1989

Liquid-liquid extraction in a column using agitation /

Charles W. Stahl
Lehigh University

Liquid-Liquid Extraction In A Column Using Agitation

By

CHARLES W. STAHL

LIQUID-LIQUID EXTRACTION IN A COLUMN USING AGITATION

by

Charles W. Stahl

This Thesis is presented to the faculty of Lehigh University in partial fulfillment of the requirements for the degree of Master of Science in Chemical Engineering.

Charles W. Stahl

CHARLES W. STAHL

Lehigh University

Bethlehem, Pennsylvania

September 1947

TABLE OF CONTENTS

LIST OF FIGURES

INTRODUCTION

The separation of two liquids by using a third liquid is reported in the literature using several different types of apparatus to gain the separation. Data are given for liquid-liquid Extraction using a Wetter-Wall Column (1), (2), (3), a Packed Column (1), (4), (5), (6), (7), (8), (9), (10), a Perforated-Plate Column (6), (11), and a Spray Column (12). With the exception of a master's thesis (13) little, if anything, is to be found in the literature on continuous liquid-liquid extraction using agitation in a baffled column or perforated plate column.

The purpose of this investigation is to determine whether or not it is plausible to use agitation as a method of liquid-liquid extraction and to design, construct, and test different types of columns to find what problems must be overcome and which type of column is best suited for using agitation as a method of extraction.

The liquid system selected for use in this investigation is benzene-acetic acid-water. The benzene used was one degree water-white, saturated with water. The wash water used was Bethlehem City tap water. The acetic acid used was Glacial Acetic acid 99.5% acetic acid.

In order to determine which design to try out first a search of the literature was made for information and characteristics of liquid-liquid systems (14), (15). Elgin, in his work on a Spray Column (12), found that all the extraction seemed to take place

in the breaking up of the liquid into bubbles in the spray nozzle itself and for a very short distance in front of the nozzle. Treybal reported work on a perforated plate tower and his results (11) indicated that in that type of column the extraction took place in the breaking up of the liquid into bubbles and for a small distance beyond the perforated plate. Treybal found that by decreasing the distance between the plates the efficiency of the column was improved. From the above reported results it was decided to construct a column with a series of impellers to break up strenuously the liquid streams flowing counter current through the tower.

To compare the efficiency of the columns designed, the results are reported in equilibrium units. An equilibrium unit is the amount of acetic acid extracted from the benzene for a given proportion of water to benzene-acetic acid solution when the mixture is at equilibrium at room temperature.*

* See Appendix (Calculations).

SUMMARY

Three different columns were designed and tested using the liquid system water-acetic acid-benzene. The first column did not function, due to surface effects brought about by the small size of the equipment used. The second column operated satisfactorily and was operated at ten different speeds using various numbers and sizes of impellers. The data gathered using the second column showed that it was possible by using agitation in continuous liquid-liquid extraction to get results of more than one equilibrium unit.

The shape of the curve when equilibrium units are plotted against RPM is an S, regardless of the number or size of the impellers. (Tables 4-8). A maximum value of equilibrium units is approached which it is not possible to exceed, using a benzene-acetic acid-water system of a given concentration, regardless of the number of impellers, the size of the impellers or the speed of the impeller shaft, if the flow rate of the benzene-acetic acid solution is constant, and that of the water is constant. (Figures 10-14).

Keeping the rate of flow constant for the water and the benzene-acetic acid solution and the concentration of the acetic acid in the benzene constant and decreasing the number of impellers of a given size, the curve is flattened out, and becomes less steep, taking more speed to reach the maximum extraction possible. When the impeller size is decreased, all other conditions being the same, the curve is flattened still farther and more speed is needed to arrive at the maximum extraction possible.

Using the second column, it was possible in all cases, except when piano wire impellers of less than five on a shaft were used, to exceed one equilibrium unit of extraction. In the large column it was possible to exceed two equilibrium units of extraction.

SUGGESTIONS FOR FUTURE WORK

Further investigation is necessary on different liquid systems. The system benzene-benzoic acid-water should be investigated. In this system the water film is controlling, while in the system benzene-acetic acid-water the benzene film controls. Systems should be tried that have distribution coefficients near one.

Investigation is necessary on the effects of varying the flow rates through the column, both on the system investigated here and on other systems.

Information is needed of temperature effects on extraction. Whether or not the extraction rate is increased by higher or lower temperatures is not known. Temperature of the incoming liquids may have little or no effect on the degree of extraction achieved.

On each new system tried, information is needed on the number of impellers, the size of the impellers, and the speed necessary to give the best degree of extraction. Speeds of over 950 RPM are not possible on the column designed for this investigation. Higher speeds should be investigated.

By modifying the design of the column used in this investigation, it would be possible to operate the column under pressure. The effect of pressure on the degree of extraction should be investigated for each system used.

New column designs are possible and should be investigated. Each system may require a little different design for the column, especially as to the size of the holes necessary, if plates are used in the column. New designs should be tried and thoroughly tested to determine the best possible design for use with all types of liquid-liquid systems.

A summary of suggestions for future work is as follows:

(1) Investigate different liquid-liquid systems.

(2) Investigate the effects of varying the flow rates of the liquid components of the different systems through the column.

(3) Investigate temperature effects on the degree of extraction obtained.

(4) Investigate the number of impellers, the size of the impellers and the RPM necessary to obtain the highest degree of extraction for each system.

(5) Modify the column's design so it can be operated under pressure and investigate the effect of pressure on extraction.

(6) Redesign the columns and make the above investigations on the new columns.

COLUMN DESIGN

Three different columns were designed and constructed for this work.

First Column Design.

The first column designed (Figure 1) was made of glass tubing with all metal work of aluminum. The glass column itself was of standard pyrex glass. The interior of the column consisted of five aluminum plates forming four partitions of equal height. In these four partitions were the impellers. The aluminum plates were of two kinds (Figure 3). The top plate and bottom plate, beside having a hole in the center for the impeller shaft and a hole on the side for the baffle rod, also had a hole for the introduction of the liquid into the top and bottom compartments. The four impellers mounted rigidly on the center shaft fell in the center of each compartment and supplied the agitation to operate the column.

The baffle rod served two purposes. The first, was to hold the plates and the impeller in a rigid position, so that the entire inside of the column could be removed from the column as a unit. The second purpose was to act as a baffle. The baffle rod was long enough to fit into holes cut in the aluminum end plates to keep it rigid.

The aluminum end plates were machined with a circular packing groove to seal the glass tubing to the end plates without leaking.

In the top plate five holes were drilled, one for the entering water, one for the exit benzene, one for a liquid seal pipe for the impeller rod to pass through entering the column, and two for the tie rods. In the bottom end plate, four holes were drilled, one for the entering benzene-acid mixture, one for the water outlet, and two for the tie rods. Besides the through holes in the end plates in the top, a shallow hole was drilled for the baffle rod and a similar hole was drilled in the bottom end plate.

The liquid entered and left the column through glass tubing bent at right angles and sealed to the aluminum end plates with sealing wax. On the entering water side, in the top, the glass tubing extended through the end plate and through the first plate so that the water entering the column would enter directly into the first stirring compartment. The exit benzene line at the top came through the aluminum end plate and ended flush with the inside edge of the end plate. For the entering benzene-acetic acid mixture, the glass tubing extended through the column end plate and through the bottom plate the same as the incoming water in the top of the column. The water outlet was the same as the benzene outlet in the top.

The packing used to prevent the joint between the glass and the aluminum from breaking was graphite treated fiber. To pull these joints up tightly and to keep the column together, two brass tie rods were used, one 180° away from the other. The rods were threaded on both ends and take-up bolts were used. By tightening or loosening these bolts the pressure on the packing could be increased or decreased.

The water seal on the top of the column allowed the impeller shaft to pass into the column without the use of a mercury or fancy pressure type seal. The longer the seal pipe that the impeller shaft passed through, the more pressure the column would operate under before water was spilled from the top of the seal pipe.

With this design it was hoped that the benzene and acetic-acid solution entering at the bottom compartment would pass up through the hole in the center of the plate, which the impeller shaft passes through to each succeeding compartment, separate from the water and form a clear area of washed benzene at the top of the column, so that clear washed benzene would be forced from the column. Water, entering the top compartment, was expected to be mixed with the benzene to be washed and pass down the column through the same center hole in each plate through which the benzene was rising. From the bottom stirring compartment, the water was to separate from the benzene and form a clear layer of water and extracted acetic acid solution in the bottom of the column, and this clear water-acetic acid solution would be forced from the bottom of the column.

The column failed to operate due to large surface tension effect in the small size equipment used. The benzene-acetic acid mix would not rise through the small holes in the plates. The water, however, did as was expected and collected at the bottom of the column in a fairly clear condition. To remedy this situation a second column was designed.

Second Column Design.

The second column design (Figure 2) did away with the five plates used in the first design, kept the same end plates, but modified the seal and substituted baffles for the plates. To each of two aluminum rods, eight pieces of sheet aluminum were welded to act as baffles (Figure 4). These baffles were fastened in place by having the aluminum rods the baffles were connected to, a trifle longer than the glass tubing of the column. Then, by drilling one additional hole in each end plate 180° away from the hole used for the baffle rod hole in the first design, a tight fit kept the baffles in place when the tie rods were tightened up.

The type of seal between the end of the glass tubing and the aluminum end plates was changed to make a more liquid-proof connection. A larger, deeper, doughnut-shaped groove was machined in each end plate. A neoprene gasket was cut exactly to fit this groove. The glass was then inserted, the tie rods tightened, and a completely liquid-proof seal was effected for both top and bottom. The impeller spacing was changed on the second column, but the same number and size of impellers was used.

In the place of glass inlets and outlets in the bottom of the tower, copper connectors were threaded in, and copper tubing was used in place of the glass tubing. This change was necessary to keep the benzene from softening the sealing wax and causing leaks around the entering and leaving lines in the bottom of the column.

This column operated well. The benzene-acetic acid solution, entering the bottom of the column, passed up through the area agitated by the four impellers, and the washed benzene separated out

in a clear layer at the top of the column. The washed benzene that was forced from the column when collected in a three liter bottle was clear and free from cloudiness. The water entering at the top passed down through the agitated area and separated from the benzene to form a relatively clear layer of water-acetic acid solution in the bottom of the column. The wash water forced from the bottom of the column was slightly cloudy but cleared on standing. However, benzene could be detected by smelling the used wash water.

The operation of this second column was considered satisfactory, and most of the data gathered for this paper were acquired using the column as it is described here, with the exception of the number and size of the impellers.

Third Column Design.

The third design (Figure 5) was based on the results and operating behavior found in operating column two. This third column was designed larger than the first two to eliminate surface effects present in the smaller apparatus.

Pyrex glass tubing was again used, so that the action inside the column could be witnessed. Copper and brass were used for the inside work in the column. Sheet steel was used for end plates, because neither copper, brass nor aluminum were available in the thickness required.

The inside of the column was divided into four compartments of almost equal size. Three perforated copper plates (Figure 6) were used to divide the column into compartments. The first plate and the third plate were each of the same size and had the same size holes.

The center plate was the same size as the inside diameter of the glass column which gave a tight fit, while the top and bottom plates were smaller than the inside diameter of the glass tubing, leaving enough space for the liquids to pass between the plates and the glass column wall. These plates each had five holes drilled in them, one center hole through which the impeller shaft passed, slightly smaller than the other four holes, and four larger holes, one spaced every 90^{o} around the plate close to the outside edge of the plate.

These plates were made fast as a unit by four longitudinal copper baffles. The copper plates were silver soldered to these four baffles. Through the center of the plates ran a brass impeller shaft on which were mounted two brass impellers. The brass impellers were soldered to the shaft in the center of the middle two compartments. These two center compartments were the mixing compartments.

The brass impeller shaft entered the column through a liquid seal similar to the type used on columns one and two. The brass rod rested on the bottom end plate in a bearing groove cut in the center of the steel plate. The top and bottom end plates had four holes drilled in them for four tie rods, and also two holes (threaded) in each plate, one for the incoming liquid and the other for the outgoing liquid. These holes were fitted with copper tubing connections. Besides these holes, the top end plate had a center hole drilled through it and threaded, into which a piece of pipe was threaded for use as the liquid seal pipe. The impeller shaft passed through this pipe.

The four tie rods were of steel rod, threaded on both ends. A rubber gasket was cut to fit around the end of the glass tubing, flat against the steel plate at both ends. The top ends of the four baffles pressed into this rubber gasket and were prevented from rotating as the impeller shaft rotated. The rubber acted as a liquid-proof seal when the column was assembled and the tie rods tightened.

In order to support the column in a vertical position, two pieces of steel rod were welded to the top and bottom end plates, so that regular ring-stand clamps and a ring-stand could be used to hold the column in a rigid vertical position.

The column operated satisfactorily and some of the data taken in this paper were taken from this large column. As in the second column, washed benzene was taken from the top of the column clear and free from cloudiness, while wash water was removed from the bottom of the column fairly clear from cloudiness. The wash water removed from this column was clearer than that removed from the second column, but still retained the benzene odor.

Tables 1, 2, and 3 contain construction dimensions for columns 1, 2, and 3 respectively.

AUXILIARY EQUIPMENT

To operate the column correctly and obtain reproducible data, the following pieces of auxiliary equipment and control instruments were needed:

1. A Milton Roy Chemical Proportionating Pump.
2. A variable speed electric laboratory mixer.
3. A constant level device.
4. Two five-gallon glass storage jugs.
5. A 55-gallon metal water storage drum.
6. A stroboscope.
7. Connecting tubing of copper, glass, neoprene and synthetic rubber.

The Milton Roy Chemical Proportionating Pump was used to supply both the water and the benzene-acetic acid solution to the column. The capacity of the pump could be varied by a hand adjusting wheel on each piston from 0 ml per second to 18 ml per second. The hand adjusting wheel is connected to a dial (Figure 7), so that any given amount of liquid can be pumped from either side by setting the dial. Calibration data for the pump are given in Table 10.

The liquid supply for the pump was from two five-gallon jugs. One jug held the washing water and the other the benzene-acetic acid solution. Since more wash water was used than benzene-acetic acid solution, a constant temperature supply of wash water was maintained from a 55-gallon steel storage drum that fed by gravity into the 5-gallon water jug.

The power to run the impeller was supplied by a variable speed electric laboratory mixer. The speed of the mixer could be adjusted to ten different speeds by varying the rheostat on the back of the mixer from settings one to ten.

A device for keeping the benzene-water interface at a predetermined and constant level in the column during a run was designed (Figure 8). The device consisted of a piece of glass tubing into the bottom of which was a two-hole rubber stopper and into the top of which was a one-hole cork stopper. The incoming wash water, after leaving the column, entered the bottom of the leveling device, the entering glass tubing extending into the leveling device only as far as the inside edge of the rubber stopper. The exiting wash liquid spilled over a glass tubing stand pipe inside the leveling device and left through the bottom rubber stopper. This stand pipe glass tubing entered the leveling device through the bottom rubber stopper and extended three-fourths the way up the inside of the leveling device. The end of the glass stand pipe into which the water spilled was slightly enlarged. The top rubber stopper hole was for a vent only. The entire leveling device could be raised or lowered easily so that by regulating the head of liquid in the column the interface level could be controlled.

The stroboscope was used to get the average speed of the impeller shaft during each run. Two marks were made in India ink on the impeller shaft. A straight series of dots about an inch long, running parallel to the vertical axis of the shaft, were drawn on one side. Half a revolution farther, a wavy line was drawn in India ink parallel to the vertical axis of the shaft and also about an inch long. By focusing the stroboscope on the impeller shaft where

these lines were located, it was possible to read the speed, in RPM, of the shaft.

The connecting tubing used in this set-up varied greatly. Saran tubing was used to connect the liquid reservoirs to the proportionating pump. Copper tubing was used to connect the pump to the inlets on the top and bottom of the columns. Rubber tubing (neoprene) was used to connect the wash water from the column to the constant level device. Glass tubing and rubber tubing were used to carry the wash water from the constant level device to the drain. Neoprene tubing was used to carry the washed benzene to the used benzene storage drum.

METHOD OF TESTING

A solution of benzene and acetic acid was made up such that a 25 ml sample would titrate about 22 ml of 0.1 normal NaOH using phenolphthalein indicator. The approximate amount of acetic acid needed for this concentration was 90 ml of glacial acetic acid to five gallons of benzene. The benzene-acetic acid mixture was continuously agitated during the entire run by using a variable speed, electric powered, laboratory stirrer.

The proportionating pump was set at seven-eighths on the outside dial for the water feed, and at two-eighths on the outside dial for the benzene-acetic acid solution feed. This gave a flow of water of 6.01 ml per second and of benzene-acetic acid solution of 1.52 ml per second. The column was then filled with benzene up to the center of the second baffle from the bottom, the remaining space being filled with water. The level adjuster was fixed to keep the interface constant at this point throughout the run.

The laboratory mixer was then set at the desired speed by using the ten speed rheostat on the back of the motor. Samples were taken from the discharged water. About five minutes was allowed for the column to arrive at equilibrium as each speed change was made. During the time that the column was coming to equilibrium, the stroboscope was used to check the speed of stirring. After about five minutes had elapsed, samples were taken in a 50 cc graduated flask. The contents were then poured into a 150 cc Erlenmeyer flask to which five drops of phenolphthalein indicator were added and titrated with about 0.1 normal sodium hydroxide solution to the end point. When three titrations were within .5 ml of each other, the

column was assumed to be at equilibrium. At least three runs were always taken.

The washed benzene exiting from the tower was collected and re-used in future runs. The wash water from the column was allowed to run down the drain.

The same procedure was followed on the large column with two exceptions. The first was that the interface of the benzene and water was kept constant at one inch below the lower plate in the column. The second exception was that a much longer time was necessary to allow the column to come to equilibrium after starting, or when the impeller speed is changed -- about 30 minutes being required for the column to come to equilibrium when it was first started up.

RESULTS

Columns two and three both gave results over one equilibrium unit. Both columns operated satisfactorily, using the system benzene-acetic acid-water and no secondary separation of the water side or the benzene side was necessary to get a clear product. By using either column two or column three, it was possible continuously to separate acetic acid from benzene by using agitation in a baffled column and obtain a better separation than one equilibrium unit.

DISCUSSION

All three columns designed and tested were made of pyrex glass. By using glass, it was possible to observe the action that took place in the column. Using the system benzene-acetic acid-water, no deposit was formed on the glass wall to restrict the view of the inside of the column.

The first column design failed to operate, because it was observed that the holes through which the benzene was to rise were not large enough to allow the benzene to pass. The wash water flowed readily through these holes. (This proved to be the correct reason for the first column failure, for when the third column was designed, plates with larger holes were used and there was no trouble in its operation.)

The second column of approximately the same diameter as the first had a different interior, baffles being substituted for plates. When operated at slow speeds, under 300 RPM, the bubbles of benzene passing up the column were broken up at each impeller level and not a great deal of horizontal throw-off from the impellers was observed. The bubble size on leaving the top impeller was smaller than the original break-up at the first impeller level, but was not small enough to form an emulsion with the water. Individual bubbles could be seen in the column and in no portion was the water cloudy. A clear layer of benzene separated at the top of the column and clear water solution was drawn off at the bottom of the column.

At 950 RPM in the second column, using four impellers, the entire column, with the exception of the layer of benzene at the top

of the column and the water layer at the bottom of the column, was a milky white emulsion. The pattern that the impellers cause was observed to be in the shape of a four leaf clover with the impeller shaft as the hub and the two leaves on each side starting at the end of the impeller and going out on different sides in an arc back to the impeller shaft along the baffles. There did not appear to be an end-to-end turn-over of the entire agitated liquid.

When fewer impellers were used, the only difference noted was at slower speeds where the bubbles were not broken up as well. At high speeds with but one impeller, the column was milky to the same extent as with four impellers, but there was not as much turbulence visible.

The capacity possible for this small column was limited by the amount of pressure possible before the liquid seal at the top of the column ran over. The rate of input to the column within the small range possible for this column design seemed to have little effect on the ability of the two liquids to separate at either end of the column.

In the third column design, it was not possible to draw off clear benzene when two impellers, three-eighths inches wide, were used at high speed, as the entire top of the column clouded up. When smaller impellers, one-eighth inch wide, were used at high speed it was possible to draw off clear benzene at the top of the column and clear water at the bottom. Agitation in this larger column was restricted to the two mixing compartments, the perforated plates and baffles, keeping the two end settling compartments calm and clear. A thin layer of water formed on the top of the center plate and it is

possible that if smaller holes or fewer holes were present this layer could be held at a predetermined depth, giving three interfaces in the column and actually dividing the column into two completely separate sections.

The pattern of agitation in each mixing compartment was the same as described in the smaller column, but the agitation was confined to the mixing compartments.

Because the system selected to study had a distribution coefficient of about nine, it was not possible to get conclusive results. The difference between two equilibrium units and three equilibrium units on this system was within the limit of error of the experimental work.

The difference in the number of equilibrium units possible, using different numbers and sizes of impellers, was probably caused by surface effects within the column. In the second column, it was observed that globules of benzene would cling to the baffles, especially near the interface, and suddenly release and float upward in the column. This was probably the cause for the variations in the number of equilibrium units possible, using different numbers and sizes of impellers.

TABLE 1

DIMENSIONS OF FIRST COLUMN

A.	Over-all.	
	1. Length of column	15-5/16"
	2. Inside diameter	1-13/16"
	3. Outside diameter	2"
B.	Plates.	
	1. Number	5
	2. Diameter	1-13/16"
	3. Thickness	1/16"
	4. Center Hole (diameter)	3/8"
	5. Feed Hole (diameter)	5/16"
	6. Fastener Rod Hole (diameter)	3/16"
	7. Bottom of bottom plate to end of fastener rod	4- 3/32"
C.	End Plates.	
	1. Top.	
	a. Diameter	3- 7/16"
	b. Thickness	7/16"
	c. Inlet Hole (diameter)	9/32"
	d. Exit Hole (diameter)	9/32"
	e. Stirring Rod Entrance Hole (diameter)	3/8"
	f. Fastener Rod Hole (diameter)	5/32"
	g. Fastener Rod Hole (depth)	1/8"
	h. Tie Rod Holes (diameter)	3/16"
	i. Gasket Groove (depth)	1/8"
	j. Gasket Groove (outside diameter)	2- 1/16"

k. Gasket Groove Inside diameter ..... 1- 3/4"

l. Pyrex Glass over flow (diameter) ..... 3/8"

m. Pyrex Glass over flow (height) ..... 3- 3/16"

n. Pyrex Glass Tubing Connection (diameter) .... 9/32"

2. Bottom.

a. Diameter ..... 3- 7/16"

b. Thickness ..... 7/16"

c. Inlet Hole (diameter) ..... 11/32"

d. Outlet Hole (diameter) ..... 11/32"

e. Copper Tubing Connections (diameter) ..... 1/2"

f. Bearing diameter ..... 3/16"

g. Bearing Depth ..... 1/8"

D. Impeller Shaft.

1. Length ..... 23"

2. Diameter ..... 3/16"

3. Bottom of Shaft to Center of Impeller ..... 5"

E. Impellers.

1. Number 4

2. Length ..... 1"

3. Thickness ..... 1/4"

4. Width ..... 1/16"

5. Distance apart in centers ..... 1- 1/2"

F. Tie Rods.

1. Number 2

2. Length ..... 16"

3. Diameter ..... 3/16"

TABLE 2

DIMENSIONS OF SECOND COLUMN

A. Over-all.

1.	Length	15-5/16"
2.	Inside diameter	1-23/32"
3.	Outside diameter	1- 7/8"

B. Baffles.

1.	No. per fastener rod	8
2.	Length	1"
3.	Width	5/8"
4.	Thickness	1/16"
5.	Distance from end of fastener rod to center of first baffle	1- 1/8"
6.	Distance center to center	1- 3/4"
7.	Distance from center to bottom baffle to end of fastener rod	1"

C. Fastener Rod.

1.	Number	2
2.	Length	14- 1/2"
3.	Diameter	3/16"

D. End Plates.

1. Top and Bottom.

Same as in Column I, except for different gasket groove.

a.	Gasket groove - outside diameter	2- 3/16"
b.	Gasket groove - inside diameter	1-11/16"
c.	Gasket groove - depth	1/4"

d. Gasket groove - depth ..... 1/4"

e. Neoprene gasket - thickness ..... 1/8"

E. Impeller Shafts.

1. Length ..... 23"

2. Diameter ..... 3/16"

3. Bottom of Shaft to center of last impeller ..... 5-1/2"

4. Distance between impellers (in center) ..... 1-3/4"

F. Impellers.

1. Number 4

2. Length ..... 1"

3. Thickness ..... 1/16"

4. Width ..... 1/4"

TABLE 3

DIMENSIONS OF THIRD COLUMN

A.	Over-all.	
	1. Length	28- 3/4"
	2. Inside diameter	3-11/16"
	3. Outside diameter	3- 7/8"
B.	Plates.	
	1. Number	3
	2. Diameter	
	a. Top and bottom plates	3- 3/32"
	b. Center plate	3-11/16"
	3. Center hole Diameter	5/8"
	4. Top and bottom plates - outside holes (diameter)	7/8"
	5. Center plate outside holes	1/2"
C.	Baffles.	
	1. Number	4
	2. Length	28"
	3. Width	2/5"
	4. Thickness	1/16"
D.	End Plates.	
	1. Top.	
	a. Diameter	6"
	b. Thickness	3/8"
	c. Tie rod holes - number	4
	- diameter	5/16"
	d. Inlet hole (diameter)	11/32"

e. Outlet hole (diameter) ..... 11/32"

f. Center hole ..... 7/16"

2. Bottom.

a. Diameter ..... 6"

b. Thickness ..... 3/8"

c. Tie rod holes - number 4

- diameter ..... 5/16"

d. Inlet hole diameter ..... 11/32"

e. Outlet hole diameter ..... 11/32"

f. Bearing diameter ..... 1/4"

g. Bearing depth ..... 1/16"

E. Impellers and Impeller Shaft.

1. Shaft diameter ..... 1/4"

2. Shaft Length ..... 34"

3. Distance from bottom shaft to last impeller center ..... 10-23/32"

4. Number Impellers 2

5. Length Impellers ..... 1- 4/5"

6. Width Impellers ..... 1/8"

7. Thickness Impellers ..... 1/16"

F. Liquid Seal Pipe.

1. Pipe Diameter ..... 1/2"

2. Pipe Length ..... 4"

TABLE 4

EXTRACTION CURVE DATA

Rate of Flow - Benzene-Acetic Acid - 1.519 ml per sec

Rate of Flow - Water - 6.010 ml per sec

No. of Impellers - 4

Size of Impellers - 1" x 1/4" x 1/16"

Benzene-Acetic Acid Concentration - 20.2 ml 0.1113N NaOH for 25 ml Sample

SPEED	RPM (AVG.)	SAMPLE EXIT H_2O IN ML	0.1113N ML NaOH(AVG.)	EQUILIBRIUM SAMPLE IN ML	0.1113N ML NaOH(AVG.)	NO. OF EQUILIBRIUM UNITS
0	0	50	7.26	25	5.1	0.712
1	311.8	50	9.00	25	5.1	0.883
2	504.2	50	10.6	25	5.1	1.040
3	541.9	50	10.8	25	5.1	1.059
4	626.1	50	10.91	25	5.1	1.070
5	707.3	50	11.21	25	5.1	1.090
6	780.6	50	11.05	25	5.1	1.083
7	867.5	50	10.9	25	5.1	1.069
8	898.6	50	11.06	25	5.1	1.083
9	-	-	-	-	-	-
10	944.5	50	11.0	25	5.1	1.078

TABLE 5

EXTRACTION CURVE DATA

Rate of Flow - Benzene-Acetic Acid - 1.519 ml per sec

Rate of Flow - Water - 6.010 ml per sec

No. of Impellers - 2

Size of Impellers - 1" x 1/4" x 1/16"

Benzene-Acetic Acid Concentration - 221 ml 0.1015 N NaOH for 25 ml Sample

SPEED	RPM (AVG.)	SAMPLE EXIT H_2O IN ML	0.1015N ML NaOH(AVG.)	EQUILIBRIUM SAMPLE IN ML	0.1015N ML NaOH(AVG.)	NO. OF EQUILIBRIUM UNITS
0	0	50	6.9	25	4.88	0.707
1	325	50	8.075	25	4.88	0.828
2	444.8	50	9.07	25	4.88	0.930
3	553.7	50	9.79	25	4.88	1.002
4	622.5	50	10.00	25	4.88	1.025
5	713.2	50	10.21	25	4.88	1.048
6	786.3	50	10.23	25	4.88	1.049
7	867.9	50	10.32	25	4.88	1.058
8	905.7	50	10.50	25	4.88	1.077
9	-	-	-	-	-	-
10	964.	50	10.45	25	4.88	1.071

TABLE 6

EXTRACTION CURVE DATA

Rate of Flow - Benzene-Acetic Acid - 1.519 ml per sec

Rate of Flow - Water - 6.010 ml per sec

No. of Impellers - 1

Size of Impellers - 1" x 1/4" x 1/16"

Benzene-Acetic Acid Concentration - 16.5 ml of 0.1258N NaOH for 25 ml Sample

SPEED	RPM (AVG.)	SAMPLE EXIT H_2O IN ML	0.1258N ML NaOH(AVG.)	EQUILIBRIUM SAMPLE IN ML	0.1258N ML NaOH(AVG.)	NO. OF EQUILIBRIUM UNITS
0	0	50	5.25	25	4.3	0.611
1	332.4	50	6.05	25	4.3	0.704
2	435.8	50	6.73	25	4.3	0.783
3	539.2	50	7.70	25	4.3	0.895
4	630.4	50	8.15	25	4.3	0.948
5	717.0	50	8.57	25	4.3	0.996
6	777.2	50	8.60	25	4.3	1.000
7	850.9	50	8.73	25	4.3	1.015
8	917.7	50	8.80	25	4.3	1.023
9	-	-	-	-	-	-
10	947.5	50	8.61	25	4.3	1.000

TABLE 7

EXTRACTION CURVE DATA

Rate of Flow - Benzene-Acetic Acid - 1.519 ml per sec

Rate of Flow - Water - 6.010 ml per sec

No. of Impellers - 1

Size of Impellers - 1/8" x 1" x 1/16"

BENZENE-ACETIC ACID CONCENTRATION - 25.5 ml 0.1015N NaOH for 25 ml Sample

SPEED	RPM (AVG.)	SAMPLE EXIT H_2O IN ML	0.1015N ML NaOH (AVG.)	EQUILIBRIUM SAMPLE IN ML	0.1015N ML NaOH(AVG.)	NO. OF EQUILIBRIUM UNITS
0	0	50	10.29	25	6.5	0.792
1	330.3	50	10.74	25	6.5	0.827
2	495.7	50	11.92	25	6.5	0.918
3	565.2	50	12.42	25	6.5	0.956
4	630.0	50	12.48	25	6.5	0.961
5	703.1	50	13.35	25	6.5	1.026
6	766.9	50	13.48	25	6.5	1.036
7	862.1	50	13.30	25	6.5	1.022
8	907.3	50	13.36	25	6.5	1.028
9	-	-	-	-	-	-
10	952.0	50	13.20	25	6.5	1.015

TABLE 8

EXTRACTION CURVE DATA

Rate of Flow - Benzene-Acetic Acid - 1.519 ml per sec

Rate of Flow - Water - 6.010 ml per sec

No. of Impellers - 1

Size of Impellers - 1/8" x 1" x 1/16"

Benzene-Acetic Acid Concentration - 22 ml 0.1015N NaOH for 25 ml Sample

SPEED	RPM (AVG.)	SAMPLE EXIT H_2O IN ML	0.1015N ML NaOH(AVG.)	EQUILIBRIUM SAMPLE IN ML	0.1015N ML NaOH(AVG.)	NO. OF EQUILIBRIUM UNITS
0	0	50	6.3	25	4.5	0.702
1	361.3	50	6.88	25	4.5	0.765
2	444.5	50	7.65	25	4.5	0.850
3	596.8	50	8.03	25	4.5	0.893
4	671.3	50	8.67	25	4.5	0.964
5	762.0	50	9.03	25	4.5	1.004
6	822.5	50	9.26	25	4.5	1.028
7	851.5	50	9.50	25	4.5	1.055
8	893.4	50	9.48	25	4.5	1.054
9	941.0	50	9.95	25	4.5	1.105
10	970.0	50	9.90	25	4.5	1.100

TABLE 9

EXTRACTION CURVE DATA

Rate of Flow - Benzene-Acetic Acid - 1.519 ml per sec

Rate of Flow - Water - 6.010 ml per sec

Size of Impellers - 1" x .047" dia.

Benzene-Acetic Acid Concentration - 18.2 ml 0.1258N NaOH for 25 ml sample

No. OF IMPELLERS	RPM (AVG.)	SAMPLE EXIT H_2O IN ML	0.1258N ML NaOH(AVG.)	EQUILIBRIUM SAMPLE IN ML	0.1258N ML NaOH(AVG.)	NO. OF EQUILIBRIUM UNITS
1	960	50	8.00	25	4.67	0.858
2	965	50	8.60	25	4.67	0.922
3	950	50	8.80	25	4.67	0.945
4	950	50	9.00	25	4.67	0.963
5	962	50	9.40	25	4.67	1.008
6	950	50	9.00	25	4.67	0.963

TABLE 10

PUMP CALIBRATION

READINGS LEFT DIAL IN EIGHTS	ML PER SEC FLOW 1	2	3	avg.	READINGS RIGHT DIAL IN EIGHTS	ML PER SEC FLOW 1	2	3	avg.
1	0.717	0.725	0.627	0.690	1	0.780	0.654	0.770	0.735
2	1.445	1.573	1.540	1.519	2	1.546	1.569	1.650	1.588
3	2.470	2.450	2.410	2.665	3	2.530	2.505	2.630	2.555
4	3.230	3.558	3.220	3.336	4	3.540	3.760	3.710	3.670
5	4.175	4.410	4.215	4.267	5	4.210	4.610	4.570	4.463
6	5.130	5.320	5.000	5.150	6	5.130	5.220	5.175	5.175
7	5.910	5.925	5.905	5.913	7	6.020	6.000	6.015	6.010
8	6.710	6.795	6.530	6.678	8	6.740	6.660	7.400	6.933

TABLE 11

EXTRACTION CURVE DATA

COLUMN NO. 3

Rate of Flow - Benzene-Acetic acid solution - 1.519 ml per sec

Rate of Flow - Water - 6.010 ml per sec

No. of Impellers - 2

Size of Impellers - Run I - 3/8" x 1-4/5" x 1/16"

Run II- 1/8" x 4/5" x 1/16"

Benzene-Acetic Acid Concentration - 20.0 ml 0.1122 N NaOH for 25 ml Sample

Speed - 950 RPM

RUN	SAMPLE EXIT H_2O IN ML	0.1122N ML NaOH(AVG.)	EQUILIBRIUM SAMPLE IN ML	0.1122N ML NaOH(AVG.)	NO. OF EQUILIBRIUM UNITS
1	50	10.8	25	4.9	over two
2	50	10.65	25	4.9	over two

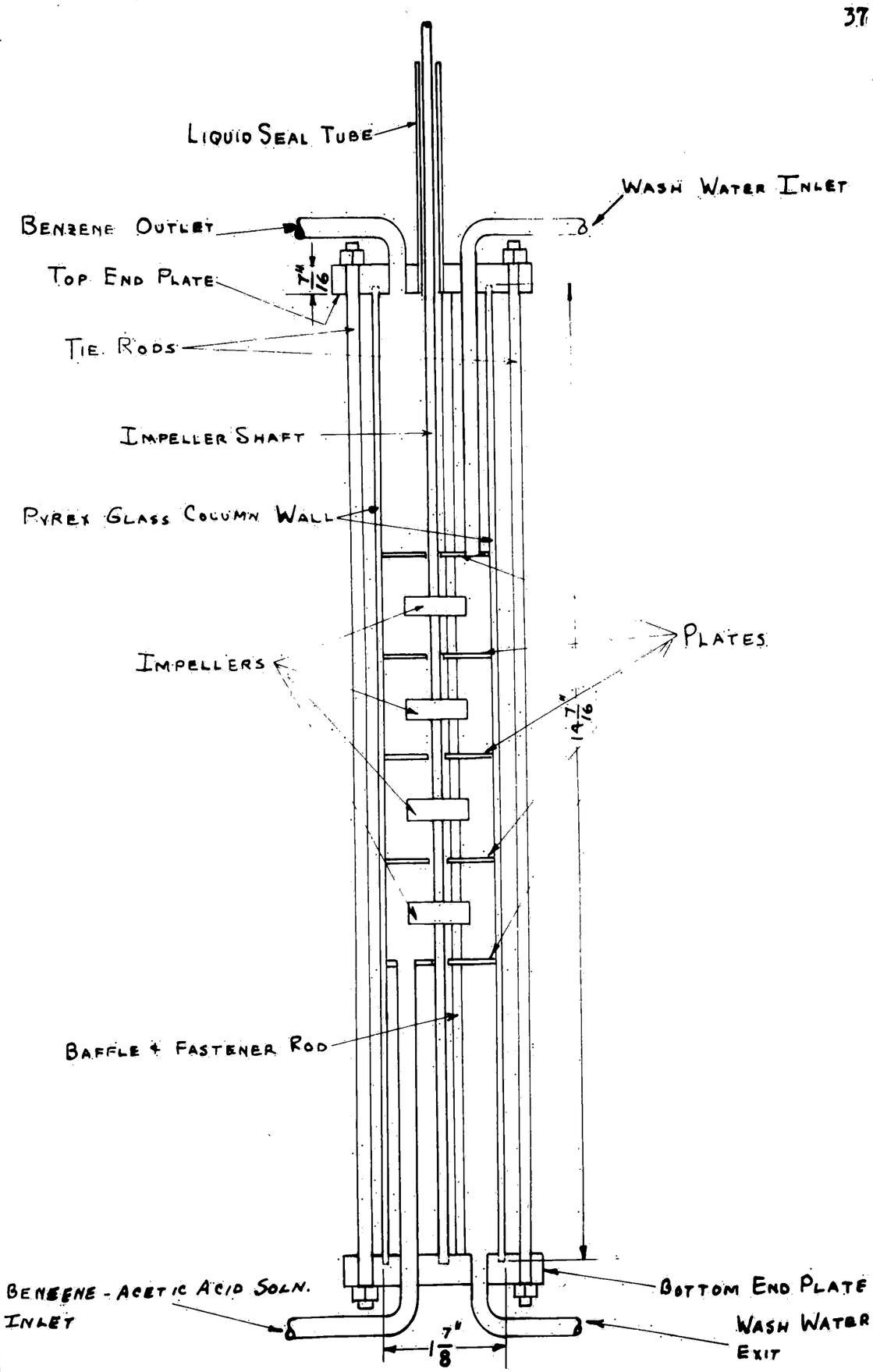


FIGURE 1 FIRST COLUMN DESIGN

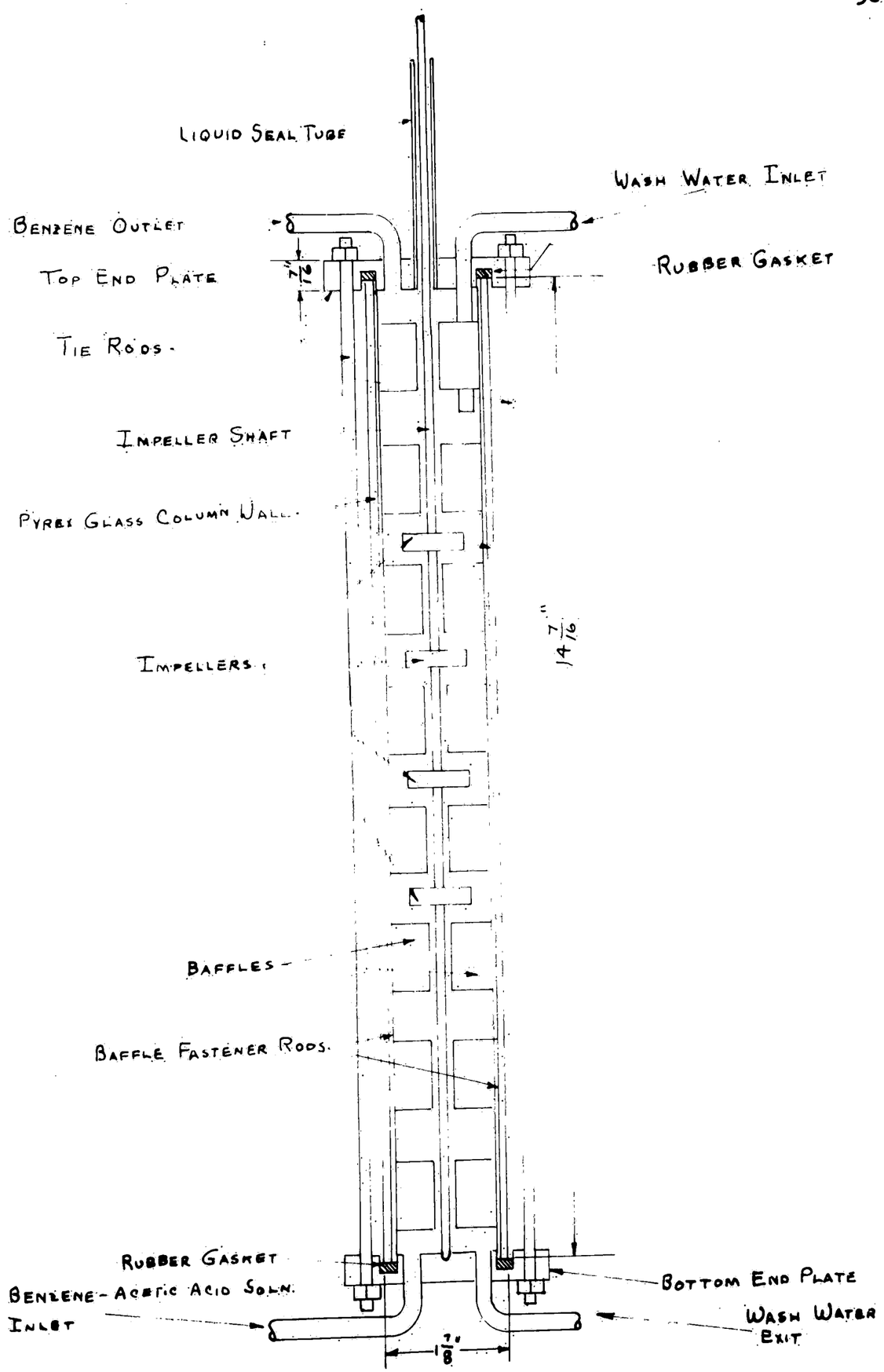


FIGURE 2 SECOND COLUMN DESIGN

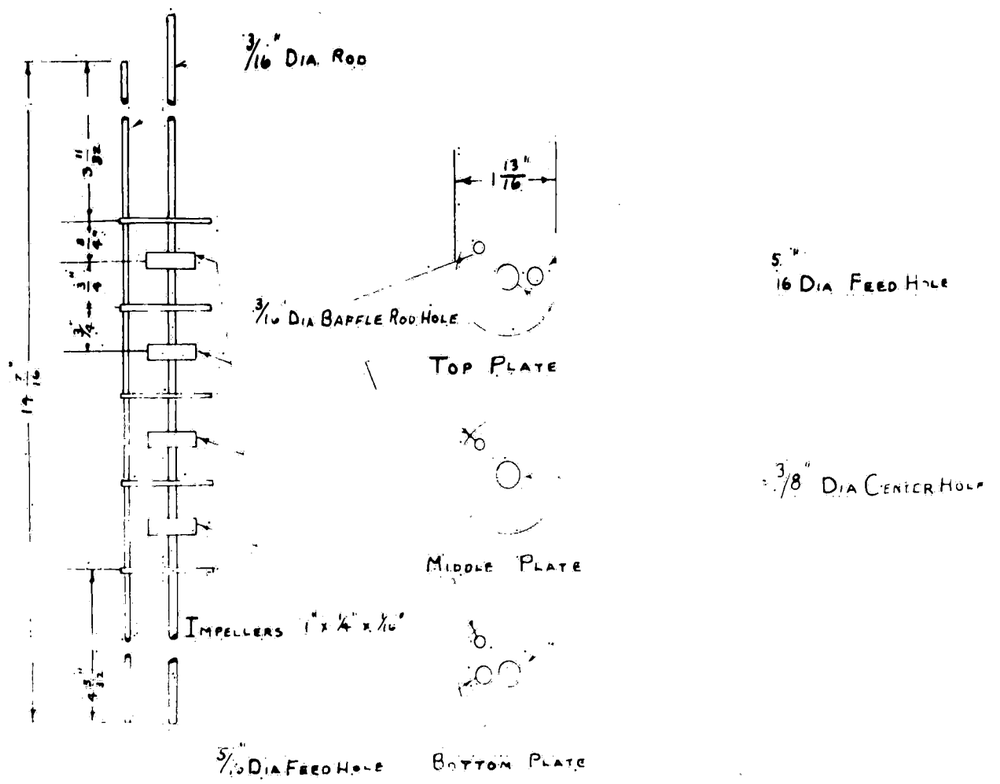


FIGURE 3 INTERNAL PARTS OF FIRST COLUMN (ALL ALUMINUM)

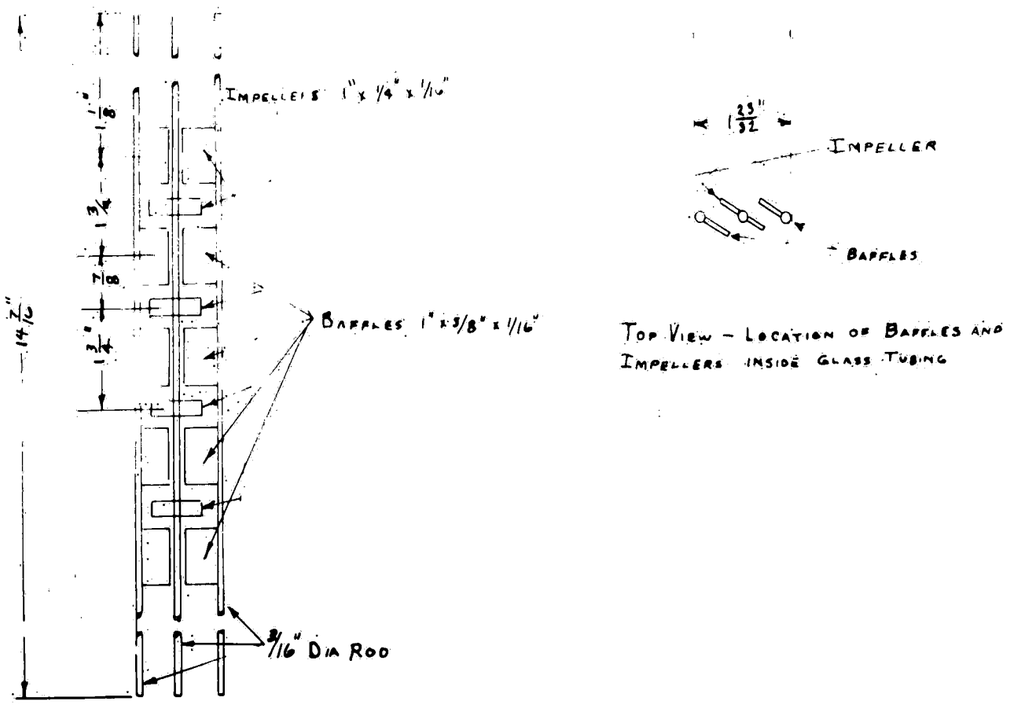


FIGURE 4 INTERNAL PARTS OF SECOND COLUMN (ALL ALUMINUM)

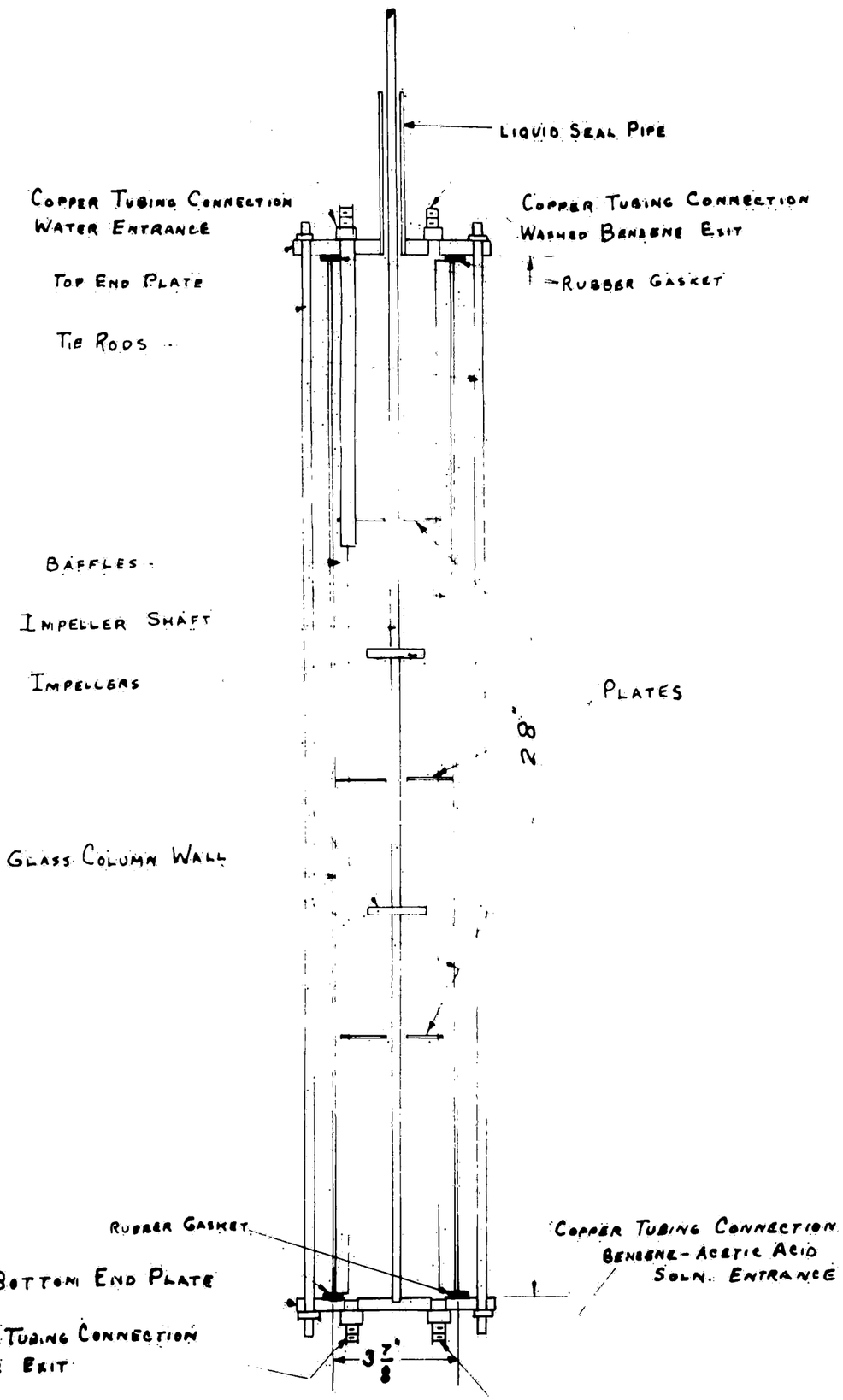


FIGURE 5 THIRD COLUMN DESIGN

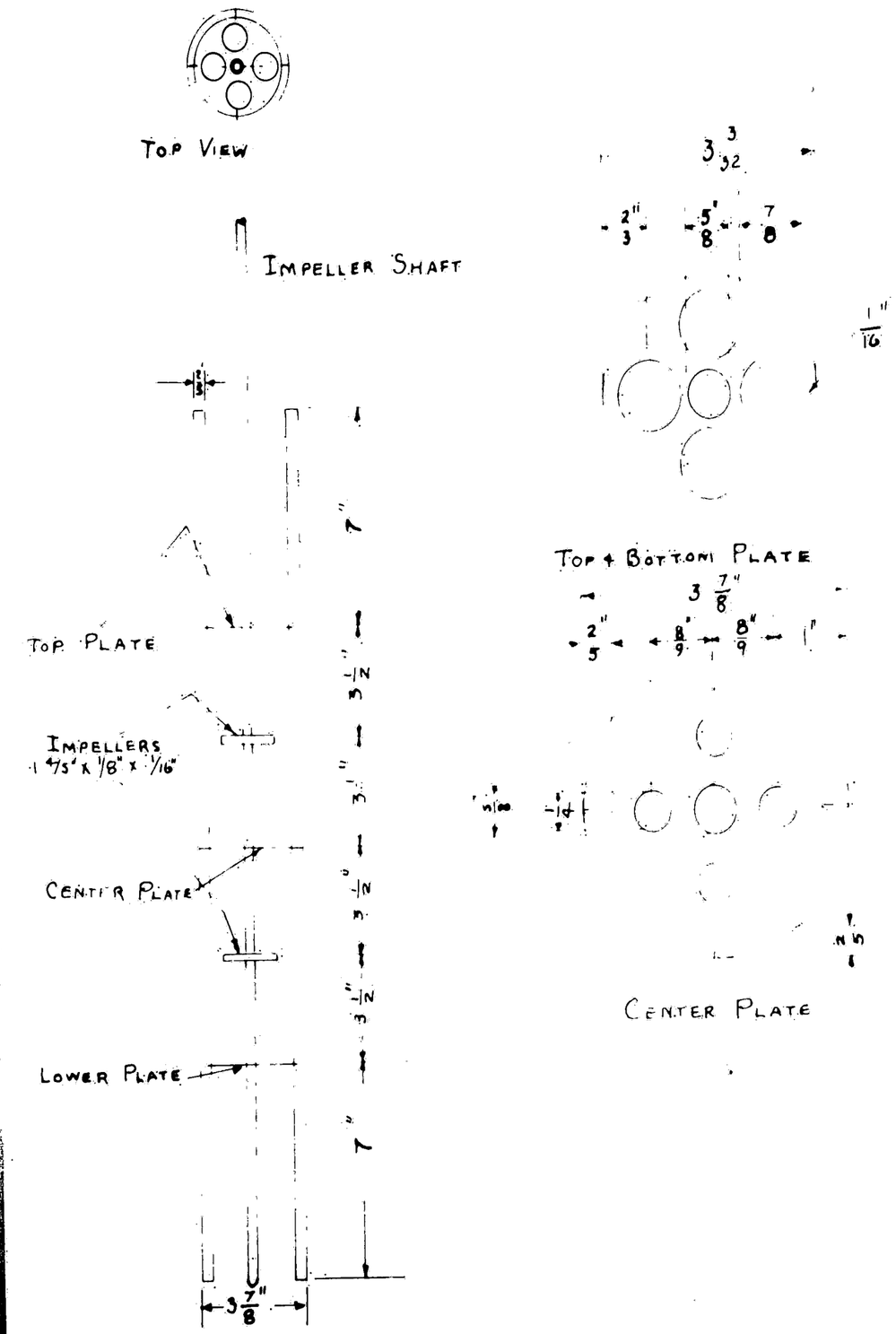


FIGURE 6 INTERNAL PARTS OF THIRD COLUMN (BRASS & COPPER)

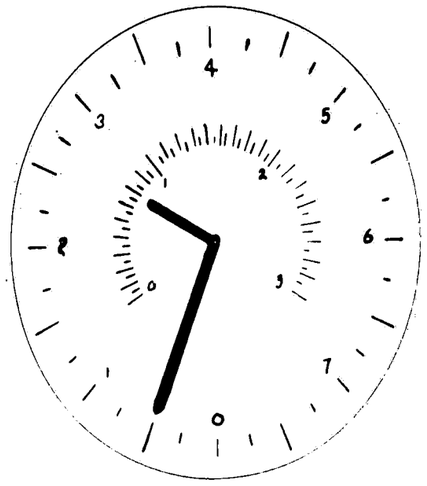


FIGURE 7 PROPORTIONATING PUMP HAND-ADJUSTING DIAL

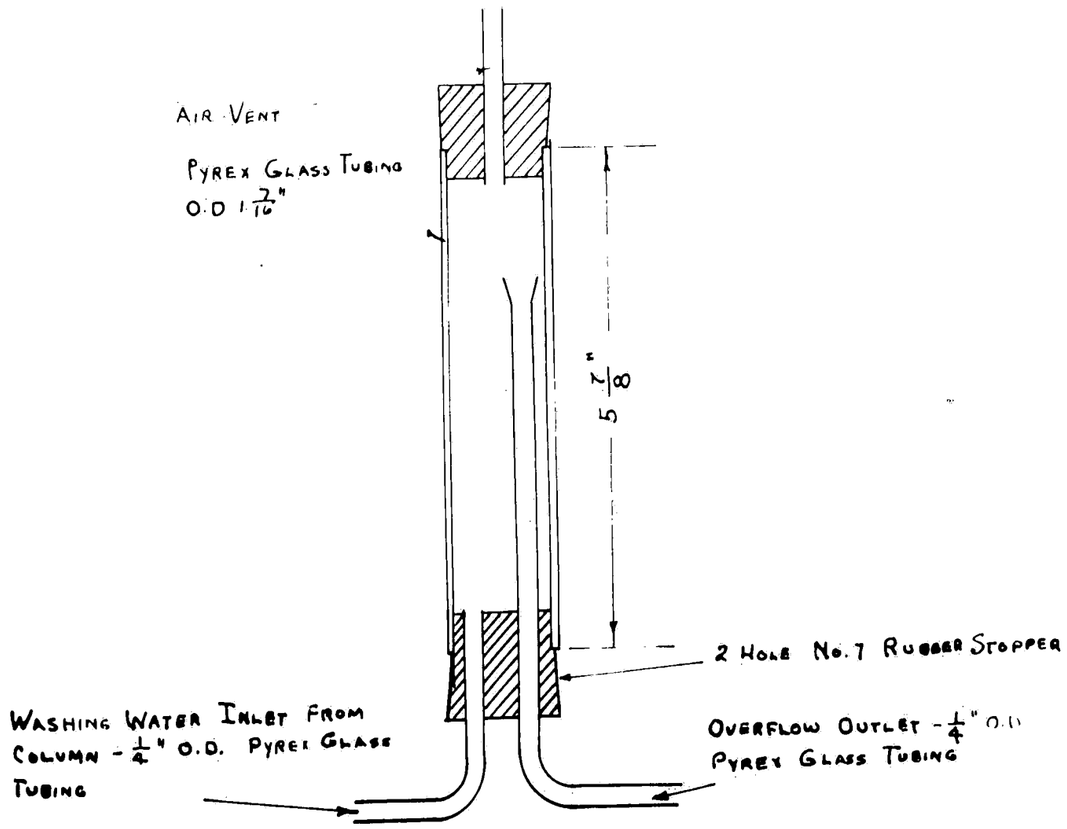


FIGURE 8 CONSTANT LEVEL DEVICE

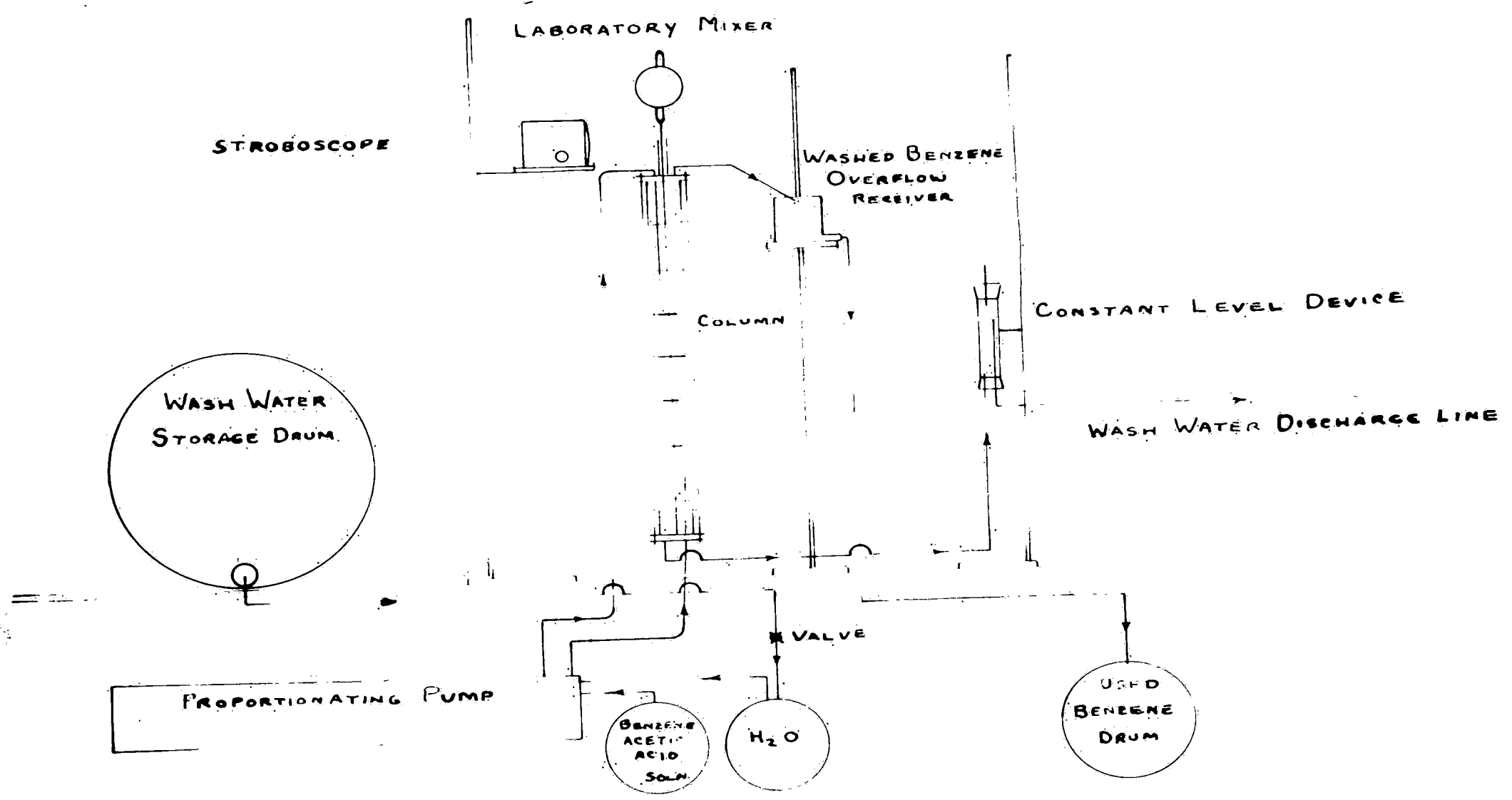


FIGURE 9 LAYOUT OF EXTRACTION EQUIPMENT

FIGURE 10

EXTRACTION CURVE

(Data of Table 4)

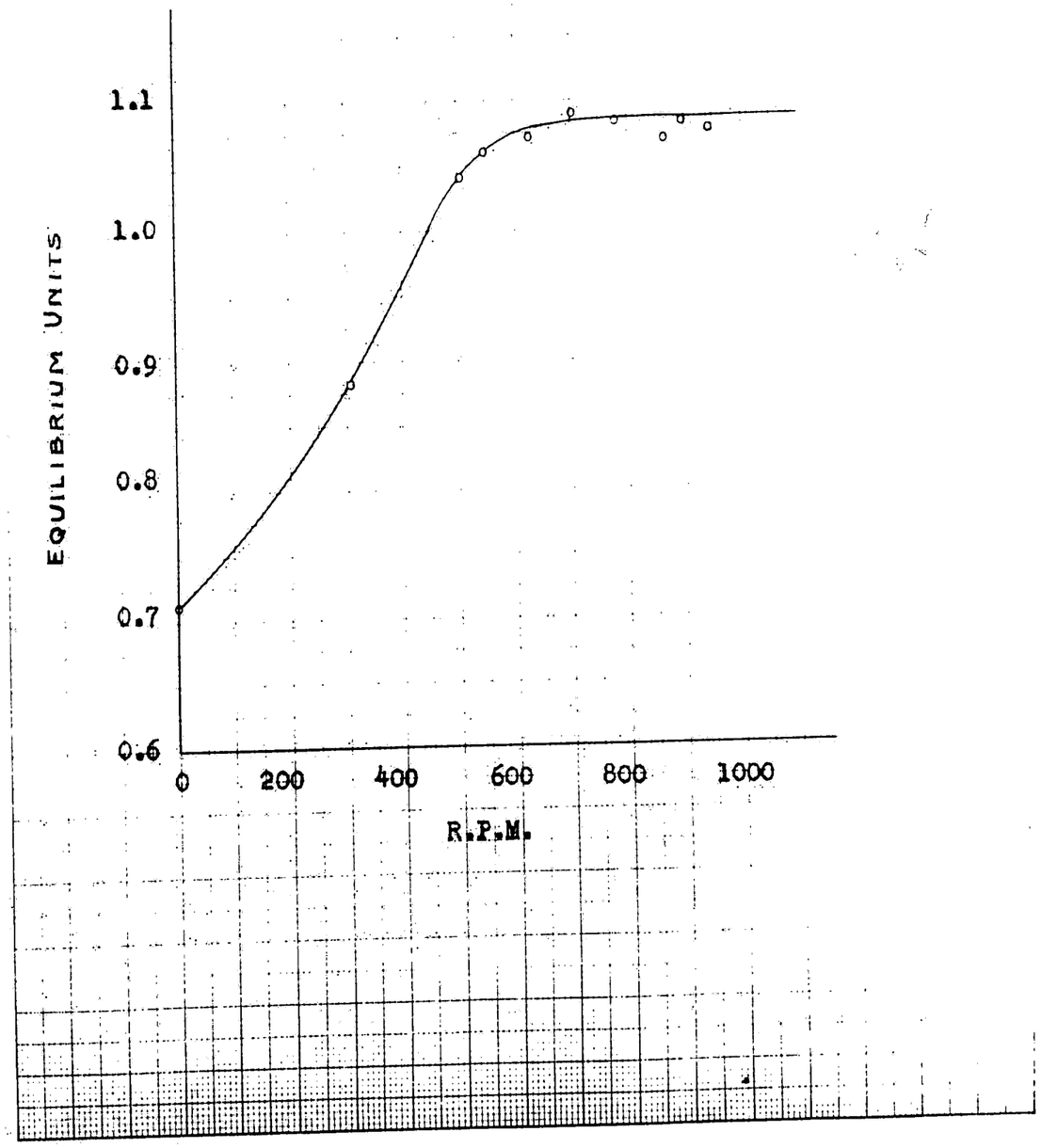


FIGURE 11

EXTRACTION CURVE

(Data of Table 5)

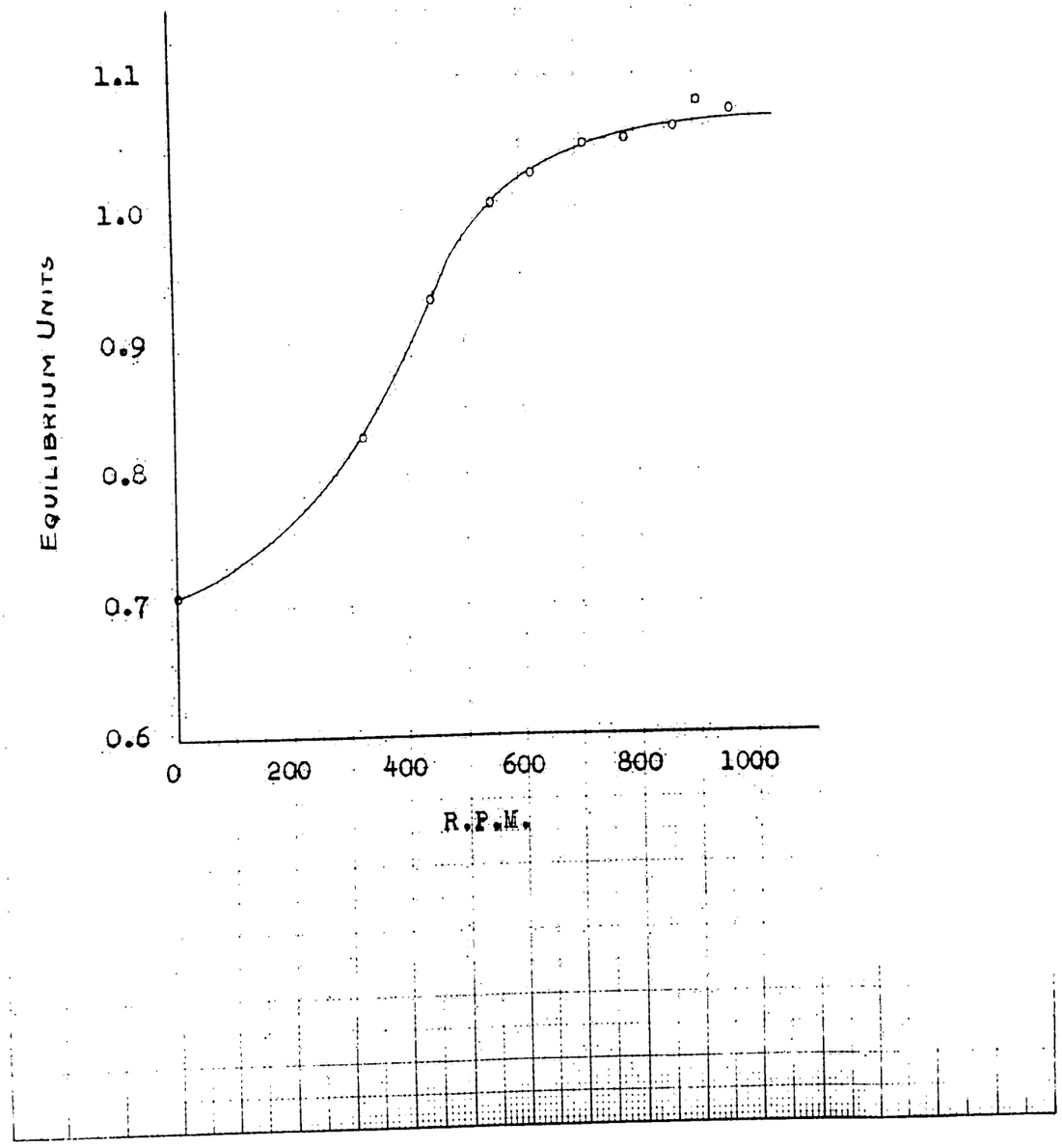


FIGURE 12

EXTRACTION CURVE

(Data of Table 6)

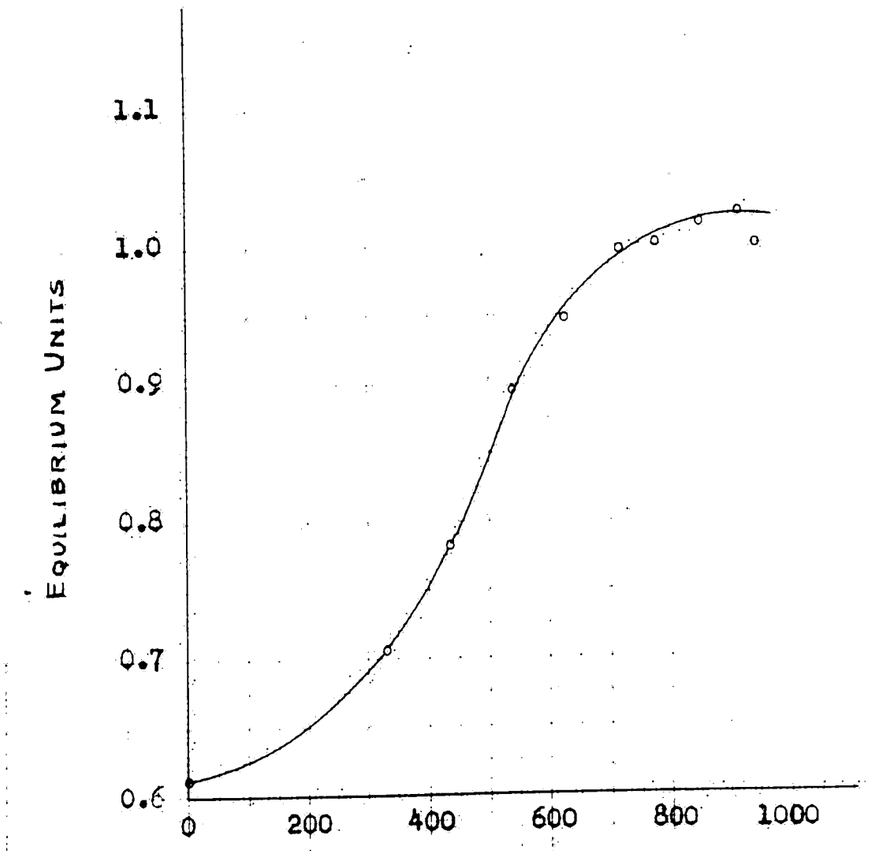


FIGURE 13

EXTRACTION CURVE

(Data of Table 7)

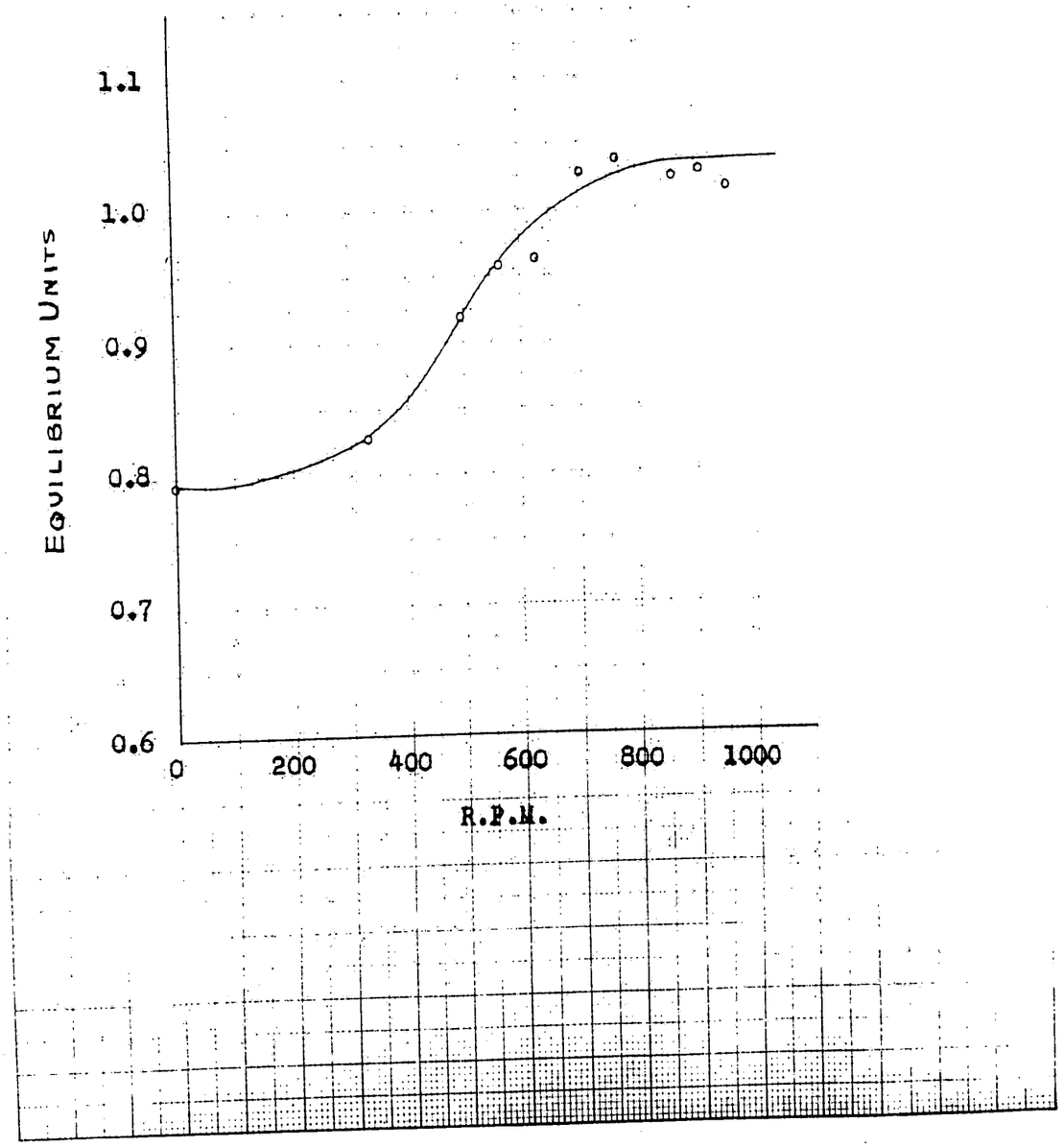


FIGURE 14

EXTRACTION CURVE

(Data of Table 8)

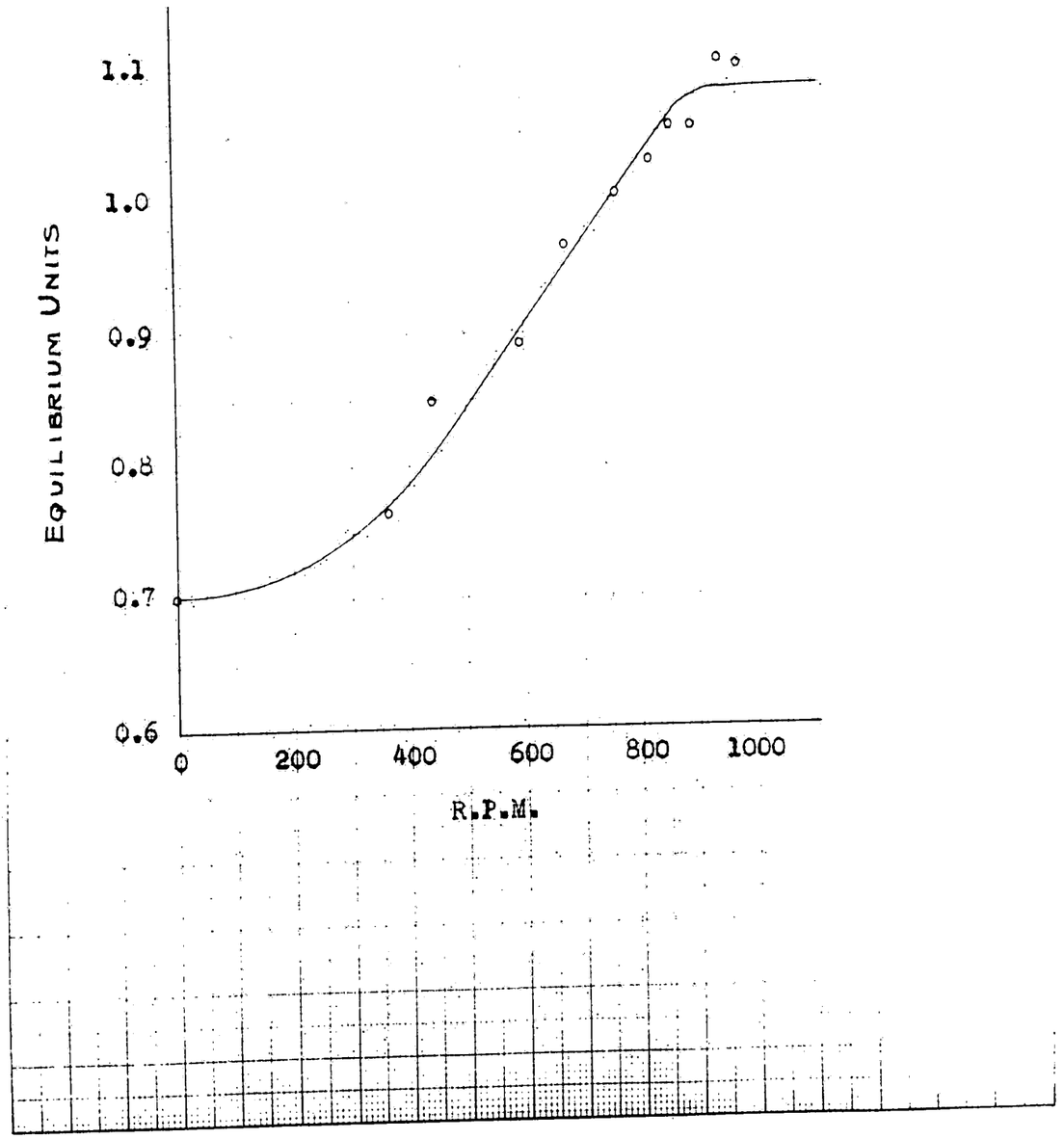


FIGURE 15

EXTRACTION CURVE

(Data of Table 9)

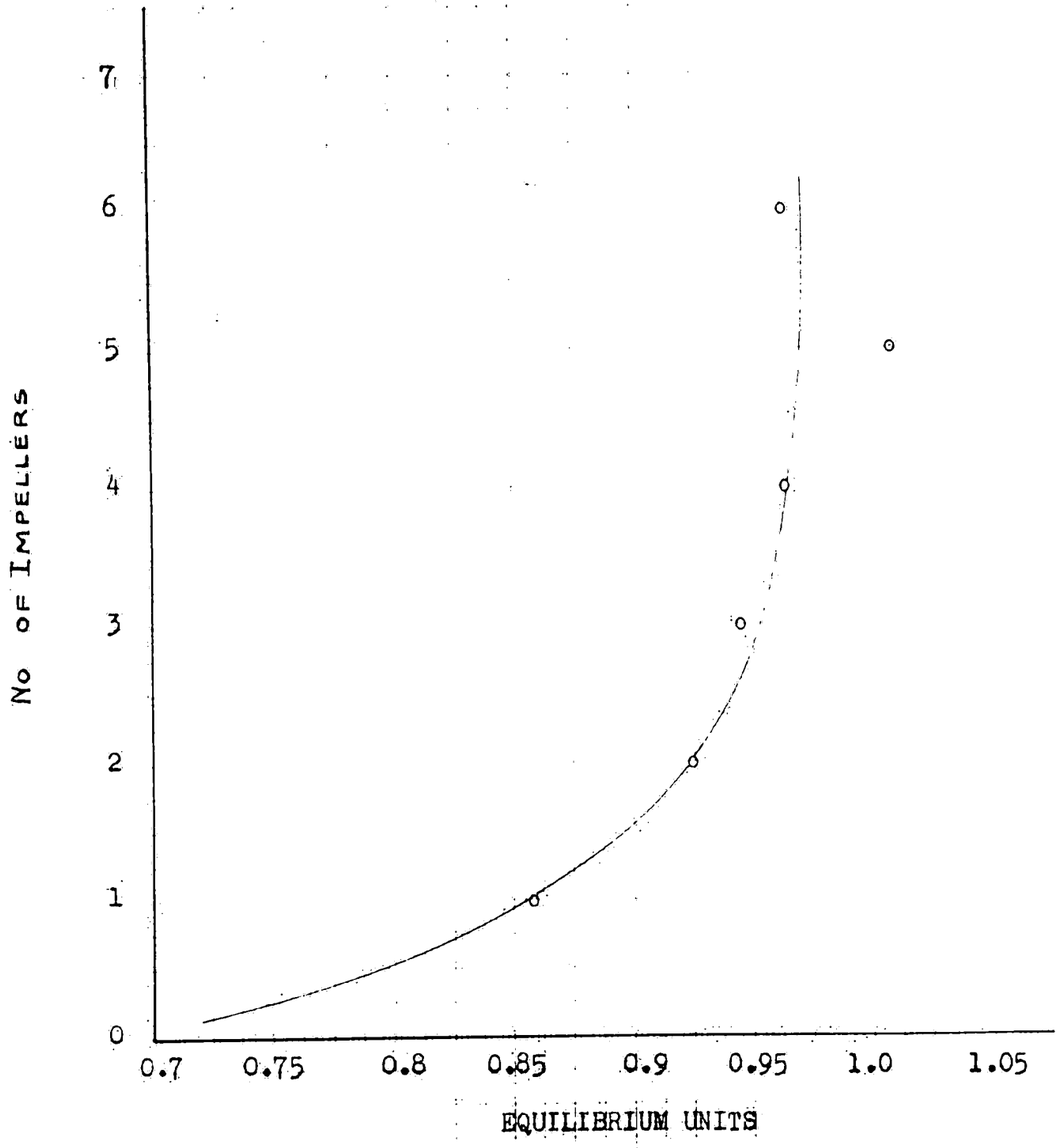
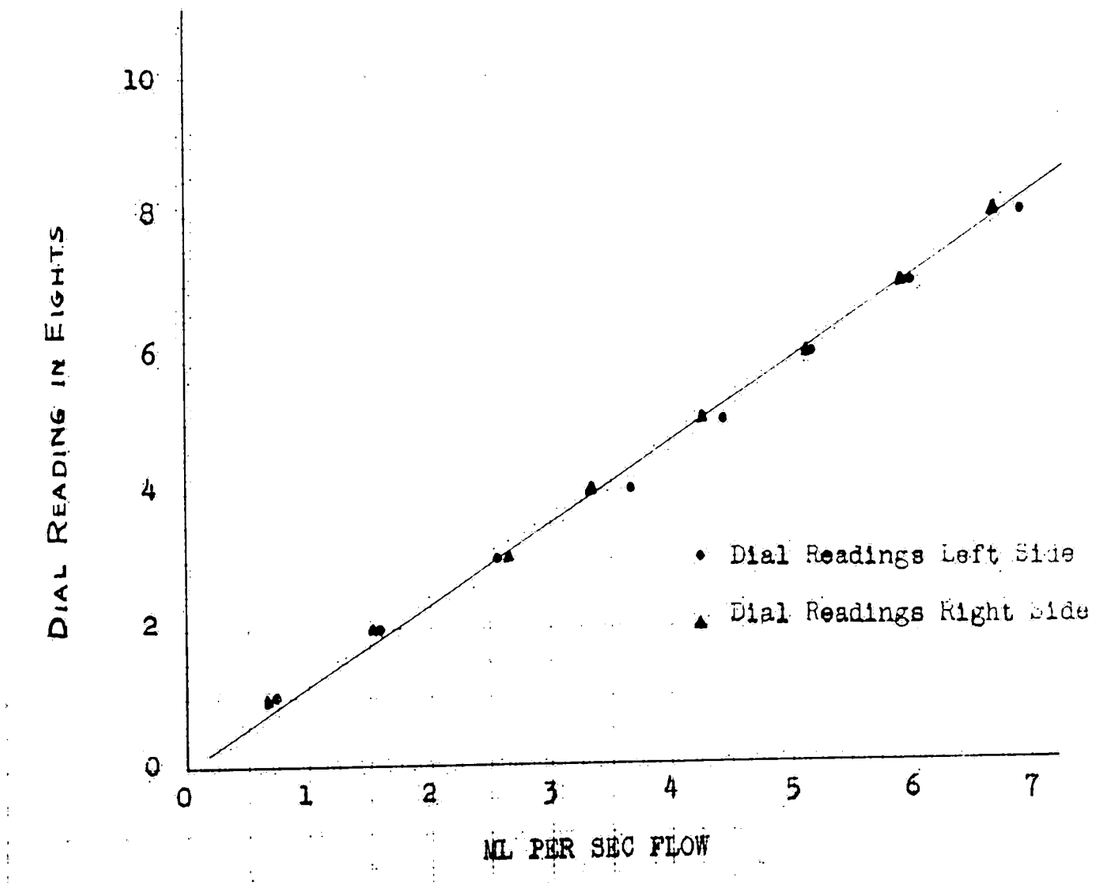


FIGURE 16

PUMP CALIBRATION CURVE

(Data of Table 10)



APPENDIX

CALCULATIONS

I. One Equilibrium Unit.

Rate of Flow of Benzene-Acetic Acid Solution - 1.519 ml per sec

Rate of Flow of H_2O - 6.010 ml per sec

$$\frac{100 \text{ ml}}{1.519 \text{ ml/sec}} \qquad \frac{6.010 \text{ ml/sec}}{} = 396 \text{ ml}$$

Equilibrium Sample Used

100 ml of Benzene-Acetic acid solution

396 ml of H_2O

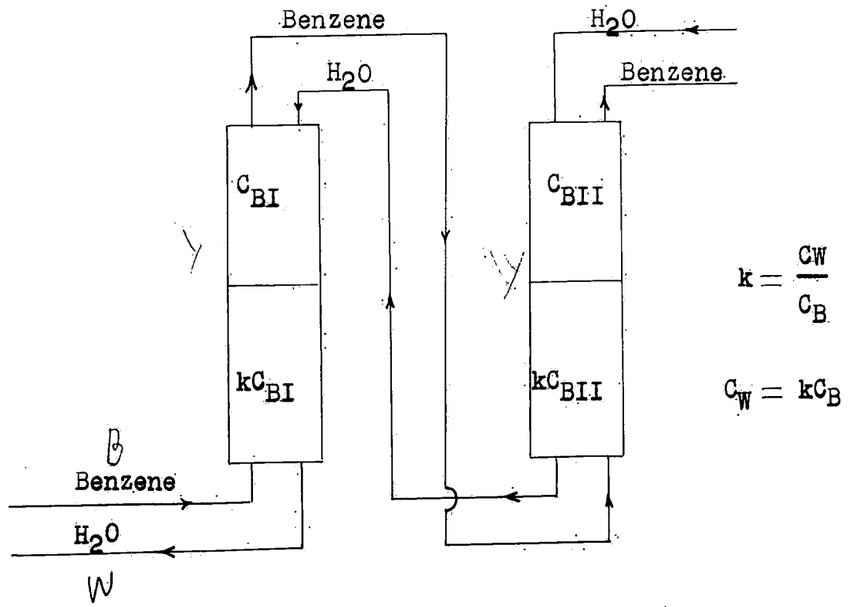
Titrating the Equilibrium Sample with .1258N NaOH

ml .1258N NaOH	ml Sample
4.5	25
4.5	25

For 50 ml sample, 1 equilibrium unit = 9.0 ml .1258N NaOH

II. Two Equilibrium Units.

Using data from Column 3, Table 11



Material Balance

Unit I $B_a + WkC_{BII} = BC_{BI} + WkC_{BI}$

Unit II $Wb^0 + BC_{BI} = WkC_{BII} + BC_{BII}$

where B = Rate of flow in ml/sec of Benzene and Acetic Acid solution

a = Concentration of Acetic Acid in benzene in ml of .1122N NaOH

W = Rate of flow of water in ml/sec

k = Distribution coefficient = $\frac{CW}{CB}$

CW = Concentration of Acetic Acid in water at equilibrium

C_B = Concentration of Acetic Acid in Benzene at equilibrium

b = Concentration of acetic acid in water in ml of .1122N NaOH

The equilibrium sample contains

100 ml of benzene-acetic acid solution

396 ml of water

Strength of benzene-acetic acid solution is 20 ml of 0.1122N NaOH for a 25 ml sample

Thus in 100 ml benzene-acetic acid solution there are

4 x 20 = 80 ml 0.1122N NaOH

At equilibrium 25 ml sample water titrated

4.9 ml 0.1122N NaOH

Thus the amount of acetic acid in the 396 ml water at equilibrium is $\frac{396}{25}$ x 4.9 = 77.8 ml of 0.1122N NaOH

Since there was 80 ml of 0.1122N NaOH originally, then there is left in the 100 ml of benzene

80 - 77.8 = 2.2 ml of 0.1122N NaOH of acetic acid

Thus $k = \frac{77.8}{3.96} \div 2.2 = 8.92 = \frac{C_w}{C_b}$

With a rate of flow for benzene and acetic acid solution of 1.519 ml/sec and for water 6.010 ml/sec, and substituting in equilations I and II and solving for

C_{BI} we get

C_{BI} = 0.02227 ml of 0.1122N NaOH per ml sample

since $C_W = k\ C_B$

$C_W = (8.92)(.0227) = 0.203$ ml of NaOH per ml sample

Thus for a 50 ml sample two equilibrium units are

50 x .203 = 10.15 ml of 0.1122N NaOH

BIBLIOGRAPHY

1. Comings, Trans. Am. Inst. Chem. Eng., 38, 143, (1942)
2. Brinsmade, Trans. Am. Inst. Chem. Eng., 39, 679, (1943)
3. Treybal, Trans. Am. Inst. Chem. Eng., 38, 203, (1942)
4. Colburn, Trans. Am. Inst. Chem. Eng., 32, 179, (1942)
5. Row, Trans. Am. Inst. Chem. Eng., 37, 559, (1941)
6. Alberton, Trans. Am. Inst. Chem. Eng., 39, 361, (1943)
7. Knight, Trans. Am. Inst. Chem. Eng., 39, 439, (1943)
8. Rushton, Ind. and Eng. Chem., 29, 309, (1937)
9. Dicks, Trans. Am. Inst. Chem. Eng., 41, 789, (1945)
10. Varterressian, Ind. and Eng. Chem., 28, 928, (1936)
11. Treybal, Ind. and Eng. Chem., 34, 709, (1942)
12. Elgin, Trans. Am. Inst. Chem. Eng., 31, 639, (1935)
13. Wehner, W. G., Master's Thesis, Lehigh University, (1946)
14. Elgin, Trans. Am. Inst. Chem. Eng., 38, 26, (1946)
15. Elgin, Trans. Am. Inst. Chem. Eng., 39, 23, (1947)