular column with rotating central shaft may be considered a variation of this type.

2. Equipment in which interface for transfer is obtained by oscillatory flow of the liquids passing through the contact equipment, as in pulsed packed columns and pulsed perforated-plate columns.

3. Equipment of the mixer-settler type in which thorough contact is achieved in individual stages by the mechanical action of an impeller or a mixing jet, and the phases are then separated by gravity or by centrifugation before entering the adjacent stages. Within the mixer-settler type many variations in design are possible. The major factors are:

(a) A common shaft for all stirrers, vs. an individual shaft for each stage. The use of a common shaft evidently requires a more direct interconnection between the stages; however, this type appears less expensive both to construct and to maintain.

(b) "Vertical" vs. "horizontal" arrangement of the stages. In general the "vertical" units possess a common shaft and the "horizontal" units do not. Also, the transfer between stages almost always occurs by gravity in the "vertical" type, while it may occur either by gravity or by pumping of one or both streams in a "horizontal" unit.

(c) Cocurrent vs. countercurrent settling within each stage. In cocurrent settling, the emulsion produced in the mixing chamber passes to a single settling chamber, from which the separated phases move to the adjacent stages. In countercurrent settling, the emulsion spreads into two settling chambers,
one above and one below the mixing chamber. From the upper settler, the light phase is carried to the next higher stage, while the heavy phase is returned to the mixing chamber; from the lower settler, the heavy phase is withdrawn and the light phase is returned to the mixer.

(d) Control of the ratio of phases in the mixing chamber, independently of the flow rates, which is permitted by some designs. This will allow the residence time to be increased for the phase in which the slow transfer step occurs. This phase control can take place only if the pressure distribution in the mixing chamber is uniform enough to permit the density of fluids to cause an equalization of densities between mixing and settling chamber; then the control of the heavy phase level in the settling chamber will control its volume in the mixing section. If the emulsion entering the settling chamber is uniform with that in the mixing chamber, then back flow of one phase must occur from the settling chamber to the mixing chamber, to permit the phase ratio to be independent of flow rate. Therefore, if the organic-to-aqueous phase-ratio is greater than the organic-to-aqueous flow ratio, a certain amount of countercurrent settling or recycling of the organic phase must be postulated, even though the over-all flow to each settling chamber is cocurrent.

Twelve industrial designs for mixer-settlers are discussed in this section. Of these, the McKittrick column (Figure 1) appears to have the soundest design. It is a vertical column with a common shaft for the stirrers, and flow is by gravity, providing simplicity of construction and operation. The settling chambers are outside of the column, hence the column is much shorter than it would otherwise be. A summary of the main features of all twelve mixer-settlers is shown in Table I.
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VERTICAL COLUMNS

McKittrick Column (1939). The apparatus in Fig. 1 is a vertical mixer-settler with a common shaft for all stirring paddles. In each stage the liquids flow cocurrently to the settling chamber. The mixing paddles are mounted on a vertical shaft extending the height of the column through the horizontal partitions, the partitions being provided with suitable packed bearings around shaft to prevent the flow of fluid between mixing stages. Each adjacent pair of chambers is interconnected by a small valve which is used for venting air from the apparatus or draining liquid to the lower chamber.

Elongated horizontal settling tubes are connected to each mixing chamber at a level near the middle of the chamber. These settling tubes are cylindrical in shape and entirely external to the mixing chambers. The settling tubes are connected to T-fittings, opening into vertical conduits which connect with the upper part of the mixing chamber above and the lower part of the mixing chamber below. The heavy liquid take-off from the bottom stage is controlled by valve and actuated by a liquid level controller operable to cause only heavy liquid to discharge at the bottom. The light liquid discharge at the top of the column is controlled by a pressure-actuated valve, arranged to maintain a predetermined pressure in the system. The heavy feed to the column enters at the top through line while the light feed enters at the bottom through line 17.

Fig. 2 shows a schematic view of the flow throughout the column. The overall flow is countercurrent while the settling flow is cocurrent.
McKittrick Column

FIG. 1

McKittrick Column

FIG. 2
MU 968
The column is assembled by bolting the mixing sections together by external flanges. Each mixing paddle must be fixed to the common shaft before the next mixing chamber is bolted into place. After assembling the mixing chambers the settling chambers may be attached externally as piping which is connected to a T leading to the next higher and next lower mixing chambers, as shown in Fig. 1.

Schöneborn Column (1934). The apparatus in Fig. 3 is a vertical mixer-settler with a common shaft for all mixers. The over-all flow is by gravity while the liquids flow cocurrently to the settling chambers. The heavier liquid enters at the top through pipe 13 and after passing through the column leaves through pipe 4; the light liquid enters through pipe 9 at the bottom and leaves through pipe 16. Each of the mixing sections 1 is separated from the section above and below by horizontal partitions, each of which has a central opening through which shaft 21 passes and the light and heavy liquids flow between stages. The partition or floor of each mixing section is provided with an upper and lower projection. The upper projection 37 in Fig. 4 serves to dam, on the bottom 35, the heavier liquid settling in the section above. As fresh liquid flows into the upper section, the heavier liquid flows over projection 37 through opening 36 into the next lower mixing section. The lower projection 38 serves a similar purpose for the lighter liquid. On the projection 38 are four tubular extensions 39 which carry the light liquid collecting beneath the floor 35 into the next higher mixing section, without hindering the downward movement of the heavier liquid. Each mixing section is provided with flanges 1a by means of which it is bolted to adjacent sections.
Figs. 4, 5, and 6 illustrate the design of the mixing device. It consists of an inner supporting member 42 secured on the shaft 21 by a key 41, and inside the housing 43. When the stirrer is rotated, fresh unmixed liquid flows into channels 44 and 44a from the sections above and below. The centrifugal force causes the mixed liquids to be thrown out of the side opening 44b into the settling chamber where they separate and are then directed to adjacent stages.

A comparison of the McKittrick and Schöneborn columns shows the settling chambers to be external to the column in the McKittrick type while they are inside the column in the Schöneborn type.

McConnell Column (1937). The apparatus shown in Fig. 7 is a vertical mixer-settler with a common shaft for all mixers. The heavy phase is pumped from stage to stage while the light liquid flows by gravity. The liquids flow concurrently while settling. The ratio of phases in the mixing chamber is relatively independent of flow rate, because the interstage pumping may be accelerated or retarded to allow one of the phases to accumulate. The lighter liquid is fed into the inlet 6 at the bottom and the heavier liquid is fed into inlet 8 at the top. After the liquids pass countercurrently through the tower, the lighter liquid is discharged at the top outlet 7, and the heavier liquid is discharged at the lower outlet 9. In travel through the apparatus, the liquids are mixed together in the impeller housings 10; passing through the top they enter the settling chamber where they separate and maintain an interface at the upper part of the compartment. The heavier phase is then pumped through outlet 14 to the bottom of the next lower mixing sectio
FIG. 4

FIG. 5

FIG. 6
where it enters with the lighter phase moving up from the section beneath. Individual temperature control in each stage is provided by coil 20.

**Scheibel Column (1946).** The vertical mixer-settler shown in Fig. 8 has a common shaft for its agitators, and the overall flow is by gravity. Mixing sections are alternated with settling sections packed with Raschig rings or other packing material, in which the liquids undergo countercurrent flow. A column of this range can be run with either phase continuous. Stage efficiencies of over 100 percent have been observed due to material transfer in the packing in addition to that in the mixing section. It has been observed experimentally that a packed section larger than the mixing section leads to better stage efficiencies.

**Bottaro Columns (Inclined) (1940).** The nearly vertical mixer-settlers shown in Figs. 9 and 11 have common shafts for the agitators, and the liquids flow in a countercurrent manner while settling as in the Scheibel column. The overall flow is by gravity with the heavy liquid fed at the top at entrance 4, while the light liquid is fed at the bottom at entrance 5. In Fig. 9 the settling zone is formed of flat plates 8. The vertical position of operation is excluded in this design since it is desirable to have the fluids pass each other in layers or sheets rather than in drops. The settling plates are placed very close together to give more complete settling together with some additional material transfer. Fig. 10 shows a cross section at II-II.

In Fig. 11, the principles are the same except that the calming section consists of cylindrical shells attached to the rotating shaft which are purported to enhance the separation of phases by centrifugal force. A cross section at V-V is shown in Fig. 12.
HEAVY LIQUID

LIGHT LIQUID

SCHEIHEL COLUMN

FIG. 8

MU 972
BOTTARO COLUMN FIG. 11

FIG. 12
MU 974
Othmer Column (1935). The vertical mixer settler shown in Fig. 13 has a common shaft for its agitators and the over-all liquid flow is by gravity. The two phases settle in a countercurrent manner. The mixing chambers are separated from each other by hollow cones with fins dividing the volume outside and inside into four sections for improved settling. The stirring shaft passes through the apex of these cones with some space left for upward flow of light liquid. The heavy liquid passes down the outside of the cones and through small openings to the next mixing chamber. Fig. 14 is a detailed drawing of this cone.

Comparison of Vertical (Common- Shaft) Mixers. The McKittrick column is a quite satisfactory design. The phase ratio in the mixing chamber is essentially independent of flow rates, which permits the phase ratio to be adjusted to give the maximum transfer rate. Moreover, the relative volumes of mixing and settling chambers may be varied widely.

The Schöneborn has the unsatisfactory feature of having a very small mixing zone, and also has the phase ratio in the mixing zone a function only of the flow rates.

The McConnell column uses pumps for the heavy phase, which cost more than gravity flow. The method of introduction of the heavy phase, so that it must travel upward with the light phase, is unsatisfactory. Such a condition might well cause some of the heavy phase to by-pass the mixer completely and pass into the next chamber. The small mixing chamber and large settling chamber may or may not be satisfactory, and will in any case require a high stirring speed.

The Schelbel column has the disadvantage of having the phase ratio in the mixing zone dependent upon the flow rate. However, it is possible to alter greatly the ratio of phases in the mixing zone by changing the continuous phase,
FIG. 14

FIG. 15

OTHMER COLUMN FIG. 13
or by varying the stirring rate and packing size. The Scheibel column is probably the least expensive to construct in this group.

The first Bottaro column (Fig. 9) is very similar to the Scheibel column except that the settling sections are parallel flat plates. These are probably more expensive to construct and assemble than the packed sections of Scheibel's. The second Bottaro column (Fig. 11) is somewhat impractical because of the high cost of fabricating the settling cylinders, and because of the heavy shaft and supports required to carry the weight of the settling sections.

The Othmer column (Fig. 13) is very similar to the preceding two (Figs. 8 and 9), except that the settling sections are arranged so that no contacting takes place while settling even though countercurrent flow takes place.

The mixing efficiency of the type of stirrer used in each design is approximately the same.

HORIZONTAL EXTRACTORS

Van Dijck Extractor (1941). The fluids flow countercurrently by gravity in all sections of the nearly horizontal mixer-settler shown in Fig. 15.13 The unit consists of a cylindrical shell with supports 2 and 3, subdivided by partitions 4a, 4b, 4c, 4d, 4a', 4b', 4c', 4d', and etc. formed of perforated plates or sieves to provide alternating mixing and settling zones. A common shaft 12 carries agitators 13a, 13b, 13a', and 13b' such as propellers or stirring rods, and is turned by pulley 14 to provide mixing in the mixing zones.

The flow through the apparatus is by gravity alone, and depends upon the tilt of the apparatus and upon the density differences of the two phases. Valves 15 and 16 control the feed to and outflow from the end settling zone 7.
while valves 17 and 18 similarly control the outflow from and feed to end settling zone 8. The light liquid enters through valve 15 and the heavy liquid enters through valve 18.

Holley Extractor (1934). The horizontal mixer-settler shown in Fig. 17 has an individual stirring shaft for each stage, with a recirculation line from the settling chamber to the mixing chamber. The rate of recirculation is provided by a valve in the recirculation line. The rate of recirculation controls the ratio of phases in the mixing chamber. The overall flow is by gravity. The light liquid enters at y and leaves the apparatus at y' after passing through several mixing stages (four are shown). The heavy liquid enters at z and leaves at z'. The emulsion from the mixing chamber flows through d to the settling chamber where the heavy phase can either be recirculated to the mixing chamber through e or sent to the next mixing section through the bottom connection g. The light phase passes from each settling section to the next mixing section through connection f. The recirculation device is intended as means for maintaining a fixed ratio of light to heavy liquid in the mixing chamber irrespective of the rate at which the respective liquids are fed. By providing a number of connecting pipes between the respective mixing and separating vessels, and by controlling the flow through them, the amount of liquid recirculated to the mixing vessel may be varied widely to meet varying practical requirements. The counterflow of the liquids is caused by gravity alone.

In Fig. 18 the mixing chamber is shown in detail. A vertical spindle h is the mixing shaft. Vanes or blades i are mounted upon the spindle, one beneath the other and at an angle one to the other, the vanes or blades
HOLLEY EXTRACTOR

FIG. 17

FIG. 18

MU 977
being mounted so that a diagonal line across them coincides with the axis of the shaft. The top blade is surmounted by an antivortex disc j transversely mounted upon the spindle h. The spindle h is located eccentrically to the vertical center line of the mixing chamber to help prevent vortex formation. It is also away from the position of the connecting pipes. The mixing vessels may be provided with covers or bells k secured to the spindles h and having downwardly extending peripheral edges adjusted for reception within an annular sealing trough l on the top plate of the vessels.

Fig. 18 also shows a device for removing foreign matter that may accumulate at the interface in a settling chamber. For this purpose a horizontal radial arm m, open or slotted, may be mounted on a vertical spindle n, the upper end of which is supportedly suitably; the lower end is mounted within a tubular socket at the bottom of the separating vessel, the socket being connected to an outlet pipe that extends beneath the vessel; the outlet may be controlled by a valve. By vertical adjustment of the upper end of the hollow rod or spindle, the horizontal arm may be set at the interface; upon rotation of the hollow rod, the arm will sweep the plane of the interface, and any accumulation of solid matter at the interface will pass out with the entrancing liquid into a skimming tank beneath the settling chamber.

Mensing Extractor (1946). The horizontal mixer-settler shown in Fig. 19 has individual stirrers and countercurrent settling flow controlled by a recycling tube which may be adjusted in height so as to recycle either emulsion or settled phases. The two phases are pumped into the mixing section by the action of the mixing propeller. The heavy liquid level in each chamber is set by an adjustable inverted u.

Fluid to be extracted enters through conduit 10, and extracting solvent enters through conduit 11. They mix in conduit 13 through which they enter
the space enclosed by sleeve 7 and become thoroughly and intimately agitated by the action of the impeller. The latter, designed for upward thrust, forces the emulsified mixture up through the sleeve, out through openings 8 into the annular space between sleeve 7 and baffle 9, down through the annular space, under the lower edge of baffle 9, and out into the main space of chamber 1. In this main chamber, the emulsion from the mixing section breaks, and the respective phases overflow through opening 16 and out of the chamber through 17 or fall through opening 18 up through conduit 19 and down through conduit 21. By adjusting the height of the inverted U formed by conduits 19 and 21, the height above the bottom of the chamber, and therefore the volume of the heavy phase, may be controlled.

It is apparent that the flow capacity through the sleeve 7 and the annular space between the sleeve and baffle is potentially much larger than through the feed conduits 10 and 11. Conduit 14 is used in this connection to recycle unseparated or partially separated emulsion. In practice it is desirable to make the inlet to conduit 14 adjustable in height so that the recycled fluid may be drawn from either separated or unseparated layers.

In Fig. 20 the apparatus consists of several chambers of the type shown in Fig. 19. A large chamber 22 is divided into three chambers of equal size, each containing the apparatus described in the single unit. Each chamber is provided with a conduit 10 for introducing the fluid to be extracted. In chamber 23 this enters from the outside, and in chambers 24 and 25 the fluid comes from the immediately preceding chamber. Each of the chambers is also provided with a conduit 11 for introducing solvent in a direction countercurrent to the flow of material to be extracted. The solvent is introduced through conduit 28 into a pocket 29 formed by baffle 30, from which pocket the solvent
is carried through a conduit down into the T connection below the impeller. Each of the chambers has a pocket, but the solvent flow from one chamber to the next is over the dividing wall which does not extend the entire height of the chambers.

In order to obtain a more uniform recirculation, conduit 14 has been replaced by the following alternative arrangement. In each of the chambers a third concentric baffle 31 is located between sleeve 7 and baffle 9 and extending about half the height of sleeve 7. This baffle is closed at the top by an annular plate. Admission into the annular space between sleeve 7 and baffle 31 is by means of several short conduits 33 spaced approximately equally around the baffle.

The liquid interface within each chamber is controlled by adjusting the distance to which conduit 36 extends up into pocket 34.

**Edeleanu Extractor (1927).** The horizontal mixer settler shown in Fig. 21, with individual stirrers, has the two phases settling in cocurrent flow. The light phase is pumped up to the next mixer while the heavy phase flows by gravity. The heavy liquid enters by line 64, and the light liquid by line 65. The liquids then proceed in a countercurrent flow pattern. Fig. 22 shows the method for introducing emulsion into the settling chamber, with minimum disturbance to the contents of the chamber.

Historically this is one of the earliest designs. In its inherent simplicity it can be seen to be the forerunner of many of the other designs, including especially the Holley and Menning models.

**Standard Oil Development Co. Extractor (1949).** The horizontal mixer-settler shown in Fig. 23 has individual stirrers with the settling phases flowing
EDELEANU EXTRACTOR  FIG. 21

FIG. 22  
MU 979
HEAVY PHASE OUTLET PORT

PLANT (SECTION C-C)

MIXING CHAMBER PARTITIONS
AGITATOR
PORT WELLS

D

ANTI-SWIRL BAFFLES

LIGHT PHASE INLET PORT

LIGHT PHASE OUTLET PORT

HEAVY PHASE INLET PORT

SETTLER END VIEW (SECTION B-B)

ELEVATION (SECTION D-D)

MIXER END VIEW (SECTION A-A)

MIXING CHAMBER PARTITIONS
AGITATOR
PORT WELLS

D

ANTI-SWIRL Baffles

LIGHT PHASE INLET PORT

LIGHT PHASE OUTLET PORT

HEAVY PHASE INLET PORT

SETTLER END VIEW (SECTION B-B)

ELEVATION (SECTION D-D)

MIXER END VIEW (SECTION A-A)

STANDARD OIL DEVELOPMENT CO EXTRACTOR FIG. 23

MU 980
cocurrently. In this unit a positive control of the phase ratio in the mixing chamber is maintained by means of a weir on the discharge side of the mixing stage. A large unit of this type would contain a number of stages side by side but turned 180° with respect to one another in a horizontal plane. This permits a very compact box-like construction with the interstage flow accomplished by means of openings cut in the partitions between stages.

The overall flow through the unit is countercurrent, but in each stage the flow is cocurrent. Each stage consists of an antechamber, a mixing zone, and a settling section. The antechamber is a quiescent zone into which the light and heavy phases enter before passing through a horizontal spot into the mixing zone. This antechamber therefore prevents back-mixing by isolating the interstage ports from the direct action of the mixing element. It also permits a perfectly symmetrical design for the mixing zone, in that the phases after mixing pass to the settler through a horizontal slot which is identical with that at the entrance of the mixing zone. Both horizontal slots are protected from the direct action of the mixer by a shielding baffle.

At the end of the settler the separated phases resume their countercurrent flow pattern by going in opposite directions to the adjacent mixer antechambers. Flow through the unit is by gravity; the head required for flow is obtained by tilting the contactor, hence no interstage pumps or valves are required.

**Gordon Extractor (1939).** The horizontal mixer-settler shown in Fig. 24 has individual stirrers with the settling phases flowing cocurrently. The two phases are pumped into the mixing chamber by the mixing paddle. The ratio of phases in the mixing zone is dependent upon the flow rates. The mixed fluids are thrown through porous plate 33 into the large cylindrical or cubic settling chamber to break the emulsion. The light fluid overflows through 69, while the heavy fluid leaves through 65. In Fig. 25 a pressure equalizing system is shown, attached to the heavy fluid exit. An adjustable valve at f
GORDON EXTRACTOR FIG. 24
may be used to control the effective height of the overflow in order to maintain the correct ratio of water layer to solvent layer within the tank. Fig. 26 shows details of the stirring device.

Because the mixing chamber is contained completely within the settling chamber, this extractor is basically similar to any one stage of the Schönborn column.

CONCLUSIONS

The mixer-settlers designed by McKittrick, Scheibel, Holley, and Standard Development (Figs. 1, 8, 17 and 23) are considered the best of the twelve shown, from an overall point of view. For large-scale operation in which initial cost and simplicity of construction are considered, the McKittrick and Scheibel designs are the best. The Scheibel column is somewhat simpler, but has the important disadvantage that the ratio of phases is dependent upon the flow rate. The McKittrick column has a phase ratio in each mixing section which is independent of the flow rate; however, it is more complicated structurally than Scheibel's. Because of the need for individual motors and individual shaft seals, and because of the smaller driving force available for gravity flow, horizontal extractors are concluded to be inferior to vertical units. A major argument in favor of a horizontal mixer-settler is the use of horizontal space when vertical room is not available.

DESIGN OF CONTINUOUS COUNTERCURRENT HORIZONTAL MIXER-SETTLERS

A need for sturdily constructed continuous-flow liquid-liquid extraction equipment has arisen in connection with the pilot-scale demonstration of the chelate process for plutonium separation from uranium and fission products.
Two twenty-stage horizontal mixer-settlers were designed and constructed for this purpose. This particular type of equipment was chosen because of a lack of vertical space, and because of the favorable experience with it by the Argonne Laboratory and the Standard Oil Development Company.

It was decided that individual motors for stirring each stage would be preferable to a belt drive because of mechanical simplicity. The shafts of these stirring motors enter the stirring chamber through liquid-tight seals. The alternative, construction using open stirrer wells, would increase the liquid hold-up in each stage when the mixer-settler is run in a tilted position. In addition, seals reduce the hazard from overflow or spray of radioactive solutions.

Since strongly acid aqueous solutions and organic solvents are used together, it was necessary to have all construction materials resistant to this combination. The materials used were stainless steel with Teflon as a gasketing material and Kel-F for windows.

Because of beta and gamma radiation through the walls of the extraction equipment, it was surrounded by a two-inch lead wall with lead-glass windows. The cave is 20 ft. long, 4 ft. wide, and 8 ft. high, and is equipped with a ventilation system which changes the air inside once each minute. The inside walls and ceiling of the cave are painted with white strip coat which can be peeled off if it becomes radioactive. The floor of the cave is covered with rectangular stainless-steel pans, which drain possible spills or washings to a collecting drum, and can be replaced readily.

The storage tanks for the radioactive material used are located inside the cave. They are equipped with stirring paddles for dispersion of any
chemicals added to the feed solutions, and hold about 16 liters apiece. In Fig. 27 two of these tanks are shown inside the cave along with one mixer-settler bank. In Fig. 28 two more of the feed tanks are shown. All feed tanks for non-radioactive materials are located outside the cave. These tanks are elliptical in shape, are mounted vertically, and hold 32 liters each.

The pumps used for both radioactive and non-radioactive solutions are diaphragm pumps. They consist of a bellows filled with oil which is actuated by a rotating cam. When the bellows is compressed by the cam, the diaphragm is distended, this pushes fluid out of pump chamber by lifting the discharge check valve. When cam pressure is released from the bellows a spring returns it to its normal position, and the diaphragm returns to its original position. This closes the discharge check valve and opens the inlet check valve, which allows fluid to fill the pump chamber. When this pump is used for radioactive solutions, the pumping chamber is inside the cave, and the bellows pressure is transmitted through an oil-filled tube to the pumping diaphragm. The pumping rate is determined by timing the flow between calibration marks on the tank sight glass, while the tank discharge valve is temporarily closed. The pumping rate can be changed by a threaded cap enclosing the pumps bellows, which changes the distance through which the bellows returns after release by the cam and thus alters the stroke length.

STIRRING MOTORS AND SHAFT SEALS

The stirring motors are variable speed, 115 volts, D.C./40 H.P., 3350 RPM, continuous duty, type NSH 13, made by the Bodine Electric Co. The mounting for one bank of 20 of these motors is shown in Fig. 29.
The liquid-tight seals on the agitator shafts were built by the Crane Packing Co. Each seal proper consists of a fixed ceramic ring and a rotating carbon ring. The faces of these two are held together by spring tension, and are in rotating contact. The ceramic ring is held in the shaft housing, while the carbon ring is secured to the rotating shaft. An exploded view of these assemblies may be seen in Fig. 30. Fig. 31 shows the carbon ring and associated parts which are attached to the stirring shaft. The carbon ring is supported by a Teflon ring, which fits snugly over the shaft and is held in place by a spring which fits into the cover for the assembly. The cover is held on the shaft by a set screw. The carbon ring is held by the cover by small steel pieces peened over the ridges.

The ceramic ring fits in a well at the bottom of the shaft housing. The Teflon ring fits snugly over the outside of the ceramic ring and is held in place and pressed tightly around the ceramic ring by a brass bushing. The pressure is supplied by the threaded steel ring which screws down on the brass bushing.

One difficulty with this type of seal is the relatively large amount of heat liberated by the friction of the two faces. Therefore it is necessary to have solution close enough to the faces to lubricate them. However, these continuously sealed seals have so far operated/sixty hours without any seizure or leakage, and the present design is believed to be quite durable.

AGITATORS AND FLOW DESIGN

In order to design a proper baffling system, the flow characteristics of the mixing chamber were studied in a prototype mixer-settler having a single stirred stage. A diagram of the prototype is shown in Fig. 32. The prototype
was built with space corresponding to three stages of the larger unit, but a stirrer was used only in the center stage. Large windows were built in both of the end stages in order to observe the flow pattern and the phase ratio in each stage. The heavy phase discharging from the lowest chamber was controlled electrically by means of the difference in conductivity of benzene and aqueous nitric acid.

It was found that a phenomenon which was termed "pullover" occurred if an on-off type of liquid level control was used to regulate the discharge of the heavy phase from the bottom of the prototype. When the air operated valve controlling the flow of heavy phase opened, a rapid surge of the heavy phase through the mixer-settler occurred. It is possible that this could cause considerable back mixing of the light phase. The difficulty was remedied by an open by-pass or by a proportional rate control valve.

Back mixing, which was particularly difficult to eliminate, is the transfer of material from the mixing chamber of one stage to the adjacent settling chamber which normally feeds into it. Various shapes of baffles were tried as shielding for the entry ports of the light and heavy phases to the mixing chamber. It was found that back mixing occurred in all types tested except those which formed an antechamber for the entering streams. The walls of the antechamber must extend the entire height of the mixing chamber and thus completely shield the entry ports from the direct action of the mixing device.

The design of the stirring paddle decided upon was a flat rectangular blade (Fig. 33) of 5/8 inch width. This proved quite satisfactory over the whole range of stirring and with the antechamber design back mixing of the heavy phase was
was eliminated completely. The flow rate of the light phase was varied from zero to 100 ml/min while the flow rate of heavy phase was varied from zero to 80 ml/min. No back mixing was observed in this series of runs with a stirring speed of 3500 RPM. The tilt of the prototype was varied from one in three to one in eleven with no noticeable change in operating characteristics. With a 7/8 inch paddle at speeds of around 3000 RPM the whole unit became filled with emulsion so that nothing was visible through the windows.

Fig. 34 shows the baffle design used in the large mixer-settlers. This design seems to cause no pumping action and no blocking of flow, in agreement with the operation of the prototype. Fig. 35 shows an unsymmetrical design which caused pumping if the stirring paddle was moving clockwise, and blocking of the flow if the stirring was counter-clockwise.

Air vents are provided in all baffles so that air will flow successively from one chamber to the one next above it and will finally escape through the vent at the top of the column. These air vents may be seen in Fig. 36 as triangular holes at the top corner of the baffles which separate the mixing and settling chambers. The central slot in the baffle nearest the mixing chamber is for the flow of emulsified liquid. After passing through this slot the emulsified liquid hits the second baffle and tends to separate into phases which then run into the settling chamber through the rectangular slots at the top and bottom of the second baffle. After entering the settling chamber, the light phase flows to the mixing chamber above it and the heavy phase to the mixing chamber below it.

Figs. 37, 38 and 39 are views of the large mixer-settler. Fig. 37 is
a top view from an angle while Fig. 38 is a side view of the unit. Fig. 39 is a top view with the Teflon gasket in place. This large unit holds six liters. It is forty-four inches long, four inches wide and two and one-half inches deep. As mentioned earlier, it has twenty stages, each consisting of an antechamber, a mixing chamber, and a settling chamber. It was designed for a total throughput of around 30 ml per minute. The flow between stages is by gravity operating through the density difference of the two phases. A weir in each settling section controls the level of the heavy phase. The ratio of phases may be altered somewhat by changing the tilt of the mixer-settler.

Figs. 40, 41 and 42 show three views of a bank of stirrers for one of the mixer-settlers. This bank fits over the Teflon gasket shown in Fig. 39.

ACCESSORY EQUIPMENT

The only control apparatus needed for the large mixer settlers consists of a means of controlling the take-off of the heavy phase from the bottom. This allows the light phase to be forced from the top of the column as it is pumped in at the bottom end. The present control system involves an electronic circuit which actuates a solenoid valve controlling the air to an air-operated valve in the discharge line at the lower end of the mixer settler. The circuit distinguishes between the phases by the difference in their conductivity. The circuit is actuated by a platinum wire probe which is enclosed in glass tubing except for a tip about 1/4 inch long which is in the solution in the mixer-settler. This probe is sealed into the unit by a compression fitting of Teflon against the glass tube. The main body of the mixer-settler comprises the other half of the circuit. Since the heavy
phase is the more highly conducting phase, when the water level reaches the
probe tip the solenoid valve is actuated closing the air supply to the
air operated valve in the heavy liquid discharge line at the bottom of the
mixer-settler. The air in the valve then bleeds slowly to the atmosphere
through a needle valve permitting the stem of the valve to rise slowly and
the flow of the heavy liquid to increase until the liquid level drops below
the probe tip. The solenoid valve is then opened allowing air to bleed
in through another needle valve and slowly close the air-operated valve.
The air-operated valve therefore fluctuates about a mean position which is
automatically set by the flow.

In a unit of this small size it has been observed that the most satis-
factory position for the actuating probe, for the heavy phase, is in the top
stage. This tends to give an inherently stable flow pattern since the
heavy phase entering the top stage is forced to flow through the column.
If the probe is placed in the bottom stage, a large amount of water is
emptied from the unit when the stirrers are turned on. This amount is so
great that several stages are essentially emptied of water.

The sampling tubing for the mixer is stainless steel of 0.025 inch I.D.
Two tubes are used for each stage to be sampled, one going to the bottom
for the heavy phase and the other ending near the top for the light phase.
This tubing is sealed in by individual compression fittings of Teflon. The
discharge ends of these tubes are sharpened and are attached to a metal
frame with each point supported about an inch from the end. Serum bottles
with vacuum-tight stoppers are evacuated, and then the sharpened point of
the sampling tube is thrust through the stopper. The vacuum in the bottle
then pulls the fluid to be sampled into the bottle. When the bottle is pulled
off, the rubber stopper wipes the tubing clean and no dripping occurs. The bottles are handled by tongs through the lead wall, when radioactive material is used; they are passed in and out of the cave in a lead block, which sits on a pivoted arm with a lead shield on each end. The lead shields are arranged to close the hole in the wall whether the pivoted arm is in the cave or out of the cave.

The temperature profile of each mixer-settler is determined by a series of iron constantan thermocouples. The wells for these thermocouples are shown in Figs. 43 and 44. They extend down into the settling chambers of ten stages in each mixer-settler. The thermocouple wires are soldered to the bottom of the well for better heat transfer. The well is filled with oil and sealed with cement to enclose the whole unit. This should prevent corrosion of the wires at the contact points. Shown in Fig. 45 is the thermocouple deck with two ten-way switches, a potentiometer, and a light-beam galvanometer. The temperature is determined from a calibration chart after balancing the galvanometer and reading the microvoltage.
FIG. 45
BIBLIOGRAPHY


