

IS - 309

A SMALL SCALE COUNTERCURRENT

LIQUID-LIQUID EXTRACTOR

by

Harley A. Wilhelm

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by

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ABSTRACT

Details of design and operation are given for a laboratory-size, 20-stage, multiple-contact, "unlimited-feed", countercurrent-flow, liquid-liquid extractor. The apparatus consists mainly of glass parts that are joined by polyethylene tubing and are mounted in a steel cradle that can rotate on its horizontal axis. The reservoirs for feed liquids are an integral part of the assembly, and proper rotation of the assembly causes the flow of liquids to, through and from the extractor. Testing and developing of, and small scale production by, extraction systems are conveniently carried out in this extractor. Stagewise and product samples can be readily obtained for study of the extraction behavior of the components of a liquid-liquid system.

Countercurrent liquid-liquid extraction is a well established operation for separation and purification of compounds on laboratory, pilot plant and large production scales of processing. Each new extraction problem, however, requires a study to determine workable conditions for effecting the desired separation or purification. Preliminary studies of single stage distribution behaviors of the components of liquid-liquid systems can give data that are basic to the development of extraction processes. Later stages in the development of a practical liquid-liquid extraction process, however, generally require that tests on the variables in the conditions of operation also be made with manipulations that simulate those of the multi-stage equipment to be employed in processing. Proper equipment for these tests may not only facilitate the derivation of conditions for laboratory or large scale processing but may also make it possible to readily obtain data that are basic to an understanding of the extraction mechanism. This article presents details on the construction and operation of small scale, 20-stage, multiple-contact, countercurrent-flow extractor in which such tests can be conveniently made.

Separation and purification of metals through liquid-liquid extraction of their compounds have received considerable attention and study in recent years. Many investigations have been directed toward

extraction systems with water and an organic liquid as the relatively immiscible solvents between which two or more metals as compounds in a mixture may differentially distribute. A number of such metal separation systems have been developed in this laboratory employing equipment of the general type presented here. The extractor described in detail in this article, however, has a number of improvements over earlier designs. Although the work here on liquid-liquid extraction has been essentially limited to studies of metal separation by such aqueous-organic systems, it appears that applications to other problems in the broader field of liquid-liquid, or solvent, extraction are entirely within the possibilities of the apparatus described in this report.

DESIGN AND OPERATION

The extractor presented here consists essentially of an orderly assembly of feeder systems, mixer-settler chambers, interstage transfer bulbs and connection tubes all made of glass with joints of polyethylene or other practically inert flexible tubing. The connections between the parts of this assembly may be so arranged as to give countercurrent flow of the two liquid phases. This extractor assembly (see Fig. 1) is mounted in an open structure steel cradle consisting essentially of two steel end plates connected by four lengths of angle iron. The cradle is supported in a horizontal position by bearings in

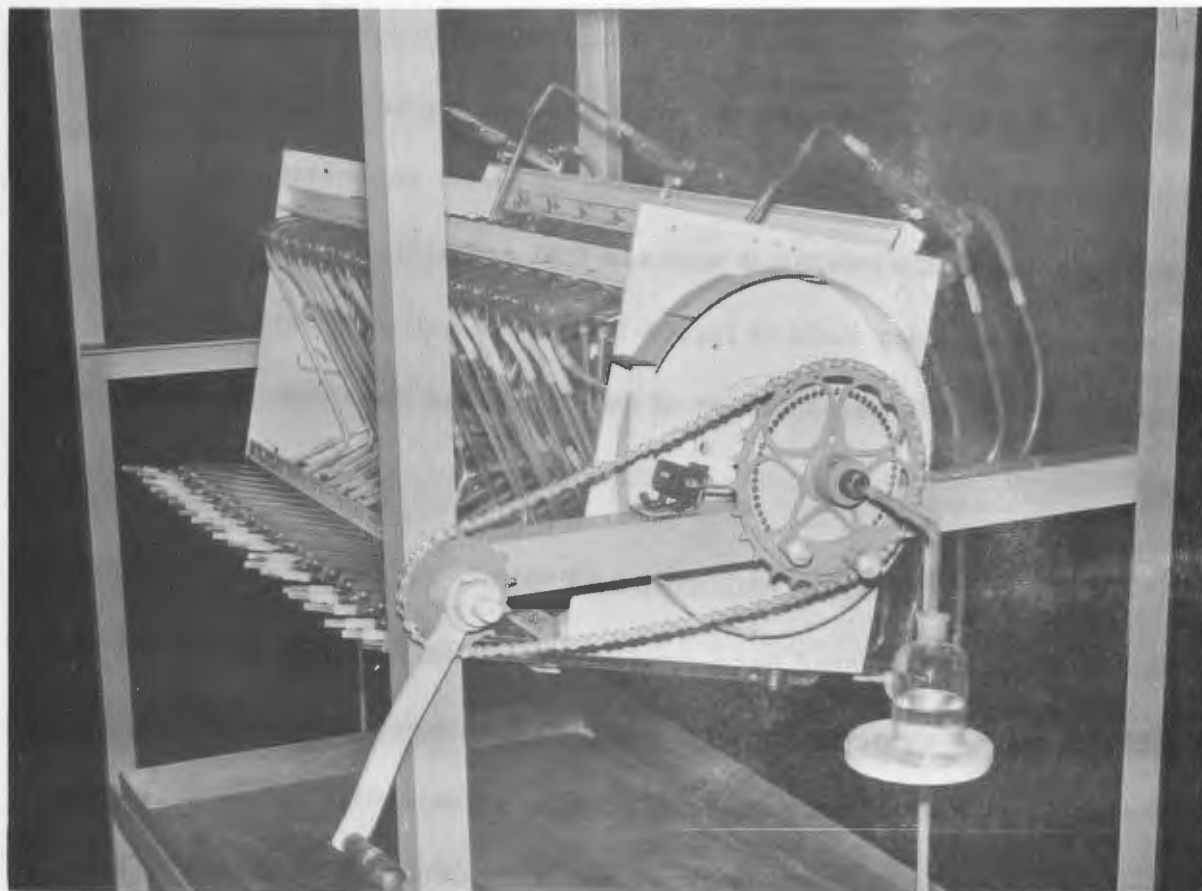


Figure 1. Photograph of extractor assembly.

a steel framework. The feeding of liquids to the extractor, the multi-stage extraction and the delivery of product solutions from the extractor are performed through rotation of the cradle and its extractor assembly on the horizontal axis. Since these operations take place repeatedly as rotations of the cradle are continued, the extractor may be referred to as of the "unlimited feed" type.

Reservoir tanks containing the liquids to be fed to the extractor at the desired stages are mounted along the axis of rotation of the cradle and are parts of the assembly. The two end shafts of the extractor cradle are hollow and they can, therefore, accommodate coaxial glass tubes for delivery of product solutions from the extractor and through glass sleeve joints to stationary receivers. A sprocket wheel fixed on one of these end shafts enables rotary motion to be imparted to the cradle by means of a hand-crank operated chain drive. Feeding of liquids to the extractor, mixing and settling and countercurrent flow of the phases in the extractor and delivery of the product solutions from the extractor are, then, effected through proper manipulation of the hand crank.

The performance of this laboratory extractor simulates that of a continuous countercurrent-flow vertical extraction column.

Extractors that have some physical resemblances to the one presented here have been described in articles by Craig,⁽¹⁾ by Lathe and Ruthven,⁽²⁾

by von Metzsch,⁽³⁾ by Verzele and Alderweireldt⁽⁴⁾ and by Wilhelm and Foos.^(5, 6) The apparatus developed by Craig and that developed by Lathe and Ruthven were, however, designed to move only one of the liquid phases in a stagewise manner; so these extractors, referred to as "limited feed" type, operate on essentially single batches rather than on continued additions of feed. All of the extractors considered here have a common feature in that they depend for their operation on repetition of a sequence of mixing, settling and separate flow of the phases in each stage. Simultaneous operation of a number of stages through this sequence of events gives, through proper connections, effectively progressive stagewise flow.

The separation of the two phases in a stage in most of the above extractors is accomplished after settling by drawing off the less dense liquid through a tube opening that is positioned at or near the liquid-liquid interface. Since the volume of the more dense liquid essentially determines the height (or position) of this interface after the settling, it consequently determines the optimum position of the opening through which the less dense phase is removed. In extraction systems set up to operate under conditions designed for production, significant concentration and volume changes often occur in each of the portions of the immiscible liquid phases as these portions make the stagewise contacts on countercurrent flow through the extractor. As

an extraction process approaches steady-state operation, any such changes in volumes of the portions of the phases in the various stages will develop somewhat gradually. Should the volume change for the more dense phase become such that the position of the interface is moved significantly, then the tube opening for carrying away the less dense phase in extractors of the type being considered may need repositioning. The extractor described in detail here has a design that permits adjustment of the positions of these openings. Consequently this extractor has broader adaptability in liquid-liquid extraction studies and operations than those described by Craig, by von Metzsch and by Verzele and Alderweireldt which do not provide for such adjustments. Also the extractor of Lathe and Ruthven, although quite different in design, lacks the broader adaptability.

Two main features of this extractor that differ from those of one extractor presented earlier from this laboratory⁽⁶⁾ are:

- 1.) Data can readily be obtained from all of the stages and not from just the alternate stages.
- 2.) The feeders and their supply tanks are conveniently mounted in the rotating assembly as an integral part of the flow system of the extractor. This last feature appears also to be a decided improvement over feeder systems used in any of the previously reported laboratory extractors that operate on somewhat related principles.

The present design has a number of improvements over that of a similar extractor reported earlier.⁽⁵⁾ The extractor is, in the main, an orderly side-by-side arrangement of 20 units; each unit corresponding to a single stage such as that sketched in Fig. 2. The operation of this extractor may be described by reference to this sketch. Assume that portions of the two immiscible liquid phases, differing in density, are in the mixer-settler chamber M. The two liquids are mixed by causing them to flow rapidly from end to end in this chamber. An oscillatory motion of this unit about its axis of rotation, between about $+20^\circ$ and -20° from the horizontal position represented in Fig. 2, will generally give proper agitation. After adequate mixing, chamber M is then held at a nearly horizontal position to permit settling of the liquid phases. The unit is then turned slowly clockwise through about 90° and as the mixer-settler chamber goes to a vertical position the light liquid flows out through tube L toward the interstage transfer bulb T. It is to be noted here that the position of the extension of tube L is adjustable within chamber M so that the separation of the liquids at the liquid-liquid interface can be properly accommodated. Further rotation of the unit in the clockwise direction to the 180° position permits the heavier liquid phase to flow from M through tube H to the interstage transfer bulb T'.

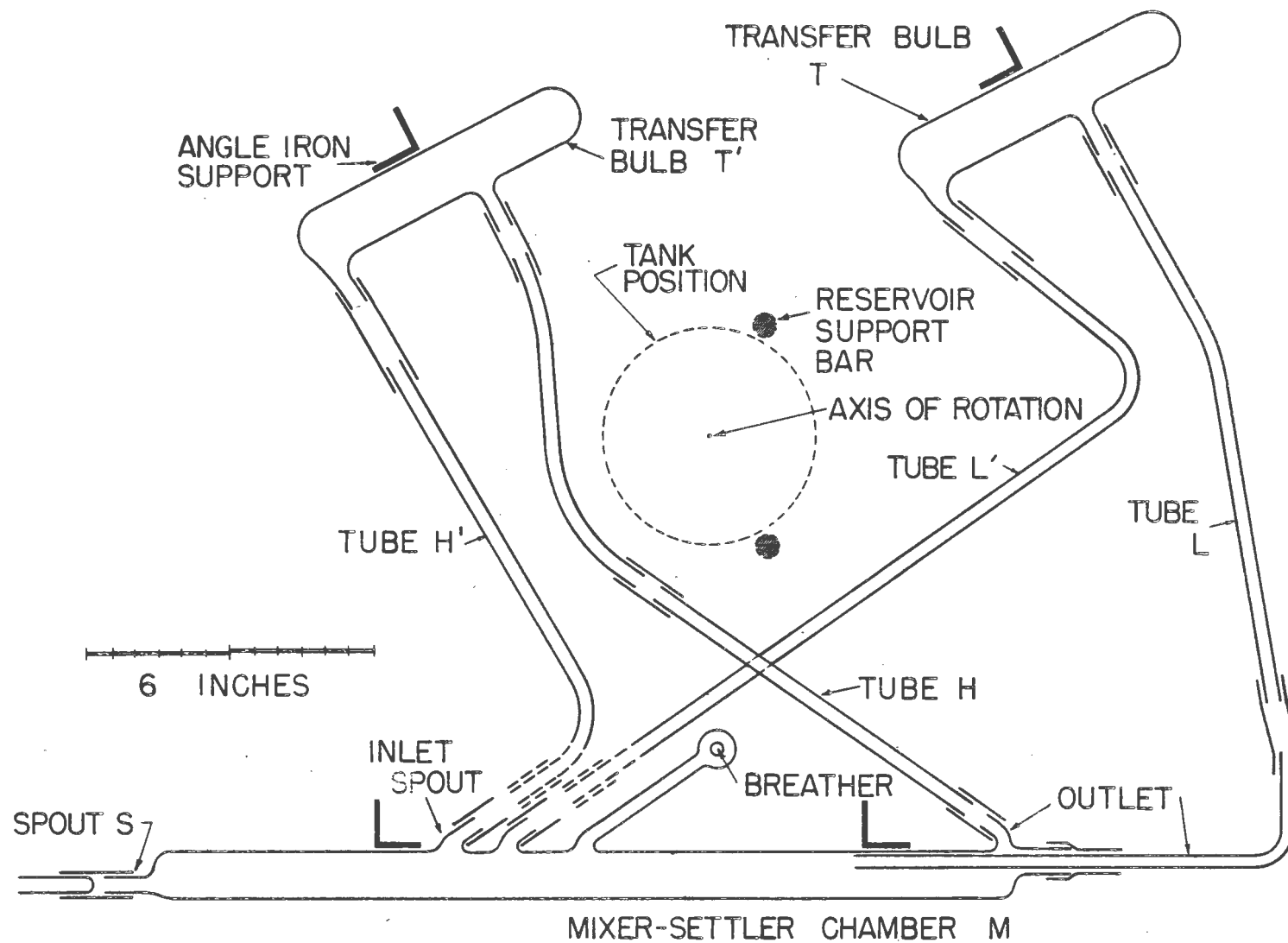


Figure 2. A sketch representing the arrangement of parts for a single stage.

At this point, 180° clockwise from the starting position, the portions of the two phases are separated and are in their respective inter-stage transfer bulbs.

The dashed sections of tube H' and tube L' in Fig. 2 indicate that in the complete assembly these tubes are not actually connected to inlet spouts on the mixer-settler chamber shown but to spouts of similar chambers on either side of this chamber. Further clockwise rotation of the unit, then, allows the separated liquids to flow from the transfer bulbs to mixer-settler chambers M+1 and M-1 that are juxtaposed to chamber M in the total assembly. This completes one cycle of operation for a single stage. For the entire assembly, similar patterns of flow are simultaneously effected for all of the stages. On each successive cycle during continued operation, then, the mixing-settling-flow sequences for all of the 20 stages take place concurrently. The net result is countercurrent flow with the more dense and less dense liquid phases being delivered from stages at opposite ends of the extractor to stationary receivers.

The feeding of liquids to the extractor is essentially automatic, and an explanation of the feeder system may be facilitated by reference to Fig. 3. Here a mixer-settler chamber is shown by dashed lines in order to clarify the arrangement of the feeder unit with respect to the extractor system. The axis of rotation, the reservoir support bars and the reservoir tank are also in the same positions that were

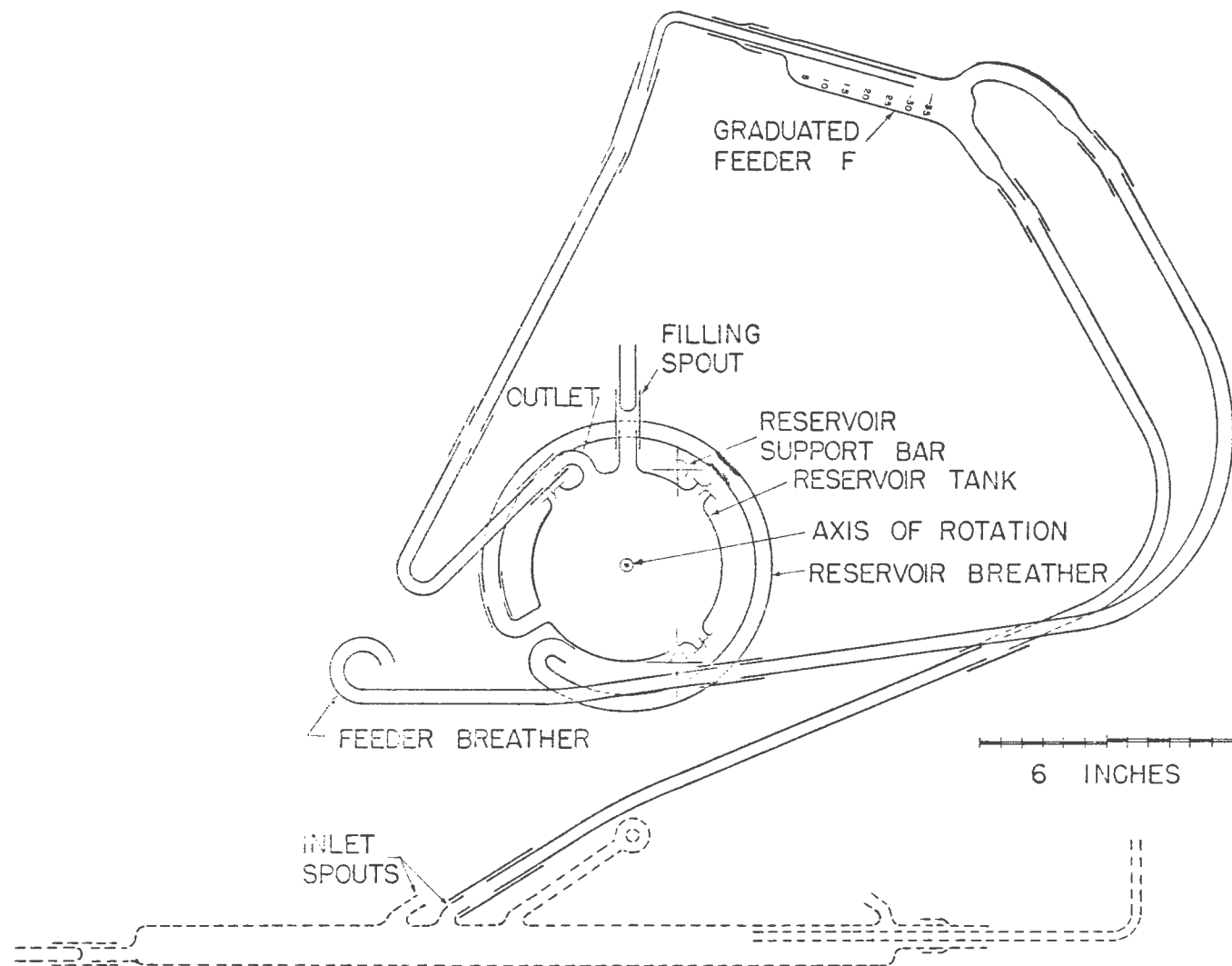


Figure 3. Sketch of a feeder system.

indicated in Fig 2. Liquid from the cylindrical reservoir tank of Fig. 3 flows into and floods the graduated feeder F at a point near or shortly beyond the 180° of clockwise rotation in the operation cycle described above. Continuation of this clockwise rotation causes excess liquid in the tubes connected to the feeder to flow back to the reservoir. When this rotation is such that the liquid level is within the upper enlarged tube section of feeder F, a short quick rotary movement is imparted to the assembly to bring the graduated section of the feeder to a vertical position. This position is held until the excess liquid has flowed from the graduated section of the feeder. By this manipulation a measured volume of liquid is retained for delivery to the extractor on completion of the cycle. It is to be noted that the position of the return flow tube in the graduated feeder is adjustable; it is the position of this tube that determines the volume of liquid delivered by the feeder.

For this extractor which simulates a vertical extraction column with countercurrent flow, three such feeder systems are generally adequate. One feeder may introduce organic solvent at one end of the extractor, one may introduce aqueous scrub at the other end and another may introduce feed solution at some intermediate stage. So on each cycle of operation of this extractor, portions of liquids are

taken from three reservoirs and delivered simultaneously to the extractor at the selected stages. The net result of the over-all operation of repeated cycling is then continued output of product liquids through countercurrent flow maintained by continued feed from the reservoirs.

In order to insure proper flow of the liquid phases during the cycle it is essential that adequate breathers, or air vents, be provided in the equipment. The breather for the mixer-settler chamber is shown in Fig. 2 as a bulb approximately 20 millimeters in diameter with the breather hole in one side. The breather for the graduated feeder is shown in Fig. 3 as merely a long curved tube that is bent sharply at the open end. The breather system for the reservoir tank is a combination of four such tubes, each of which almost completely encircles the tank. A constriction at about the mid point of each of these four breather tubes helps to restrict the rate of flow of liquid in this breather system. Although the connections of these four circular breathers to the tank are positioned as indicated in Fig. 3 at about 90° intervals around the cylindrical tank axis, only one of these breathers is represented in this figure. Figure 4 is a more detailed sketch of the reservoir with the tank, breathers, filling spout and outlet shown in perspective.

In the operation of this extractor the flow pattern is such that each portion of liquid introduced at a stage progresses

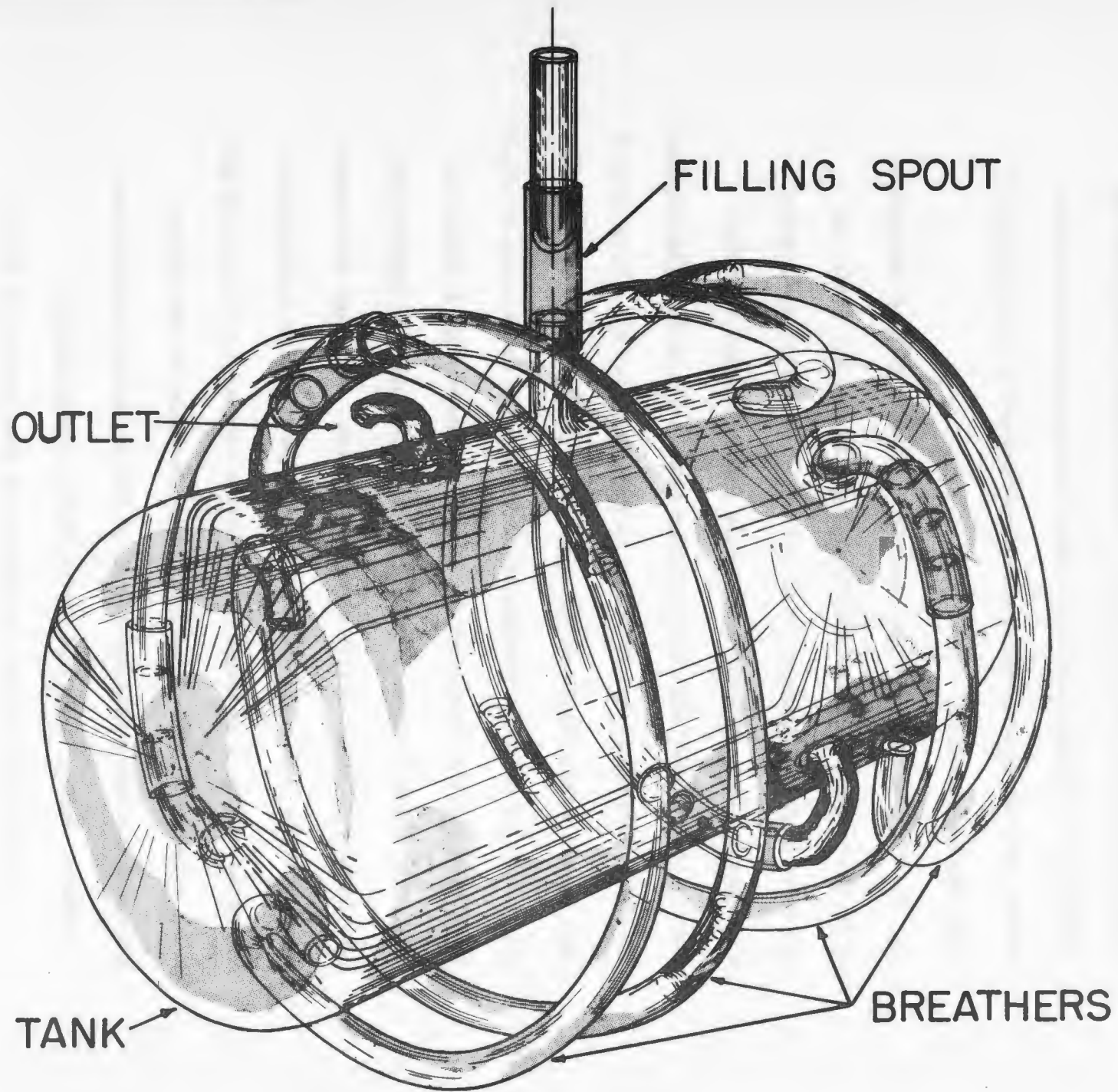


Figure 4. Sketch showing, in perspective, some details of a feeder system reservoir.

in essentially one direction by one stage only on each successive cycle until it is discharged from an end stage. It is obvious that since the two phases move countercurrently on each cycle of operation there are portions of the light phase and heavy phase that do not contact one another. Alternate portions of the light phase will contact only alternate portions of the heavy phase. However, each portion of either phase contacts a portion of the other phase in each of the stages on its way to discharge. In effect then, this extractor is equivalent to two extractors of a type described earlier⁽⁶⁾ working in parallel but offset by one stage. The net result of a stagewise analysis then would correspond to an analysis of a complete series of stages in a countercurrent-flow liquid-liquid extraction column.

The capacities and dimensions of the parts of this extractor were chosen for the particular laboratory scale of operation desired. Each mixer-settler chamber will conveniently accommodate a volume of about 60 milliliters of the combined liquid phases per cycle through the mixing, settling and separation operations. The same general design could readily be employed in construction of an extractor of much greater capacity. Smaller scale equipment of this design could be constructed; however, when the sizes of parts are so small that the proper flow of liquids is restricted through surface tension effects the operation could be unsatisfactory.

The main body of each of the mixer-settler chambers (refer to Fig. 2) for the extractor being described is 18 inches in length and was made from 25-millimeter diameter glass tubing. The transfer bulbs are 6.5 inches in length and were made from 30-millimeter tubing. All spouts and connecting glass tubing indicated in Fig. 2 are 8 millimeter size except for a 13-millimeter spout and its 7-millimeter insert tube that are parts of the adjustable connection at the delivery end of the mixer-settler chamber.

The reservoir tank in the feeder system of Fig. 3 is a cylinder about 4.5 inches in diameter and 9.5 inches in length. It has an operating capacity of about two liters of liquid. The filling spout and the breathers to this tank were made from 10-millimeter tubing. The main body of the graduated feeder (graduated in milliliters) was made from 25-millimeter glass and is about 5.5 inches in length; all spouts sealed to the body of this feeder are of 10-millimeter glass tubing.

MISCELLANEOUS DETAILS

The cradle that supports the glass extractor assembly (see Fig. 1) consists essentially of two 14-inch by 17-inch by one-quarter-inch thick steel end plates connected by four 43-inch long sections of one-inch angle iron welded in position. The relative positions and tilts of these four sections of angle iron are indicated in Fig. 2. Clamps are used

in bolting the glass parts to the angle-iron supports; plastic separators prevent contact of the glass and metal. Another section of one-inch angle iron that is bolted to the end plates is used to support the graduated feeders as shown in Fig. 1. The two reservoir support bars indicated in Fig. 3 are removable one-half-inch diameter steel rods that are parallel to the axis of rotation and are supported by the end plates. The tanks are strapped to these two bars. The hollow end shafts are one-half-inch inside diameter and were made from one-inch diameter steel rod. The end plates of the cradle have mounting hubs for the hollow end shafts that provide the means for support of the cradle in bearings (pillow blocks) mounted on members of the over-all steel framework of the extractor.

This steel framework (see Fig. 5) is constructed of two-inch angle iron and is 53 inches long by 34 inches wide and, as mounted on four four-inch casters, stands 60 inches high. The extractor assembly is covered by a sheet of three-quarter-inch thick plywood that is mounted on top of this framework. For convenience in charging the reservoir tanks, holes about one inch in diameter are drilled through this plywood at points above the filling spouts of the reservoir tanks. A stainless steel pan for catching liquids from washing or from any leaks that may develop is supported in the framework and below the cradle assembly.

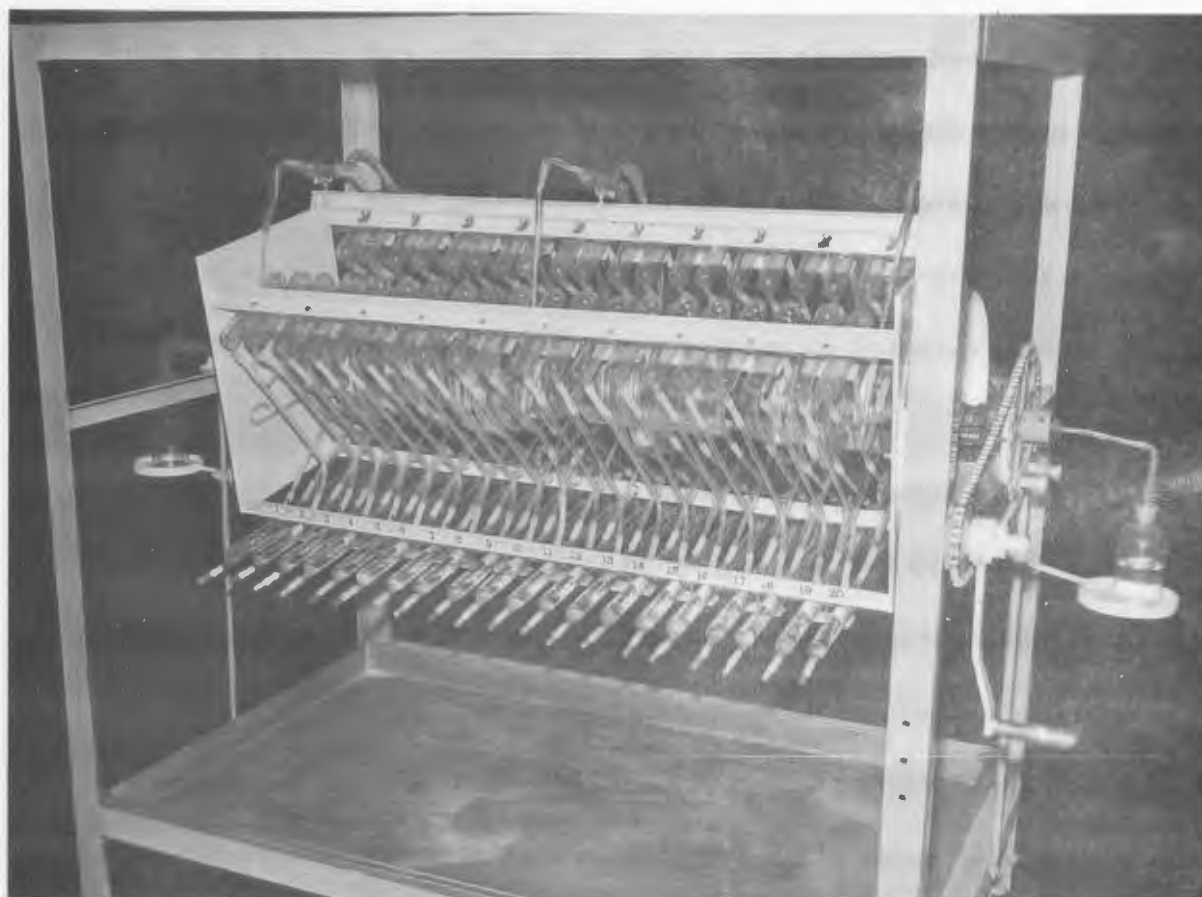


Figure 5. Photograph showing steel framework of extractor.

A number of other miscellaneous details in the design and construction of the particular extractor contribute to facilitating its operation. The small sprocket to which the hand crank is attached (see Fig. 1) is specially made with half as many sprockets as the larger wheel that is secured to one end shaft of the cradle. On repeated cycling during an extraction operation, mixing and settling and flow of liquids recur then at certain positions of the hand crank. The position of the small sprocket wheel axle can be adjusted on the steel framework in order to properly tighten the chain of the drive.

A circular plate that is secured stationary to the bearing support and mounted back of the larger sprocket wheel has a number of holes drilled on a circle near its circumference. Two pins that are mounted on this sprocket wheel can be inserted into these holes to lock the cradle in position. Since these two pins are separated by a distance equal to that for nine and one-half holes on the circular plate, the number of possible lock positions are double the number of holes in the plate. Another small pin on the back side of the large sprocket wheel and near its chain track activates the rotation, or cycle, counter that is secured to the framework.

In moving the extractor through its 360° cycle there are positions of oscillation, hold, slow movement, intermediate movement and fast movement. The operator can observe the equipment and follow

the proper sequence; however, a two-inch-wide steel band that is bolted to the end plate of the cradle near the chain drive is color coded for proper manipulation. A pointer mounted on the framework extends to this color coded band; this combination can help keep the operator informed as to the particular manipulation being approached.

If the two inlet spouts on each mixer-settler chamber were in the same plane as indicated by the sketch in Fig. 2, a congestion of tubes, connections and breathers would be experienced on each mixer-settler near these spouts. In actual construction of a mixer-settler chamber, however, these spouts are attached so that they are spread by about 20° with respect to the circular cross-sections of this chamber. This arrangement not only avoids the congestion but points these inlet spouts more nearly in the direction of the tubes leading from their respective transfer bulbs (see Fig. 5).

Polyethylene tubing connections generally can be forced over slightly larger glass tubing more readily if they are near the temperature of boiling water when installed. Certain connections, especially those between two greatly different sizes of glass such as at the outlet end of the mixer-settler chamber of Fig. 2, may require pre-shaping. One method for forming such preshaped connectors is to select polyethylene tubing of the proper size to fit the smaller of the two sizes of glass, heat a short section at one end of this polyethylene

tubing in boiling water and then force it over a lubricated steel form of about the same size as the larger glass. On cooling, the polyethylene retains the size of the steel form. Cutting the polyethylene connector to the proper length, inserting in place and reheating with a steam jet can give the proper fit. A small amount of a lubricant such as a silicone grease may be needed between the glass and polyethylene for the adjustable connections.

FILLING, STARTING AND ADJUSTING THE EXTRACTOR

Starting an extraction with this equipment, as well as with any other extractor, requires that some workable plan be followed. One systematic procedure that may be used with the extractor described here will be given in some detail. Assume that the plan of the extraction is to employ an organic (less dense) liquid that is to enter at one end of the extractor, an aqueous scrub that is to be introduced at the stage at the opposite end of the extractor and an aqueous feed, that is a solution of two materials to be separated, is to be fed to the extractor at some intermediate stage. Inter-stage connections are such that the aqueous phase and organic phase move stagewise in a countercurrent fashion through the extractor.

The adjustable tubes in all of the mixer-settler chambers are set so that their open ends (see Fig. 2) will be well above the estimated liquid-liquid interface levels when these chambers arrive at the

vertical position where the separations of phases occur on the first cycle. For this preliminary adjustment the open ends of these tubes could be set to be above the calculated levels for the combined phases in their respective stages but they must not be set so as to be below the liquid-liquid interfaces.

The graduated feeders are next adjusted to deliver the desired volumes of the three liquids to the extractor. Connections are made between the three feeders and the selected stages. The three reservoir tanks are then charged with their specified liquids. To do this, the cradle is locked so as to hold the feeder units in a position near that represented by Fig. 3. The extended stems of three funnels are passed through the holes in the plywood cover of the extractor and inserted into the filling spouts of the reservoir tanks. The liquids are then introduced through funnels that are above the plywood cover. No tank should be filled more than about 80 percent of its total volume, that is, to a level of not greater than three-fourths on the vertical diameter of the tank cylinder. Filling to much above this level could cause interference in the tank breathing and liquid flow. The funnels are removed and the filling spouts to the tanks are then plugged.

The extractor cradle assembly is next rotated clockwise about 45° where the position of the mixer-settler chambers is still below the axis of rotation but at an angle of negative 45° with respect

to the horizontal as viewed from the sprocket end. The cradle is then locked with the chambers in this position for the preliminary charging.

The plugs of the spouts (see spout S, Fig. 2) are removed and volumes of aqueous scrub equivalent to the total volume of aqueous phase to be treated in each of the stages per cycle of operation are placed in the respective chambers through their spouts. Those stages that will operate with both aqueous scrub and aqueous feed will receive at this time a volume of aqueous scrub equivalent to the volume of both aqueous liquids. Next a volume of organic, roughly equal to the planned volume to enter the extractor on each cycle, is introduced into each of the mixer-settler chambers. The plugs to the spouts are inserted and the cradle rotated counterclockwise to the position corresponding to that of Fig. 2. Receiving flasks are then placed at both ends of the cradle, as indicated by Fig. 5, for deliveries from the extractor. The extractor is now properly charged and ready for starting through the first cycle of operation.

The mixing of the portions of the two phases in all of the 20 stages is accomplished simultaneously by the oscillatory motion described earlier. Twenty to thirty seconds of good mixing of the phases by this repeated motion is adequate time to reach essentially

equilibrium distribution conditions for many systems. At the end of a mixing operation, any liquid that may be in the outlet tubes of the mixer-settler chambers should be allowed to drain back; then these chambers are moved slowly to near the horizontal position, indicated in Fig. 2, for settling.

When the phase settling is essentially complete in all stages, the cradle is rotated clockwise (very slowly at first to minimize remixing) for about 90° to attain the position where the mixer-settler chambers stand vertically. The adjustable outlet tube in each of the 20 chambers is then positioned where its open end stands about one-half-inch above the liquid-liquid interface in the chamber. In the next 90° of clockwise rotation that continues in this cycle, a considerable amount of the organic phase in each stage is, then, allowed to flow with the aqueous phase. The filling of the graduated feeders takes place during the third 90° of clockwise rotation and the procedure as described above for this operation is followed. Another 90° of this rotation completes the first cycle.

At the proper point in subsequent cycles the position of the tube opening of the adjustable outlet in each mixer-settler is inspected and corrected if needed in order to insure that none of the heavier, aqueous phase will flow with the organic phase to the next stage. As steady-state conditions are approached, the position of the open ends

of these outlets are moved nearer to, but always maintained above, the liquid-liquid interfaces. This condition allows a small amount of the organic phase in each stage to go with the aqueous phase to the next stage. There, this amount of organic is combined with the regular flow to that stage and on the next cycle returns to the stage from which it came. In effect, then, a small amount of backmixing of the organic takes place. The amount of this backmixing at each stage is determined by the position of the adjustable outlet tube with respect to the liquid-liquid interface at the separation position in the cycle. As a final adjustment, a safe position for the open end of the outlet tube in this extractor is about three millimeters above the interface. The backmixing of the organic phase will then in general be relatively small, but it is essential in order to insure that backmixing of the aqueous phase, which could apparently throw subsequent stages out of adjustment, does not take place. It should be noted that there is no bypassing of stages by either phase in the operation of this extractor.

USE OF THE EXTRACTOR

After charging the extractor as described above, a number of cycles of operation will be required before any detectable effect of the feed material from the solution that enters at an intermediate stage can become evident in the discharge liquid at either end of the

extractor. After this point is reached for a discharge liquid, however, the amount of the product delivered per cycle at that end of the extractor may increase rather abruptly. As the cycles are continued the amounts of products as well as their purities will tend to level off at values determined by the conditions of the extraction. When the point of essentially no change in the products is reached on repeated operation, the system may be considered as operating at steady state.

Since the reservoir tank in each feeder system for the particular size of equipment described here will accommodate about two liters of liquid, one or two chargings of these tanks can in general supply liquids for enough cycles of operation to bring the extraction system to essentially steady state for the condition selected. However, if the major conditions of operation are shifted as the cycles progress, as might be expected in the development phase of an extraction process, then further charging of some tanks may be required before steady state is attained with the final conditions. Assuming equilibrium in the mixing, the major conditions of operation are constitution of the liquids, feeder-stage connections and relative flow rates, or flow ratios, of the liquids to the extractor. The behavior of the system in the extractor and analytical data on the product solutions are generally used as guides in adjusting the conditions of operation. Relatively

small amounts of materials, however, are usually adequate in the use of this extractor for testing and developing extraction conditions for a potential liquid-liquid system.

This small extractor is also convenient to use where the supply of material has limitation considerations or where only a small amount of product is desired. Continued operation, however, yields more product; and extractors of this design but with larger capacity parts can be used to produce rather significant amount of products.⁽⁶⁾ The small extractor can usually supply adequate amounts of samples for making, by ordinary analytical procedures, a number of determinations on the constituents of interest in the liquids.

At the completion of an extraction test it is possible to get detailed stagewise data on the liquid-liquid system. Volumes of the phases and concentrations of the species in each phase in each of the stages constitute data that may be obtained and used in interpretation of the behavior of the constituents in the over-all extraction process. If stagewise data are to be obtained, it is essential that the stages be sampled only after thorough mixing of the liquids that are in the mixer-settler chambers at the end of the last complete cycle of operation.

An extractor of the design described here allows an experiment to be interrupted and then resumed at the operator's convenience without interfering with the proper functioning of the extractor. This is an especially desirable feature when delays for analyses as well as

periods of unattention by the operator are necessary during an extraction experiment that may intermittently extend for a number of days. Only in case the liquid-liquid system itself presents a time dependent condition of instability may the convenience of intermittent operation not be fully realized.

For experimental work with the extractor, hand operation has been quite satisfactory; however, if such an extractor were to be employed in somewhat routine productions, a programmed motorized drive that would move the extractor assembly through the sequence of the various cycle phases repeatedly might be of interest.

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