1964

The effect of unequal tray spacing on flooding capacties of a pulsed column

Richard A. Knazek
Lehigh University

Recommended Citation

Knazek, Richard A., "The effect of unequal tray spacing on flooding capacties of a pulsed column" (1964). *Theses and Dissertations*. 5037.
https://preserve.lehigh.edu/etd/5037

THE EFFECT OF UNEQUAL TRAY
SPACING ON FLOODING CAPACITIES
OF A PULSED COLUMN

by

Richard A. Knazek

A THESIS

Presented to the Graduate Faculty
of Lehigh University
in Candidacy for the Degree of
Master of Science

Lehigh University
1964

CERTIFICATE OF APPROVAL

This thesis is accepted and approved in partial fulfillment of the requirements for the degree of Master of Science.

(date)

Professor in Charge

Head of the Department

ACKNOWLEDGEMENTS

I would like to thank Professor Curtis W. Clump for his advice during the course of this investigation and Miss Virginia Edwards for typing the final draft of this paper. The suggestions and assistance of Joseph Hojsak and the chemical engineering graduate students were also deeply appreciated.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

ABSTRACT

The flooding capacities of a pulsed liquid-liquid extraction column were determined for three different plate arrangements. The TBP, kerosene-benzoic acid-water system was used to achieve flooding in a column that was one inch in diameter and four feet in length.

All runs were made with a 1.25 inch pulse amplitude over a frequency range of 14 to 76 cycles per minute.

The maximum permissible flow rate through a plate arrangement with uniform one-inch spacing was 700 gallons per hour per square foot but increased to 1500 gallons per hour per square foot when the plate spacing of the upper portion of the column was increased to two inches. The flooding capacity of the column with uniform two-inch spacing was observed to be nearly identical to that of the graded column.

INTRODUCTION

Removal of impurities from liquids may be effected by any one of many types of chemical processes. Distillation, evaporation, adsorption, and liquid-liquid extraction are just a few of the possibilities. Of course the choice of procedure depends mainly on the physical and chemical properties of the system involved and the amounts of material to be handled.

The advent of nuclear process industries and the subsequent demand for techniques to process radioactive fuels and wastes has led to an increased interest in liquid-liquid extraction processes. Unfortunately, standard liquid-liquid extraction procedures have two inherent disadvantages. The first of these is that a third component must be introduced to extract the undesired substance from the process stream. This introduces the burden of solvent recovery, if the third component is expensive, or its removal if the impurity is the desired product of the process. There is little that can be done to overcome this problem since it is the basis of all liquid-liquid extraction processes.

The second disadvantage incurred is the relatively inefficient behavior of all liquid-liquid extraction procedures. The height equivalent to a theoretical stage of an extraction operation may approach fifteen feet, whereas the HETS of a distillation operation may be as little as two feet. This vast difference in the efficiency of these processes stems from two phenomena.

The first of these phenomena is that the boiling action within a distillation column causes a repeated formation and condensation of vapor bubbles. This renewal of the interfacial surface area continuously exposes fresh surface to the continuous phase thereby increasing the effective inter-phase driving force, thus, increasing the overall efficiency of the operation. In fact, it has been shown by Sherwood, Evans, and Longeor (33) that as much as 50% of extraction from a single droplet occurs during the formation of the droplet. Others (20, 39) have since indicated that the percent extraction during droplet formation is closer to 20%. In either case though, the frequent reformation of droplets is shown to be quite desirable in all extraction processes.

The second reason for the large difference in efficiency between distillation and liquid-liquid extraction lies in the amount of mixing achieved within the column. In columns possessing no mechanical mixing devices, the only source of turbulence is the potential energy supplied by the density difference of the two phases (37). The large difference in densities between the liquid and vapor phases in distillation processes indicates that these operations are always exceedingly well mixed, whereas, the slight difference in density between phases in liquid-liquid extraction processes induces only a mild degree of mixing (40). That is, the buoyant force acting on the dispersed phase as it flows through the continuous phase is insufficient to provide a high degree of turbulence in liquid-liquid extraction processes.

Thus, the two main reasons for the inefficient operation of liquid-liquid extraction processes are: 1) inadequate mixing, and 2) infrequent droplet formation (31). However, these difficulties may be circumvented by increasing the amount of contact per unit energy available and/or supplying additional energy from an external source.

Whereas trays and packings have been used to achieve increased contact area for many years, little work had been done to increase mixing in liquid-liquid extraction operations prior to 1949. The Podbielniak centrifugal contactor (38), the Scheibel column (38), and the Van Dijck reciprocating plate column (14), use mechanically powered devices to achieve intimate mixing of the two phases. However, the presence of moving parts introduces maintenance problems - problems that are especially undesirable if the process streams are of a corrosive or radioactive nature. However, in 1930 Van Dijck suggested that increased turbulence could also be achieved by pulsing the process liquids through stationary plates. The first unclassified performance data on such a unit were presented in 1953 by Cohen and Beyer (12). They showed that the pulsating action of the liquid streams did indeed reduce the height of a transfer unit by a factor of two. This apparatus, termed a "pulsed column" (8), has since received a great amount of attention, stemming from the demands of the relatively new field of nuclear processing. Production of high purity fuels and the subsequent processing of radioactive wastes requires liquid-liquid extraction procedures that entail a sizeable amount of equipment that must be shielded by some type of cubicle and operated by remote control. Clearly, any reduction in equipment size leads to considerable savings in the cost of the unit and simplifies the remote operation (35). The effect of pulsing has been shown to reduce the size of equipment needed to attain a given extraction, hence, its desirability in the nuclear processing field. Many articles have listed applications of pulsed columns to industrial scale nuclear processes (1,4,6,7,8,13,15,19, 20,21,23,24,32,34).

THEORY

General Operating Characteristics

Inspection of the counter-current flow patterns of pulsed sieve plate columns shows that there are four distinct modes of behavior: 1) mixer-settler, 2) emulsion-type, 3) unstable, and 4) flooding. Operation within any one of these regions depends upon the rate of flow and the amount of pulsation applied. A qualitative description of this dependence is presented in Figure 1 (31) and described more completely in the following paragraphs.

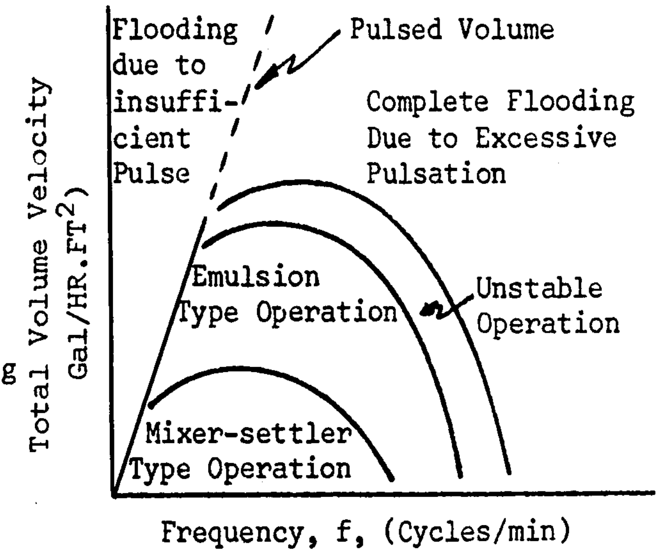


Figure 1

Mixer-settler-type Operation

Mixer-settler operation occurs at low throughput rates and mild pulsations. It is characterized by the coalescence of both phases to form two distinct layers in the inter-plate region during the quiescent portions of the pulse cycle. That is, during the extreme portions of the pulse stroke a layer of the light phase forms beneath the plates while a layer of heavy phase rests directly beneath it and on top of the plate below. The formation of these layers is possible since the plate perforations are too small to permit flow between adjacent inter-plate regions. As illustrated in Figure 2, the upward pulse stroke forces the light phase layer through the plate in the form of droplets that rise through the heavy phase layer and coalesce beneath the next plate during the quiescent portion of the stroke. The reverse process is obtained during the downstroke portion of the cycle; the heavy phase being drawn downward through the plate in droplet form and settling atop the plate below. This procedure is repeated with each pulse cycle and causes a net counter-current flow of the two phases through the column.

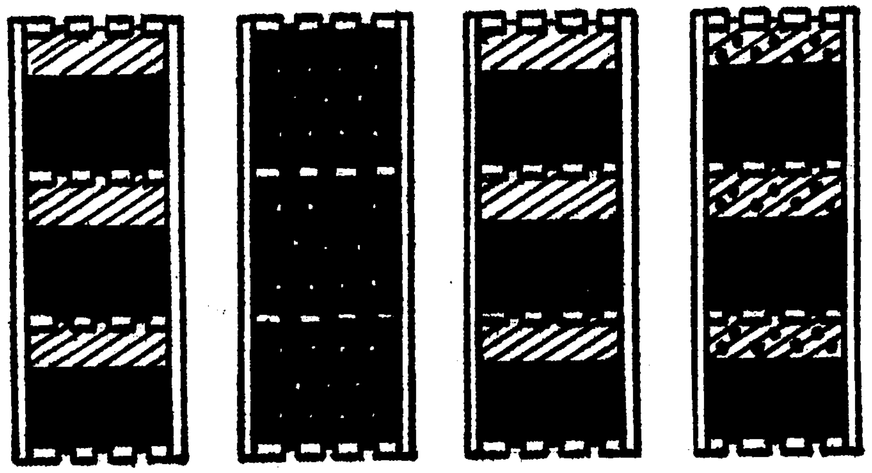


Figure 2

Although this type of operation is very stable, it is quite inefficient since little transfer occurs during the quiescent portion of the pulse. The inefficiency of this mode of operation serves to reiterate a statement made in an earlier segment of this paper. Although the time period of droplet dispersion in a pulsed column operating in the mixer-settler region is shorter than that of a spray tower of comparable size, an increase in efficiency is obtained when the pulsed column is used. This, then, must result from the more turbulent nature of the flow and continual exposure of fresh inter-phase surface by the repeated formation of dispersed phase droplets (12). While this operation is more efficient than standard liquid-liquid extraction techniques, it is not the most efficient form of pulsed column operation and should be avoided.

Emulsion-type Operation

Emulsion-type operation occurs at higher throughputs and stronger pulsing conditions than does the mixer-settler type of operation. It is characterized by a fairly uniform dispersion of small droplets that do not coalesce until reaching the terminal portions of the column. These droplets are repeatedly forced through the plates as they move up and down the column causing considerable deformation and internal agitation of the droplet. In addition, the passage of the dispersed phase through the plate perforation tends to strip off any stagnant film of continuous phase that might adhere to the droplet (4). Thus, with the great amount of interfacial contact area per unit volume and highly turbulent flow, an efficient operation would be expected in this region. Indeed this is the case: the highest efficiencies of pulsed column operation are obtained within the emulsion-type region.

Unstable Operation

Unstable operation of a pulsed sieve plate column occurs at still higher flow rates and pulse conditions. Indication of this type of behavior is far more nebulous than the two aforementioned regions, in that the presence of any one of several phenomena may define instability. Mixtures of fine and coarse droplets, formation of irregularly shaped globules of dispersed phase, and periodic reversals of continuous phase are symptoms of unstable operation. These conditions both reduce the column efficiency and cause it to fluctuate widely during operations making it undesirable to utilize this mode of operation.

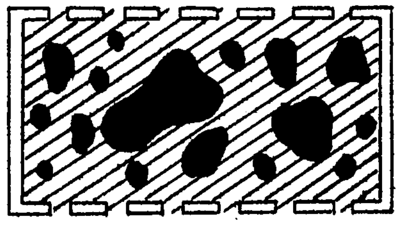


Figure 3

Flooding

There are several types of flow behavior that constitute flooding of a pulsed column: flooding due to, 1) inadequate pulsation, 2) impairment of flow by a honeycomb-like plug of fine droplets, and 3) formation of emulsions.

The first of these, flooding due to inadequate pulsation, occurs when the frequency-amplitude product, or pulsed velocity, is insufficient to force a given flow through the column. When this occurs, there is a gradual buildup of light phase beneath the bottom plate and/or heavy phase above the top plate with an eventual emission of one or both of the feed streams from the column at the end opposite its intended point of exit. Edwards and Beyer (16) have proposed a model of inadequate pulsation and described it by the following equation:

$$G + L = V_p (\cosh \theta) + G/2$$

where: G = light phase flow rate, cc/min
L = heavy phase flow rate, cc/min
V_p = pulsed volume velocity, cc/min, $v \cdot f$
f = pulse frequency, cycles/min
v = pulse volume, the volume displaced during the pulse movement of the fluid contents of the column from one extreme position to the other, cc/cycle
$\theta = G/\pi V_p$

This correlation has been shown to be fairly accurate and quite useful when sizing equipment for construction of pulsed column units.

The second type of flooding occurs when the total throughput and/or pulsation is increased to a point where the turbulence of the system is sufficient to form an agglomeration of droplets within the extraction section of the column. Unlike those obtained in the emulsion-type of operation, these droplets do not rise through the column but rather stay in one section of the column and act as a resistance to the flow of the two streams. With their movement hindered, the phases collect on both sides of this plug - the light phase beneath it and the heavy phase above it. This increased hold-up causes a noticeable reduction in flow, and eventually, forces the light phase from the column via the heavy phase exit and the heavy phase through the light effluent line.

The third type of flooding is due to excessive emulsification of the process streams or primary interface. This phenomenon is characterized by extensive entrainment of the light phase in the heavy effluent and/or entrainment of the heavy phase in the light effluent. Like the above mentioned flooding, this type of behavior occurs due to intense mixing. The products are then in the form of colloidal dispersions that would require impractically large disengagement sections or other complex and cumbersome pieces of equipment to produce clear products.

It is quite apparent, then, that operation of any type of extraction equipment is impossible if flooding conditions prevail -- an indication that this mode of operation must be avoided at all times.

Because of the low efficiency of the mixer-settler region, it is highly improbable that there are any industrial applications that can utilize this mode of operation. For this reason, flooding due to inadequate pulsing appears to be of little interest and will be ignored in this investigation.

However, flooding of pulsed sieve plate columns due to intense mixing within the column can pose a major problem in extraction procedures. This is especially true if the system being processed is easily emulsified or if high flow rates are employed. Clearly, since flooding reduces the production capacity and lowers the effectiveness of an extraction unit, any technique that will alleviate this problem can be of significant value. The investigation of this particular problem is the subject of this paper.

Observation of emulsion-type operation shows that the variation of droplet size over a short length of column is negligible. However, inspection of the droplet dispersions near the top and bottom of the column reveals a marked difference in droplet size. Depending upon the system and choice of continuous phase, the dispersion will become more or less coarse as it travels through the column. Therefore, since an extraction column becomes more efficient as the interfacial surface area is increased, it becomes apparent that not all portions of the column operate with the same efficiency. It is desirable, therefore, to increase the degree of dispersions in other sections of the column and thereby increase the over-all efficiency of the column.

The coarsely dispersed region of operation may be refined by increasing either the strength of pulsation or the total flow rate. While the increased turbulence does achieve a finer dispersion, it also causes the previously acceptable dispersion to either emulse or to form a tight, immovable plug of droplets. In other words, an attempt to obtain a uniform dispersion throughout the column by an increase in turbulence is likely to result in flooding.

Since the flooding capacity of a column is controlled by the region of tight dispersion, any method that would lower the degree of dispersion without affecting other sections of the column should increase the operational limits. Geier (20) has indicated that this can be achieved if the regions of tight dispersions are allowed greater opportunity to coalesce. Thus, any revision of the column geometry that will reduce the turbulence of a tightly dispersed region should increase the flooding capacity of a pulsed column. This can be achieved in several ways: greater plate free area, larger hole diameters, and/or increased plate spacing.

Geier has stated that an increase in plate spacing does increase the flooding capacity of a column. However, since this statement was based only on two data points, any conclusions reached can only serve as a vague indication of operational behavior and disallow any far-reaching conclusions to be made. For this reason, a more thorough investigation was initiated to verify the trends indicated and conclusions reached by Geier. The description and results of this investigation are the subject of this paper.

APPARATUS

A schematic diagram of the extraction unit used in this investigation is shown in Figure 4 and its key presented in Table I.

Extraction Column The column consisted of a pyrex pipe, four feet in length with a one-inch inner diameter. The disengagement sections were one-to-two-inch glass bell reducers, six inches in length and fitted with teflon gaskets. The column was sealed at both ends with two sheets of stainless steel plates pierced only by the feed, product, and pulse lines.

Sieve Plates The sieve plates were made from 18 gauge stainless steel and perforated with 44 holes, 0.067 inches in diameter, to provide a 22 percent plate free area. The plates were strung on a stainless steel welding rod and separated by sections of 1/4-inch stainless steel tubing. Forty-five plates were used with one-inch plate spacing, thirty-eight plates were used in the graded column, and twenty-three plates were used with two-inch plate spacing. The hole arrangement is depicted in Figure 5.

Pulse Pump Sinusoidal pulsations were applied to the continuous phase by a Milton Roy Simplex Controlled Volume pump. The pump was powered by a variable speed motor having a frequency range of 10 to 76 cycles per minute. When used in conjunction with this column, the pulse amplitude could be varied from 0 to 1.25 inches within the extraction section. The pulse was exerted on the process fluids through a 1/4-inch stainless steel tube that barely pierced the bottom of the disengagement section. This held entrainment of the light phase in the pulse line to a minimum.

Feed Pumps A Milton Roy Duplex Proportioning pump served as the feed pump for the aqueous and organic streams. The maximum flow was 500 cc per minute for each feed stream. The flows were set by adjustment of the pump stroke length and monitored on the two rotameters mounted on the control panel based on Figures 7-10. Both feeds were kept in ten-gallon stainless steel tanks and pumped through $2\frac{1}{2}$ gallon stainless steel surge tanks that partially removed the pulsations caused by the feed pumps.

Miscellaneous All lines for both the aqueous and organic streams were 1/4-inch stainless steel tubing with the exception of the aqueous effluent line which was teflon tubing.

The organic stream entered the column just below the bottom plate. This plate was situated two inches above the lower disengagement section to allow greater opportunity for coalescence of the organic entrained in the aqueous product. The organic product was withdrawn from the upper-most portion of the top disengagement section and stored in the ten-gallon stainless steel organic product tank.

The aqueous stream was supplied just above the top plate. It was taken off as aqueous effluent at the very bottom of the column through the jackleg, the function of which was to stabilize the position of the interface by balancing the static head in the column. The product was then stored in the ten-gallon stainless steel aqueous product tank.

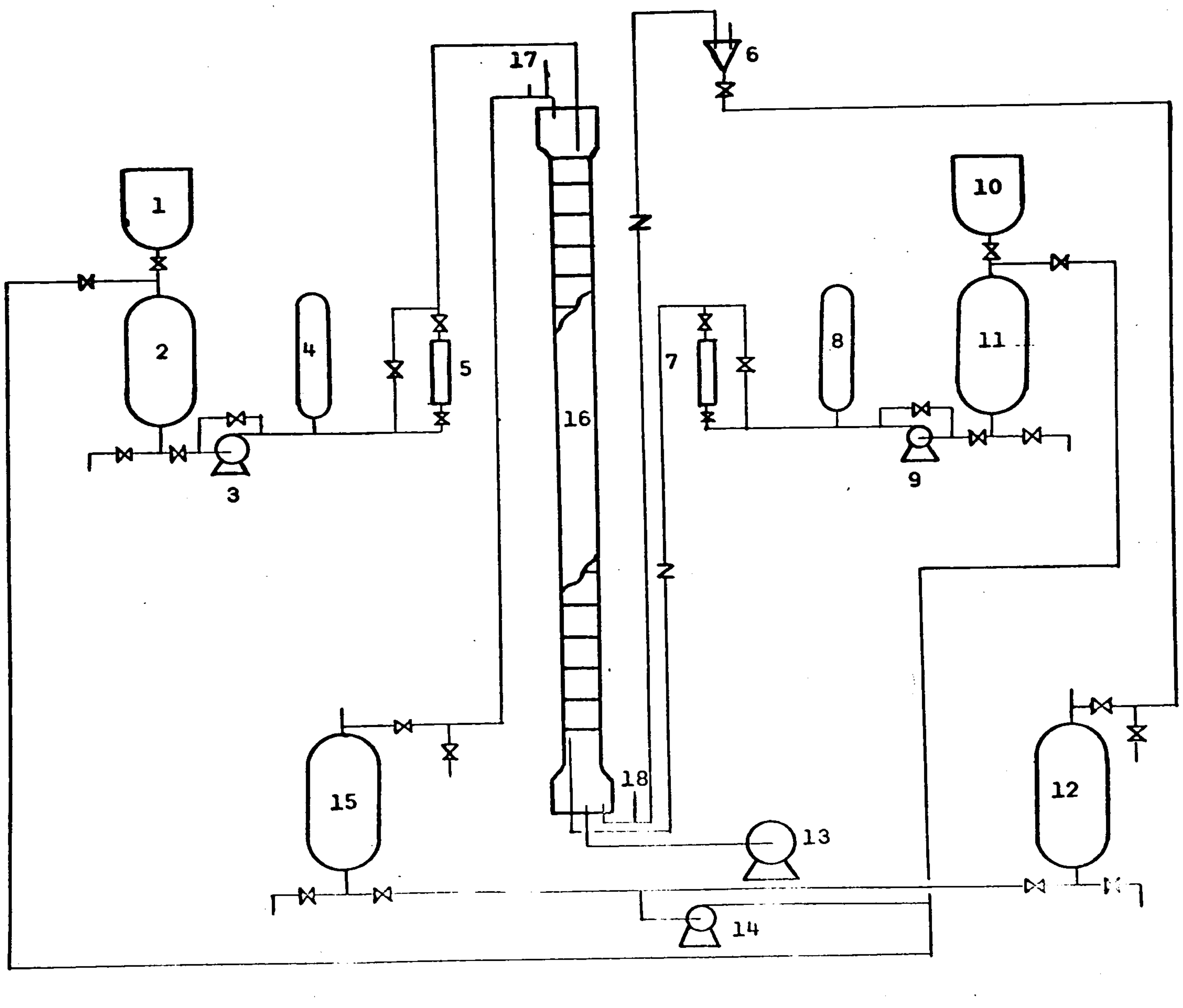
Materials A thirty percent solution of FMC tri-butyl-phosphate in Shell industrial grade kerosene was used as the basis of the organic feed. It was then acidified to 0.16 N with commercial grade benzoic acid and saturated with distilled water.

The aqueous feed used was distilled water previously saturated with kerosene and tri-butyl-phosphate.

The organic solution was used repeatedly but the aqueous product was discarded after each run.

PULSED EXTRACTION UNIT

Figure 4



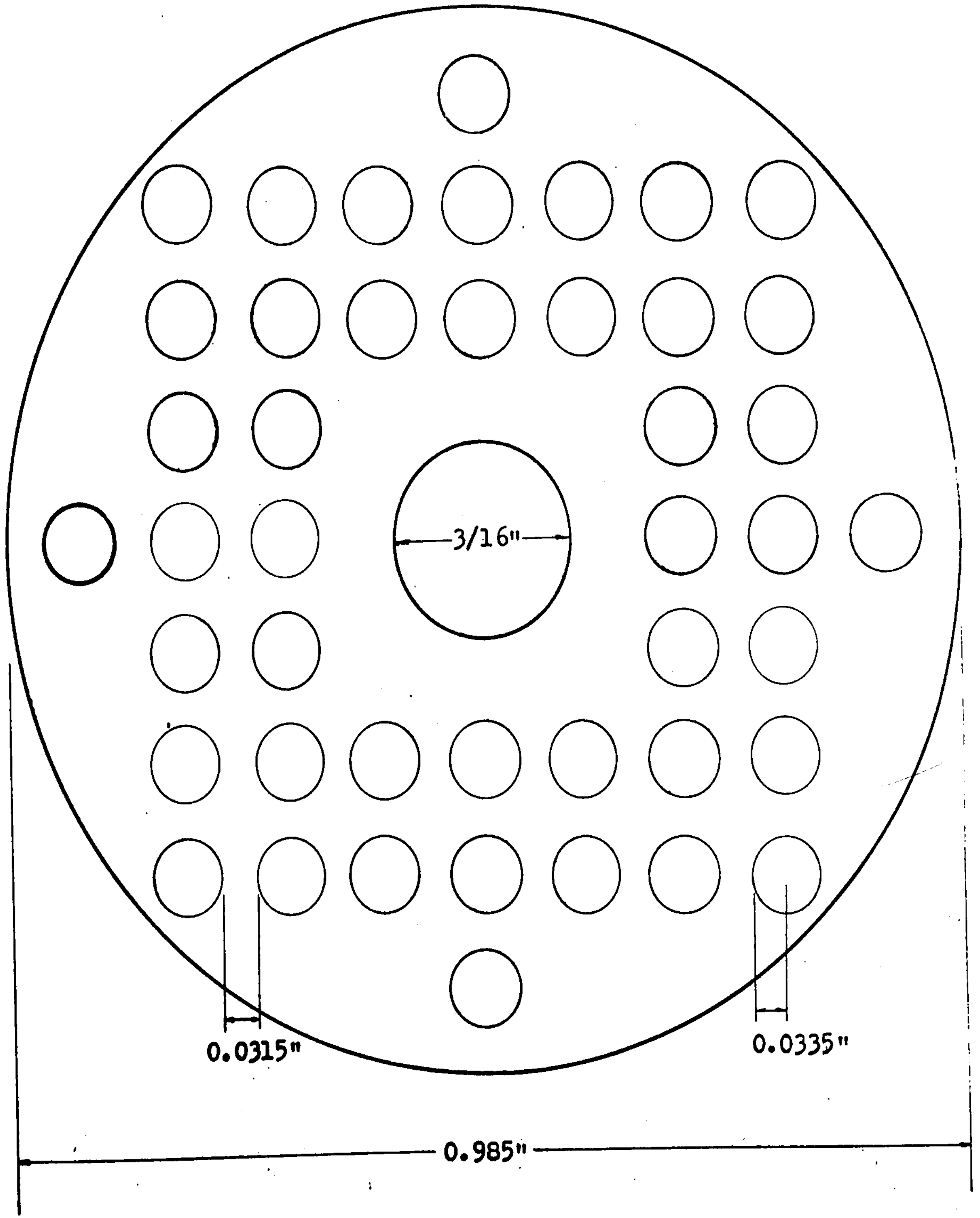
Key to Figure 4

TABLE I

1 - Aqueous mixing tank
2 - Aqueous feed tank
3 - Aqueous feed pump
4 - Aqueous surge tank
5 - Aqueous rotameter
6 - Primary interface controller (jack-leg)
7 - Organic rotameter
8 - Organic surge tank
9 - Organic feed pump
10 - Organic mixing tank
11 - Organic feed tank
12 - Aqueous product tank
13 - Pulse pump
14 - Auxiliary materials handling pump
15 - Organic product tank
16 - Extraction column
17 - Top thermocouple
18 - Bottom thermocouple

Diagram of Sieve Tray

Figure 5



PROCEDURE

A thirty volume percent solution of tri-butyl-phosphate in kerosene was prepared in the organic mixing tank. The specific gravity was measured with a Westphal Balance indicating whether more kerosene or TBP was added to provide a feed specific gravity equal to 0.855. Approximately three liters of distilled water were added to twenty liter portions of the organic and stirred vigorously for fifteen minutes in an attempt to obtain a saturated solution. Benzoic acid was then added to the solution until a normality of 0.16 was reached. The mixing tank was then covered and drained into the feed tank. The feed was allowed to stand for a half-hour after which the excess water was drained from the bottom of the feed tank.

The aqueous feed was prepared by saturating distilled water with one liter of the thirty percent TBP-kerosene solution in the aqueous mixing tank. This solution was then drained into the aqueous feed tank. The excess organic remained in the feed tank, requiring that the tank not be pumped dry during the operation.

The organic rotameter valve was closed and the aqueous phase rotameter valve opened completely. The organic pump dial was then set at zero, the aqueous pump dial set at 100, and the feed pump started.

After a small amount of the aqueous feed had collected in the bottom disengagement section, the pulsing unit was started and the desired frequency selected. When the column was filled with aqueous feed, the feed rates were set on the organic and aqueous pump controls (Figures 9 and 10), the organic phase rotameter opened completely, and the jackleg placed near the top of the column.

When the flow became constant, the jackleg was adjusted to keep the primary interface in the upper disengagement section, thereby maintaining the aqueous stream as the continuous phase.

The flows were kept constant during a run and their ratio as close to unity as possible; determined by measuring the volume obtained in a given length of time.

The time allotted for one run was fifteen minutes. If flooding did not occur during that run, the pulse frequency was increased and the procedure repeated. The frequency was measured and recorded for each run. Since the pulse amplitude was kept constant for all of the runs, only the frequency, flow, and description of the flow behavior were recorded.

After flooding was obtained within one series of runs the feed pump controls were changed to provide a different flow, the frequency lowered, and the entire procedure repeated.

When the organic feed tank emptied, the fluid in the organic product tank was pumped into the organic mixing tank via the auxiliary handling pump, brought up to the correct specific gravity and pH, and drained into the feed tank.

Since the aqueous product was discarded, the procedure in preparing additional aqueous feed was identical to the preparation of the initial feed.

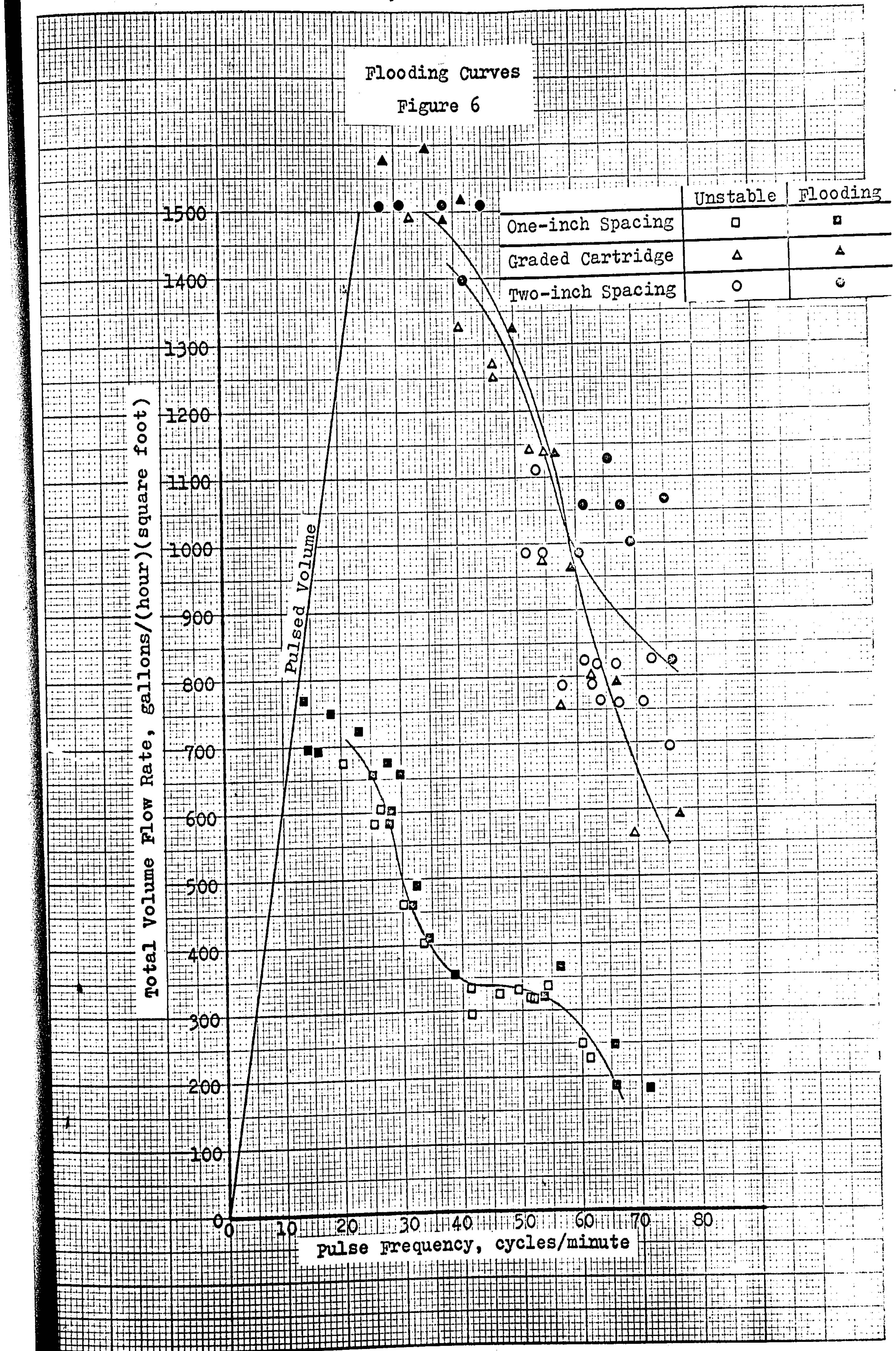
PRESENTATION AND DISCUSSION OF RESULTS

The purpose of this investigation was to observe the effect of plate spacing on the flooding capacity of a pulsed sieve plate column. This was achieved by determining the strength of pulsation required to achieve flooding at various flow rates. A complete series of runs covering the entire range of permissible operating conditions were made for three different plate arrangements and it is according to these arrangements that the runs are grouped.

The pulse amplitude was kept constant at 1.25 inches, the ratio of organic to aqueous flows close to unity, and the aqueous phase continuous for all runs.

The flooding curves obtained for the various plate arrangements are presented in Figure 6. The unfilled points indicate unstable operating regions while the areas to the left and right of the curve represent the stable and the flooding conditions. The data on which these curves are based are presented in Table II.

One-inch Spacing The first group of runs was made with a uniform plate spacing of one inch throughout the entire column that provided a total of 45 plates in the contacting section of the column. The flow was kept constant for each series of runs while the pulse frequency was slowly increased until the column became flooded. Enroute to the flooding point, the flow patterns progressed through the three modes of behavior: mixer-settler, emulsion-type, and finally the unstable type of operation.



Flooding Curves
Figure 6

The flooding observed in every series of runs within this group was caused by the formation of an immovable "plug" of droplets within the column. This, as described earlier, prevented the counter-current flow of the two feed streams and caused a build-up of the aqueous phase above and the organic phase below the "plug". This dispersion of droplets formed fifteen plates below the upper disengagement section for all of the flow rates investigated, indicating that if the column were "opened" above this point the flooding capacity would be increased due to the reduced turbulence within this region.

The maximum permissible flow for this particular plate arrangement was 700 gallons per square foot-hour at a frequency of 20 cycles per minute. The flooding capacity then dropped rapidly as the frequency of pulsing was increased to 40 cycles per minute. At this point the decrease in allowable flow became more gradual, tapering off to approximately 340 gallons per hour-square foot before dropping rapidly again when the pulse frequency reached 50 cycles per minute.

Each series of runs for the three plate arrangements formed an emulsed aqueous product at the bottom of the column when the pulse frequency reached 48 cycles per minute. This occurred at nearly the same frequency for all flow rates and was attributed to entrainment of the organic feed in the pulse line. Since this phenomenon depended on the location of the pulse line entrance rather than upon the plate arrangement, it was not considered flooding. It seems that the formation of this emulsion would be avoided if the lower disengagement section was lengthened to reduce the probability of organic feed being drawn into the pulse line.

Graded Cartridge Visual observations made during the runs described above indicated that removal of alternate plates in the section 15 inches below the upper disengagement section would raise the flooding capacity of the column. This would be a direct result of the reduced turbulence within this section of the column. Rearrangement was carried out using 30 one-inch and 7 two-inch plate spacers that provided 38 sieve plates in the contacting section of the column. The investigation of the flooding characteristics of this arrangement comprised the second group of runs.

The maximum flow through this unequally spaced plate arrangement, or graded cartridge, was 1500 gallons per square foot-hour, or more than twice the maximum flow rate attainable with the uniform one-inch plate spacing.

This result is in agreement with the trends indicated by Geier. It cannot, however, be compared quantitatively to the results of Geier because of differences in plate arrangement, column diameter, and column height.

As expected, the flooding capacity decreased rapidly as the pulse frequency was increased. However, the rate of change of decrease in allowable flow was more constant over the range of frequencies investigated. If the point of inflection observed in the first group of runs does occur with this plate arrangement, it does so at pulse frequencies in excess of those attainable with the equipment available for this investigation.

As in the first group of runs, the emulsion of the aqueous product occurred when the pulse frequency reached 48 cycles per minute, lending support to the thought that the emulsion was a result of organic entrainment within the pulse line.

The flooding observed in this group of runs was also caused by the build-up of a network of droplets to form an immovable "plug", the location of which was three plates below the "opened" section of the column.

Two-inch Spacing The third series of runs utilized a two-inch spacing throughout the entire column. The flooding curve obtained was, within experimental accuracy, the same as the curve obtained with the graded column. However, with this arrangement, flooding was not caused by a plug of droplets, but rather by an emulsion of aqueous phase in the organic product at the top plate. It appears that the replacement of the one-inch plate spacers by two-inch plate spacers again reduced the intensity of inter-phase mixing to an extent that the organic phase was required to travel a greater distance to become sufficiently dispersed and form a "plug" of droplets. In this case, the flooding occurred just above the top plate, with an emulsion-like mixture being formed in the upper disengagement section. Of course, this phenomenon prevented the maintenance of a stable interface and forced heavy entrainment of the aqueous stream in the organic product.

It is reasonable to expect then, that if the contacting section of this column had been longer, as was the Geier apparatus, the plug of droplets would have formed four feet from the bottom plate and caused the type of flooding observed in the first two groups of runs.

All runs mentioned above were made using an organic feed acidified to 0.16 N with benzoic acid. However, several trial runs were made using two-inch plate spacing and very low flows with an unadulterated TBP-kerosene feed. Stable operation was impossible even at the lowest frequencies because of a rapid build-up of a honeycomb-like network of organic bubbles throughout the entire column. Whether this was a result of a decrease in interfacial surface tension or, as Geier proposes, related to the amount of mass being transferred between phases, is impossible to ascertain since the organic feed normality was held constant throughout the investigation.

An investigation of the dependency of flooding characteristics upon feed composition would answer this question.

CONCLUSIONS

1. The flooding capacity of a pulsed sieve plate column will increase as the inter-plate spacing is increased.

2. The maximum operational flow through the uniform two-inch plate arrangement was more than twice the maximum flow obtained using a uniform one-inch plate spacing.

3. It is not necessary to increase the inter-plate spacing throughout the entire column to increase the flooding capacity. An increase of the inter-plate spacing in the region of tight dispersion will achieve the same result while still providing a large number of plates to enhance intra-phase mass transfer.

4. The flooding curves of the pulsed column for both the graded cartridge and the uniform two-inch arrangement are nearly identical.

5. Because of the nature of the 30 percent TBP in kerosene-water system, some substance must be added to the organic feed to stabilize the dispersed organic droplets. Lack of this stabilizing agent will produce honeycomb-like formations within the column making the column operation unstable.

RECOMMENDATIONS

1. A similar investigation should be carried out using the toluene-acetone-water system. While this system may not cause flooding as readily as the TBP, kerosene-benzoic acid-water system, it will certainly lend itself to straightforward and consistent product analyses. The equilibrium data for this system are presented by Othmer (29).

2. The toluene-acetone-water system should be used to gather more extensive HTU data for graded columns ranging from a uniform two-inch plate spacing to a uniform one-inch plate spacing. This will allow the advantages of higher flooding capacity to be weighed against the subsequent decrease in extraction efficiency as more and more sieve plates are removed from the column.

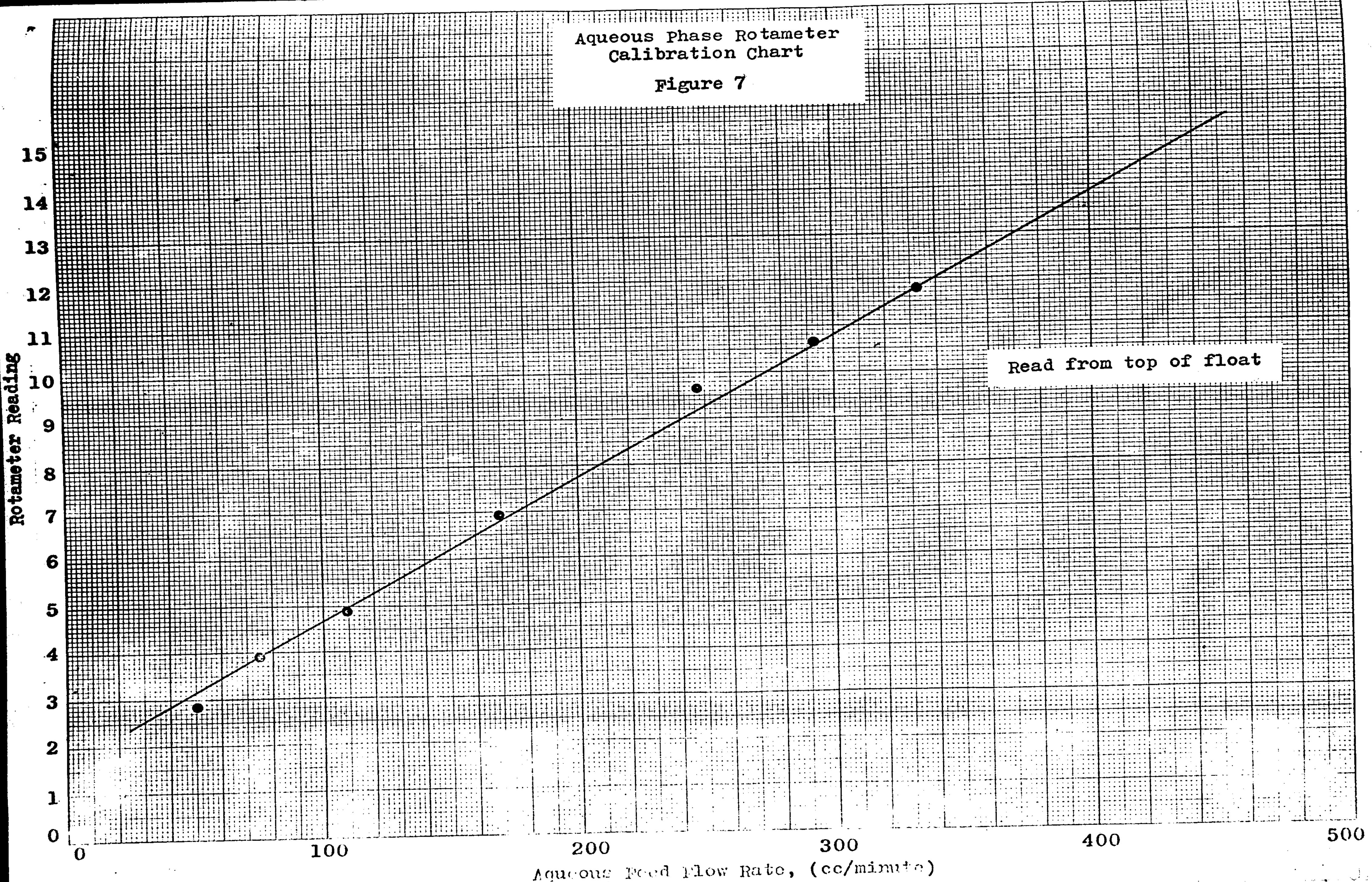
3. Flooding curves should be prepared for organic feeds of various compositions to relate flooding characteristics to either the amount of intra-phase mass transfer or physical properties of the feed.

4. Variation of flow during a series of runs was not uncommon. This problem would be alleviated and more rapid equilibration would be attained if the feed streams

were gravity fed via constant head feed tanks rather than pumped through the surge tanks employed.

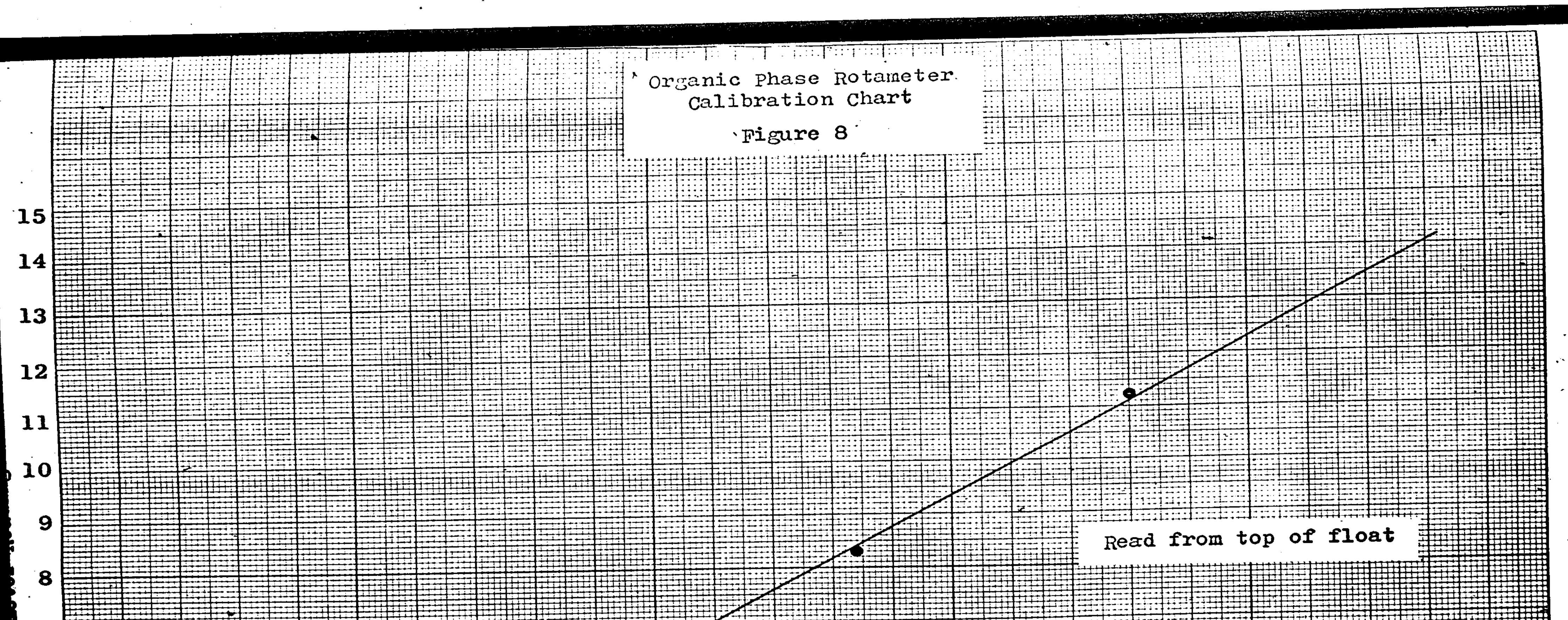
5. Longer disengagement sections should replace those now in use. This would allow greater ease in maintaining the primary interface and reduce the likelihood of organic entrainment in the aqueous product at high pulse frequencies.

APPENDICES



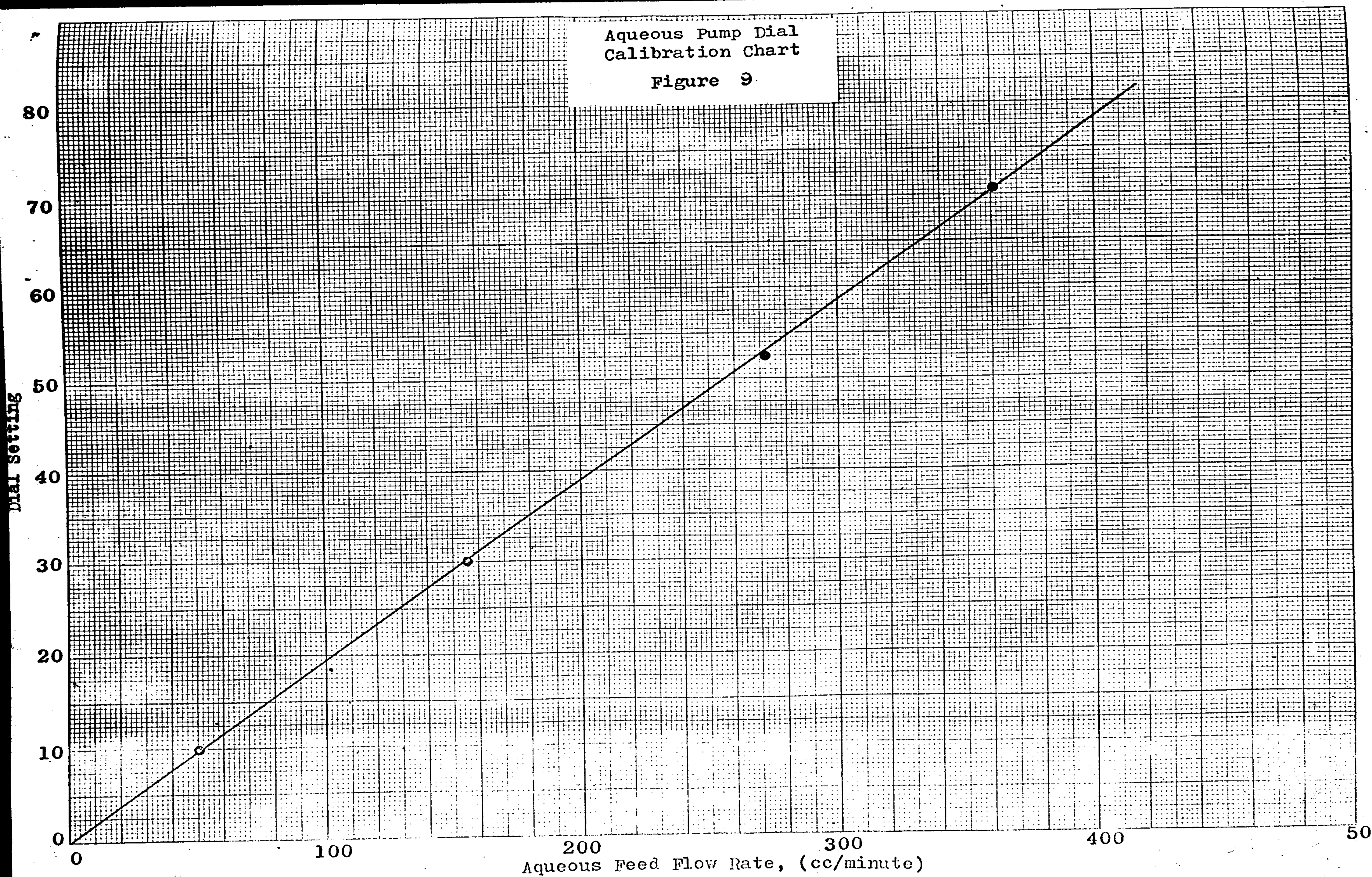
Aqueous Phase Rotameter Calibration Chart

Figure 7



Organic Phase Rotameter Calibration Chart

Figure 8



Aqueous Pump Dial Calibration Chart

Figure 9

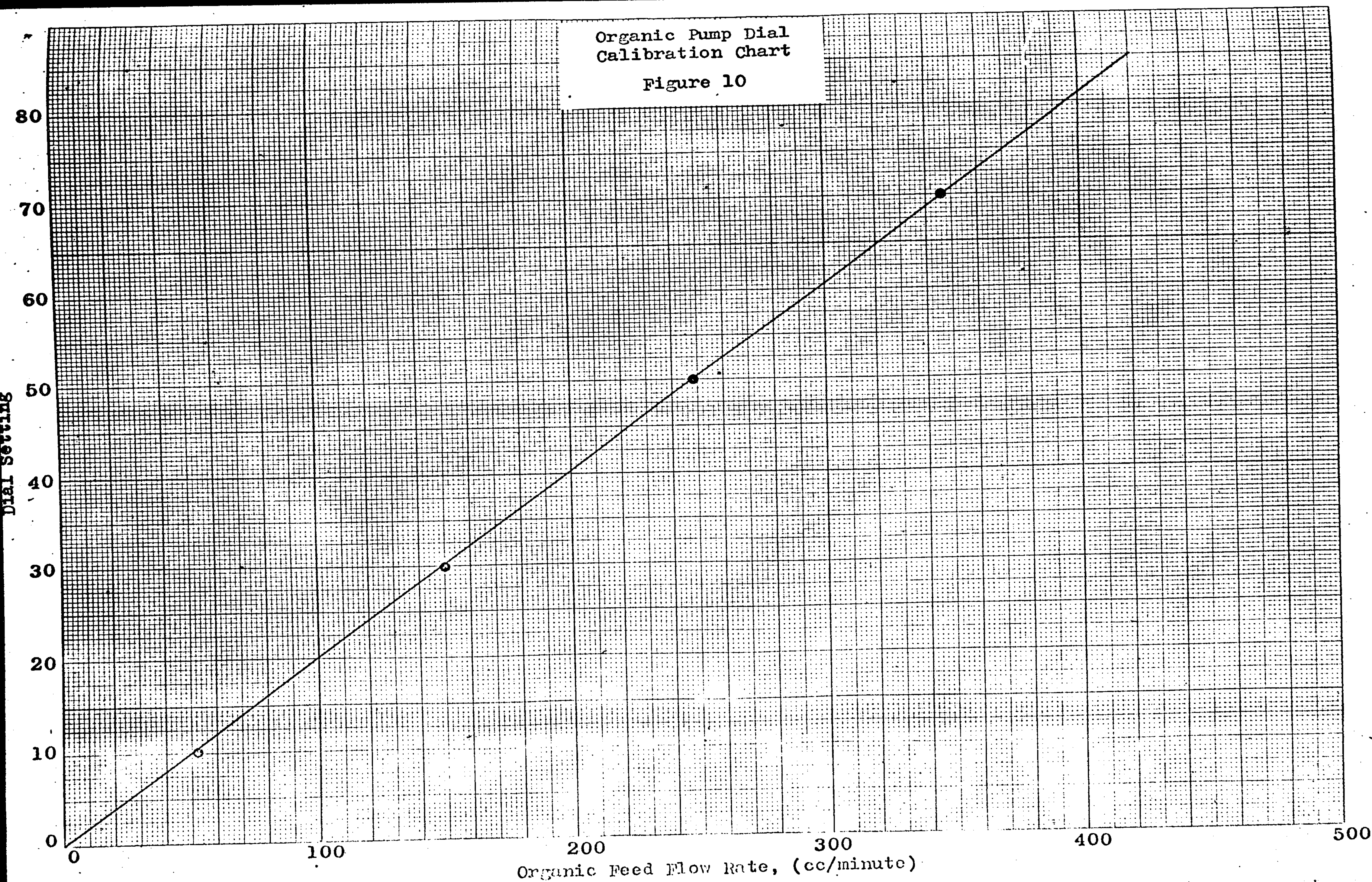
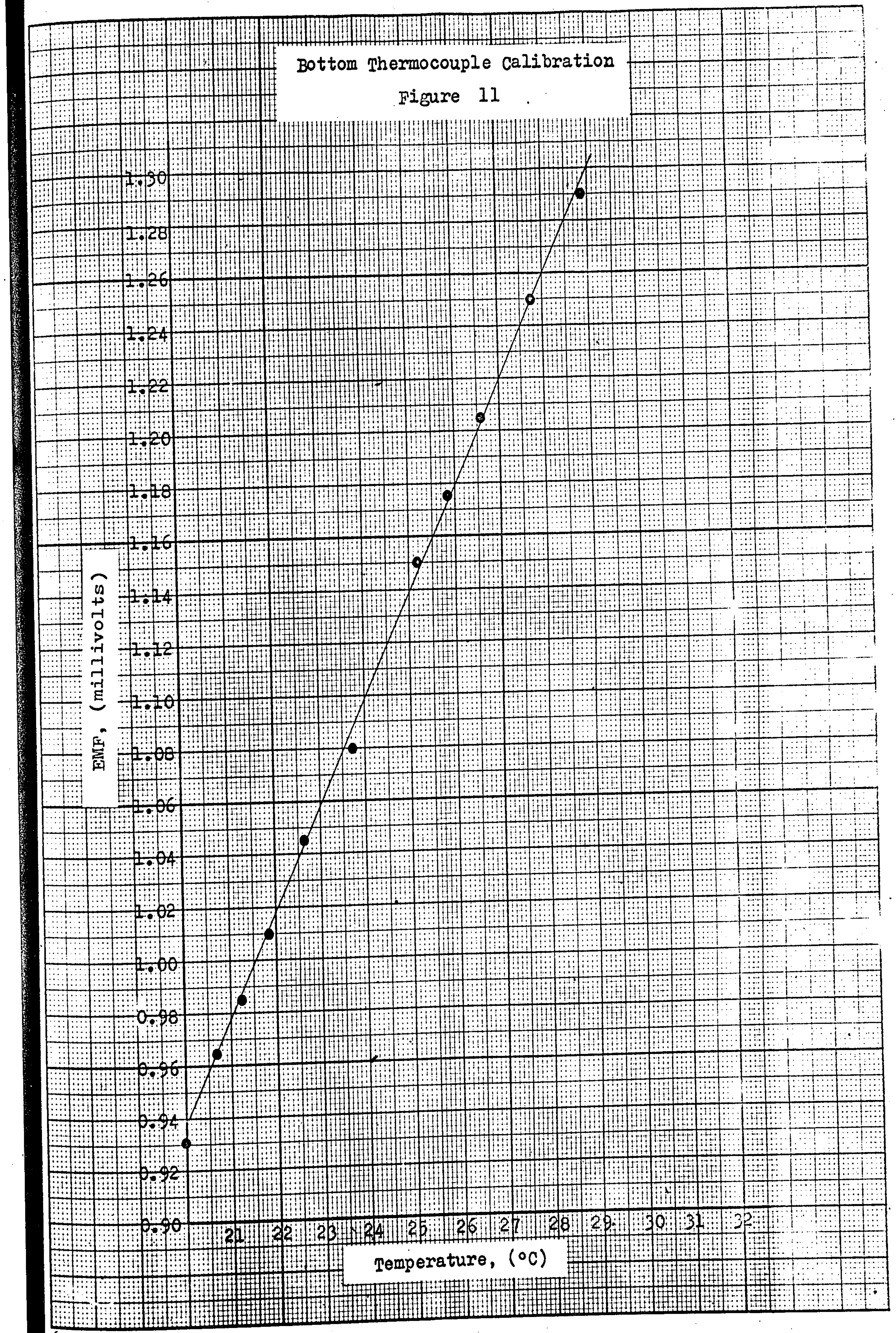
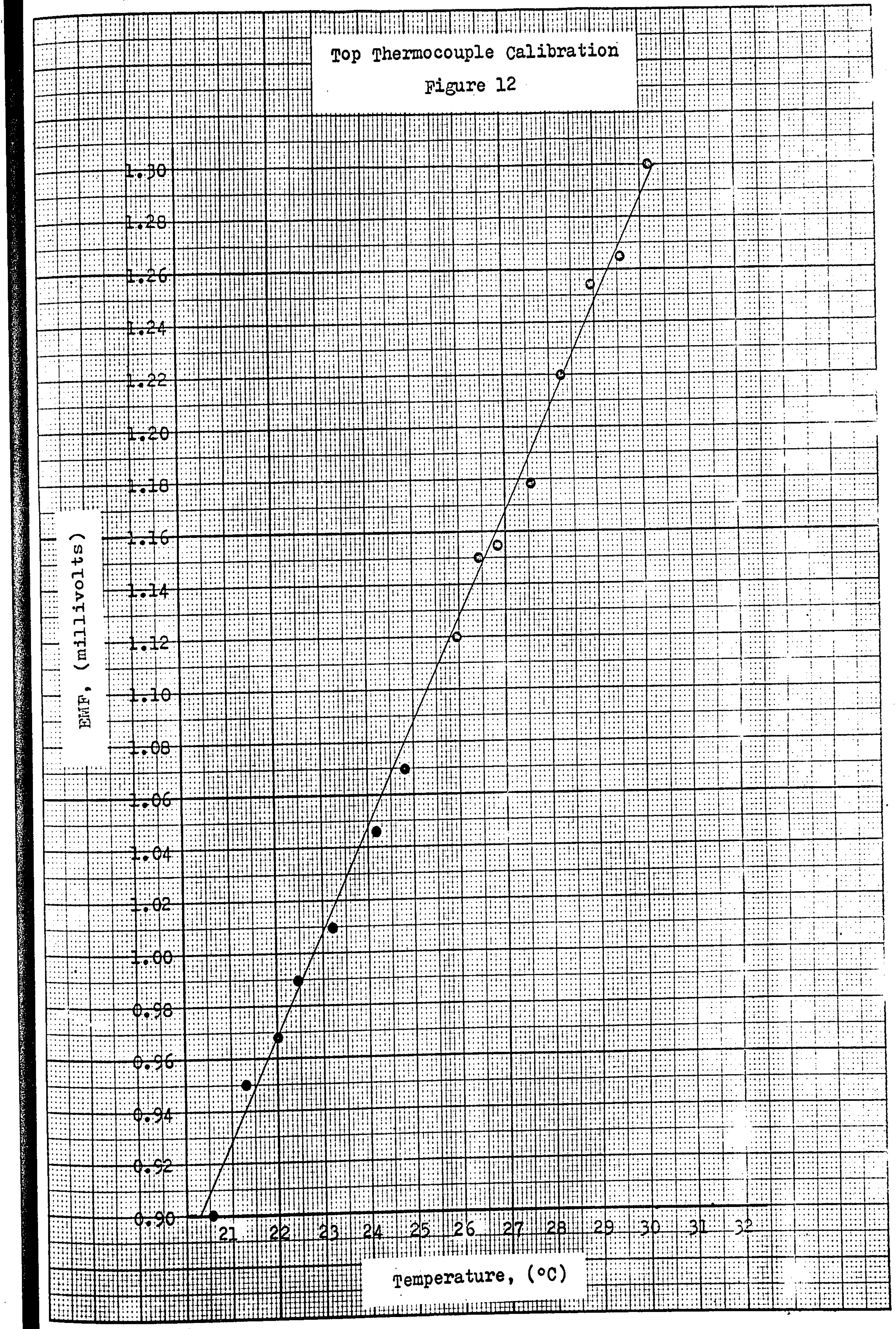

Organic Pump Dial
Calibration Chart

Figure 10



Bottom Thermocouple Calibration
Figure 11
EMF, (millivolts)
1.30
1.28
1.26
1.24
1.22
1.20
1.18
1.16
1.14
1.12
1.10
1.08
1.06
1.04
1.02
1.00
0.98
0.96
0.94
0.92
0.90
21
22
23
24
25
26
27
28
29
30
31
32
Temperature, (°C)



Top Thermocouple Calibration

Figure 12

FLOODING DATA

Table II

Run	f	Flow	*	Run	f	Flow	*	Run	f	Flow	*
2	30.9	456	S	6h	44.4	164	S	8a	42.5	216	S
2a	38.5	468	S	6i	50.8	199	S	8b	53.1	202	S
2b	48.4	468	F	6j	50.8	246	S	8c	61.2	228	U
2e	81.1	392	F	6k	54.1	249	S	8d	71.8	181	F
2f	41.1	292	U	6l	60.0	249	U	8e	65.9	187	F
3	25.2	658	F	6m	65.2	249	F	8f	38.2	333	S
3a	30.0	658	F	7	32.9	357	S	8g	44.8	333	S
3b	26.3	1047	F	7a	38.6	357	F	8h	49.4	322	U
3c	23.0	1047	F	7b	33.2	401	U	8i	51.7	322	U
4	18.3	503	S	7c	34.3	403	F	8j	52.2	322	U
4a	22.9	503	S	7d	34.3	363	S	8k	53.6	322	F
4b	27.6	491	S	7e	37.0	322	S	15	38.5	860	S
4c	32.9	491	F	7f	40.0	322	S	15a	42.8	965	S
4d	34.4	977	F	7g	46.1	322	U	15b	32.6	1491	U
4e	30.3	977	F	7h	49.2	333	U	15c	38.2	1491	F
4f	22.5	977	S	7i	52.2	333	U	15d	28.0	1579	F
4g	13.5	772	F	7j	54.5	339	U	15h	25.1	854	S
4h	13.5	713	S	7k	56.1	363	F	15i	33.0	763	S
4i	13.5	643	S	7l	21.3	579	S	15j	44.8	631	S
4j	18.3	749	F	7m	24.0	579	S	15k	52.2	631	S
4k	15.9	696	F	7n	27.9	585	F	15l	58.3	567	S
4l	14.6	696	F	7o	22.6	585	S	15m	69.8	567	U
4m	13.5	649	S	7p	25.4	579	U	15n	76.9	591	F
5	24.2	444	S	7q	20.6	602	S	15o	47.6	684	S
5a	26.4	444	S	7r	24.4	602	S	15p	53.6	784	S
5b	28.6	462	S	7s	26.8	608	U	15q	57.7	760	U
5c	30.2	462	U	7t	28.0	608	F	15r	62.5	801	U
5d	31.6	462	F	7u	20.4	678	U	15s	66.7	792	F
6	35.7	339	S	7w	13.9	836	F	15t	41.4	947	S
6a	41.7	339	U	7x	10.3	690	S	15u	51.3	982	S
6b	41.7	257	F	7y	22.9	620	S	15v	54.5	977	U
6c	33.1	211	S	7z	25.0	620	S	15w	59.4	965	F
6d	43.8	199	S	7a'	28.0	678	F	15x	44.3	1117	S
6f	38.7	175	S	7b'	23.0	725	F	15y	48.0	1117	S
6g	38.7	231	S	7c'	20.3	725	S	15z	50.0	1117	S
				8	38.7	216	S	15a'	52.2	1140	U

Run	f	Flow	*
15b'	55.0	1140	U
15c'	56.6	1135	F
15d'	42.9	1251	S
15e'	46.5	1251	U
15f'	46.5	1275	U
15g'	50.0	1327	F
15h'	40.5	1327	U
15i'	41.7	1520	F
15j'	35.9	1597	F
15k'	35.3	1597	F
15l'	29.0	1743	F
1	33.0	322	S
1a	44.4	322	S
1b	58.2	322	S
1c	65.2	322	S
1d	75.0	322	S
1e	75.9	322	S
1f	55.0	462	S
1g	61.2	462	S
1h	51.2	474	S
1i	58.2	468	S
1j	65.9	468	S
1k	73.2	444	S
1l	75.2	456	S
1m	55.0	567	S
1n	61.8	567	S
1o	64.5	567	S
1p	41.6	795	S
1q	48.4	795	S
1r	54.0	789	S
1s	57.7	789	U
1t	62.5	789	U
1u	63.8	760	U
1v	66.7	760	U
1w	71.4	760	U
1x	75.9	696	U
2	48.3	947	S
2a	53.1	947	S
2c	61.9	1059	F
2d	67.4	1059	F
2i	65.9	1129	F
2m	67.3	1053	F
2n	75.4	1064	F
3	35.3	988	S
3b	46.5	988	S
3c	51.2	988	U
3d	54.5	988	U
3e	60.6	988	F
3g	61.2	825	U
3h	63.9	825	U
3i	66.6	825	U
3j	72.4	825	U
3k	75.9	825	F
3l	53.0	1111	F
3m	41.9	1398	F
3o	38.2	1509	F
3p	31.6	1509	F
3q	27.6	1509	F
4	69.8	1000	F
4a	44.5	1510	F

* = Description of Operating Mode: S - stable, U - unstable, F - flooding.

f = Frequency, cycles/minute

Flow = Total Flow Rate, Gallons/(Hour)(Square Foot)

TRIBUTYL PHOSPHATE SPECIFICATIONS

TABLE III

Manufacturer	FMC Corporation
Specific Gravity @20°C/20°C	0.978 ± 0.003
Moisture (A.S.T.M. D95-46)	Maximum 0.2%
Color A.P.H.A. Pt-Co (A.S.T.M. D 268-46/4B). . .	Maximum No. 50
Odor	Normal; characteristic
Acidity (A.S.T.M. D 268-46/16) . . .	Maximum 0.05% as acetic acid (0.03% as phosphoric acid)

BIBLIOGRAPHY

1. Adams, H. W., et. al., U.S. Atomic Energy Commission Report HW - 33053 (1954).

2. Behmoiras, J., K. J. Bril, and P. Krumholz, Industrial and Engineering Chemistry Process Design and Development 1, #1 "Performance of Pulsed Sieve-Plate Extraction Columns during the Separation of Uranium from Thorium", 64 (1962).

3. Belaga, M. W. and J. E. Bigelow, AEC Declassified Report KT-133, (1952).

4. Benedict, M. and T. H. Pigford, Nuclear Chemical Engineering, McGraw-Hill, New York, (1957).

5. Biery, J. C. and D. R. Boyland, Industrial and Engineering Chemistry Fundamentals 2, #1, "Dynamic Simulation of Liquid-liquid Extraction Column", 44 (1963).

6. Bril, K., S. Holger, and B. Rethy, Analytical Chemistry 31, 1353 (1959).

7. Bril, K. and P. Krumholz, Interamerican Symposium Peaceful Application Atomic Energy, 3rd, Brazil T/022 (1960).

8. Burns, W. A., C. Groot, and C. M. Slansky, U.S. Atomic Energy Commission Report HW-14728 (1949).

9. Chantry, W. A., R. L. Van Berg, and H. F. Wiegandt, Industrial and Engineering Chemistry 47, #6, "Application of Pulsation to Liquid-liquid Extraction", pp. 1153-9, (1955).

10. Chemical Engineering, Oct., 1955, p. 112.

11. Choffe, B. and Y. L. Gladel, Journal of Applied Chemistry 8, "Liquid-liquid Extraction: Efficiency of a Perforated-plate Pulsed Column", 580 (1958).

12. Cohen, R. M. and G. H. Beyer, Chemical Engineering Progress 49, 279 (1953).

13. Cooper, V. R. and M. T. Walling, Jr., Proceedings of the U.N. Conference on the Peaceful Uses of Atomic Energy, 2nd, Geneva, 17, pp. 291-323, (1958).

14. Dijck, W. J. van, U.S. Patent 2,011,186 (1935).

15. Durandet, J., D. Defives, B. Choffe, and Y. L. Gladel, Proceedings of the U.N. Conference on the Peaceful Uses of Atomic Energy 2nd, Geneva, 17, pp. 180-191 (1958).

16. Edwards, R. B., and G. H. Beyer, AIChE Journal 2, #2, "Flooding Characteristics of a Pulse Extraction Column", (1956).

17. Ellison, C. V., U.S.A.E.C. Report ORNL-912, (1951).

18. Foust, A. S., L. A. Wenzel, C. W. Clump, L. Maus, and L. B. Andersen, Principles of Unit Operations, John Wiley & Sons, Inc., New York, (1960).

19. Geier, R. G., U.S.A.E.C. Report TID-7534, 1, 107 (1957).

20. Geier, R. G., Proceedings of the U.N. International Conference on the Peaceful Uses of Atomic Energy, 2nd, Geneva, 17, 192-9 (1958).

21. Harrington, C. D., and A. E. Ruehle, Uranium Production Technology, Van Nostrand, Princeton, N.J., (1959).

22. Jealous, A. C., and H. F. Johnson, Industrial and Engineering Chemistry 47, "Power Requirements for Pulse Generation in Pulse Columns", #6, p. 1159, (1955).

23. Krumholz, P., K. Bril, and N. U. Camargo, U.S.A.E.C. Report LPO-1, (1959).

24. Lang, G. P., U.S.A.E.C. Report MCW-1373, (1955).

25. Li, W. H., and W. M. Newton, AIChE Journal 3, "Liquid-liquid Extraction in a Pulsed Perforated-plate Column", 56, (1957).

26. Licht, W., and J. B. Conway, Industrial and Engineering Chemistry, 42, 1151, (1950).

27. Logsdail, D. H., and J. D. Thornton, Transactions of the Institute of Chemical Engineers, 35, "The Effect of Column Diameter upon the Performance and Throughput of Pulsed Plate Columns", 331, (1957).

28. McGair, Gilbert, "The Design and Testing of a Pulsed Sieve Plate Extraction Column", Master's Thesis, Lehigh University, (1960).

29. Othmer, D. F., R. E. White, and E. Truger, Industrial and Engineering Chemistry 33, 825, (1941).

30. Sehmel, G. A., and A. L. Babb, Industrial and Engineering Chemistry, Process Design and Development, 2, #1, "Holdup Studies in a Pulsed Sieve-Plate Solvent Extraction Column", 38 (1963).

31. Sege, G., and F. N. Woodfield, Chemical Engineering Progress, 50, "Pulse Column Variables", 396 (1954).

32. Sege, G., and F. N. Woodfield, Chemical Engineering Progress Symposium Series No. 13, 14, (1954).

33. Sherwood, T. K., J. E. Evans, and J. V. A. Longeor, Industrial and Engineering Chemistry, 31, 1144 (1939).

34. Shelton, S. M., E. D. Dilling, and J. H. McClain, Proceedings of the International Conference on the Peaceful Uses of Atomic Energy, 8, "Zirconium Metal Production", p. 505, United Nations, New York, (1956).

35. Sobotik, R. H., and D. M. Himmelblaw, AIChE Journal 6, #4, "The Effect of Plate Wetting Characteristics on Pulse Column Extraction Efficiency", p. 619-24, (1960).

36. Stephenson, R., Pulse Columns, presented at Toronto Meeting of the AIChE, April 26-29, 1953.

37. Thorton, J. D., Transactions of the Institute of Chemical Engineers, 35, "The Effect of Pulse Wave-form and Plate Geometry on the Performance and Throughput of a Pulsed Column", 316, (1957).

38. Treybal, R. E., Mass Transfer Operations, McGraw-Hill Book Company, New York City, (1955).

39. West, F. B., P. A. Robinson, A. C. Morgenthaler, Jr., T. R. Beck, and D. K. McGregor, Industrial and Engineering Chemistry, 43, 234, (1950).

40. Wiegandt, H. F., and R. L. Van Berg, Chemical Engineering, "Key to Better Extraction", p. 183, (1954).

VITA

The author was born on March 23, 1942, the son of Aloysius E. and Agnes B. Knazek. While a resident of Cleveland, Ohio, he attended Saint Joseph High School, graduating in 1958. The next four years were spent at Case Institute of Technology where he received a Bachelor of Science degree in chemical engineering in 1962.