

Studies in Agitation  
Development of a Method of Sampling

A Thesis  
Submitted in Partial Fullfillment  
of the Requirements for the  
degree of

Master of Science  
in  
Chemical Engineering

Submitted by  
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### Acknowledgment

This work was carried out under the direction of Prof. Harold Bunger of the Chemistry Department. The writer wishes to express his appreciation of the helpful suggestions offered by Prof. Bunger during the course of the investigation.

The writer is also very appreciative of the help given by Prof. I. H. Gerks of the Electrical Engineering Department in developing the amplifying apparatus.

## Introduction

Mixing and agitation— in spite of the fact that it is one of the most frequently used unit operations of chemical engineering—, from a standpoint of theory, design, and operation, is one of the least studied and least understood processes.

The literature, although very meagre, does contain several papers on this subject. Most of these papers are reports of investigations in which either immiscible solids or miscible liquids were mixed with liquids. However, there is one published research in which insoluble solids were suspended in liquids.

Probably the first investigation, certainly the first of importance, of a mixing operation was that of Wood, Whittemore, and Badger (1). They mixed miscible liquids and followed the progress of the mixing by conductivity measurements. As a result of this work it was shown that the common paddle agitator is an efficient mixing device. It was also shown that mixing was practically complete in a very short time. Their results did not justify a mathematical treatment.

(1) Wood, Whittemore & Badger: Chem. & Met.; 27, 1176  
(1922)

Following this work several papers appeared on problems in mixing. The results of these researches indicated that mixing takes place rapidly.

The first comprehensive study of mixing and agitation was that of Hixon & Crowell (2). In these investigations an attempt was made to isolate, as much as possible, and study, each of the numerous variables such as design and size of paddle, speed of stirrer (r.p.m.), relative position of paddle, etc., on the rate of dissolution of solids in liquids. Data are reported for the solution of naphthalene in benzene and for sodium chloride in water. The progress of the mixing operation was followed by direct sampling; that is, samples were dipped out in the latter case, and in the former, naphthalene balls, which acted as stirrers, were removed and the amount dissolved determined by weighing. On the basis of the results, a relation connecting the rate of solution with the amount of agitation was advanced; the so-called "cube-root" law. The results also indicated that mixing was rapid.

The latest work on this subject, that of A. M. White (3), was concerned directly with the suspension

- (2) Hixon & Crowell: Ind. & Eng. Chem. 23; 923, 1003,  
1164 (1931)
- (3) White, Sumerford, Bryant, & Lukins: Ind. & Eng.  
Chem. 24; 1160 (1932)

of insoluble solid in liquids; specifically, sand in water. In this work a simple paddle agitator was used. Sampling was accomplished by means of glass tubes inserted into the side of the mixing chamber at different levels and to various distances. In this manner the amount of sand per unit volume was determined at different points. The results showed that uniform sand distribution was never realized; that the height of the paddle had a marked influence on the distribution curves, and that the size of the suspended sand particles at any given point, was intimately associated with the velocity of the liquid at that point.

It may be seen from this brief review of the literature that mixing takes place very rapidly, and that in the above researches, in order to measure the progress or state of the mixing, samples were obtained by introducing foreign elements such as glass tubes or electrodes into the system being mixed. This practice constitutes an objection to previous researches because of the disturbance produced, in the mixture, by the sampling device itself. Further most of these devices were unsuitable for making instantaneous measurements.

Consequently it is seen that the method of sampling is very important. The requirements of a

sampling device are as follows: first, it must be amenable to rapid manipulation, and second, it must give an accurate picture of the state of the mixture. A few of the methods of sampling, which have been used conformed to one of these requirements (the first) but none met both the first and the second. A method, to comply with the second requirement, must be arranged in such manner that sampling may be effected without introducing tubes, wires or other disturbing factors into the mixture.

The method of sampling to be presented in this paper is designed to fulfill the above requirements. It is based on the fact that the amount of light being transmitted through a system is inversely proportional to the optical resistance offered by that system.

Therefore, the apparatus about to be described is, simply, a set-up with which varying amounts of light, passing through a mixture of solid particles and water, can be measured under varying conditions of agitation and concentration of solid particles.

## Apparatus

### Tank and Stand:

A cylindrical sheet-iron tank (A), 37 inches in diameter, 48 inches high and of about 200 gallons capacity, was mounted on a wooden stand as shown in Figures 1, 2, & 3 and the photographs of page 10. Circular openings (B), 1 inch in diameter, were cut, in the same vertical line at 12 inch intervals, in one side of the tank. These openings were fitted with converging lenses. The tank was equipped with a valve at the bottom.

### Stirring Device:

The stirring device was simply an iron paddle, 16 inches over-all length, made of 1 in. X 5/16 in. material, which was clamped on a vertical shaft (C) situated at the center of the tank.

### Shaft:

The shaft (C) was a 1½ inch extra heavy pipe, 8 feet long. It was supported at the top by two bearings (D and E) 18 inches apart. The shaft was fitted with lamps, slip-rings and a ring gear as shown in Figure 4.

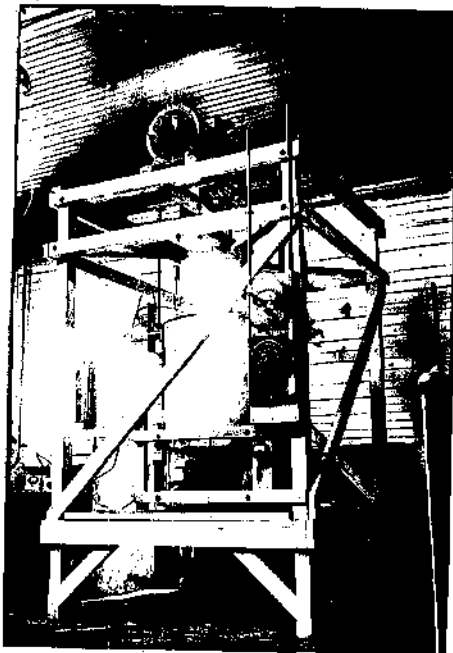


**Motor and Accessories:**

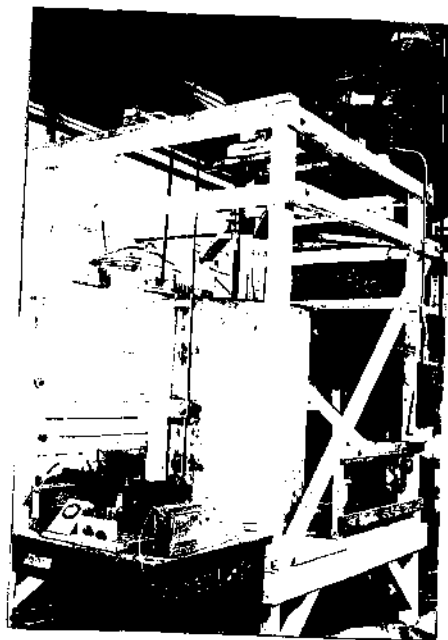
A shunt-wound D.C. motor (G) was connected by a jack-shaft and pinion (F) to the ring-gear on the shaft. A starting box (H) was provided for the motor and in order to have available a wide range of speeds a large resistance (I) was connected in series with the armature of the motor.

The above letters in parenthesis refer to Figures 1, 2, & 3 unless otherwise stated.

Photographs of Apparatus



Front View



Side View

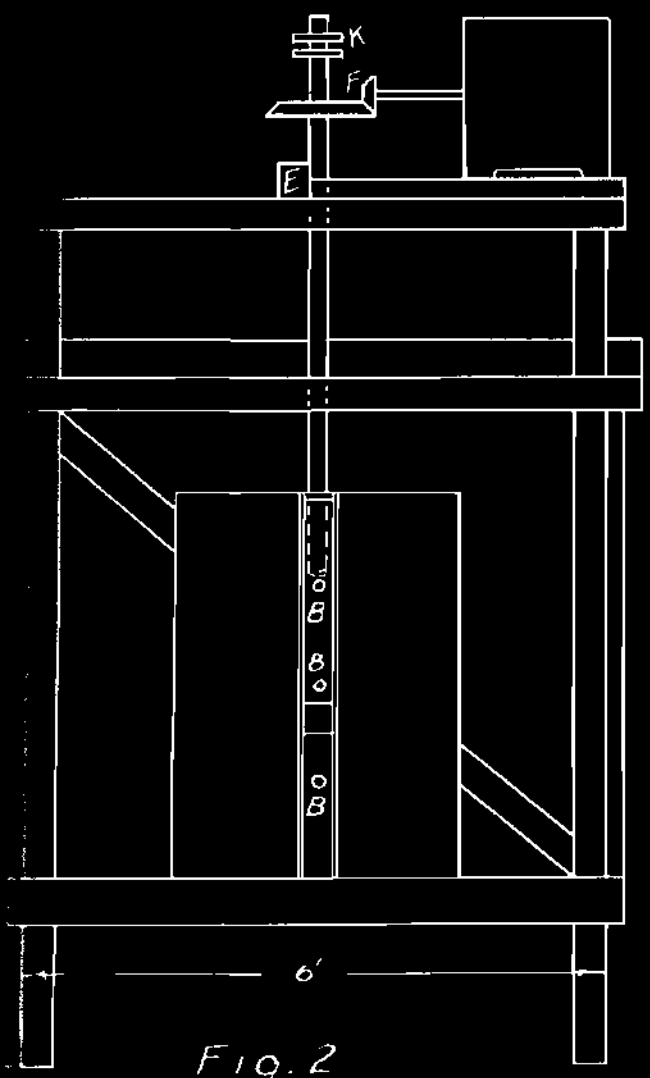


Fig. 2  
Side Elevation

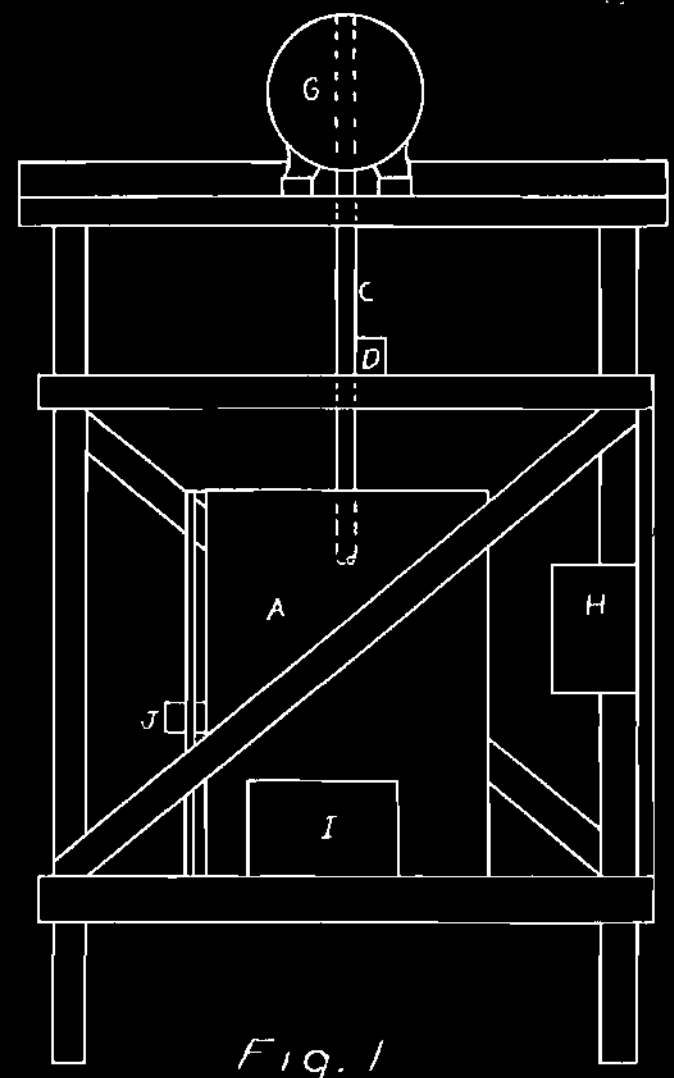


Fig. 1  
Front Elevation

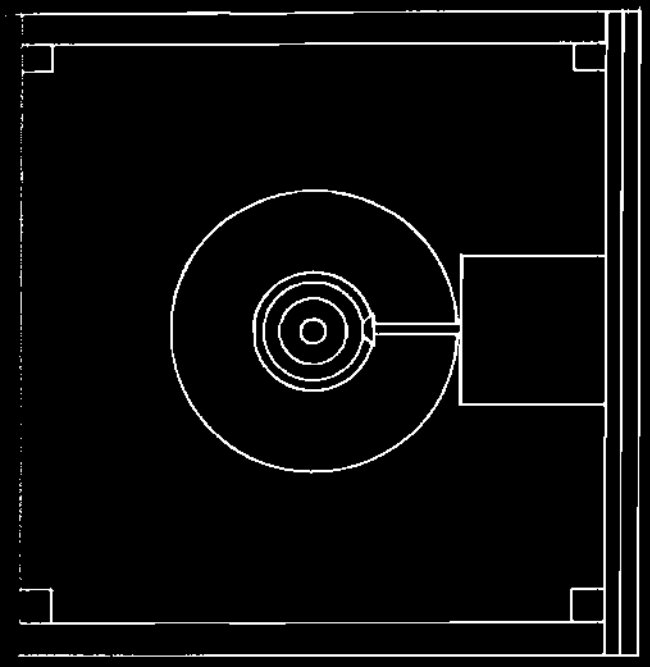


Fig. 3  
Plan

General View of Set-up

Scale: 1/2" = 1'

Legend

- A - Tank
- B - Windows
- C - Shaft
- D and E - Bearings
- F - Ring Gear and Pinion
- G - Motor
- H - Starting BOX
- I - Resistance
- J - Phototube Container
- K - Slip-rings

#### Lights and Connections:

The lamps (Fig. 4), 50 C.P. Automobile head-lamps, were connected in series, through a resistance directly to a 110 volt D.C. line. The current was transmitted to the lights by means of brushes and slip-rings. This circuit also included a switch.

#### Sampling Apparatus:

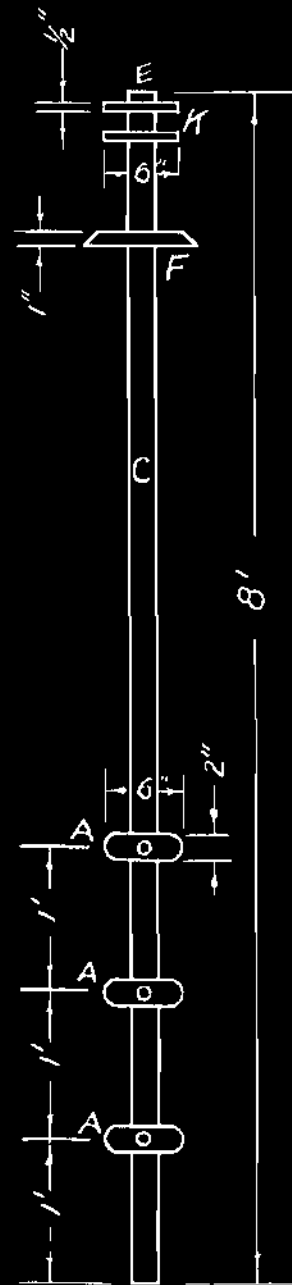
The hook-up of the apparatus for measuring the variation of light intensity is shown in Fig. 5. The photoelectric cell (G.E.; P.J. — 23) was placed in a small box (J in Fig. 1 & 2) arranged in such manner that any light striking the cell must pass through the windows of the tank. The box was mounted on a movable base.

#### Current Amplifier:

The amplifying device (Fig. 5) was constructed as shown. The meter used for measuring currents was a Weston rectifying micro-ammeter type 301-A.

#### Solid used for Suspension:

The solid used for suspension was fired, thoroughly washed grog of uniform size. The grog was screened through 20 and 30 mesh screens. The portion used was collected on the 30 mesh screen.

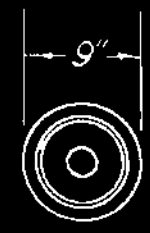


*Details of Shaft*

*Scale:  $\frac{3}{4}'' = 1'$*

*Legend*

- K - Brass Slip-rings*
- F - Ring Gear*
- C -  $\frac{1}{2}''$  Extra Heavy Iron Pipe*
- A - 50 C.P. Automobile Head-lamps—Sets of four*  
*Wiring for Lamps enters at E*



*Fig. 4*

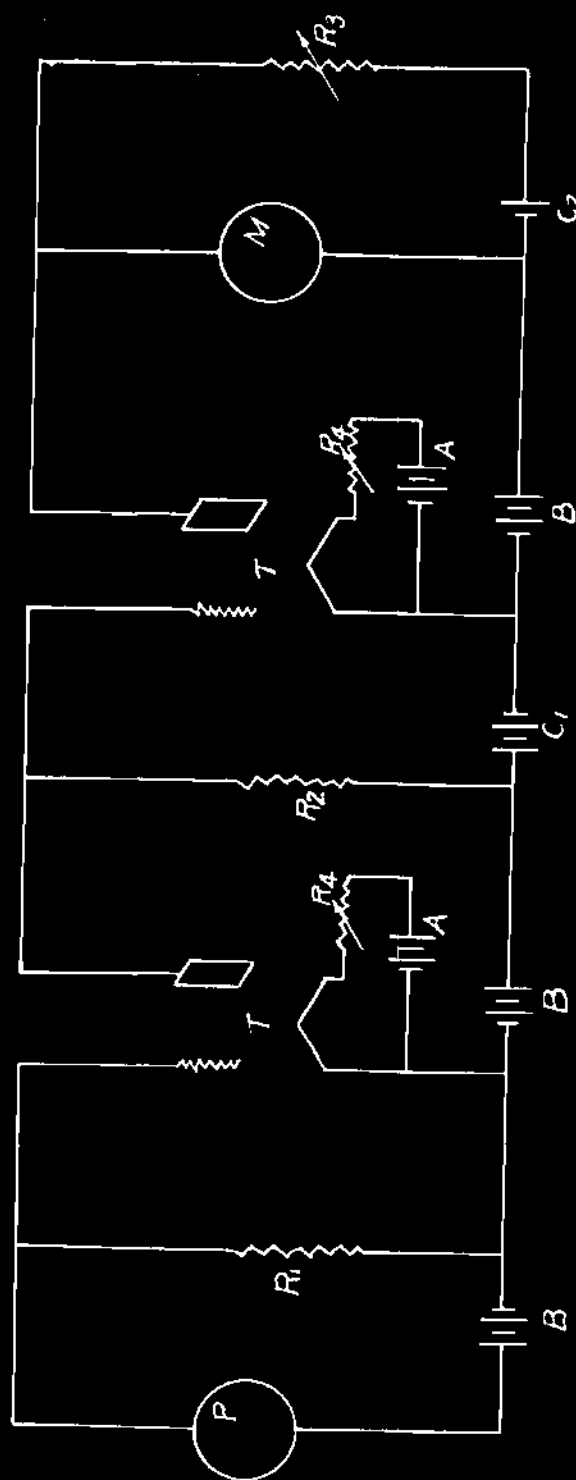


Diagram of Phototube and Amplifier

Legend

- P - Phototube (G.E. P.J-23)
- M - Weston Micro-ammeter (Type 301-A)
- T - Thermionic Tubes (RCA 201-A)
- A - 6 V. "A" Batteries
- B - 45 V. "B" Batteries
- C<sub>1</sub>, C<sub>2</sub> - 9 and 4½ V "C" Batteries, respectively
- R<sub>1</sub> - 2 Megohm Resistance
- R<sub>2</sub> - 2000 Ohm Resistance
- R<sub>3</sub> - 5000 Ohm Variable Resistance
- R<sub>4</sub> - 30 Ohm Variable Resistances

Fig. 5

#### Auxiliary Sampling Apparatus:

In Fig. 6 is shown an apparatus used for sampling, which is similar in principle to the method employed by A. M. White. It consists of seven glass tubes fixed to a movable base. The tubes are in a straight line, two inches apart at the bottom. When in position for use they removed samples from the path of the light through the tank. Vacuum was applied to the tubes to withdraw samples.

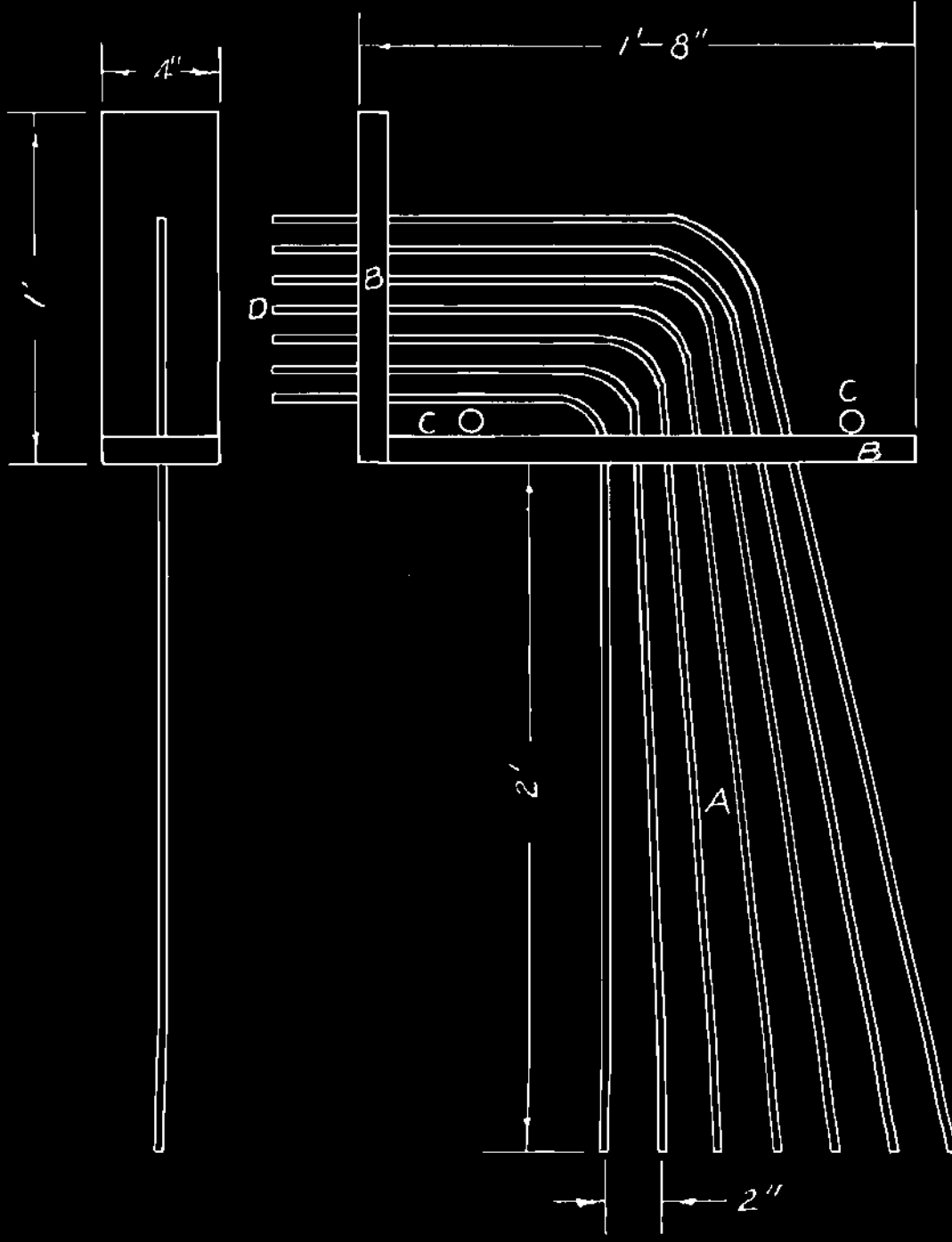


Fig. 6

*Auxiliary Sampling Apparatus*

Scale : 2" = 1'

*Legend*

- A - Glass Tubes (3/16")
- B - Boards (1" X 4")
- C - Clamps
- D - Vacuum applied



### Procedure

The procedure followed for collection of data was quite simple. The tank was filled, to a level of  $3\frac{1}{2}$  feet, with water and the stirrer started and adjusted to the desired speed (65 r.p.m. for these experiments). The lights were turned on and the ammeter reading recorded. Other readings were obtained by adding various amounts of grog noting the meter reading and then inserting the device of Fig. 6 and withdrawing samples, at 2 inch intervals from the path of the light. The tubes and connections were drained between runs.

The amounts of water and grog in the samples were determined.

The paddle was 8 inches from the bottom of the tank in these tests.

All samples and ammeter readings were taken at a height of 2 feet above the bottom of the tank.

It was found that the thermionic tubes of the amplifying apparatus required about thirty minutes warming-up before a constant reading could be obtained.

## Results

The experimental results are given in Tables I to VI inclusive. The first column in each is the tube number; number 1 tube was about 3 inches from the center and number 7 tube was .2 inches from the wall of the tank. The second column shows the total volume of water and the third, the total weight of solid in a particular sample. The last column gives the results when calculated as parts of grog per million parts of water, by weight.

The first run (Run #1) was made with zero concentration of grog. The ammeter reading was 220 micro-amperes.

Table I

Run No. 2	Meter reading—200(micro-amps.)		
Tube No.	Vol. water (C.C.)	Wgt. grog (gms.)	m.g. of grog per liter
1	255	0.0000	0.0
2	260	.0002	0.8
3	285	.0110	38.8
4	255	.0002	.8
5	215	.0003	1.0
6	310	.0162	52.2
7	295	.0100	33.9
<b>Average</b>			18.2

Table II

Run No. 2		Meter reading—170(micro-amps.)	
Tube No.	Vol. water (C.C.)	Wgt. grog (gms.)	m.g. of grog per liter
1	255	0.0015	5.9
2	250	.0012	4.8
3	300	.0030	10.0
4	255	.0016	6.3
5*	300	.0202	67.1
6	285	.0180	63.3
7	310	.0409	132.3
Average			41.4

Table III

Run No. 4		Meter reading—130(micro-amps.)	
Tube No.	Vol. Water (C.C.)	Wgt. grog (gms.)	m.g. of grog per liter
1	280	0.0004	1.4
2	245	.0002	0.8
3	305	.0021	6.8
4	210	.0159	72.3
5	300	.0131	48.6
6	290	.0231	78.3
7	305	.0369	121.0
Average			47.0

Table IV

Run No. 5		Meter reading--90(micro-amps.)	
Tube No.	Vol. water (C.C.)	Wgt. grog (gms.)	m.g. of grog per liter
1	280	0.0041	14.6
2	245	.0014	5.7
3	305	.0046	14.9
4	210	.0228	109.6
5	300	.0252	84.0
6	290	.0675	232.7
7	305	.1100	360.6
Average			117.4

Table V

Run No. 6		Meter reading--50(micro-amps.)	
Tube No.	Vol. water (C.C.)	Wgt. grog (gms.)	m.g. of grog per liter
1	255	0.0065	25.3
2	235	.0053	22.6
3	280	.0082	29.3
4	260	.0101	38.7
5	280	.0444	159.7
6	265	.0875	327.7
7	290	.1278	442.2
Average			149.3

Table VI

Run No. 7	Meter reading--0(micro-amps.)		
Tube No.	Vol. water (C.C.)	Wgt. grog (gms.)	m.g. of grog per liter
1	270	0.0061	22.6
2	250	.0058	23.2
3	300	.0216	72.5
4	285	.0153	54.1
5	300	.0960	320.0
6	285	.1436	502.1
7	300	.2433	805.6
Average			257.6

In Table VII the Run No. with the corresponding meter reading and the average grog content are collected.

Table VII

Run No.	Meter reading (micro-amps.)	Average wgt. of grog (m.g./l)
1	220	0.0
2	200	18.2
3	170	41.4
4	130	47.0
5	90	117.4
6	50	149.3
7	0	257.1

The meter readings are plotted against the amounts of grog (m.g./l) in Figure 7.

The average weight of grog (m.g./l) found in the six samples from any tube are listed in Table VII, together with the distance from the center of the tank.

Table VIII

Tube No.	Average amount of grog (m.g./l)	Distance from center
1	11.7	3 inches
2	9.6	5 "
3	28.7	7 "
4	46.9	9 "
5	113.6	11 "
6	209.4	13 "
7	315.9	15 "

The values in the last two columns of Table VIII are plotted in Figure 8.

*Effect of "Grog" Concentration on  
The Amount of Transmitted Light*

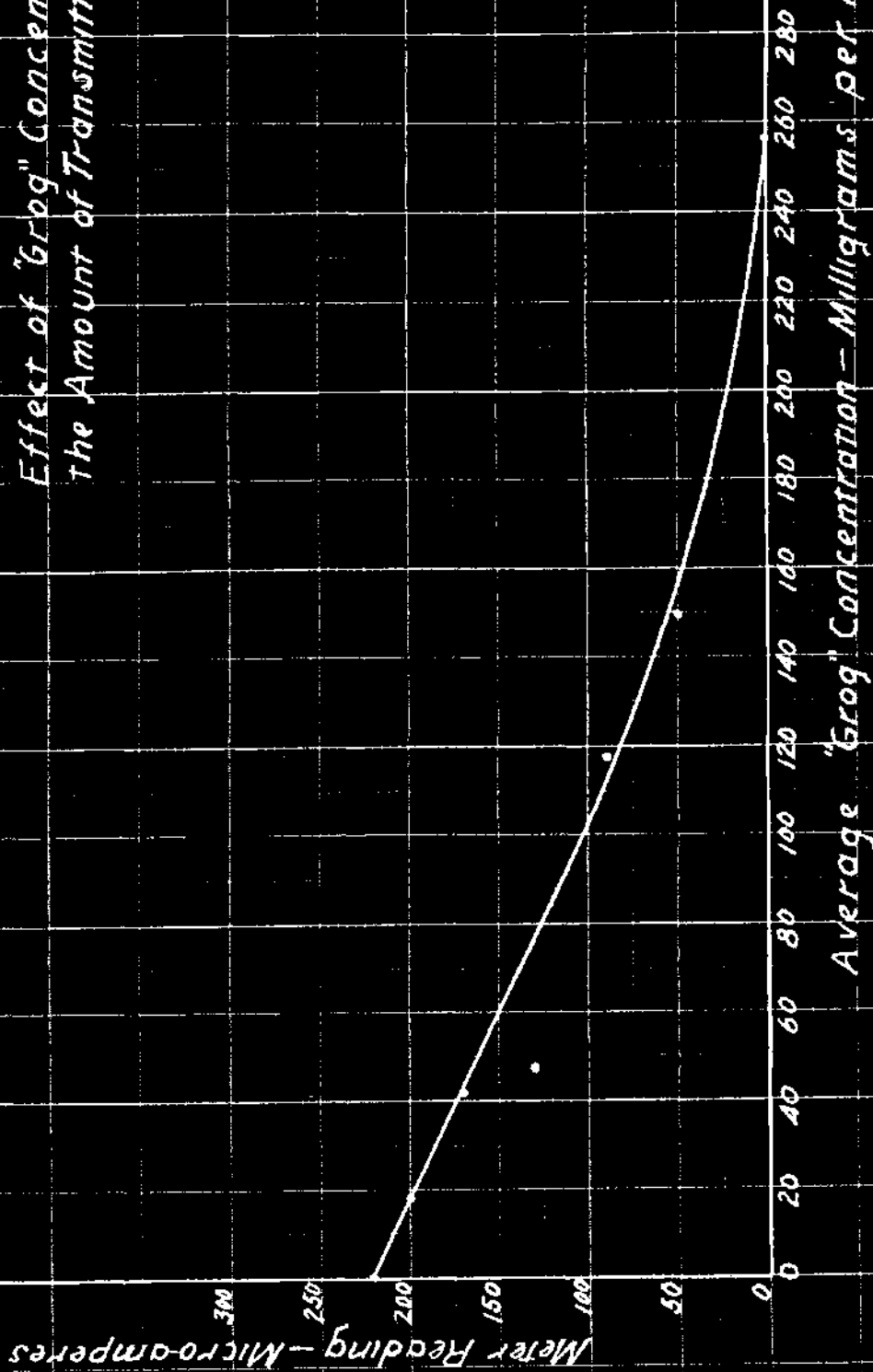
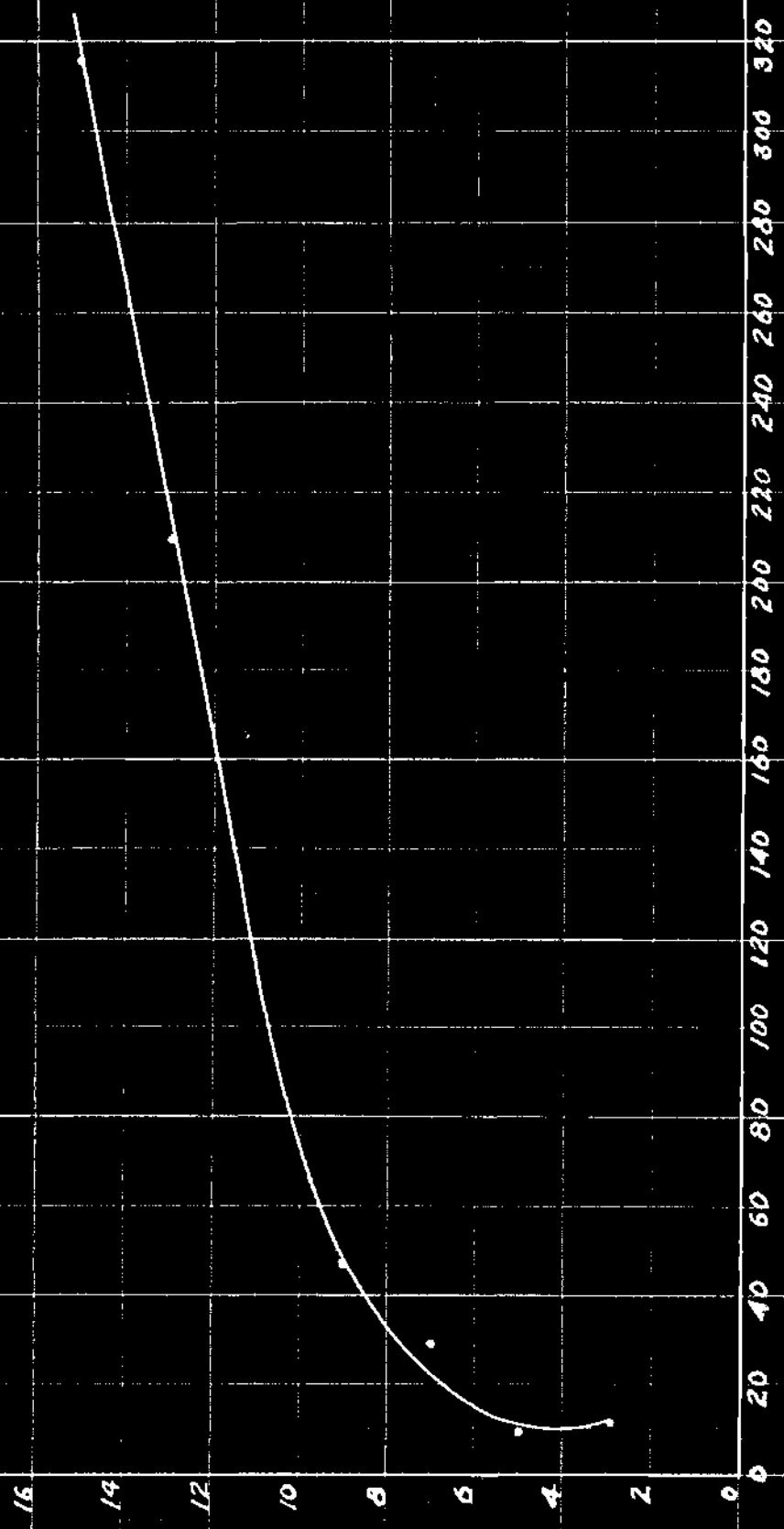


Fig. 7

Distance of tubes from center of tank — Inches

*Distribution of Solid Particles*



Average of all samples from the different tubes — Milligrams per liter

Fig. 8



## Discussion of Results

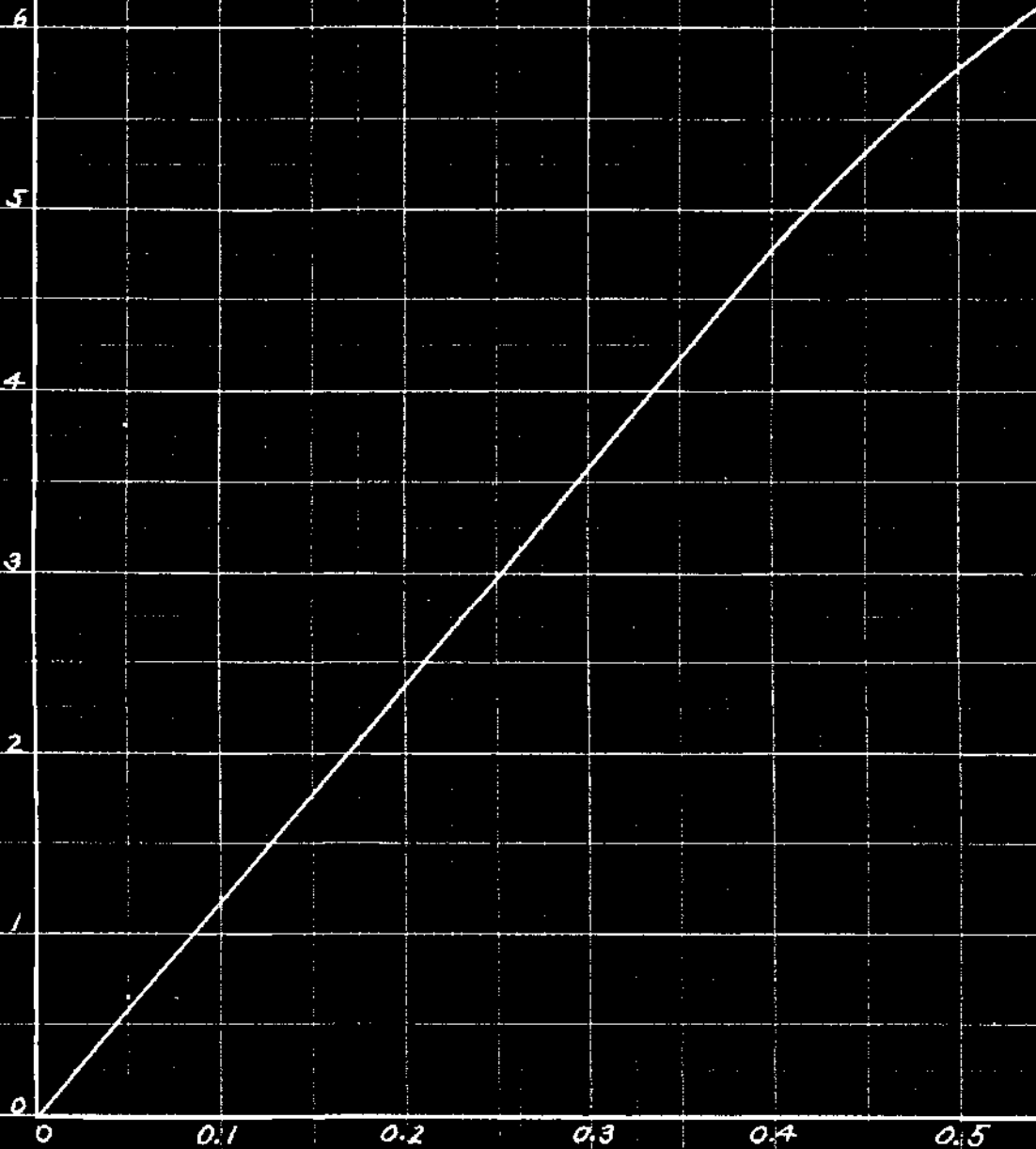
The data of Table VII as pictured graphically in Figure 7 show that the amount of light passing through the mixture of grog in water, decreases as the concentration of grog increases. This curve indicates clearly that the quantity of light which can pass through the mixture is some function of the solid concentration. It follows, then, that this method is suitable for measuring the concentration of solids in liquids.

Figure 9 shows that the photoelectric current is proportional to the amount of light incident upon the phototube. Therefore the terms, amount of light and photoelectric current are inter-changeable.

The data of Table VII might be subjected very simply, to an approximate mathematical treatment, but it is obvious that any relation arrived at, would be valid only when applied to the particular apparatus as used in this work. Any other light intensity or solid particles of different sizes

Curve for G.E. PJ-23  
Phototube at constant  
Anode Voltage of 45 v.

Anode current in Micro-amperes



Light in Lumens

Fig. 9

and densities would not give the same results. Therefore, Fig. 7 is not intended as a calibration curve, for this method of sampling, except as applied to the set-up of apparatus which was used in this research. But the form of this curve will be valid in any case.

The data of any one of Tables I through VI show very clearly that the solid particles are never uniformly distributed. If the dispersion were uniform the curve of Figure 8 would be a straight line; this plot may be used as a rough measure of the distribution of the grog. These results confirm the work of A. M. White on this phase of the suspension of insoluble solids in liquids.

No experimental work was carried out on the solution of solids in liquids, but it is believed that this method of sampling offers an excellent means of studying such a system.

It was hoped that the power consumption and time elements of mixing could be studied by this method, but the apparatus used was found to be unsuitable for such work. Under more favorable conditions these factors can probably be investigated.

### Summary

An apparatus for sampling a system during the process of agitation and mixing has been described.

It has been shown that the total amount of light passing through a mixture of grog and water is a straight line function of the grog concentration at moderate values, above this the ratio does not remain constant.

Further it has been shown that the distribution of the solid is not uniform.

Also it has been pointed out, that by use of the method given in this paper, future work on several aspects of mixing and agitation problems should be somewhat simplified and made more accurate.