

LIQUID-LIQUID EXTRACTION
IN A CENTRIFUGAL PUMP

A THESIS

Presented to

the Faculty of the Division of Graduate Studies
Georgia Institute of Technology

In Partial Fulfillment
of the Requirements for the Degree
Master of Science in Chemical Engineering

by

Jackson Arthur Ransohoff

June 1951

Cleveland
7/10

LIQUID-LIQUID EXTRACTION
IN A CENTRIFUGAL PUMP

Approved:

[Handwritten signature]

Date Approved by Chairman *May 30, 1951*

ACKNOWLEDGEMENTS

For his aid and guidance in the prosecution of this work, I wish to express my sincere appreciation to Professor Wm. Meese Newton. I should also like to thank Professor F. O. Nottingham, of the School of Electrical Engineering, for designing and providing the system for measuring impeller speeds; and Messrs. C. L. Brown, J. L. Chaille, C. E. Collum, R. W. Galphin, H. M. Gammage, J. H. Gore, D. E. Griffith, D. L. Lowi, G. E. Mansfield, H. A. McGee, L. C. Thayer, and J. G. Spraul for their help in obtaining the data.

TABLE OF CONTENTS

| | |
|---|-----|
| Acknowledgements | iii |
| List of Tables | v |
| List of Figures | vi |
| Summary | 1 |
| Chapter | |
| I. Introduction | 2 |
| II. Development of a Criterion for Mixing | 16 |
| III. Equipment, Materials, and Operating Procedure | 20 |
| IV. Calculation of Results | 26 |
| V. Results and Discussion | 29 |
| VI. Conclusions | 32 |
| Appendix I. Tables | 33 |
| Appendix II. Curves | 49 |
| Appendix III. Sample Calculation | 58 |
| Suggestions for Further Work | 60 |
| Bibliography | 63 |

LIST OF TABLES

| | Page |
|--------------------------------------|------|
| Table I. Experimental Data | 33 |

LIST OF FIGURES

| Figure | Page |
|--|------|
| 1. Schematic Drawing of Experimental System | 21a |
| 2. Front View of Experimental System | 21b |
| 3. Top View of Experimental System | 21c |
| 4. Impellers | 22a |
| 5. Orifice Calibrations | 49 |
| 6. Audio Frequency Oscillator Calibration | 50 |
| 7. Equilibrium Curves For Benzoic Acid Distributed Between Toluene and Water | 51 |
| 8. Relative Area Produced at Zero RPM vs Flow Rate . | 52 |
| 9. Relative Area Produced by Impeller No. 1 at a Flow Rate of $0.00075 \text{ Ft}^3/\text{Sec.}$ vs Impeller RPM . . | 53 |
| 10. Relative Area Produced by Impeller No. 2 at a Flow Rate of $0.00075 \text{ Ft}^3/\text{Sec.}$ vs Impeller RPM . . | 54 |
| 11. Relative Area Produced by Impeller No. 3 at a Flow Rate of $0.00075 \text{ Ft}^3/\text{Sec.}$ vs Impeller RPM . . | 55 |
| 12. Relative Area Produced by Impeller No. 4 at a Flow Rate of $0.00075 \text{ Ft}^3/\text{Sec.}$ vs Impeller RPM . . | 56 |
| 13. Average Relative Area Produced by Impeller No. 1 at Varied Flow Rates vs Impeller RPM | 57 |
| 14. Proposed Liquid-liquid System Using Centrifugal Pumps Driven From One Shaft as Mixing Stages . . . | 62 |

SUMMARY

Using a system of benzoic acid distributed between toluene and water, the affect of a centrifugal pump upon mixing was studied. It was found that, contrary to popular conception, equilibrium in a liquid-liquid system can be attained easily in one pass through a centrifugal pump, and it is suggested that knowledge of impeller design be applied toward designing an impeller which would make use of such a pump in liquid-liquid extraction processes economical.

From the mass transfer equation, a relationship was developed which enabled calculation of the effective interfacial area produced by the pump, and it was proposed that this area be used as a criterion for effective mixing. Using this concept, mixing was found to be a function of the impeller speed cubed. This relationship, when combined with the fact that power is approximately a function of the cube of the impeller speed, supports the concept that mixing accomplished is a linear function of power consumption.

I

INTRODUCTION

Although liquid extraction has received much attention recently, due primarily to the widespread use of solvent extraction in the production of lubricating oils, it is by no means a new field. It is rather one of the older of the chemical engineering process methods, growing from the leaching operations long used in winning many metals from their ores, and the extraction processes employed by the sugar beet industry.

Liquid extraction may be subdivided into two major categories; solid-liquid extraction, where a substance is dissolved from a solid by a suitable solvent, and liquid-liquid extraction, where a solute is transferred from one liquid to another. The extracting liquid is called the extract, and the other liquid the raffinate.

Fundamental Concepts

In accordance with W. G. Whitman's (1) two film concept, whenever two liquid phases or a gas and a liquid phase are in contact, there are two fluid films present, one on each side of the interface. It is believed that equilibrium is attained at the interface, but the two films which meet at the interface resist mass transfer. Transfer through the films is diffusional in nature, and

the rate of diffusion is proportional to the activity gradient across the film. Rate of transfer is also proportional to the interfacial area which forms between the two phases.

Since knowledge of conditions at the interface is usually not available, it has become common practice to combine the effects of both films into an overall mass transfer coefficient; and rate of mass transfer from the main body of one liquid to the main body of the other may be expressed by the equation,

$$\frac{dM}{d\theta} = KA(a_r - a_e) \quad (I:1)$$

where, M is the mass transferred across the interface in time θ ,

K is the overall mass transfer coefficient,

A is the interfacial area between the phases, and

a_r and a_e are the activities of the solute in the main body of the raffinate and extract layers respectively.

Due to the difficulty of measuring activities, it is desirable to convert the activities of equation (I:1) into concentrations. Accordingly, $a_r = \gamma_r C_r$ and $a_e = \gamma_e C_e$, where the γ 's are the respective activity coefficients, and the C's are the respective concentrations in the two layers. Moreover, it is possible to express the concentration difference in terms of the concentration in one phase since at equilibrium, $a_r = a_e$; so $\gamma_r C_r$ may be converted into $\gamma_e C_e^*$ where C_e^* is the concentration of solute in the extract

layer which would be in equilibrium with the raffinate layer. Substituting in equation (I:1) yields,

$$\frac{dM}{d\theta} = KA Y_e (C_e^* - C_e) \quad (I:2)$$

Over small changes in temperature and concentration, Y_e will remain constant, and may therefore be incorporated into the overall mass transfer coefficient, giving,

$$\frac{dM}{d\theta} = kA (C_e^* - C_e) \quad (I:3)$$

k , though it varies with temperature and the velocity of the liquids relative to each other, is primarily dependent upon the properties of the liquids involved, and the extent to which its value can be increased is limited. Large interfacial areas, however, are readily produced by several means, and the object in the design of extraction equipment is to provide for the most economical production of such area, since the rate of mass transfer varies directly with the interfacial area.

Liquid-liquid Extraction Equipment

Liquid-liquid extraction equipment falls into two broad categories; continuous and stepwise. The continuous operations are, of course, all countercurrent, as are also the stepwise processes when more than one equilibrium stage is desired. The greatest advantage of continuous equipment

is the combining of several equilibrium stages into one piece of equipment.

Most of the continuous operations are conducted in columns, wherein the heavy liquid is introduced at the top, and allowed to flow by gravity past the lighter fluid fed into the bottom. For effective column operation, one of the two liquids must be dispersed in the other, thereby increasing the interfacial area. The dispersed liquid is referred to as the discontinuous phase, while the other is called the continuous phase. The simplest method of achieving this end is to spray the liquid which has been chosen as the discontinuous phase into the column through nozzles. The liquid so introduced is dispersed into droplets, and passes through the counterflowing liquid to the other end of the column. Such a method is especially adaptable to systems where there is a possibility of sludge formation or precipitation since the otherwise empty column is easily cleaned. However, liquids with high interfacial tensions cannot be used, since the droplets formed at the nozzles would tend to coalesce too readily in passing through the continuous phase.

A rather successful modification of the spray column is the sieve plate column. Sieve plates spaced at equal intervals from top to bottom act as orifices in an otherwise empty column, and serve to keep the liquids dispersed by causing them to change velocity as they pass through

the plates. The acceleration and subsequent deceleration of the liquids as they pass through the orifices causes the formation of many droplets in both phases. Bubble cap columns were found to be less effective than the simpler sieve plate columns, and are not used for liquid-liquid extraction.

The use of packed columns has proved fairly satisfactory, and they are quite common. Like the other columns, they vary in height and diameter, depending upon their application, and are packed with materials which are inert to the liquids being treated. Some of the materials commonly used for packing are various metals, stoneware and earthenware, glass, spun glass, and quartz pebbles. Spun glass, of course, is used as a fiber, and the other materials are most commonly used in the form of rings, or saddles. The effectiveness of a packed column depends upon the viscosities of the liquids, the difference in their densities, the interfacial tension, the type of packing, and the flow rates. Simple packed columns cannot be used efficiently if the liquids have even moderately high viscosities, if the densities of the liquids are similar, if the interfacial tension is too high, or if the liquids tend to form a sludge or precipitate.

Schiebel (2) has greatly improved the efficiency of packed columns by introducing agitation to the liquids while in flow through the column. Throughout the column

there are short spaces with no packing in which the liquids flowing are thoroughly mixed by impellers which are fastened to a shaft running vertically through the center of the column, and driven by a motor from one end. The dispersion thus formed recombines in the adjoining sections, and the process is repeated throughout the column.

The principal drawback of all extraction columns is the low flooding velocities under which operation must be conducted. An extraction column is "flooded" when the combined flow of liquid is such that some raffinate flows from the column with the extract, or vice-versa.

Podbielniak, Inc., produces a continuous countercurrent apparatus which is fairly small in size, and yet operates at reasonably high flow rates. Heavy liquid is fed into the center of the machine, which rotates at high speeds, and flows outward by centrifugal force through an inwardly directed stream of the lighter fluid. As many as five equilibrium stages have been achieved in a single pass through this apparatus, but it is quite expensive.

However, in the face of all the improvements to continuous countercurrent equipment, most liquid-liquid extraction work is still conducted in series of single stage mixers with intermediate decanters. Under such conditions of operation, the liquids to be treated are introduced to a mixing tank in which a rotating impeller

supplies the energy for mixing. From the tank, the contents go to a settling unit called a decanter in which separation under the influence of gravity occurs. It is highly desirable to achieve equilibrium in each stage, thereby reducing to a minimum the number of stages required to effect a given degree of extraction. The mixer-decanter units may easily be arranged for countercurrent operation, the raffinate from a decanter going to the mixer on one side, and the extract going to the mixing unit in the other direction.

Mixing Equipment

It is possible to mix two immiscible fluids somewhat by pumping them through the same pipe, but most mixing operations used in conjunction with extraction processes are performed in a tank with some kind of mechanically driven impeller providing the mixing motion. The simplest type of impeller is a flat paddle, which is frequently modified by setting the blades at an angle. These paddles are suitable for many batch operations, but are not considered active enough to be used for mixing fluids in continuous flow through the mixing vessel.

From angled paddles have developed marine propellers type impellers which give better mixing with the same amount of power input, but are not nearly as easily made as

the angled paddles. The marine propeller type is somewhat more easily adapted to continuous flow and is frequently used in mixing equipment for extraction processes. By far the most used impeller for extraction work is the turbine type impeller (Scheibel's column uses that type too). It may consist of any number of blades, curved or straight, and gives the liquids it processes a radial motion. Most tanks using turbine impellers are equipped with stator rings which alter the direction of liquid flow to give greater dispersion than would otherwise occur. Turbine type impellers with stator rings are widely used due to their ability to provide mixing for liquids in continuous flow without the disadvantage, common to most impeller systems, of having severe short circuiting of unmixed liquid through the system.

Centrifugal pumps are often used for blending several miscible liquids which are apportioned as they enter the pump, but are not widely used in mixing operations. The prejudice against their use seems to be due to their high power requirement, and short retention time (3). The reason for exorbitant power requirements of centrifugal pumps is that they are so frequently operated below their rated capacity, and accordingly must work against an abnormally high head. Church (4), claims that impeller dimensions may be altered so that a pump may be designed to

deliver a given throughput against a specified head at any desired impeller speed within the range of the motor and the capacity of the pump. Therefore, if it can be shown that equilibrium can be attained easily in one pass through a centrifugal pump, it would certainly be advisable to investigate modification of impeller design to cut power requirements, since mixing operations can be conducted against a very low downstream head. Admittedly, if the experimental pump must be operated at extremely high speeds and low flow rates in order for equilibrium to be attained in one pass, the impeller dimensions might have to be reduced, in order to satisfactorily reduce power requirements, to a point where the amount of mixing accomplished would fall off drastically. However, if equilibrium can be attained at moderate impeller speeds, and reasonable flow rates against a low downstream head, it is probable that the necessary reduction of impeller dimensions to permit economical operation would not greatly affect the amount of mixing produced. One purpose of this work is to show that equilibrium can be obtained in one pass through the pump against low head at reasonably high flow rate and moderate impeller speeds.

Methods of Evaluating Mixing

The study of the effect of various variables upon

mixing processes and impeller performance has suffered noticeably from the lack of a concise criterion for degree of agitation. Serner (5) notes the general acceptance of power consumption as indication of the mixing work performed. Accordingly, for a given impeller, acting upon a given system in a particular tank, an increase in power input is considered as an increase in effective mixing. Two papers by Chaddock (6) and Serner (5) further demonstrate quite vividly the need for a criterion for mixing effectiveness. On a nomograph, Chaddock (6), resorted to the adjectives, "fair", "moderate", "vigorous", and "intense" to describe various degrees of agitation. Two years later, Serner (5), apparently unsatisfied with Chaddock's description, denoted regions of "mild", "medium", and "violent" mixing on a graph relating peripheral impeller velocity and horsepower/specific gravity. Though such terminology, while somewhat confusing, is probably accurate enough for rough design calculations, it is far too nebulous for use as a basic study of mixing problems. For that matter, the field of impeller design would undoubtedly also profit from the establishment of a concise criterion for mixing.

Hixson and Smith (7) have attempted to establish an "extraction coefficient" which would indicate mixing efficiency in liquid-liquid systems. They began with

equation (I:3), but instead of using the overall mass transfer coefficient K , they used the individual film coefficients and the distribution coefficient; thus,

$$\frac{dM}{d\theta} = \frac{Dk_r k_e}{k_r + Dk_e} A(C_r - C_r^*) \quad (\text{I:4})$$

where, D is the distribution coefficient, and is equal to γ_r/γ_e ,

k_e and k_r are the respective film coefficients, and C_r^* is the raffinate composition that would be in equilibrium with the extract composition.

Considering batch operations in a beaker using iodine dispersed between H_2O and CCl_4 , they proceeded from this equation to derive the relationship,

$$-\frac{a'}{K'} \log(1 - M_e/M_e^*) = \theta \quad (\text{I:5})$$

where, M_e is the weight of solute in the extract layer,

M_e^* is the weight of solute in the extract layer that would put it in equilibrium with the raffinate,

θ is the time of operation,

$a' = V_e D / (V_r + V_e D)$, V_r and V_e being the volumes of raffinate and extract, respectively,

and $K' = k_e AD(1 - K/k_r)/V_r$ (I:6)

where K is the overall mass transfer coefficient.

Since the complete derivation is quite lengthy it has been omitted here. A plot of θ against $(1 - M_e/M_e^*)$ on semi-log paper yielded a straight line, thus justifying the use of the equation developed. The slope of the line was taken to be $-a'/K'$. a' is constant for a particular system, and K' , which may be altered by changing conditions of operation, was accordingly proposed as an extraction coefficient. K' was determined for runs at various impeller speeds in several different systems, and was found to vary as the square of the impeller speed.

Power-RPM Relationships

Due to power cost, and to the generally accepted concept that power input is essentially a linear function of the mixing accomplished in a particular system, and to the fact that impeller speed is an obvious factor in mixing, much work has been done to relate impeller speed with power consumption. Rushton (8), working with marine propeller type impellers, found the power consumption to vary as the cube of impeller RPM. Most of the literature relating power input with impeller speeds was summed up in a paper by Olney and Carlson (9) who also offered much of their own work. In this paper the relationship,

$$P = (\phi)(L^3 N^2 \mu) \text{ was established } \quad (I:7)$$

where, L = impeller diameter,

N = impeller speed,

and μ = viscosity.

Furthermore, ϕ was found to be a function of a modified Reynold's number,

$$\text{Re} = \frac{\rho N d^2}{\mu} \quad (\text{I:8})$$

where, ρ = density in gm/cc.,

N = impeller speed in revolutions per second,

d = impeller diameter in cm.,

and μ = viscosity in poises.

For most impellers, at Reynold's numbers of from 10^2 to 10^4 ,

$$\phi = f(N)^{0.81}, \quad (\text{I:9})$$

making

$$P = f(N)^{2.81} \quad (\text{I:10})$$

and for Reynolds numbers of 10^4 to 10^6 ,

$$\phi = f(N)^{0.86}, \quad (\text{I:11})$$

making

$$P = f(N)^{2.86} \quad (\text{I:12})$$

Therefore power dependence upon impeller speed lies between the 2.81 and 3.0 power of that speed for most cases.

Purpose of this Work

In view of these correlations of impeller speed

with power consumption, Hixson and Smith's (7) criterion of mixing efficiency and the acceptance of a linear relationship between power and mixing are contradictory. So, despite considerable work done to remedy the situation, a clear cut criterion of mixing efficiency is still lacking.

As previously stated in this chapter, one purpose of this investigation is to determine the feasibility of using a centrifugal pump as a mixing stage in extraction work. To accomplish this objective, a system of benzoic acid distributed between toluene and water was selected. Using the same system, it was deemed desirable to investigate the effect of impeller speed upon mixing, and in conjunction with this work, a new criterion for mixing efficiency has been developed.

II

DEVELOPMENT OF A CRITERION FOR MIXING

The need of a concise criterion for mixing was noted in Chapter I. In this section the development of such a criterion from equation (I:3) is derived for conditions of a flow process.

Development of Operating Equation

Equation (I:3) developed in Chapter I states that,

$$\frac{dM}{d\theta} = kA(C_e^* - C_e) \quad (\text{II:1})$$

where, M is the mass transferred during the time θ ,

A is the interfacial area,

k is the overall mass transfer coefficient,

C_e^* is the concentration of benzoic acid in water that would be in equilibrium with the toluene layer, and

C_e is the concentration of benzoic acid in water.

The original concentration of benzoic acid in water is zero, so,

$$C_e = M/W \quad (\text{II:2})$$

where, W is the amount of water flowing through the system in time θ .

Since, over a short length of arc, the equilibrium curve approaches a straight line,

$$C_e^* = mC_r + b \quad (\text{II:3})$$

where, C_r is the concentration of benzoic acid in toluene, m is the slope of the equilibrium line, and b is the intercept on the benzoic acid-in-water axis.

Furthermore, all benzoic acid entering the water layer must come from the toluene layer, so

$$C_r = C_r^0 - M/T \quad (\text{II:4})$$

where, C_r^0 is the original concentration of benzoic acid in toluene, and

T is the amount of toluene flowing through the system in time θ .

Substituting equations (II:2), (II:3), and (II:4) in equation (II:1) and separating variables, we obtain,

$$\int_0^M \frac{dM}{mC_r^0 + b - M\left(\frac{m}{T} + \frac{1}{W}\right)} = \int_0^{\theta_c} kA d\theta \quad (\text{II:5})$$

which, when integrated, yields,

$$\ln \left[\frac{mC_r^0 + b - M\left(\frac{m}{T} + \frac{1}{W}\right)}{mC_r^0 + b} \right] = -kA\theta_c \left(\frac{m}{T} + \frac{1}{W}\right) \quad (\text{II:6})$$

where, θ_c is the time the mixture remains in the pump system.

Resubstitution of equations (II:3), (II:4), and (II:2) in equation (II:6) yields,

$$\ln \left[\frac{mC_r + b - C_e}{mC_r^o + b} \right] = -kA \left[\frac{m\theta_c}{T} + \frac{\theta_c}{W} \right] \quad (\text{II:7})$$

But T/θ_c is equal to the toluene flow rate, t ;

and W/θ_c is equal to the water flow rate, w .

Substituting these equivalents and rearranging,

$$kA = \frac{\ln \left[\frac{mC_r^o + b}{mC_r + b - C_e} \right]}{\frac{m}{t} + \frac{1}{w}} \quad (\text{II:8})$$

Presentation of the Criterion

Since the temperature varied only eight degrees over all runs, k will not vary significantly on that account. It is realized that k will be affected by the velocity of the droplets of the discontinuous phase relative to the continuous liquid. However, it was assumed that the whole dispersion will travel in slugs from the impeller with the result that the relative velocity of the particles in one phase to the other would be substantially constant, and low enough to be neglected, over the range of impeller speeds studied. Though admittedly this

assumption may be open to attack, it was held to be reasonable enough to allow k to be assumed constant. Accordingly, A , the interfacial area produced, is hereby proposed as a criterion for mixing efficiency. No value of k will be assumed here, thereby leaving A in relative units of area.

III

EQUIPMENT, MATERIALS, AND OPERATING PROCEDURE

At the beginning of this work it was thought that low flow rates at very high pump speeds were required for achieving equilibrium in one pass through a centrifugal pump. This opinion was supported by a statement in Perry (3), and, was in line with the general feeling prevalent in the field. Accordingly, Eastern Engineering Company's Model B-1 laboratory centrifugal pump, which will operate effectively between 1800 and 15,000 RPM at flow rates from zero to 0.0042 cubic feet per second, was selected. Emulsions were expected to be formed in the process of attaining equilibrium, and accordingly provision was made to draw the samples for analysis from the line through a high speed centrifuge to immediately separate the emulsion formed. Surprisingly, it was found that equilibrium could be reached at speeds as low as 2000 RPM, and without the coincident formation of an emulsion. The centrifuge was therefore removed from the system, and since the motor did not operate effectively below 1800 RPM, a system of gears operating at a 4.8 to 1 ratio was installed between the pump and the motor. Under the new conditions, the range of operation of the pump was 500 to

2500 RPM.

Measurement and Control Equipment

An electronic method for measuring motor speed was found to be most satisfactory. A flashlight bulb installed behind the cooling fan of the motor activated a photocell, which was aimed at the light source. When one of the fan blades was not cutting in front of the light, the photocell produced an impulse which was amplified and used as the horizontal component of an oscillograph. A calibrated audio frequency oscillator (See Figure 6, Appendix) supplied the vertical impulse to the oscilloscope, and when the two frequencies were identical, a standing wave resulted. This system provided highly satisfactory measurement of RPM, and had the added advantage of always indicating even when not in balance the proximity of the pump to the desired speed.

At first flow rates were measured for the individual liquids in separate flow through a pair of calibrated orifices (See Figure 5 in Appendix) which were $13/64$ inch sharp edged plates in a $21/64$ inch line. However, lower flow rates than originally deemed feasible were decided upon, and it became necessary, for greater accuracy, to pass the liquid mixture through one orifice. The orifice calibrated for water was used, and pressure drop across it

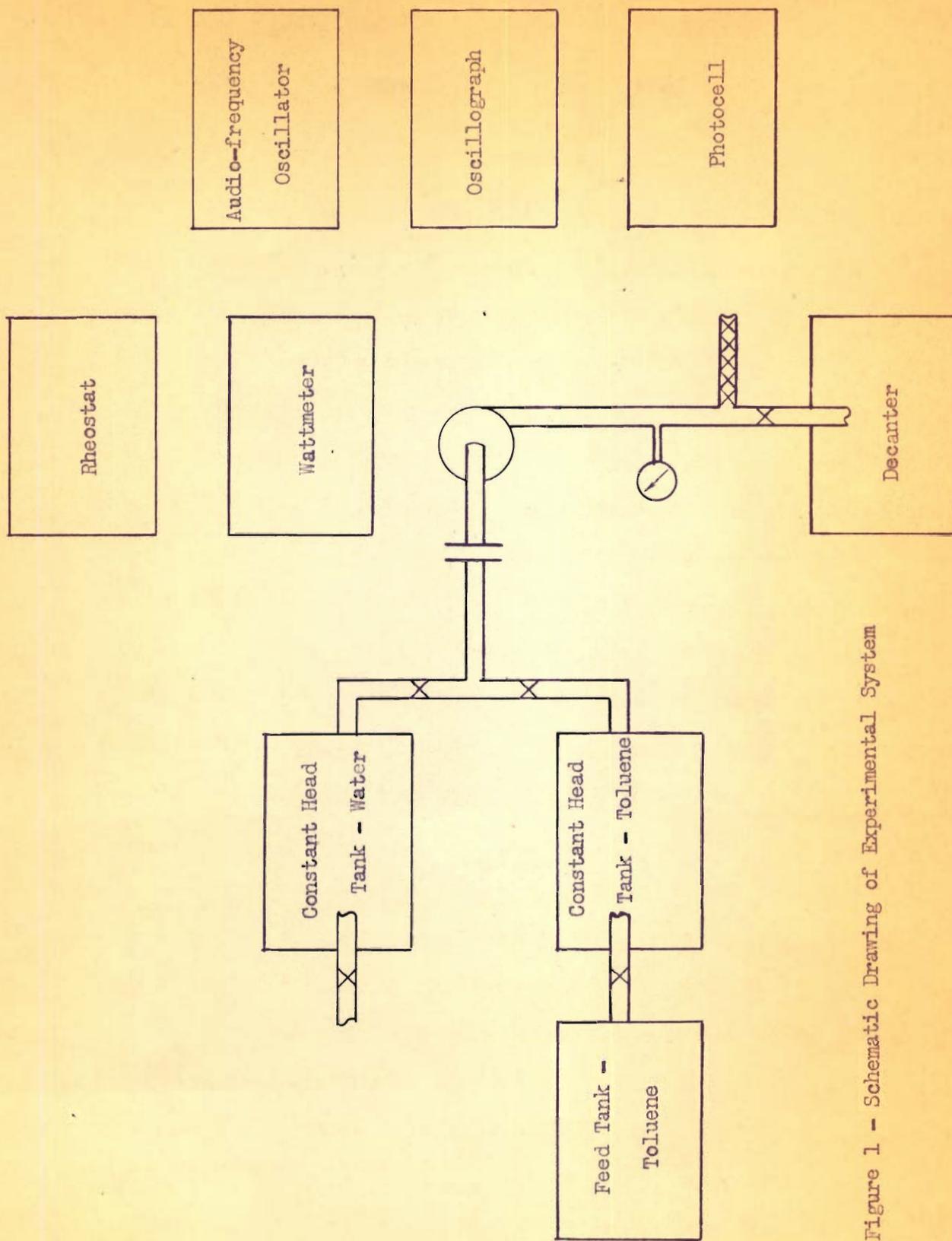


Figure 1 - Schematic Drawing of Experimental System

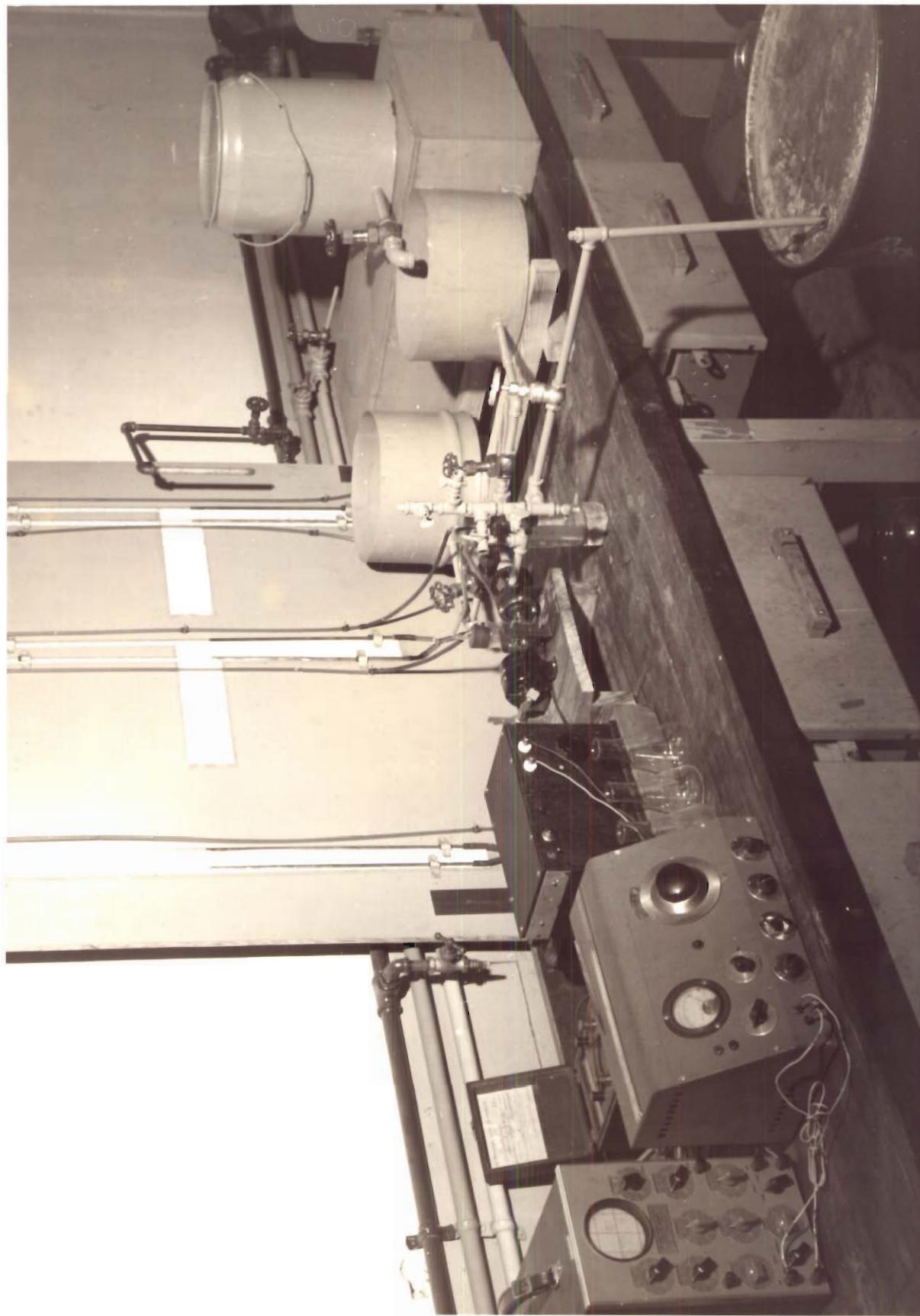


Figure 2 - Front View of Experimental System.

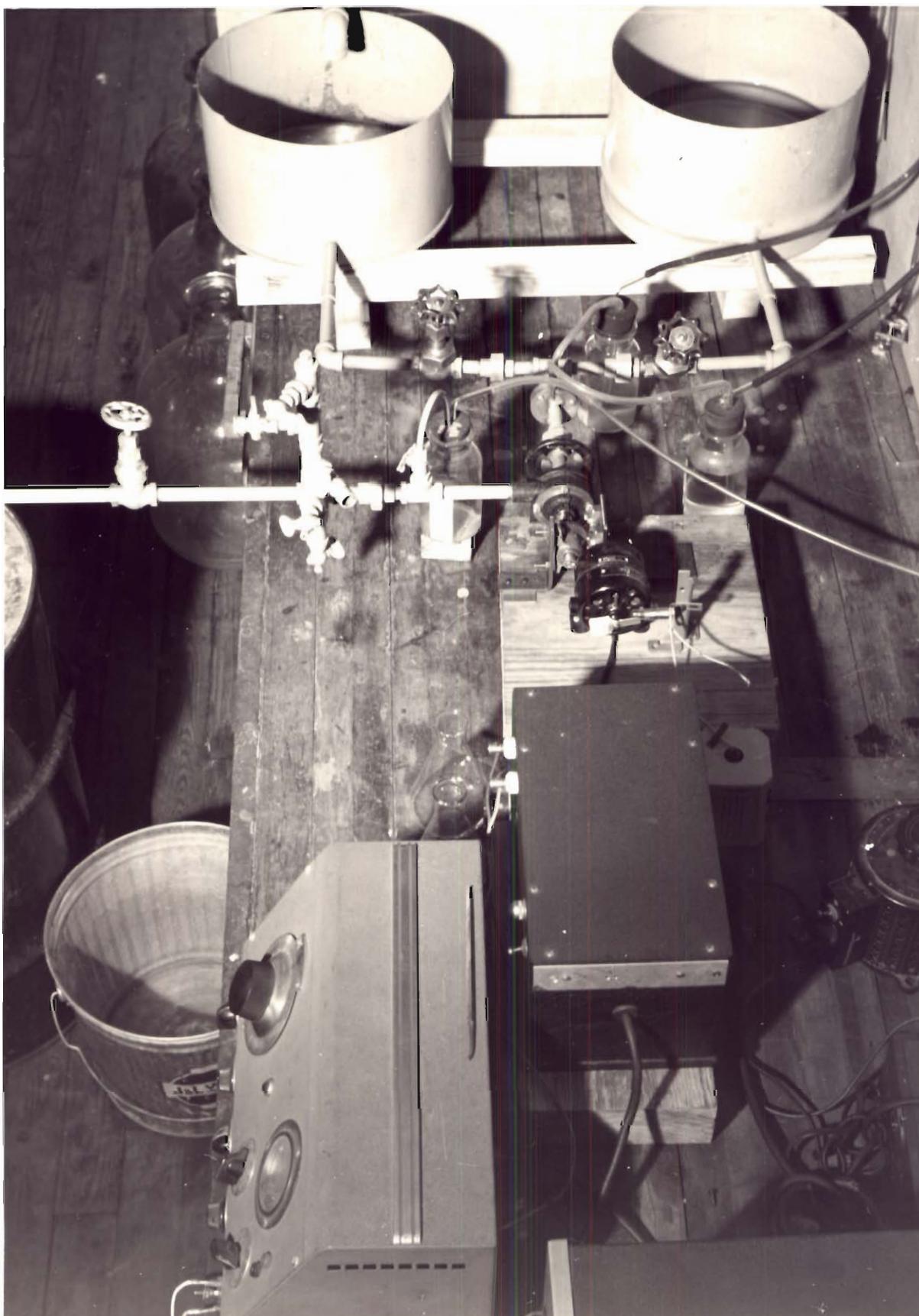


Figure 3 - Top View of Experimental System.

was measured by a water manometer. The pressure from the orifice taps to the manometer legs was transmitted by air lines.

The discharge pressure of the pump was measured from a pressure tap by means of an open water manometer; and a wattmeter installed in the line between the rheostat and the motor measured power input to the motor. Constant head tanks provided a means of obtaining constant flow, and three valves controlled the ratio and amount of liquid which flowed through the system in 1/4" standard iron pipe to a decanting drum.

A series of sampling cocks was arranged vertically so that samples could be taken from the system at a height above the line approximately equal to the line pressure. This measure reduced to a minimum the mixing effect which would have occurred had the liquid mixture been allowed to flow through the cocks under an appreciable pressure drop. A diagrammatic sketch and photographs of the equipment are shown in Figures No. 1, 2, and 3.

Impellers

Several different impellers, shown at 8/10 actual size in Figure No. 4, were used during this work. Impeller No. 1, shown center and lower right, was the impeller designed for use in the pump; it is 1.5" in diameter.

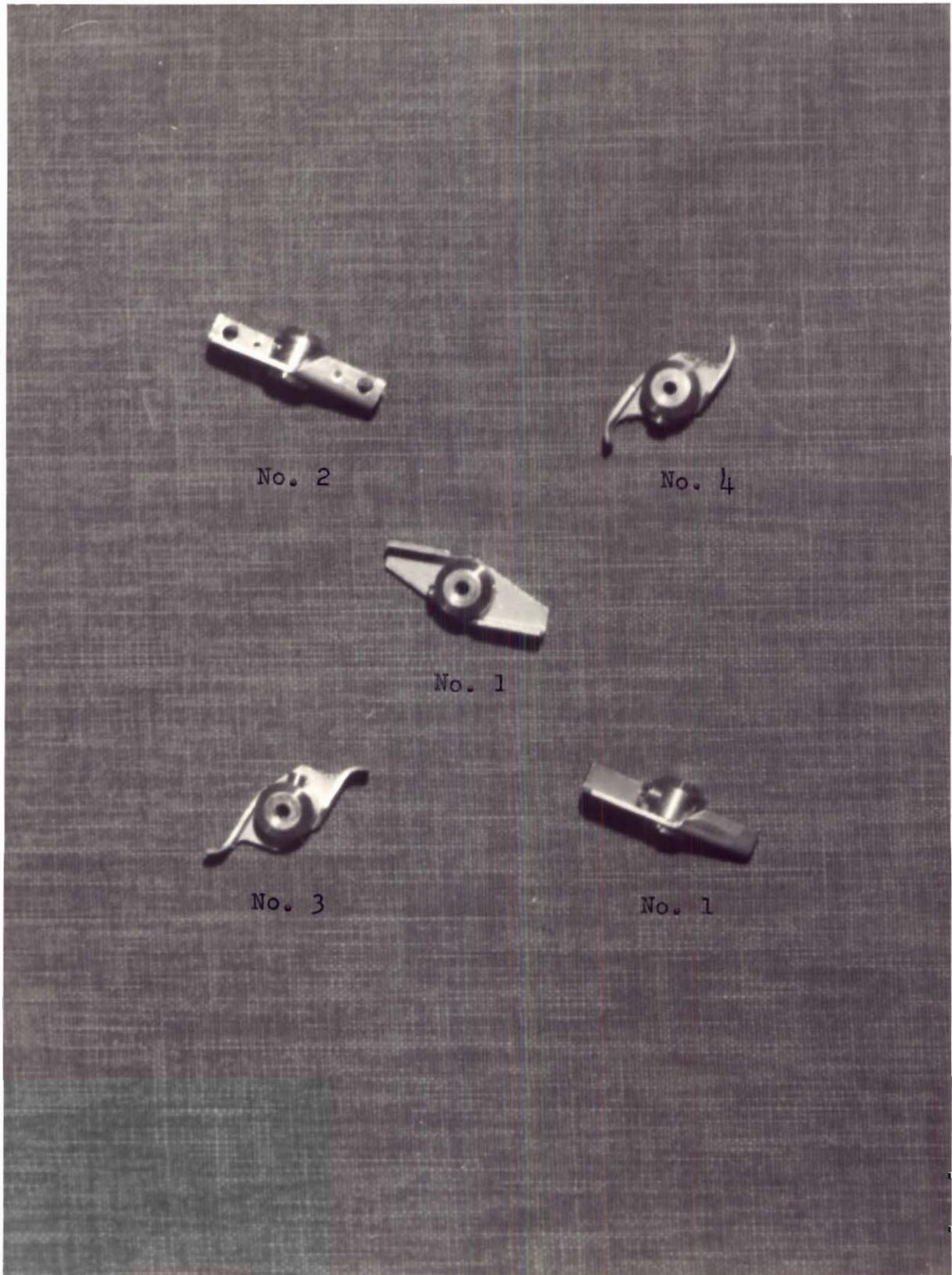


Figure 4 - Impellers

Impellers No. 2, 3, and 4 were all made by modifying impeller No. 1; on No. 2, shown upper left, the blades were perforated but otherwise unchanged; on No. 3, shown lower left, they were curved forward; and on No. 4, shown upper right, they had a backward curve.

Materials

The chemicals used in this work were nitration grade toluene, c. p. grade benzoic acid, and Atlanta city water. Equilibrium curves were established for the distribution of benzoic acid in the liquids actually used for experimental work (Figure No. 7 - Appendix). The water was checked from time to time and was found to be slightly basic. The toluene, which was reused from day to day, suffered some discoloration during the course of the work, probably due to impurities dissolved from the inside of the decanting drum. However, the equilibrium curves were checked, using the discolored solvent, and showed no change due to whatever impurities caused the discoloration. The isopropyl alcohol used in the course of analysis contained enough acidity to neutralize 0.3 cc of 0.0124 N NaOH per 10 cc of alcohol.

Operating Procedure

Use of equation (II:8) for determining effective area required that runs be made at constant flow rate, impeller speed, and water/toluene ratio. Accordingly, all

samples were withdrawn only after the system had reached a steady state of operation. Runs were begun with the constant head tanks full, the control valves wide open, and the motor running slowly. The pump RPM was first established by setting the audio frequency oscillator at the calibration corresponding with the desired motor speed. The power input was then varied through the rheostat until the pump was operating close to the desired speed. The flow was then adjusted by increasing the downstream pressure, and the rheostat was reset to give the exact pump speed desired. The flow rate was adjusted with the appropriate sample cock open since opening a cock during a run would cause a slight change in the downstream pressure, and a noticeable change in flow rate, thereby requiring resetting of the pressure valve and the rheostat.

It was attempted to set the water/toluene ratio at 1.0 for all runs by adjusting the valves in the feed lines to the pump, and the levels in the constant head tanks. However, although the ratio during any one run remained constant, it was not possible to control the value of the ratios so that they were very nearly equal to 1.0 for all runs. Over the entire period of operations, wide variance in ratios were observed, but the ratios for most runs were such that very seldom were there two parts of one liquid to but one part of the other.

Sampling Procedure

Once stable conditions had been reached, the samples were collected from the open cock in erlenmeyer flasks, and allowed to stand a few seconds to allow for complete separation of the phases by settling. The temperatures were immediately recorded, and measured samples of each layer were drawn by pipette from the flasks and placed in testtubes where they remained until analysed. The ratios of water to toluene were determined by volume measurement with allowance being made for the volume of the samples already withdrawn for analysis.

Analytical Procedure

All analyses were made by titrating with standardized NaOH. The water layer samples were titrated directly, using phenolphthalein as an indicator. To each of the toluene layer samples was added 20 cc of isopropyl alcohol per 3.0 cc of sample; this brought the toluene into solution with the aqueous solution of NaOH, and the endpoint was again determined by the change in color of phenolphthalein. The NaOH equivalent of the alcohol was of course subtracted from all toluene analyses.

IV

CALCULATION OF RESULTS

Method of Calculation

Using equation (II:8) the effective interfacial areas in runs No. 68 through 193 were calculated. In order to use the equation, toluene and water flow rates first had to be calculated from manometer readings and flow ratios. Originally it was intended that a density correction be made on the manometer readings for each run to allow for the difference in density between water and the toluene-water mixture flowing through the water calibrated orifice. However, it was decided that the accuracy of measurement was such that the data did not warrant such treatment. Accordingly, the density correction was made for all runs on the basis of a toluene-water ratio of 1.0, or a mixture of density equal to 0.93 grams/cc. The error involved in this approximation is less than 3% for most runs, and subsequently, all manometer readings were divided by 0.93 before being converted to flow rates by use of Figure No. 5. From the calculated total flow, individual flow rates were determined by use of the actual flow ratio for that particular run.

It was felt that the interfacial area produced by flowing the mixture through the system without the pump run-

ning might be considerable; and accordingly areas were calculated for various flow rates without the impeller running, and the results are plotted in Figure No. 8 of the Appendix. Hereafter these areas will be referred to as "blank" flow areas.

For impellers No. 1, 2, 3, and 4, runs were made at a flow rate of 0.00075 cubic feet per second over the complete operable range of impeller speeds. Total relative areas produced were calculated for each impeller speed, and the area produced by "blank" flow for that flow rate subtracted. The resultants were taken to be the relative areas produced by the impeller, and plotted against impeller RPM in Figures No. 9 through 12 - Appendix. In addition, for impeller No. 1, runs were conducted at various flow rates, and again the "blank" flow area for the particular rate of flow in use was subtracted from the total area produced. Then the areas produced by the impeller at each impeller speed for various throughputs were averaged, and the average area plotted against impeller speed in Figure No. 13 - Appendix. A sample calculation may be found on page 58 in the Appendix.

Evaluation of Data

Of the data used, points were discounted for several reasons; points which showed very poor material balance -

on the basis that all the benzoic acid gained by the water had to be lost by the toluene - were omitted from the plots. Where "blank" flow area was more than 60% of the total area produced, the points were discounted since a small error in determining either the "blank" flow area or the value of total area produced would cause undue error in the area calculated to have been produced in the pump, it being the difference of the two experimental values. Furthermore, as equilibrium was approached, very small error in analysis, equilibrium data, or selection of the slope of the equilibrium curve caused undue error in the calculation of area produced since it is the result of difference operations and subsequent division. Therefore, where data approached equilibrium conditions, the calculated points were also discounted in fitting the curves. The total number of points discarded by these methods did not exceed 12% of the number of determinations made.

Although pressure and power data were obtained and recorded, no correlation was attempted with regard to either of them. Some sort of relationship involving power consumption would be highly desirable, but such a large portion of the power input was consumed by motor losses, which were variable, depending upon the tightness of the packing gland, and gear alignment, that these data are really of little value.

V

RESULTS AND DISCUSSION

The first sixty-seven runs showed conclusively that equilibrium is easily attained in one pass through a centrifugal pump with the system used. As discussed in Chapter III, the first few runs showed that the system was over-designed for the work to be done, but even after this was realized, attempts were made to get non-equilibrium data using the original system. Motor speeds were not constant at the necessarily low speeds however, and therefore the data from runs No. 1 through 67 are useless, and are not recorded here other than to note that equilibrium was easily attained without coincident formation of an emulsion.

Runs No. 68 through 193 provided applicable data, and the relative areas produced in the system by these runs were calculated by use of equation (II:8). The effect of impeller speed upon areas produced was studied by plotting relative area against pump RPM on log-log paper (See Figures No. 9 through 13 - Appendix). Although the impeller used affected the position of the curves on the plots, all curves had a slope of 3.0, and accordingly,

$$A = f(N)^3 \quad (V:1)$$

Most of the plots had considerable scatter except for Figure No. 13, for which the average relative area for several runs at each impeller speed was used.

No attempt will be made to justify the relationship expressed in equation (V:1) theoretically. It is worth while noting, however, that it can be concluded from the review of the literature in Chapter I that

$$f(N)^{2.81} \leq P \leq f(N)^{3.0} \quad (V:2)$$

where, P is power consumption. Since A, the effective interfacial area produced, and P, power consumed, are similar functions of impeller speed, the use of A as a criterion for mixing accomplished is in agreement with the concept that mixing produced is a linear function of power consumption in any particular system.

Hixson and Smith (7) have proposed that their K' , which varies with the square of the impeller speed, be used as an "extraction coefficient" which they expect to serve the same purpose as the mixing criterion proposed herein. Clearly both of the criteria cannot be correct since one varies as the cube and the other as the square of the impeller speed. It is felt that although the relationship between K' and impeller speed which Hixson and Smith have developed is probably valid, the use of K' as an "extraction coefficient" does not necessarily

follow. Moreover, K' is such a conglomeration of terms that its significance is difficult to evaluate, whereas it does seem reasonable that effective interfacial area produced would be a good critique of effective mixing.

Reliability

In Chapter III the inability to operate with a water-toluene ratio of 1.0 was discussed. It was originally thought that this ratio might be a factor in the mixing accomplished by the pump, so area produced was plotted against ratio with other variables held constant. No correlation was noted, so ratio was deemed not to be a factor over the short range in which it varied during this work, and failure to control it more closely did not affect the reliability of these results.

Flow rates, which affect both the amount of "blank" flow area and the calculation of total area, were subject to an error of from five to ten percent, and the curves in Figures No. 9 through 12 do show considerable scatter. However, for the points on the curve in Figure No. 13, experimental error was compensated for by the fact that each average area was the result of calculations from at least four runs. The excellent correlation of the points on this curve to the relationship expressed in equation (V:1) markedly strengthens the trends shown in Figures No. 9 through 12.

VI

CONCLUSIONS

The following conclusions can be drawn from this investigation.

1. Contrary to popular belief, equilibrium is easily obtained in one pass of a liquid mixture through a centrifugal pump operating at moderate speeds. By using an impeller expressly designed to do mixing work, centrifugal pumps could probably be used economically in liquid-liquid extraction work.

2. The interfacial area produced in a continuous mixing process is a suitable criterion for expressing the mixing accomplished.

3. The amount of mixing produced in a given system is a function of the impeller speed cubed.

4. The contention that mixing varies linearly with power consumption for a given system is approximately valid.

APPENDIX I

TABLE I

| Run No. | Temp. °C. | Pump RPM | Power Input to Motor Watts | Combined Flow Rate Ft ³ /sec. x 10 ² | Flow Ratio Ft ³ H ₂ O per Ft ³ C ₇ H ₈ | Gage Press. at Pump Outlet Inches H ₂ O | Final C ₇ H ₆ O ₂ Conc. in C ₇ H ₈ phase lb. mols/ft ³ x 10 ³ | Final C ₇ H ₆ O ₂ Conc. in H ₂ O phase lb. mols/ft ³ x 10 ⁴ | Relative Interfacial Area Produced at Zero RPM |
|---------|--------------|----------|-------------------------------|---|--|---|---|--|--|
| 132 | 24.8 | 0 | 0 | 0.134 | 1.0 | 0.4 | 2.86 | 1.28 | 2.24 |
| 133 | 24.8 | 0 | 0 | 0.108 | 0.33 | 2.8 | 2.85 | 1.71 | 1.32 |
| 134 | 24.8 | 0 | 0 | 0.080 | 0.79 | 4.8 | 2.90 | 1.53 | 1.42 |
| 135 | 24.8 | 0 | 0 | 0.060 | 0.537 | 7.2 | 2.93 | 1.71 | 0.94 |

In Runs No. 132 - 5, Original C₇H₆O₂ Concentration in Toluene was 3.02 x 10⁻³ #-mols/ft³
Impeller No. 1 was used.

| Run No. | Temp. °C. | Pump RPM | Power Input to Motor Watts | Combined Flow Rate Ft ³ /sec. x 10 ² | Flow Ratio Ft ³ H ₂ O per Ft ³ C ₇ H ₈ | Gage Press. at Pump Outlet Inches H ₂ O | Final C ₇ H ₆ O ₂ Conc. in C ₇ H ₈ phase lb. mols/ft ³ x 10 ³ | Final C ₇ H ₆ O ₂ Conc. in H ₂ O phase lb. mols/ft ³ x 10 ⁴ | Relative Interfacial Area Produced at Zero RPM |
|---------|--------------|----------|-------------------------------|---|--|---|---|--|--|
| 47 | 19.8 | 0 | 0 | 0.057 | 2.0 | 0.2 | 5.30 | 1.17 | 0.80 |
| 48 | 20.0 | 0 | 0 | 0.030 | 2.0 | 0.9 | 5.23 | 1.28 | 0.45 |
| 49 | 20.1 | 0 | 0 | 0.037 | 1.5 | 0.5 | 5.26 | 1.33 | 0.54 |
| 113 | 26.5 | 0 | 0 | 0.076 | 0.81 | 1.0 | 3.47 | 1.06 | 0.86 |
| 114 | 26.2 | 0 | 0 | 0.047 | 0.84 | 0.1 | 3.56 | 1.67 | 0.83 |
| 127 | 25.4 | 0 | 0 | 0.142 | 0.519 | 0.1 | 3.29 | 1.89 | 2.30 |
| 128 | 25.2 | 0 | 0 | 0.121 | 1.00 | 3.6 | 3.24 | 1.63 | 2.36 |
| 129 | 25.0 | 0 | 0 | 0.100 | 1.24 | 4.5 | 3.21 | 1.32 | 1.72 |

In Runs No. 47 - 49, Original C₇H₆O₂ concentration in Toluene was 5.38×10^{-3} #-mols/ft³
Impeller No. 1 was used.

In Runs No. 113 - 4, Original C₇H₆O₂ concentration in Toluene was 3.70×10^{-3} #-mols/ft³
Impeller No. 1 was used.

In Runs No. 127 - 9, Original C₇H₆O₂ concentration in Toluene was 3.39×10^{-3} #-mols/ft³
Impeller No. 1 was used.

| Run No. | Temp. | Pump RPM | Power Input to Motor | Combined Flow Rate | Flow Ratio | Gage Press. at Pump Outlet | Final $C_7H_6O_2$ Conc. in C_7H_8 phase | Final $C_7H_6O_2$ Conc. in H_2O phase | Relative Interfacial Area Produced by the Pump |
|---------|-------|----------|----------------------|---|--|----------------------------|--|--|--|
| | °C. | | Watts | Ft ³ /sec. x 10 ² | Ft ³ H ₂ O per Ft ³ C ₇ H ₈ | Inches H ₂ O | lb. mols/ft ³ x 10 ³ | lb. mols/ft ³ x 10 ⁴ | |
| 68 | 26 | 730 | 26 | 0.089 | 1.28 | - 0.3 | 2.49 | 1.63 | 2.72 |
| 69 | 26 | 730 | 25 | 0.078 | 1.44 | 3.1 | 2.56 | 1.47 | 1.98 |
| 70 | 26.5 | 730 | 23.5 | 0.050 | 6.0 | 5.3 | 2.63 | 1.70 | 1.92 |
| 71 | 26.5 | 730 | 25 | 0.059 | 1.46 | 1.9 | 2.60 | 1.28 | 1.13 |
| 72 | 27 | 730 | 24 | 0.035 | 2.5 | 3.8 | 2.42 | 1.74 | 1.61 |
| 73 | 27.5 | 520 | 22.5 | 0.077 | 1.5 | - 0.4 | 2.56 | 1.16 | 0.32 |
| 74 | 27 | 520 | 23 | 0.032 | 2.2 | 1.6 | 2.70 | 1.55 | 0.52 |
| 75 | 26.5 | 625 | 23.2 | 0.037 | 1.0 | 1.9 | 2.88 | 2.16 | 0.51 |
| 76 | 27.5 | 625 | 25 | 0.084 | 1.21 | - 0.5 | 2.56 | 1.63 | 1.01 |

In Runs No. 68 - 76, Original $C_7H_6O_2$ concentration in Toluene was 2.88×10^{-3} #mols/ft³ Impeller No. 1 was used.

| Run No. | Temp. °C. | Pump RPM | Power Input to Motor Watts | Power Input Combined Flow Rate Ft ³ /sec. x 10 ² | Flow Ratio Ft ³ H ₂ O per Ft ³ C ₇ H ₈ | Gage Press. at Pump Outlet Inches H ₂ O | Final C ₇ H ₆ O ₂ Conc. in C ₇ H ₈ phase lb. mols/ft ³ x 10 ³ | Final C ₇ H ₆ O ₂ Conc. in H ₂ O phase lb. mols/ft ³ x 10 ⁴ | Relative Interfacial Area Produced by the Pump |
|---------|-----------|----------|----------------------------|--|---|--|--|---|--|
| 77 | 25.5 | 625 | 15 | 0.071 | 1.65 | 2.1 | 2.46 | 1.24 | 0.33 |
| 78 | 25.8 | 625 | 15 | 0.112 | 2.58 | 0.1 | 2.48 | 1.16 | 0.56 |
| 79 | 26.3 | 625 | 15 | 0.052 | 0.783 | 3.0 | 2.42 | 1.70 | 0.41 |
| 80 | 26.6 | 520 | 13.5 | 0.105 | 1.93 | 0.3 | 2.48 | 1.08 | 0.30 |
| 81 | 27.1 | 520 | 9 | 0.060 | 3.91 | 1.1 | 2.11 | 1.08 | 0.53 |
| 82 | 27.3 | 520 | 9 | 0.047 | 1.83 | 2.0 | 2.25 | 1.86 | 1.05 |
| 83 | 26.7 | 520 | 9 | 0.087 | 0.32 | 1.4 | 2.56 | 1.55 | - |
| 84 | 27.1 | 730 | 12 | 0.142 | 1.79 | 0.2 | 2.45 | 1.47 | 1.39 |
| 85 | 27.1 | 730 | 11 | 0.092 | 2.76 | 1.4 | 2.42 | 1.16 | 0.51 |
| 86 | 27.0 | 730 | 10 | 0.068 | 0.353 | 2.5 | 2.50 | 2.32 | 0.32 |

In Runs No. 77 - 86, Original C₇H₆O₂ Concentration in Toluene was 2.74 x 10⁻³ #mols/ft³ Impeller No. 1 was used.

| Run No. | Temp. | Pump RPM | Power Input to Motor | Combined Flow Rate | Flow Ratio | Gage Press. at Pump Outlet | Final $C_7H_6O_2$ Conc. in C_7H_8 phase | Final $C_7H_6O_2$ Conc. in H_2O phase | Relative Interfacial Area Produced by the Pump |
|---------|-------|----------|----------------------|---|--|----------------------------|--|--|--|
| | °C. | | Watts | Ft ³ /sec. x 10 ² | Ft ³ H ₂ O per Ft ³ C ₇ H ₈ | Inches H ₂ O | lb. mols/ft ³ x 10 ³ | lb. mols/ft ³ x 10 ⁴ | |
| 87 | 20.0 | 830 | 13 | 0.103 | 0.68 | 3.2 | 2.25 | 1.47 | 0.48 |
| 88 | 20.0 | 830 | 12 | 0.113 | 0.635 | 2.6 | 2.17 | 1.65 | 0.95 |
| 89 | 20.0 | 830 | 12 | 0.052 | 1.44 | 4.1 | 2.20 | 1.78 | 1.12 |
| 90 | 20.3 | 940 | 13 | 0.073 | 0.80 | 4.6 | 2.16 | 1.86 | 1.21 |
| 91 | 20.3 | 940 | 13 | 0.092 | 0.56 | 4.3 | 2.25 | 1.78 | 0.69 |
| 92 | 20.3 | 940 | 12 | 0.137 | 0.86 | 1.0 | 2.20 | 1.63 | 1.57 |
| 93 | 20.4 | 1040 | 14 | 0.126 | 0.53 | 3.2 | 2.20 | 1.78 | 0.95 |
| 94 | 20.5 | 1040 | 14 | 0.087 | 1.07 | 4.3 | 2.13 | 2.04 | 2.40 |
| 95 | 20.5 | 1040 | 14 | 0.060 | 1.00 | 5.1 | 2.14 | 2.18 | 1.80 |
| 96 | 20.5 | 1040 | 14 | 0.161 | 0.63 | 0.2 | 2.19 | 1.93 | 2.15 |

In Runs No. 87 - 96, Original $C_7H_6O_2$ concentration in Toluene was 2.34×10^{-3} #-mols/ft³
 Impeller No. 1 was used.

| Run No. | Temp. | Pump RPM | Power Input to Motor | Combined Flow Rate | Flow Ratio | Gage Press. at Pump Outlet | Final C ₇ H ₆ O ₂ Conc. in C ₇ H ₈ phase | Final C ₇ H ₆ O ₂ Conc. in H ₂ O phase | Relative Interfacial Area Produced by the Pump |
|---------|-------|----------|----------------------|---|--|----------------------------|---|--|--|
| | °C. | | Watts | Ft ³ /sec. x 10 ² | Ft ³ H ₂ O per Ft ³ C ₇ H ₈ | Inches H ₂ O | lb. mols/ft ³ x 10 ³ | lb. mols/ft ³ x 10 ⁴ | |
| 97 | 22 | 1150 | 18 | 0.167 | 0.706 | 0.1 | 2.55 | 2.40 | 3.20 |
| 98 | 21.5 | 1150 | 15 | 0.127 | 1.19 | 2.2 | 2.57 | 2.24 | 3.04 |
| 99 | 22.1 | 1150 | 15 | 0.103 | 0.70 | 5.6 | 2.60 | 2.49 | 2.07 |
| 100 | 22.5 | 1150 | 15 | 0.0565 | 0.186 | 7.8 | 2.72 | 3.67 | 1.65 |
| 101 | 23 | 1250 | 18 | 0.134 | 0.94 | 2.9 | 2.63 | 2.50 | 3.33 |
| 102 | 22.9 | 1250 | 17.5 | 0.121 | 1.11 | 6.1 | 2.56 | 2.48 | 3.55 |
| 103 | 23 | 1250 | 18 | 0.050 | 0.79 | 9.4 | 2.55 | 3.17 | 2.24 |
| 104 | 23 | 1350 | 20 | 0.050 | 1.125 | 9.5 | 2.50 | 3.05 | 2.62 |
| 105 | 23 | 1350 | 19 | 0.121 | 0.96 | 7.4 | 2.55 | 2.79 | 5.36 |
| 106 | 23 | 1350 | 19 | 0.153 | 1.07 | 3.5 | 2.55 | 2.64 | 6.42 |

In Runs No. 97 - 106, Original C₇H₆O₂ concentration in Toluene was 2.83×10^{-3} lb. mols/ft³
 Impeller No. 1 was used.

| Run No. | Temp. °C. | Pump RPM | Power Input to Motor | Combined Flow Rate | Flow Ratio | Gage Press. at Pump Outlet | Final C ₇ H ₈ phase Conc. in C ₇ H ₈ | Final C ₇ H ₆ O ₂ Conc. in H ₂ O phase | Relative Interfacial Area Produced by the Pump |
|---------|-----------|----------|----------------------|---|--|----------------------------|--|--|--|
| | | | Watts | Ft ³ /sec. x 10 ² | Ft ³ H ₂ O per Ft ³ C ₇ H ₈ | Inches H ₂ O | lb. mols/ft ³ x 10 ³ | lb. mols/ft ³ x 10 ⁴ | |
| 107 | 24 | 1460 | 24 | 0.213 | 0.83 | 0.9 | 3.32 | 3.40 | 6.90 |
| 108 | 25 | 1460 | 22 | 0.150 | 1.04 | 5.0 | 3.25 | 3.33 | 6.01 |
| 109 | 25 | 1460 | 22 | 0.087 | 0.83 | 8.9 | 3.19 | 3.88 | 2.94 |
| 110 | 25.3 | 1560 | 23 | 0.087 | 1.33 | 11.0 | 3.19 | 3.77 | 5.63 |
| 111 | 25 | 1560 | 23 | 0.170 | 0.714 | 7.4 | 3.32 | 3.79 | 7.50 |
| 112 | 25.5 | 1560 | 23 | 0.20 | 0.764 | 3.1 | 3.24 | 3.90 | 10.2 |
| 115 | 26.7 | 730 | 15 | 0.121 | 1.00 | 1.0 | 3.37 | 1.77 | 0.76 |
| 116 | 27 | 730 | 15 | 0.078 | 1.20 | 2.3 | 3.64 | 1.99 | 0.63 |

In Runs No. 107 - 112, Original C₇H₆O₂ concentration in Toluene was 3.70 x 10⁻³ lb. mols/ft³ Impeller No. 1 was used.

In Runs No. 115 - 116, Original C₇H₆O₂ concentration in Toluene was 3.70 x 10⁻³ lb. mols/ft³ Impeller No. 1 was used.

| Run No. | Temp. °C. | Pump RPM | Power Input to Motor Watts | Combined Flow Rate Ft ³ /sec. x 10 ² | Flow Ratio Ft ³ H ₂ O per Ft ³ C ₇ H ₈ | Gage Press. at Pump Outlet Inches H ₂ O | Final C ₇ H ₆ O ₂ Conc. in C ₇ H ₈ phase lb. mols/ft ³ x 10 ³ | Final C ₇ H ₆ O ₂ Conc. in H ₂ O phase lb. mols/ft ³ x 10 ⁴ | Relative Interfacial Area Produced by the Pump |
|---------|--------------|----------|-------------------------------|---|--|---|---|--|--|
| 117 | 21.8 | 625 | - | 0.074 | 0.60 | 0.2 | 3.16 | 1.74 | 0.18 |
| 118 | 22.3 | 730 | 23 | 0.074 | 0.444 | 2.5 | 3.19 | 2.24 | 0.34 |
| 119 | 22.2 | 830 | 24 | 0.074 | 0.428 | 2.0 | 3.16 | 2.63 | 0.73 |
| 120 | 22.5 | 940 | 25.5 | 0.074 | 0.74 | 4.5 | 3.14 | 2.59 | 1.44 |
| 121 | 22.4 | 1040 | 27.0 | 0.074 | 0.625 | 4.1 | 3.16 | 2.82 | 1.51 |
| 122 | 22.6 | 1150 | 28.0 | 0.074 | 1.00 | 5.7 | 3.11 | 2.94 | 2.58 |
| 123 | 22.6 | 1250 | 28.0 | 0.074 | 0.782 | 6.6 | 3.11 | 3.33 | 3.07 |
| 124 | 23.0 | 1350 | 28 | 0.074 | 0.84 | 8.8 | 3.01 | 3.72 | 4.85 |
| 125 | 22.8 | 1460 | 31 | 0.074 | 0.84 | 9.2 | 3.09 | 3.95 | 5.75 |
| 126 | 23.0 | 1560 | 34 | 0.074 | 0.71 | 11.5 | 3.04 | 3.98 | 5.55 |

In Runs No. 117 - 126, Original C₇H₆O₂ concentration in Toluene was 3.34×10^{-3} lb. mols/ft³ Impeller No. 1 was used.

| Run No. | Temp. °C. | Pump RPM | Power Input to Motor | Combined Flow Rate | Flow Ratio | Gage Press. at Pump Outlet | Final C ₇ H ₈ phase C ₇ H ₆ O ₂ Conc. in H ₂ O phase | Final C ₇ H ₆ O ₂ Conc. in H ₂ O phase | Relative Interfacial Area Produced by the Pump |
|---------|-----------|----------|----------------------|---|--|----------------------------|--|--|--|
| | | | Watts | Ft ³ /sec. x 10 ² | Ft ³ H ₂ O per Inches H ₂ O x 10 ³ Ft ³ C ₇ H ₈ | Inches H ₂ O | lb. mols/ft ³ x 10 ³ | lb. mols/ft ³ x 10 ⁴ | |
| 130 | 25.2 | 1670 | 29 | 0.074 | 3.42 | 21.0 | 2.14 | 3.32 | 7.97 |
| 131 | 25.3 | 1980 | 38 | 0.074 | 5.0 | 30.0 | 1.79 | 3.10 | 8.25 |
| 136 | 25.0 | 2290 | 39 | 0.074 | 1.08 | > 36 | 2.57 | 3.92 | 8.18 |

In Runs No. 130 - 131, Original C₇H₆O₂ concentration in Toluene was 3.39 x 10⁻³ lb. mols/ft³ Impeller No. 1 was used.

In Runs No. 136, Original C₇H₆O₂ concentration in Toluene was 3.02 x 10⁻³ lb. mols/ft³ Impeller No. 1 was used. F

| Run No. | Temp. °C. | Pump RPM | Power Input to Motor Watts | Combined Flow Rate Ft ³ /sec x 10 ² | Flow Ratio Ft ³ H ₂ O per Ft ³ C ₇ H ₈ | Gage Press. at Pump Outlet Inches H ₂ O | Final C ₇ H ₆ O ₂ Conc. in C ₇ H ₈ phase lb. mols/ft ³ x 10 ³ | Final C ₇ H ₆ O ₂ Conc. in H ₂ O phase lb. mols/ft ³ x 10 ⁴ | Relative Interfacial Area Produced by the Pump |
|---------|--------------|----------|-------------------------------|--|--|---|---|--|--|
| 138 | 26.1 | 625 | 9 | 0.074 | 0.76 | 0.5 | 2.52 | 1.39 | 0.15 |
| 139 | 25.5 | 830 | 10 | 0.074 | 0.89 | 1.0 | 2.55 | 1.59 | 0.45 |
| 140 | 25.0 | 1040 | 12 | 0.074 | 1.05 | 2.1 | 2.46 | 2.21 | 1.75 |
| 141 | 25.0 | 1250 | 13 | 0.074 | 1.14 | 4.2 | 2.45 | 2.32 | 1.99 |
| 142 | 24.8 | 1460 | 14 | 0.074 | 1.45 | 7.7 | 2.24 | 2.67 | 3.97 |
| 143 | 24.7 | 1670 | 17 | 0.074 | 3.67 | 10.9 | 1.51 | 2.59 | 8.25 |
| 144 | 24.5 | 1880 | 19 | 0.074 | 0.75 | 16.7 | 2.42 | 3.79 | 9.0 |
| 145 | 24 | 2080 | 23 | 0.074 | 0.57 | 21 | 2.40 | 3.87 | 11.6 |
| 146 | 24.5 | 730 | 9 | 0.074 | 0.89 | 3.6 | 2.35 | 1.55 | 0.36 |
| 147 | 24.5 | 940 | 10 | 0.074 | 1.18 | 5.2 | 2.22 | 2.83 | 4.30 |

In Runs No. 138 - 147, Original C₇H₆O₂ concentration in Toluene was 2.63×10^{-3} lb. mols/ft³
Impeller No. 2 was used.

| Run No. | Temp. | Pump RPM | Power Input to Motor | Combined Flow Rate | Flow Ratio | Gage Press. at Pump Outlet | Final $C_7H_6O_2$ Conc. in C_7H_8 phase | Final $C_7H_6O_2$ Conc. in H_2O phase | Relative Interfacial Area Produced by the Pump |
|---------|--------------|----------|----------------------|-------------------------|-------------------------------|----------------------------|---|---|--|
| | $^{\circ}C.$ | | Watts | $Ft^3/sec. \times 10^2$ | $Ft^3 H_2O$ per $Ft^3 C_7H_8$ | Inches H_2O | lb. mols/ $ft^3 \times 10^3$ | lb. mols/ $ft^3 \times 10^4$ | |
| 148 | 19 | 940 | 23 | 0.074 | 1.93 | 2.0 | 2.65 | 1.70 | 1.51 |
| 149 | 19 | 1150 | 24 | 0.074 | 2.20 | 3.3 | 2.53 | 2.04 | 2.57 |
| 150 | 19 | 1350 | 24 | 0.074 | 2.14 | 5.1 | 2.40 | 2.38 | 3.81 |
| 151 | 19.2 | 1560 | 26 | 0.074 | 1.93 | 10.0 | 2.42 | 2.74 | 5.05 |

In Runs No. 148 - 151, Original $C_7H_6O_2$ concentration in Toluene was 2.98×10^{-3} lb. mols/ ft^3 53
 Impeller No. 2 was used.

| Run No. | Temp. | Pump RPM | Power Input to Motor | Combined Flow Rate | Flow Ratio | Gage Press. at Pump Outlet | Final $C_7H_6O_2$ Conc. in C_7H_8 phase | Final $C_7H_6O_2$ Conc. in H_2O phase | Relative Interfacial Area Produced by the Pump |
|---------|--------------|----------|----------------------|-------------------------|-------------------------------|----------------------------|---|---|--|
| | $^{\circ}C.$ | | Watts | $Ft^3/sec. \times 10^2$ | $Ft^3 H_2O$ per $Ft^3 C_7H_8$ | Inches H_2O | lb. mols/ $ft^3 \times 10^3$ | lb. mols/ $ft^3 \times 10^4$ | |
| 152 | 20.4 | 625 | 11 | 0.074 | 0.70 | 0.3 | 2.63 | 1.32 | 0.11 |
| 153 | 20.0 | 730 | 12 | 0.074 | 1.25 | 1.1 | 2.47 | 1.47 | 0.98 |
| 154 | 19.8 | 830 | 13 | 0.074 | 1.11 | 2.2 | 2.60 | 1.55 | 0.64 |
| 155 | 20.0 | 940 | 15 | 0.074 | 1.13 | 2.7 | 2.66 | 1.70 | 0.98 |
| 156 | 19.9 | 1040 | 15 | 0.074 | 1.24 | 5.4 | 2.58 | 1.86 | 2.10 |
| 157 | 20.0 | 1150 | 16 | 0.074 | 1.11 | 5.7 | 2.55 | 2.09 | 1.84 |
| 158 | 20.0 | 1250 | 18 | 0.074 | 0.90 | 6.2 | 2.58 | 2.24 | 1.92 |
| 159 | 20.0 | 1350 | 20 | 0.074 | 0.88 | 9.6 | 2.55 | 2.71 | 3.20 |
| 160 | 19.6 | 1460 | 22 | 0.074 | 1.22 | 9.5 | 2.48 | 2.48 | 3.23 |
| 161 | 19.8 | 1670 | 25 | 0.074 | 1.05 | 12.4 | 2.45 | 2.94 | 4.92 |

In Runs No. 152 - 161, Original $C_7H_6O_2$ concentration in Toluene was 2.73×10^{-3} lb. mols/ ft^3 Impeller No. 3 was used. 17

| Run No. | Temp. | Pump RPM | Power Input to Motor | Combined Flow Rate | Flow Ratio | Gage Press. at Pump Outlet | Final $C_7H_6O_2$ Conc. in C_7H_8 phase | Final $C_7H_6O_2$ Conc. in H_2O phase | Relative Interfacial Area Produced by the Pump |
|---------|--------------|----------|----------------------|-------------------------|-------------------------------|----------------------------|---|---|--|
| | $^{\circ}C.$ | | Watts | $Ft^3/sec. \times 10^2$ | $Ft^3 H_2O$ per $Ft^3 C_7H_8$ | Inches H_2O | lb. mols/ $ft^3 \times 10^3$ | lb. mols/ $ft^3 \times 10^4$ | |
| 162 | 22.5 | 730 | 11 | 0.074 | 0.95 | 1.7 | 2.36 | 1.62 | 0.88 |
| 163 | 22.0 | 1040 | 14 | 0.074 | 1.00 | 4.4 | 2.19 | 2.56 | 3.49 |
| 164 | 22.0 | 1560 | 23 | 0.074 | 1.41 | 14.0 | 2.07 | 2.71 | 5.30 |
| 165 | 22.0 | 1460 | 20 | 0.074 | 1.30 | 12.9 | 2.07 | 2.86 | 6.00 |
| 166 | 21.8 | 1250 | 19 | 0.074 | 1.11 | 10.2 | 2.12 | 2.71 | 4.55 |
| 167 | 21.5 | 1700 | 22 | 0.074 | 1.25 | 17.5 | 2.14 | 3.33 | 10.8 |
| 168 | 21.5 | 1910 | 27 | 0.074 | 1.18 | 20.5 | 2.09 | 3.33 | 9.3 |
| 169 | 22.0 | 2080 | 35 | 0.074 | 1.00 | 24.2 | 2.12 | 3.41 | 9.8 |

In Runs No. 162 - 169, Original $C_7H_6O_2$ concentration in Toluene was 2.48×10^{-3} lb. mols/ ft^3 Impeller No. 3 was used.

| Run No. | Temp. | Pump RPM | Power Input to Motor | Combined Flow Rate | Flow Ratio | Gage Press. at Pump Outlet | Final $C_7H_6O_2$ Conc. in C_7H_8 phase | Final $C_7H_6O_2$ Conc. in H_2O phase | Relative Interfacial Area Produced by the Pump |
|---------|--------------|----------|----------------------|-------------------------|-------------------------------|----------------------------|---|---|--|
| | $^{\circ}C.$ | | Watts | $Ft^3/sec. \times 10^2$ | $Ft^3 H_2O$ per $Ft^3 C_7H_8$ | Inches H_2O | lb. mols/ $ft^3 \times 10^3$ | lb. mols/ $ft^3 \times 10^4$ | |
| 170 | 27 | 625 | 16 | 0.075 | 0.625 | 0.3 | 2.68 | 1.52 | 0.09 |
| 171 | 27 | 830 | 18 | 0.075 | 1.00 | 0.1 | 2.68 | 1.86 | 0.86 |
| 172 | 27 | 1040 | 20 | 0.075 | 2.00 | 1.3 | 2.58 | 1.97 | 1.70 |
| 173 | 27 | 1250 | 16 | 0.075 | 1.44 | 4.5 | 2.61 | 2.03 | 1.52 |
| 174 | 27 | 1460 | 17 | 0.075 | 0.808 | 6.5 | 2.64 | 2.56 | 1.78 |
| 175 | 27 | 1670 | 17 | 0.075 | 0.40 | 9.0 | 2.60 | 3.56 | 2.71 |
| 176 | 27 | 1880 | 19 | 0.075 | 1.00 | 12.8 | 2.52 | 3.07 | 3.63 |
| 177 | 27 | 2080 | 23 | 0.075 | 1.05 | 7.0 | 2.44 | 3.38 | 5.25 |

In Runs No. 170 - 177, Original $C_7H_6O_2$ concentration in Toluene was 2.84×10^{-3} lb. mols/ ft^3
 Impeller No. 4 was used.

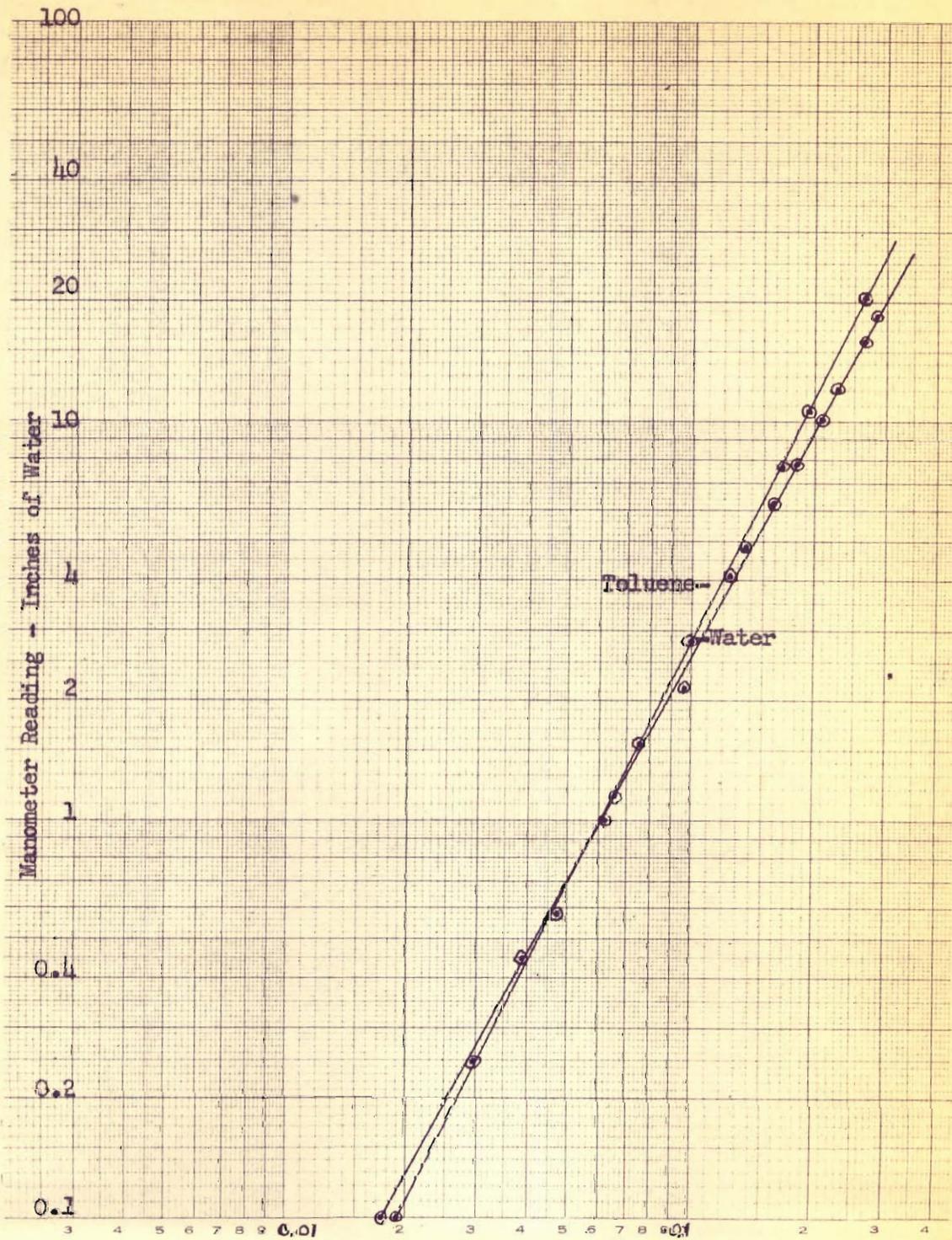
| Run No. | Temp. °C. | Pump RPM | Power Input to Motor Watts | Combined Flow Rate Ft ³ /sec. x 10 ² | Flow Ratio Ft ³ H ₂ O per Ft ³ C ₇ H ₈ | Gage Press. at Pump Outlet Inches H ₂ O | Final C ₇ H ₆ O ₂ Conc. in C ₇ H ₈ phase lb. mols/ft ³ x 10 ³ | Final C ₇ H ₆ O ₂ Conc. in H ₂ O phase lb. mols/ft ³ x 10 ⁴ | Relative Interfacial Area Produced by the Pump |
|---------|--------------|----------|-------------------------------|---|--|---|---|--|--|
| 178 | 21.5 | 940 | 15 | 0.074 | 1.32 | 0.5 | 2.47 | 1.26 | 0.16 |
| 179 | 21.7 | 730 | 13 | 0.074 | 0.67 | 0.2 | 2.45 | 1.36 | 0.44 |
| 180 | 21.5 | 1150 | 16 | 0.074 | 2.14 | 3.3 | 2.35 | 1.40 | 1.14 |
| 181 | 21.7 | 1350 | 17 | 0.074 | 1.67 | 5.0 | 2.40 | 1.78 | 1.66 |
| 182 | 21.5 | 1560 | 20 | 0.074 | 1.26 | 6.6 | 2.36 | 2.17 | 2.39 |
| 183 | 21.5 | 1770 | 22 | 0.074 | 1.11 | 11.2 | 2.39 | 2.42 | 2.80 |
| 184 | 21.5 | 1980 | 27 | 0.074 | 1.86 | 15.0 | 2.08 | 2.97 | 6.75 |
| 185 | 21.5 | 2290 | 36 | 0.074 | 1.50 | 22.8 | 2.14 | 3.17 | 9.3 |

In Runs No. 178 - 185, Original C₇H₆O₂ concentration in Toluene was 2.60 x 10⁻³ lb. mols/ft³
Impeller No. 4 was used.

| Run No. | Temp. °C. | Pump RPM | Power Input to Motor Watts | Combined Flow Rate Ft ³ /sec. x 10 ² | Flow Ratio Ft ³ H ₂ O per Ft ³ C ₇ H ₈ | Gage Press. at Pump Outlet Inches H ₂ O | Final C ₇ H ₆ O ₂ Conc. in C ₇ H ₈ phase lb. mols/ft ³ x 10 ³ | Final C ₇ H ₆ O ₂ Conc. in H ₂ O phase lb. mols/ft ³ x 10 ⁴ | Relative Interfacial Area Produced by the Pump |
|---------|--------------|----------|-------------------------------|---|--|---|---|--|--|
| 186 | 23.5 | 625 | 11 | 0.074 | 1.32 | 1.3 | 2.27 | 1.39 | 0.86 |
| 187 | 23.5 | 830 | 12 | 0.074 | 1.35 | 2.4 | 2.19 | 2.16 | 1.81 |
| 188 | 23.5 | 1040 | 14 | 0.074 | 1.12 | 3.4 | 2.30 | 2.46 | 2.96 |
| 189 | 24.0 | 1250 | 14 | 0.074 | 1.00 | 7.1 | 2.20 | 2.97 | 5.06 |
| 190 | 24.0 | 1460 | 15 | 0.074 | 1.00 | 9.3 | 2.25 | 3.20 | 5.20 |
| 191 | 23.5 | 1670 | 18 | 0.074 | 1.00 | 12.6 | 2.20 | 2.58 | 3.40 |
| 192 | 24.0 | 2080 | 23 | 0.074 | 0.895 | 21.8 | 2.26 | 3.34 | 7.02 |
| 193 | 24.0 | 2500 | 41 | 0.074 | 0.89 | 32.0 | 2.26 | 3.39 | 7.58 |

In Runs No. 186 - 193, Original C₇H₆O₂ concentration in Toluene was 2.58 x 10⁻³ lb. mols/ft³
Impeller No. 1 was used.

APPENDIX II



Flow Rate - lbs./Sec at 25°C

Figure 5 - Orifice Calibrations



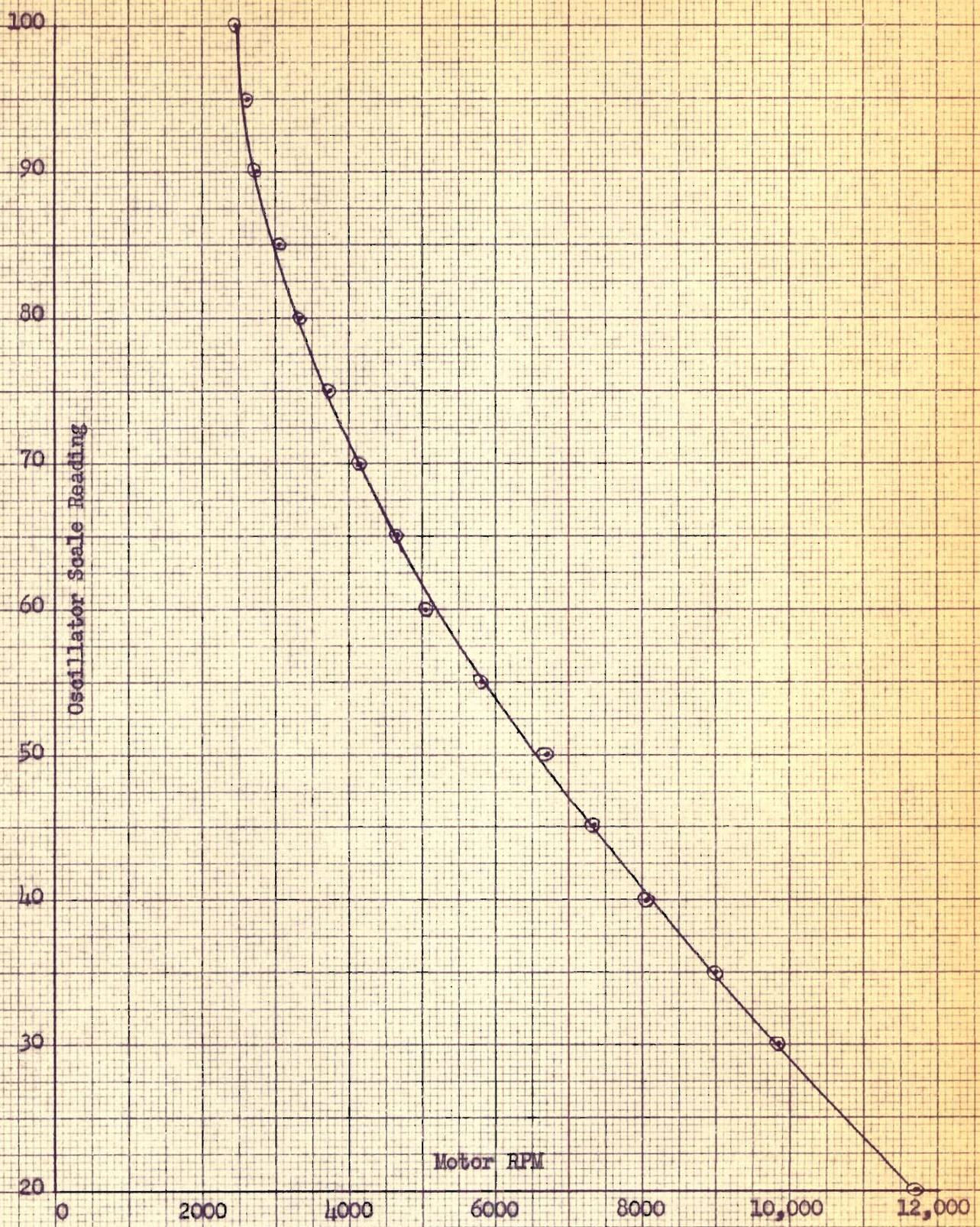


Figure 6 - Audio Frequency Oscillator Calibration

10 X 10 TO THE 24 INCH, 240 LINES ACCELERATOR.
MADE IN U. S. A.

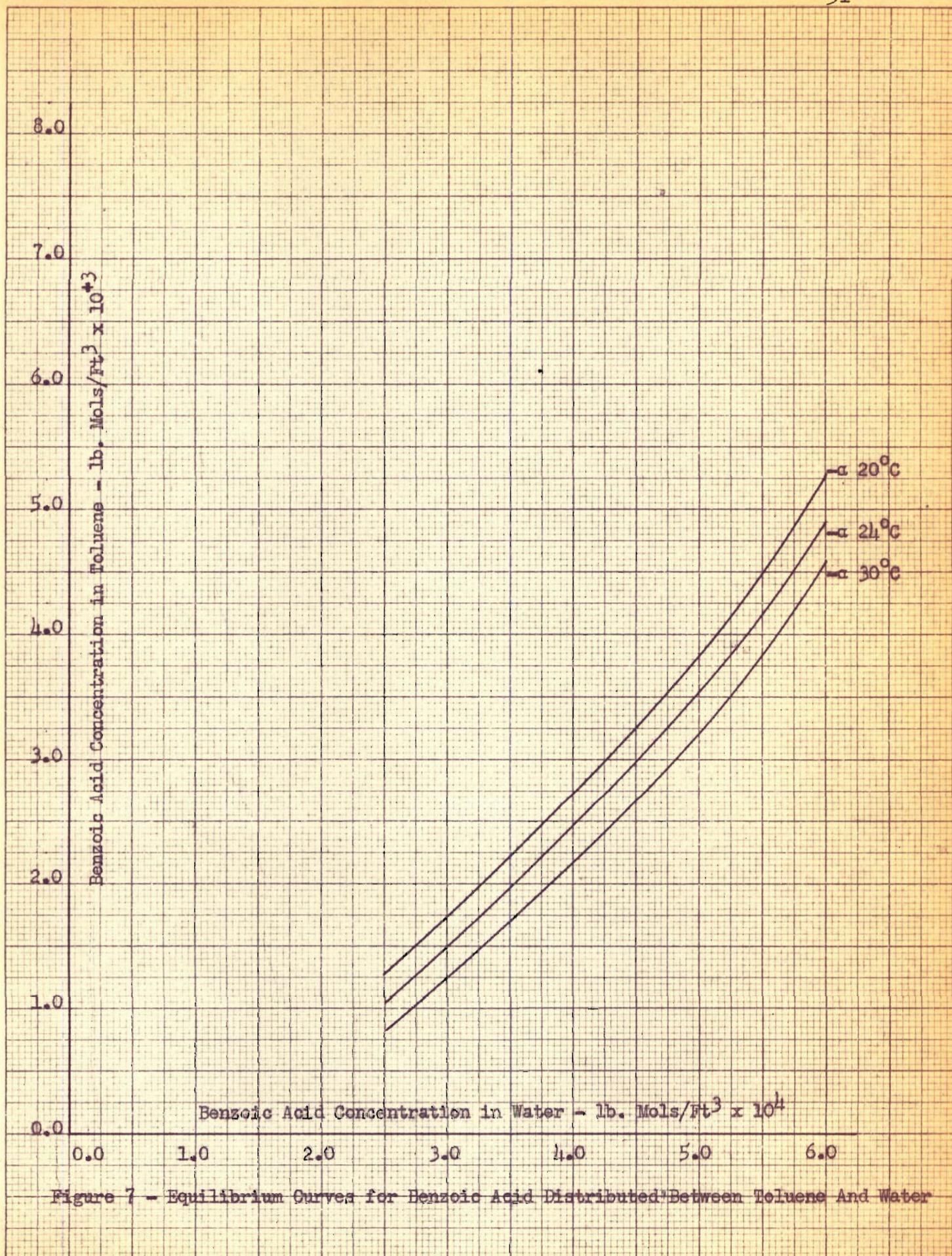


Figure 7 - Equilibrium Curves for Benzoic Acid Distributed Between Toluene And Water

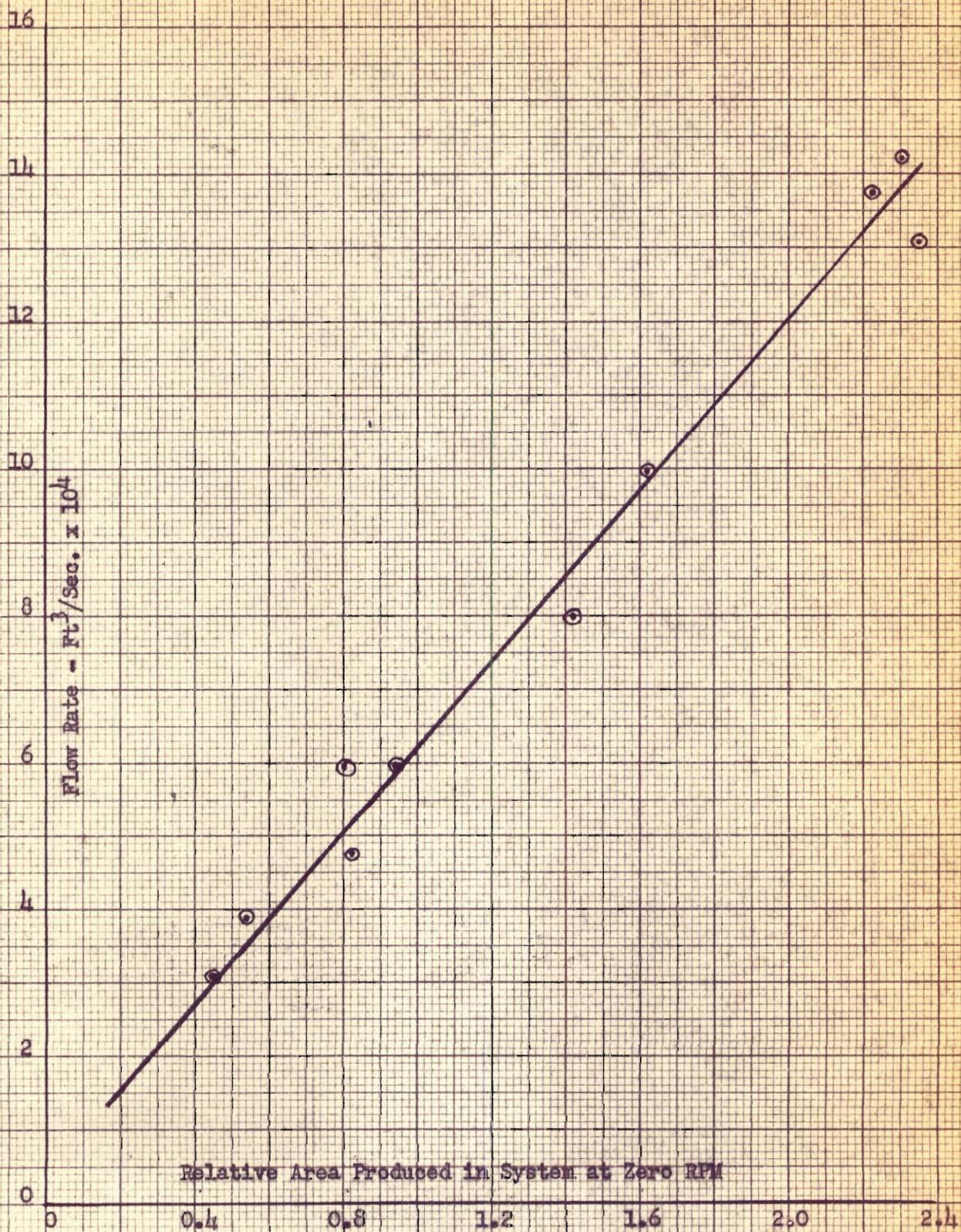


Figure 8 - Relative Area Produced at Zero RPM vs. Flow Rate

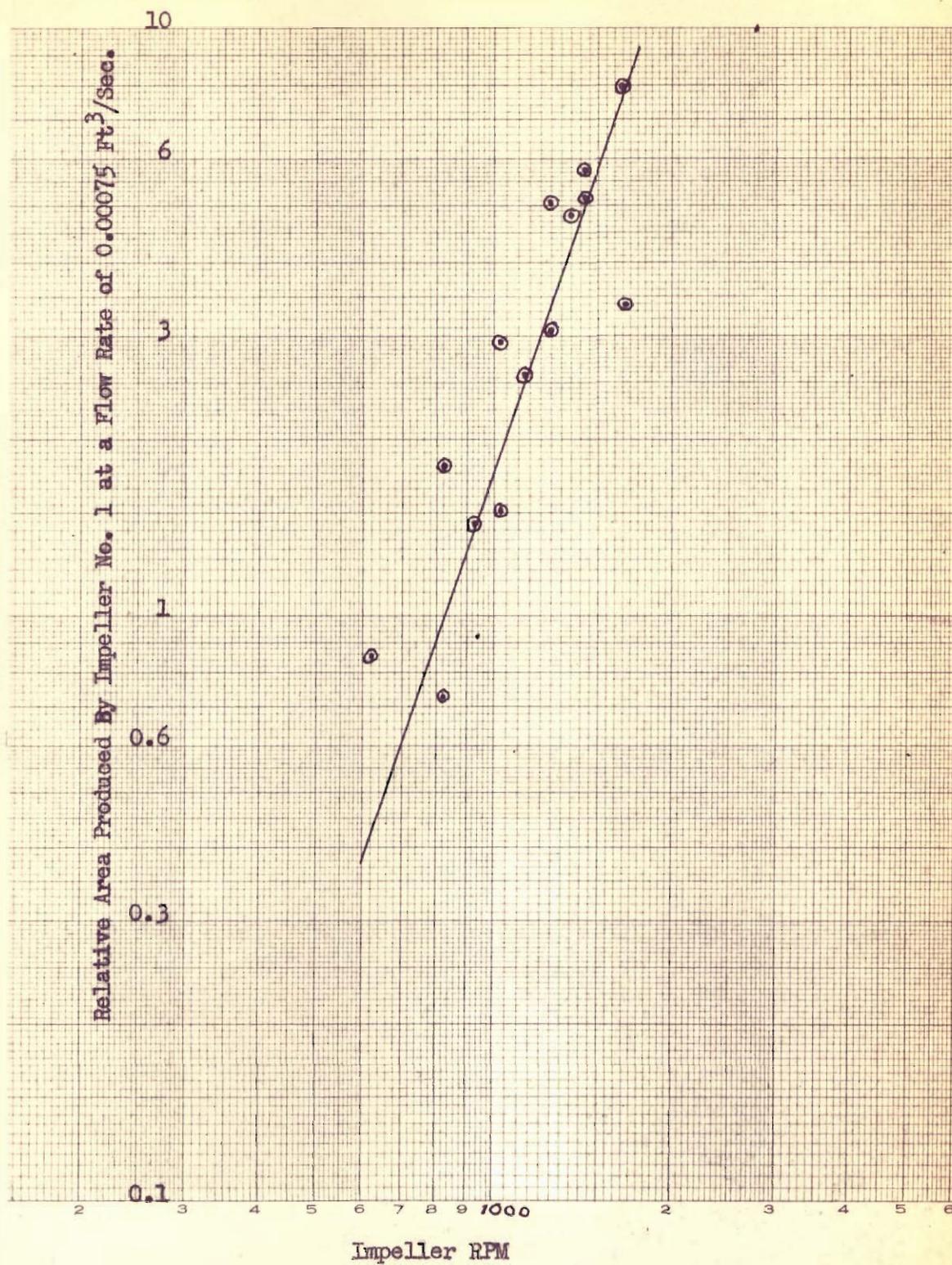


Figure 9 - Relative Area Produced By Impeller No. 1 at a Flow Rate of 0.00075 Ft³/Sec. vs. Impeller RPM

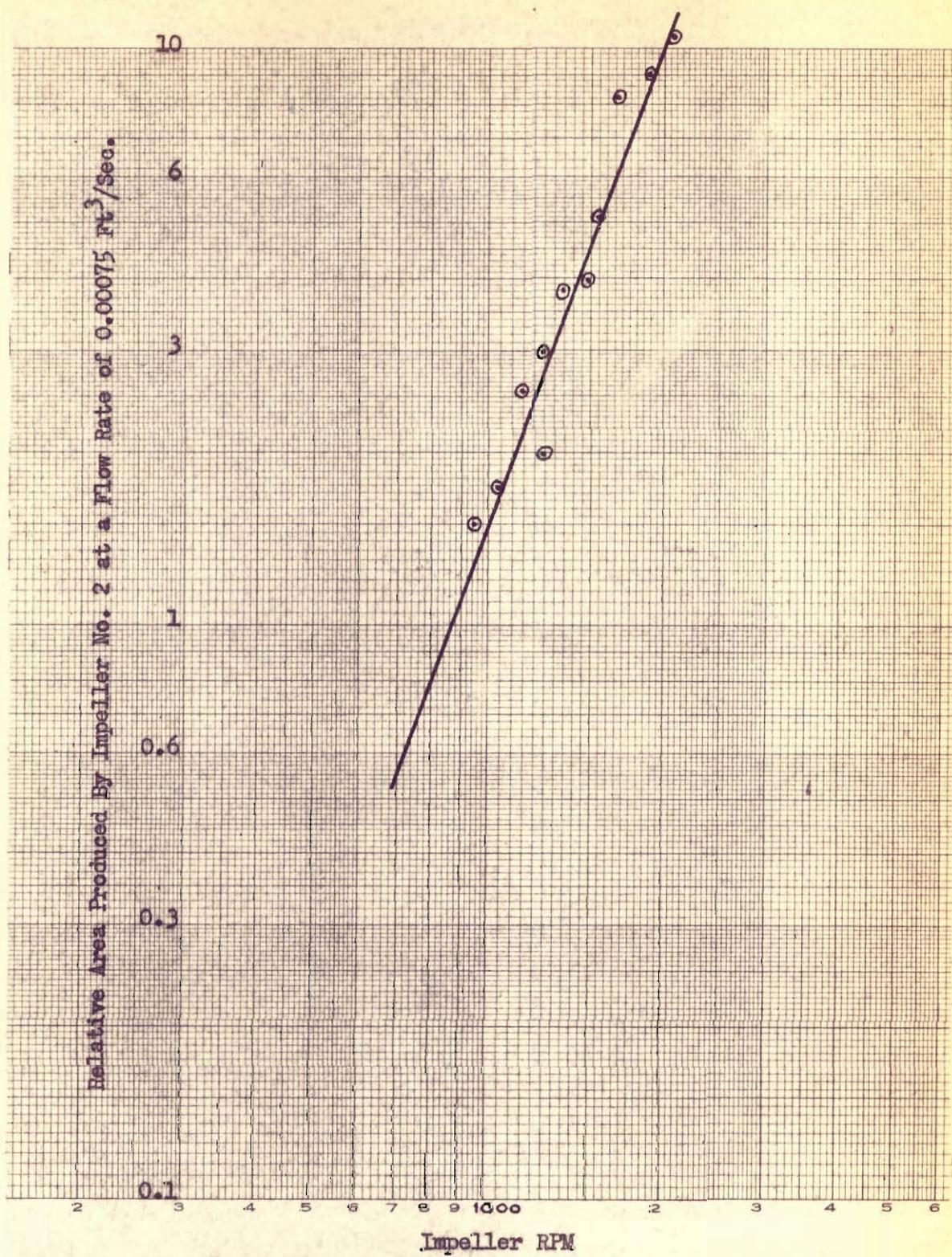


Figure 10 - Relative Area Produced By Impeller No. 2 at a Flow Rate of 0.00075 Ft³/Sec. vs. Impeller RPM

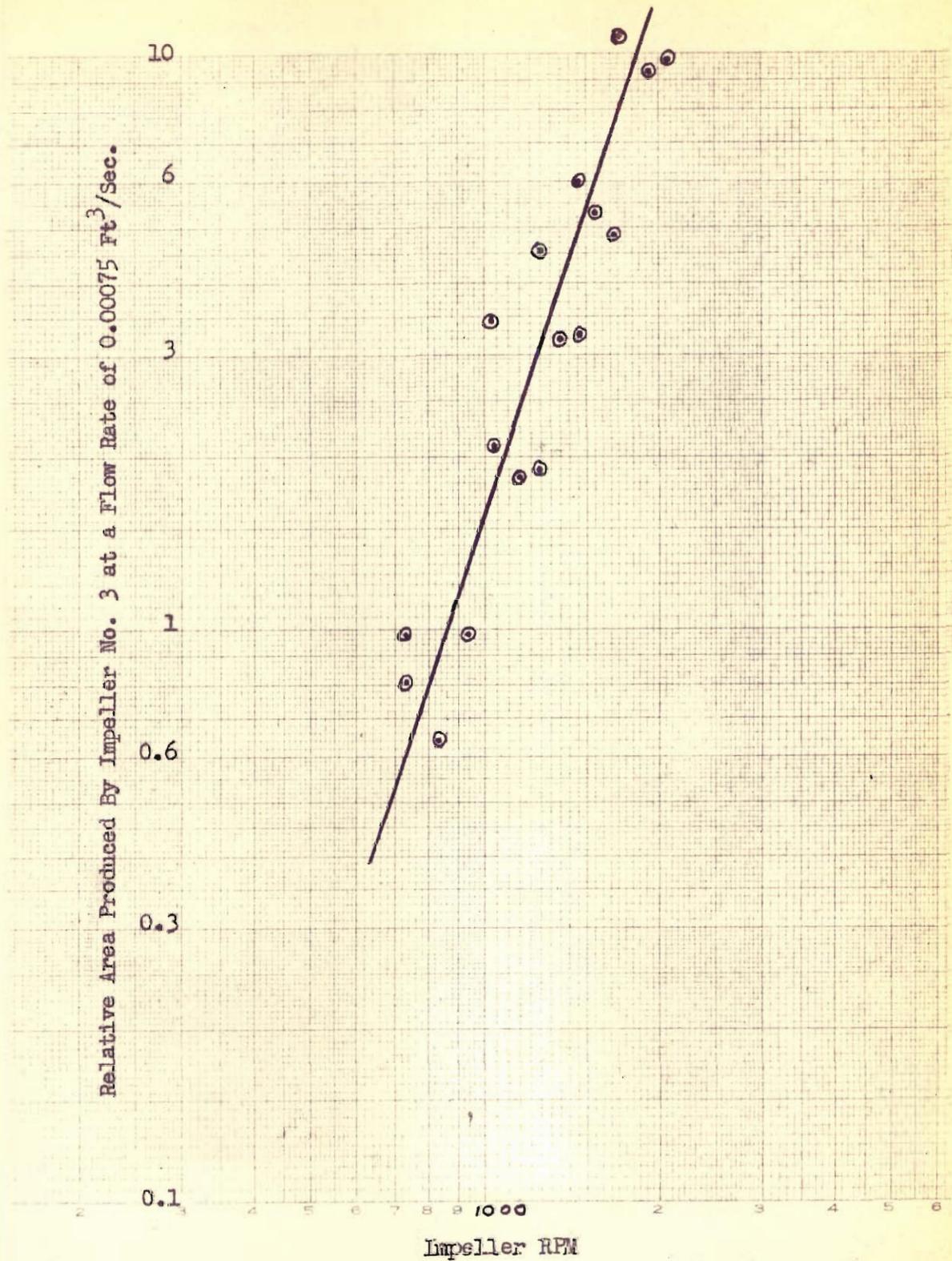


Figure 11 - Relative Area Produced By Impeller No. 3 at a Flow Rate of 0.00075 Ft³/Sec.
vs. Impeller RPM

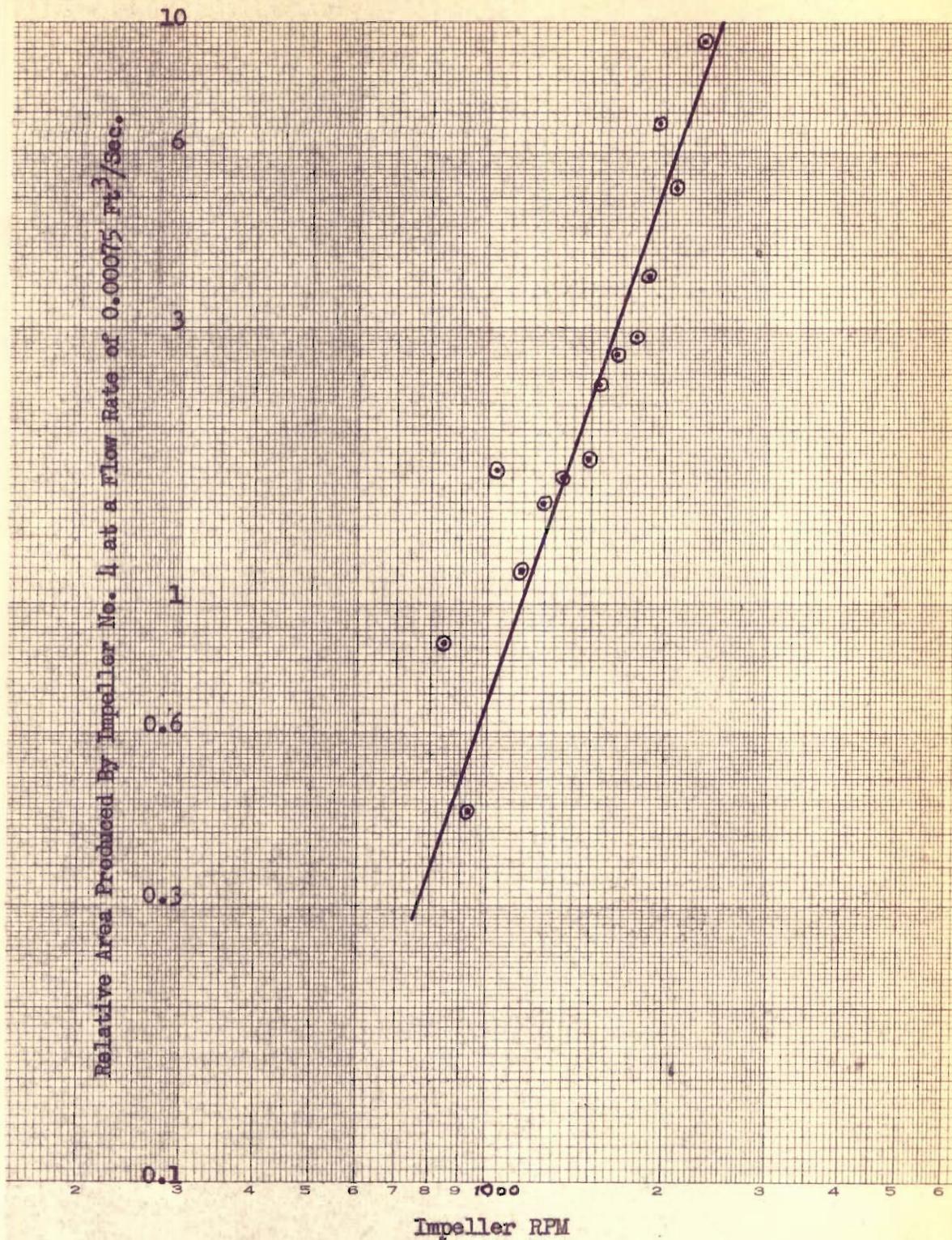


Figure 12 - Relative Area Produced By Impeller No. 4 at a Flow Rate of 0.00075 Ft³/Sec. vs. Impeller RPM

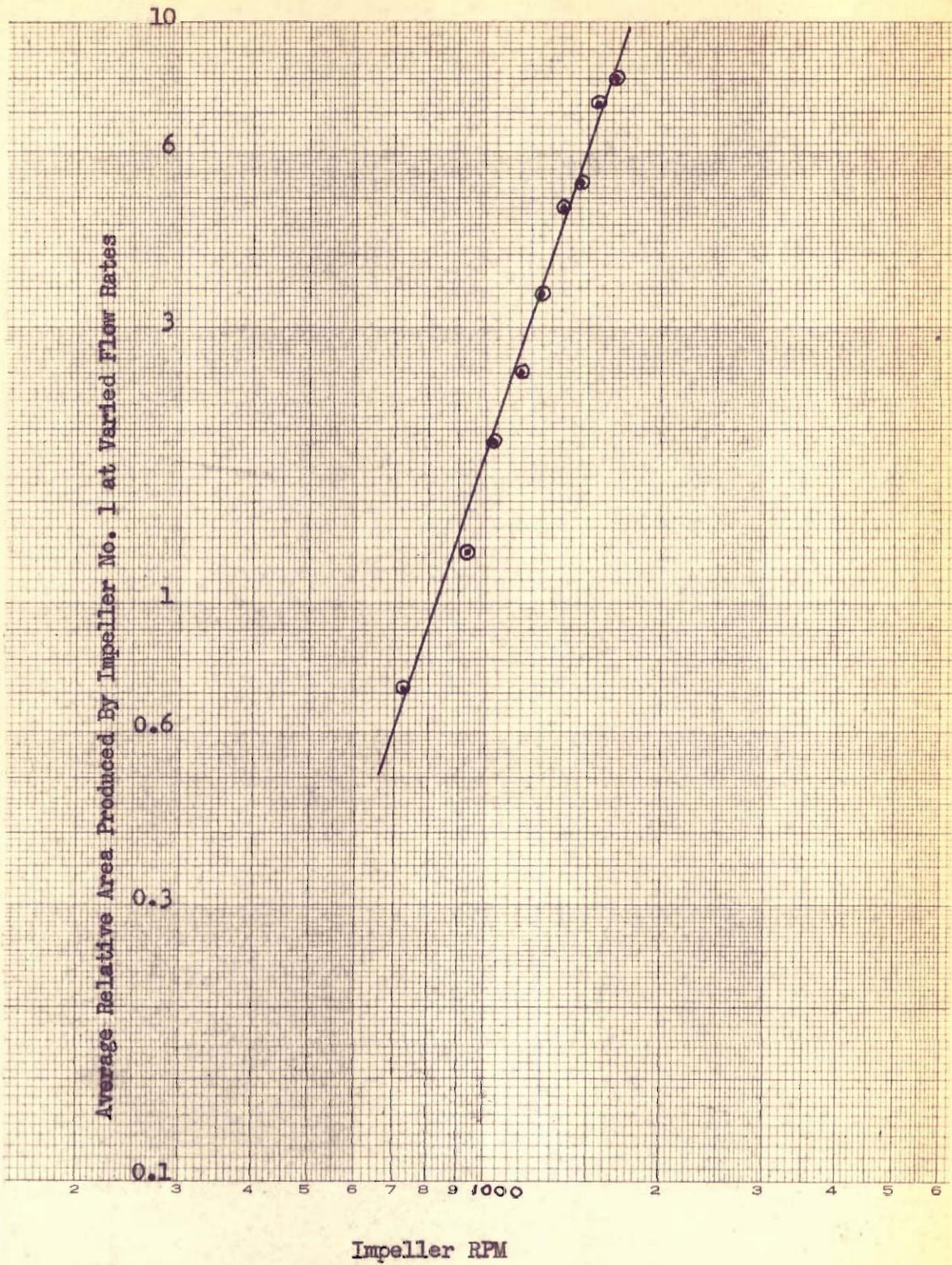


Figure 13 - Average Relative Area Produced By Impeller No. 1 at
Varied Flow Rates vs. Impeller RPM

APPENDIX III

Sample Calculation

Run No. 176 was chosen for this sample calculation. First the total area produced in the system was calculated from equation (II:8). The equation states,

$$kA = \frac{\ln \left[\frac{mC_r^0 + b}{mC_r + b - C_e} \right]}{\frac{m}{t} + \frac{1}{w}}$$

The data:

C_r^0 , the original toluene phase concentration of benzoic acid, = 2.84×10^{-3} lb. Mols/Ft³.

C_r , the final toluene phase concentration of benzoic acid, = 2.52×10^{-3} lb. Mols/Ft³.

C_e , the final water phase concentration of benzoic acid, = 3.07×10^{-4} lb. Mols/Ft³.

Combined flow rate = 0.00075 Ft³/Sec.

Flow ratio = 1.0 Ft³ water/Ft³ toluene.

Impeller No. 4 at 1880 RPM

Temperature = 27.0°C

Drawing a tangent to the 27° equilibrium curve in Figure No. 7 so that it touched the curve at the mean toluene phase concentration, 2.68×10^{-3} , it was found that

$m = 0.090$, and

$b = 1.87 \times 10^{-4}$.

Since the flow rate is $0.00075 \text{ Ft}^3/\text{Sec.}$, and equal volumes of toluene and water are flowing,

$$w = 0.000375, \text{ and}$$

$$t = 0.000375 \text{ Ft}^3/\text{Sec.}$$

Substituting the proper values in equation (II:8),

$$A_t = \frac{\ln(4.43/1.07)}{2940 k}$$

$$= \frac{4.83 \times 10^{-4}}{k}$$

From Figure No. 8, the area produced in "blank" flow at a flow rate of $0.00075 \text{ Ft}^3/\text{Sec.}$,

$$A_o = \frac{1.23 \times 10^{-4}}{k}$$

The area produced by the pump is the difference of the two areas, A_t and A_o , and accordingly,

$$A = 3.60 \times 10^{-4}/k.$$

Since k was assumed constant for all runs, A was given a relative value of 3.60, and plotted on Figure No. 13 on the ordinate representing 1880 RPM.

SUGGESTIONS FOR FURTHER WORK

Lack of appreciation of the ease with which equilibrium could be approached with the system used in this work, effected improper design of equipment. As a result, both the experimental data and the calculated results are not as accurate as they could be. Before attempting future work with this equipment, the pipe through which the liquids flow in contact with each other must be reduced to a minimum length to decrease the "blank" flow area produced. Also, the individual liquids should either be passed through separate orifices with smaller diameters, or through rotameters for more accurate measurement. Additional accuracy would result from using a system with a much lower value for k ; such a system would require higher impeller speeds for equilibrium to be attained, and would not be affected as much by "blank" flow, which, under these conditions, would become negligible.

With regard to further investigations into the use of centrifugal pumps in mixing operations, this work definitely suggests the use of a pump with an impeller which is substantially reduced in size so that a relatively little amount of liquid could be pumped at high impeller speeds without the buildup of a large downstream head. A study of impeller design would facilitate the overall design of a centrifugal pump system capable of

of achieving equilibrium mixing under conditions of relatively high flow against a low downstream head. It is proposed that for operations where more than one equilibrium stage is required, the pumps be arranged so that several impellers would be driven by one motor from the same shaft, thereby reducing total motor losses to a minimum (See Figure No. 14). The decanters would be sealed so that there will be no power consumed due to work against pressure head; that is, the head which resists flow from one pump would force liquid into the next. Once an impeller is found which satisfies the conditions stated earlier in this paragraph, such a system may be designed, and pilot plant data obtained. After obtaining pilot plant data for power required per unit of effective mixing, the proposed system could be evaluated with respect to its economic feasibility.

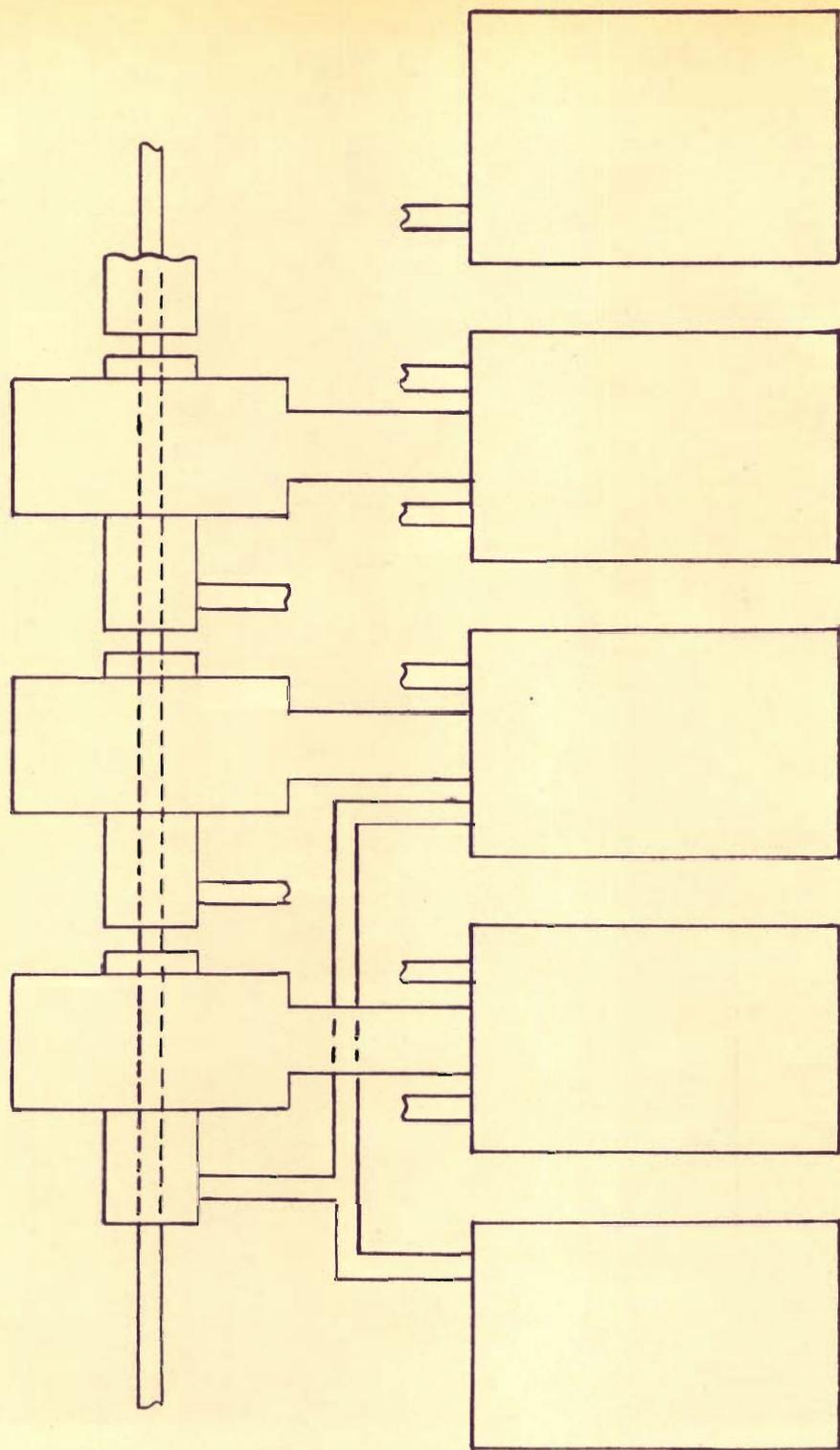


Figure 14 - Proposed Liquid-Liquid System Using Centrifugal Pumps
Driven From One Shaft As Mixing Stages.

BIBLIOGRAPHY

References

1. Whitman, W. G., "The Two Film Theory of Gas Absorption", Chemical & Metallurgical Engineering, Vol. 29, 1923. pp. 147-9.
2. Scheibel, E. G., "Fractional Liquid Extraction", Chemical Engineering Progress, Vol. 44, 1948. pp. 681-4.
3. Perry, J. H., Ed., Chemical Engineers' Handbook, 3rd Edition, Section 17, "Mixing of Material", McGraw-Hill, 1950.
4. Church, A. H., Centrifugal Pumps and Blowers, New York, N. Y., John Wiley and Sons, 1944. pp. 308.
5. Serner, H. E., "Simplified Approach to Mixing Problems", Chemical Engineering, Vol. 55, Jan. 1948. pp. 127-9.
6. Chaddock, R. E., "Practical Aspects in Design of Liquid Agitators", Chemical Engineering, Vol. 53, Nov. 1946. pp. 151-4.
7. Hixson, A. W. and M. I. Smith, "Mass Transfer in Liquid-liquid Agitation Systems", Industrial and Engineering Chemistry, Vol. 41, 1949. pp. 973-81.
8. Rushton, J. H., "Technology of Mixing", Canadian Chemical Process Industries, Vol. 30, May 1946. pp. 55-61.
9. Olney, R. B., and G. J. Carlson, "Power Absorption in Mixers", Chemical Engineering Progress, Vol. 43, Sept., 1947. pp. 473-6.

Additional Bibliography

"Agitation and Mixing Symposium", Industrial and Engineering Chemistry, June 1944.

Asquith, J. P., "The Unit Process of Mixing", The Industrial Chemist, Vol. 21, 1945. pp. 203-11, 262-9.

Chemical Engineering Catalog, 1950-51, Reinhold Publishing Corp., New York, N.Y.

Elgin, J. C., "Solvent Extraction" - A review, Industrial And Engineering Chemistry, Vol. 38 - 43, Jan. 1946-51.

Folsom, R. G., and C. K. Ferguson, "Jet Mixing of Two Liquids", Transactions of the American Society of Mechanical Engineers, Vol. 71, 1949. p. 73.

Olsen, J. F., and E. J. Lyons, "Technical Aspects of Continuous Process Systems", Chemical and Metallurgical Engineering, Vol. 52, May 1945. p. 118.

Rushton, J. H., "Mixing" - A Review, Industrial and Engineering Chemistry, Vol. 38 - 43, Jan. 1946-51.

White, A. M., and S. D. Sumerford, "New Data on Design of Simple Paddle Agitators", Chemical and Metallurgical Engineering, Vol. 43, 1936. p. 370.