THE WAGNER TURBIDIMETER
AS AN INSTRUMENT FOR THE DETERMINATION
OF
SPECIFIC SURFACE AND PARTICLE-SIZE DISTRIBUTION
OF
PORTLAND CEMENT

AN INSTRUMENT FOR THE MEASUREMENT
OF THE
THERMAL CONDUCTIVITY OF WATER,
AND FOR THE
INVESTIGATION OF PHENOMENA IN HEAT TRANSFER
IN THE LIQUID AND AT THE LIQUID-SOLID BOUNDARY

Thesis by

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ABSTRACT

The Wagner Turbidimeter has given promise of being a suitable instrument for use in the determination of the surface exposed by the particles in samples of Portland cement. Since the extent of this surface is an important factor in controlling or predicting the behavior of cements in the production of concrete, a simple but accurate method for its determination is highly desirable in connection with investigations and acceptance tests.

The purpose of the present investigation was to make a critical study of the advantages and short-comings of this instrument and to recommend modifications of apparatus or procedure which might improve its performance. The various steps in the procedure and the possible sources of uncertainty and error in the results have been examined in detail.

Several modifications of the apparatus have been found beneficial and numerous changes in the procedure to be used have been found essential for obtaining satisfactory results. With the suggested improvements, the instrument appears to offer a reasonably satisfactory method for routine tests of specific surface (usually expressed as square centimeters of surface per gram of cement), and also for approximation of the distribution of the sizes of the particles of the cement.

Although the results are not entirely accurate and reproducible, they are within useful limits and the instrument is recommended for use in acceptance test work on specific surface measurement, until a more accurate method, utilizing instruments of reasonable cost and with simple operating procedure has been developed. For research or investigational purposes, where cements of varying chemical and physical characteristics (including optical properties) are being studied, it is believed that the turbidimeter method may not be sufficiently accurate and should not be depended upon, unless special precautions are observed and great care taken in the work.

Foreword

The type of turbidimeter used in this investigation was developed at the United States Bureau of Standards in Washington by L.A.Wagner of the Cement Reference Laboratory. The method has been described by Mr. Wagner in a paper entitled, "A Rapid Method for the Determination of the Specific Surface of Portland Cement," in Volume 33, Part II of the Proceedings of the American Society for Testing Materials (1933). Further information is included in a typewritten booklet entitled, "Turbidimeter Instructions," furnished with the instruments, which are obtained through the Cement Reference Laboratory. A copy of this booklet is included in this report at Appendix I.

The investigation here reported consisted in the installation of the turbidimeter according to the instructions given, followed by careful studies of each step of the procedure to determine its effectiveness and any errors or uncertainties likely to enter the determination through its use. On the basis of the findings of these studies, modifications of apparatus and method were devised in an attempt to overcome the difficulties found. Comparative tests of the original and modified methods are included in this report. Turbidimeter results are also compared with those obtained by means of the microneter described by Knapp (Industrial and Engineering Chemistry, Analytical Edition, January, 1934).

The report is subdivided into the following parts:

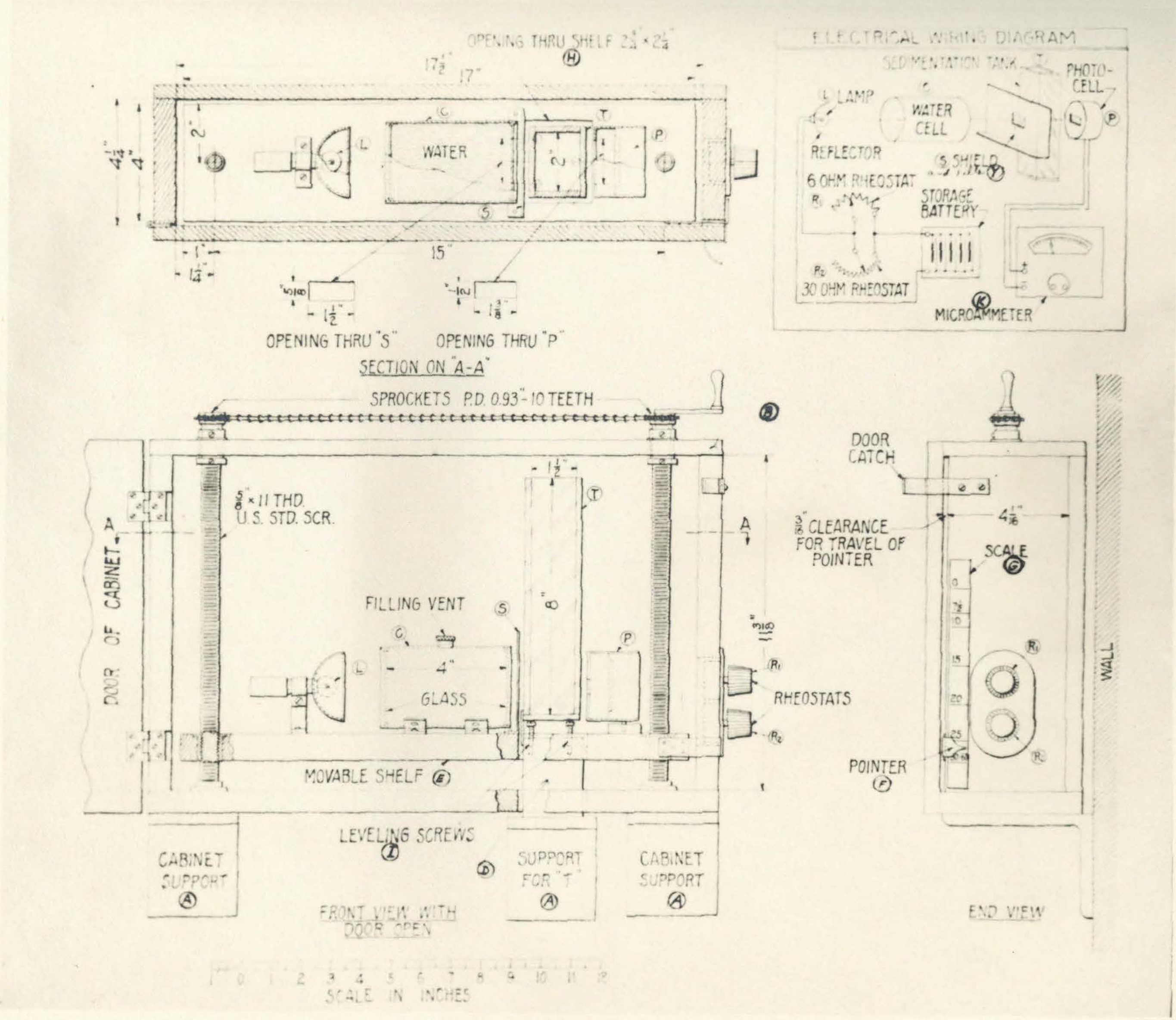
PART I

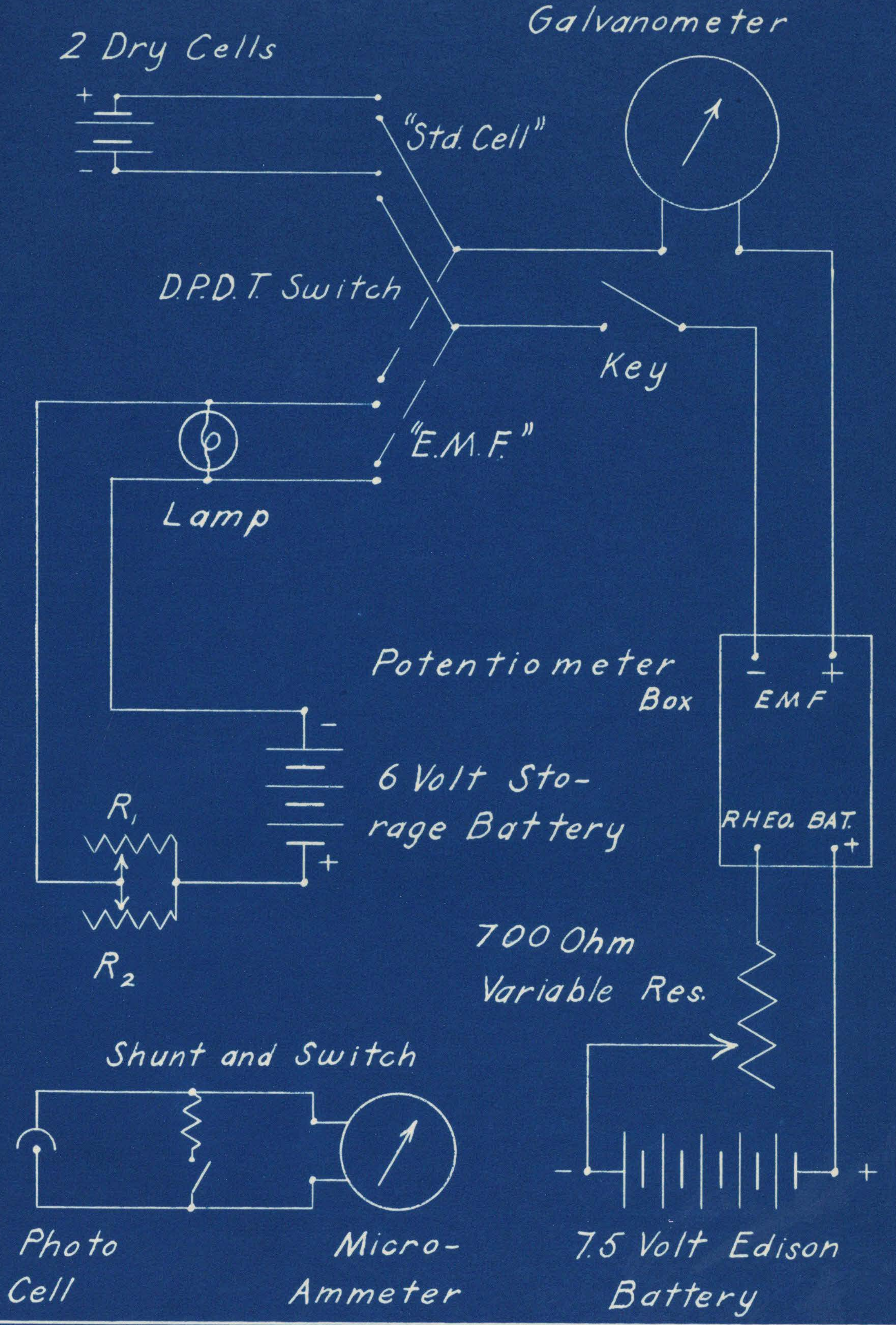
Recommended Procedure
for Operation of
The Modified Wagner Turbidimeter

I. List of Parts of the Apparatus

3	Supporting Brackets
1	Turbidimeter Cabinet
1	Base for Tank
1	Microammeter and Shunt
1	Photoelectric Cell
1	Sedimentation Tank
1	Stirring Motor and Brush
1	7-inch Funnel
1 pkg	15-inch Filter Paper
1	6-volt Lead Storage Battery
1-	Bottle of Oleic Acid and Dropping Stopper
1	Can of Filter Charcoal
1	Stirrer and Mounting
1	6-inch Büchner Funnel and Lead Ring
1 pkg	Filter paper to fit Büchner Funnel
2	5-gal. Bottles
1	1-gal. Wide-mouth Bottle
1 lb	Anhydrous $CaCl_2$ (4-mesh)
45"	Glass tubine with Stopcock and Enlarged End
5 gal	Kerosene
1	Wash Bottle and $CaCl_2$ Tube
1	Leeds and Northrup Potentiometer "students' type" No. 7651
1	Leeds and Northrup Galvanometer No. 2320
1	Variable Resistance, 0-700 ohms
1	Stopwatch
1	No. 200 Sieve, Tyler Standard, with Cover and Pan

1	Ostwald Viscosity Pipette
1	Pyknometer with Thermometer, capacity 10 cc.
1	Key for Potentiometer Circuit
1	7.5-volt Edison Storage Battery
2	4-inch Funnels
2	600-cc. Beakers
1	Test tube
1	Analytical Balance and Weights
1	Thermometer, 0°_50°C by tenths
2	Dry cells (new)
1	Desiccator
1	Camels Hair Brush
1	Piece Black Oilcloth, 2 feet square
1	D.P. D.T. Switch
1	Filter Stand
1	bottle Brombenzene
1	pkg Filter paper, 25 cm.


OPENING THRU SHELF 2¼" × 2¼"
Ⓗ
17½"
17"
4¼"
4"
2"
WATER
Ⓒ
Ⓛ
Ⓣ
Ⓟ
Ⓢ
15"
1"
1¼"
1½"
1⅜"
OPENING THRU "S"
OPENING THRU "P"
SECTION ON "A-A"
ELECTRICAL WIRING DIAGRAM
SEDIMENTATION TANK Ⓣ
PHOTO-CELL Ⓟ
Ⓛ LAMP
Ⓒ WATER CELL
REFLECTOR
Ⓢ SHIELD
6 OHM RHEOSTAT R₁
STORAGE BATTERY
R₂ 30 OHM RHEOSTAT
Ⓚ MICROAMMETER
SPROCKETS P.D. 0.93" - 10 TEETH
Ⓑ
⅝" × 11 THD. U.S. STD. SCR.
A
DOOR OF CABINET
1½"
8"
FILLING VENT
4"
GLASS
11⅜"
R₁
R₂
RHEOSTATS
MOVABLE SHELF Ⓔ
LEVELING SCREWS Ⓘ
Ⓓ
CABINET SUPPORT Ⓐ
SUPPORT FOR "T" Ⓐ
FRONT VIEW WITH DOOR OPEN
0 1 2 3 4 5 6 7 8 9 10 11 12
SCALE IN INCHES
DOOR CATCH
⅜" CLEARANCE FOR TRAVEL OF POINTER
4⅟₁₆"
SCALE Ⓖ
0
7½
10
15
20
25
POINTER Ⓕ
WALL
END VIEW



REVISED WIRING DIAGRAM

Drawn by D.L.H. 1/30/34

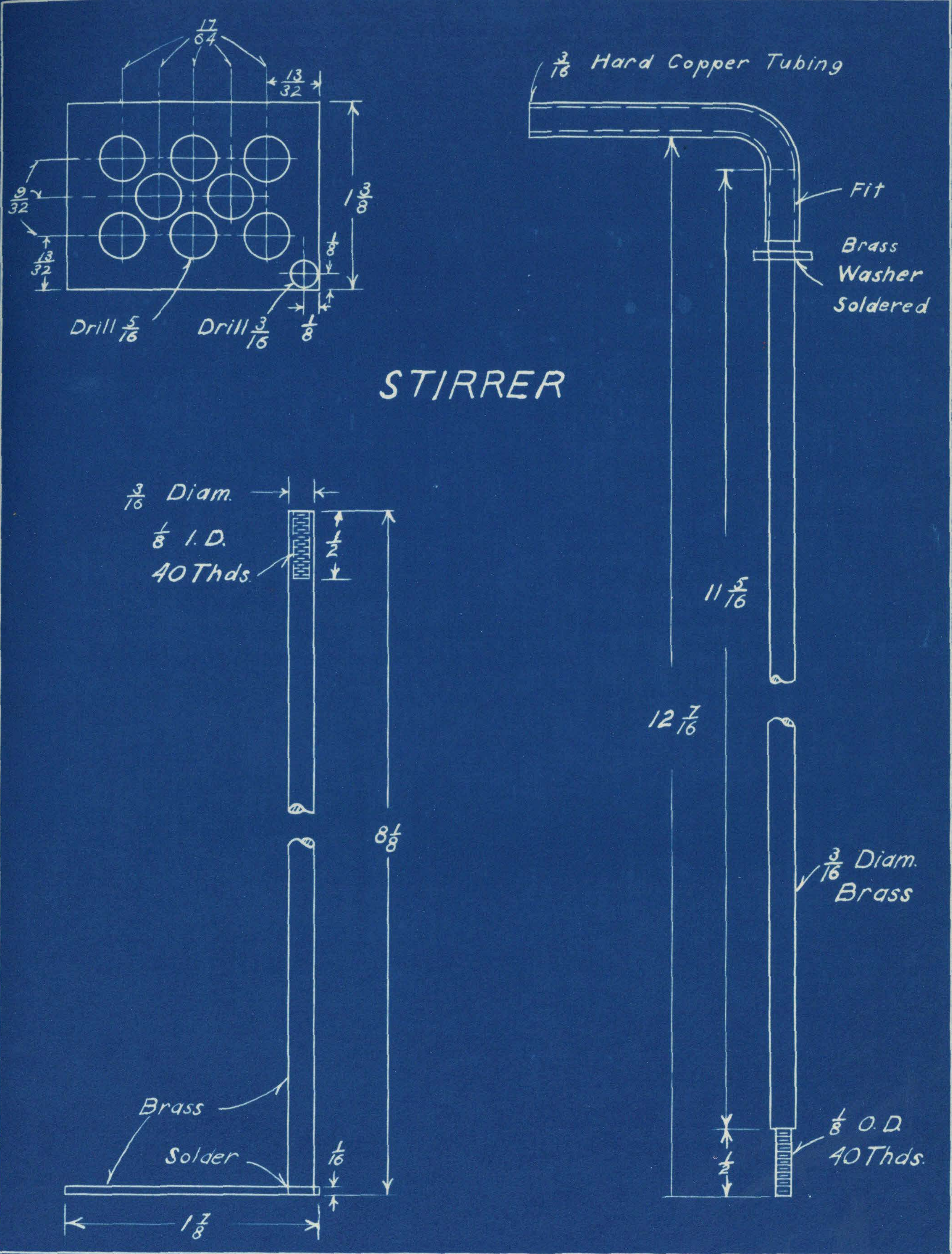


FIG. I

Scale: 1 Inch = 1 Inch

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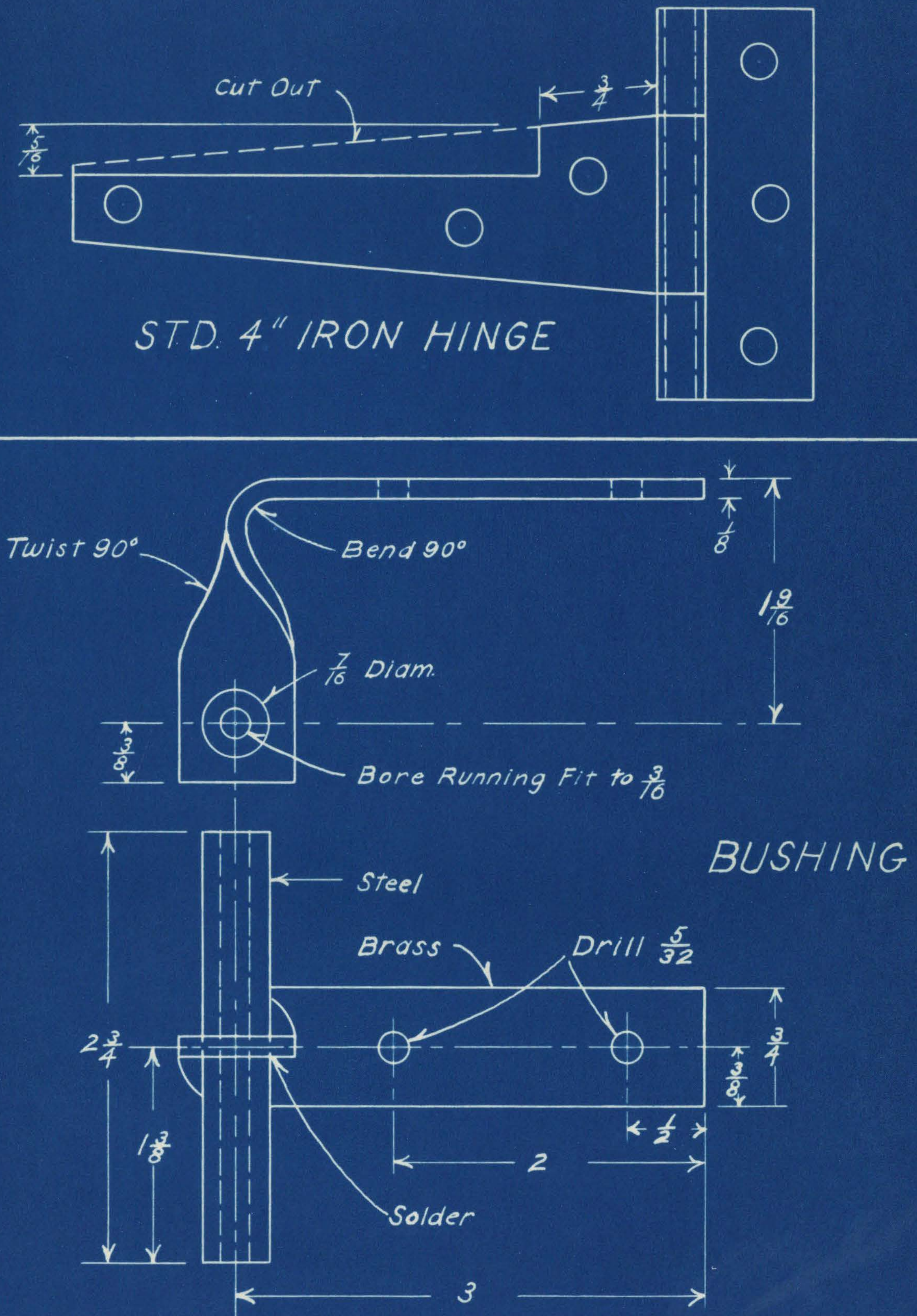

STD. 4" IRON HINGE

BUSHING

FIG II

Scale: 1 Inch = 1 Inch

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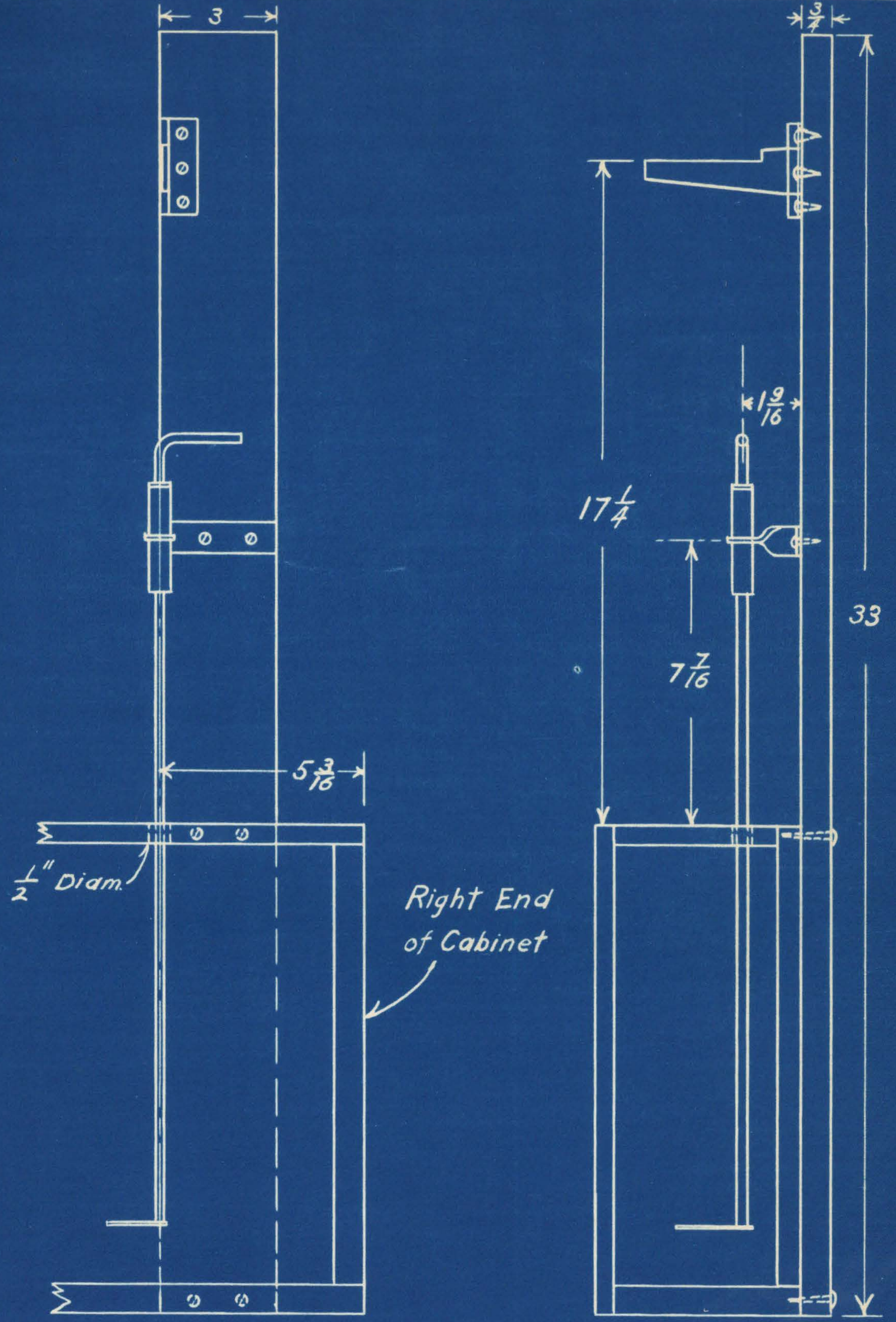


FIG. III

Showing Location and Mounting of Stirring Rod and Hinge

Scale: $\frac{1}{4}$ Inch = 1 Inch

Drawn By D.L.H. 1/15/34

II. Instructions for Installation

1. Attach the supporting brackets (A), see drawing, for the cabinet (B) and base (D) for the tank (T) to a masonry or concrete wall, column, or pier. Attach tank bracket 3/4 inch lower than cabinet brackets, Level and mount cabinet and tank base on the brackets.

2. Adjust height of tank base (D) so that the surface of the liquid in the tank (containing 335 ml.) is exactly 1 3/8 inches above the top of the shelf (E) when the pointer (F) is at the 0.0 mark on the scale (C).

3. Lower shelf until the pointer is at the 30-60 position on the scale and adjust position of tank so that the tank has equal clearance from all sides of the slot (H) in the shelf.

4. Adjust leveling screws (I) until tank has equal clearance from all sidesof shelf slot with shelf at 0.0 level.

5. Connect lamp leads to storage battery, using soldered joints or binding posts at the battery, not clips.

6. Connect leads from lamp to potentiometer, connect Edison cell, dry cells, key, resistance and galvanometer. See diagram of connections.

7. Connect leads from photocell (P) to microammeter and shunt (E) being sure to get correct polarity (see diagram of connections); try it out with a very dim light.

8. Mount stirring motor and connect to 110 volts a.c. or d.c., attach brush to stirring motor.

9. Clean the bottles and tubing and dry them; put 3/4 pound of anhydrous $CaCl_2$ in the kerosene and allow to stand for several days, shaking occasionally. Decant kerosene from $CaCl_2$ and add two tablespoons of activated charcoal, shake and let stand over night. Filter off charcoal

allowing filtrate to run into the other bottle, from which the $CaCl_2$ has been removed and washed out with some of the first filtrate. When the filtration is over put the $CaCl_2$ in the empty bottle, after cleaning our charcoal, and fit the Büchner funnel in the latter. Fill the enlarged part of the glass tubing with $CaCl_2$, using glass wool to hold it, and bend the tubing into a syphon. Fit this syphon and the 7-inch conical funnel into the full bottle. All stoppers should be cork.

10. Mount the stirring rod assembly on the back of the cabinet (see drawing). The stirring plate should have clearance from the tank on all sides. Connect a grounded wire to the tank base and the stirrer mounting.

11. Fill $CaCl_2$ tube using glass wool to hold it in, and fit wash bottle together.

12. Place some $CaCl_2$ in the desiccator.

III. General Operating Instructions.

1. Size of sample.- Use a sample of sufficient size so that the initial reading will be close to 20.0 microamperes. As a rule 0.3 gm. for fine cement (more than 95% passing a No. 200 sieve), 0.4 gm. for a medium cement (85-95%), and 0.5 gm. for a coarse cement (less than 85% passing the 200 sieve). The sample is taken from the throughput of the 200 mesh determination. This residue is removed from the pan after the dust has settled and rolled on a piece of oilcloth to thoroughly mix it. After rolling back and forth a dozen times the cement is transferred to a porcelain dish, which is placed in a desiccator until ready for use.

2. Preparation of suspension.- Place weighed sample in test tube containing 10-15 ml. of kerosene and 5 drops of oleic acid. Stir

the mixture thoroughly with rotating brush for at least a minute until no lumps appear on the bottom of the test tube while the stirring takes place. Transfer to tank, which must be perfectly clean, using the wash bottle to wash out the test tube. Put 15 ml. more kerosene in the test tube, add one drop of oleic acid, and stir again to wash off the brush. Add these washings to the tank, and use wash bottle again for the test tube. Finally adjust the volume of the suspension to 335 ml.

Note: With very fine cements a drop or two more of oleic acid may be necessary. This will be the case when a mirror-like layer of the finest particles remains adhering to the walls of the tank after the suspension is poured out. Wipe this mirror off and repeat the run, using more oleic acid.

Note: The volume is most easily adjusted by putting the tank in position and raising the shelf to the 7.5 level, when the light will enable the meniscus to be seen clearly.

3. Agitation of tank to bring about a uniformity of suspension.- Place the stirring rod in the tank and place the tank in position in the turbidimeter with the shelf at the 30-60 level. Connect the stirring rod to its upper part. Swing the hinge out so it acts as a stop to prevent the stirring rod from being lifted from the liquid. Give ten quick short strokes at the bottom of the tank and then raise the stirring rod as far as it will go allowing it to settle back by gravity. Do this twenty times and on the last stroke swing the hinge away, lift the stirrer from the suspension, swing the hinge back letting the handle of the stirrer rest on the top of the hinge to keep it out of the suspension. Push the stirrer against the side of the tank for a few seconds to allow the kerosene on the stirrer to drain off.

4. Adjustment of intensity of the light.- With the filter (Y) in the path of the light beam, but with the tank removed, adjust rheostats (R_1) and (R_2) until mocroammeter reads 20.0 microamperes. Allow the system at least 15 minutes to come to equilibrium, then with the reading at 20.0 measure the voltage drop across the lamp with the potentiometer. The potentiometer reading then becomes the standard of intensity and the rheostats are adjusted to keep it constant.

5. Determination of I_o.- After the run is over, pour the contents of the tank into the 4-inch funnel fitted with the 25-cm. filter paper; allow to drain into a 600-cc. beaker. Wash out the tank with the wash bottle, collecting washings in the 1-gal. bottle, and proceed with the next run. After the runs are over place the first filtrate in the clean tank, put the tank in the turbidimeter, close the shunt switch, and remove the colored filter. Allow the system twenty minutes to come to equilibrium, using the potentiometer to hold the intensity constant. Read the microammeter and record the value of I_o with its corresponding run. Remove the tank from the turbidimeter, pour the liquid into the 1-gal. bottle, and fill the tank with the filtrate from the next run. Place the tank back in the turbidimeter and read the I_o value at once. This procedure is repeated until all the I_o's have been measured. By doing them all at once much time is saved, since it is necessary to allow twenty minutes for each measurement if they are done at odd times. The value of I_o is about the same as the intensity without tank or colored filter in the path. Do not use the colored filter at all in this procedure, the shunt protects the microammeter.

6. Measurement of the density and viscosity of kerosene.- To determine the density of the kerosene, clean out the pyknometer with alcohol and ether and dry it with air. Fill it with distilled water, wiping the

outside dry and absorbing any overflow with a piece of filter paper, note the temperature at which the water level just comes to the mark on the side-arm. Weigh the pyknometer and water on the balance. Empty and clean it with alcohol and ether and dry with air. Weigh the pyknometer alone. Fill it with kerosene and, noting the temperature as above, weigh the pyknometer and kerosene.

W_o = wt. of pyknometer
W_1 = " " " and water
W_2 = " " " and kerosene

$(W_1 - W_o)$ = volume of pyknometer in ml. = V
ρ_t = density of water from t from table below
$\frac{(W_2 - W_o)}{V}$ = density of kerosene in grams/ml. = ρ_2

Unless the room temperature fluctuates over ten degrees, this value for the density of the kerosene is sufficiently accurate.

Table of Viscosity and Density of Distilled Water from 20° to 20°C

Temp. °C	millipoises	grams/ml.
20	10.09	0.998
21	9.84	
22	9.61	
23	9.39	
24	9.16	
25	8.95	0.997
26	8.75	
27	8.55	
28	8.36	
29	8.18	
30	8.00	0.996

Interpolation may be used

Clean the viscosity pipette with hot cleaning solution ($K_2Cr_2O_7$ in conc. H_2SO_4), then wash it out with distilled water. Dry it by blowing air through it, The air should be passed through a cotton filter first to eliminate dust. The pipette should then be clamped to a ring stand and lowered into a beaker of water, used as a means of temperature control. By means of a measuring pipette introduce distilled water into the large side of the viscosity pipette. Allow to stand for several minutes for the temperature to become equalized, and then determine the temperature (°C) of the water in the beaker. By means of a rubber tube apply suction to the capillary side of the pipette and draw the water in it up above the upper mark. Remove the rubber tubing, breaking suction, and start the stop-watch the instant the level passes the upper mark, stopping it as the level passes the lower mark. Repeat the procedure to check the first run; it should check to 2/5 of a second at the same temperature. Next wash out the pipette with alcohol followed by ether and dry by passing clean air. Clamp the pipette in the beaker and fill the beaker with water at 2°C below room temperature. Introduce the same volume of kerosene as water in the first run, by means of the measuring pipette. Take the time for the kerosene between the marks as above and repeat to check. Then raise the temperature of the water in the beaker to 2°C above room temperature and make two runs. From the table of the viscosity of water substitute the value at the temperature of the readings for water, in the formula: $\mu = \mu_W \frac{T \rho_2}{T_W}$, where μ_W and T_W are the viscosity and time for water and T is the time for kerosene. A solution of this equation gives the viscosity in millipoises of the kerosene at the temperature at which T was read. Plot the two values of the viscosity of kerosene as ordinates and the temperature at which the viscosities were measured as abscissae, using a sufficiently large scale so that tenths of degrees and millipoises may be read. Draw a straight line through the two points. If the normal room temperature fluctuates over more than four degrees Centigrade, it will be well to determine more points.

7. Measurement of density of cement.- Fill the pyknometer half full of brombenzene. Weigh out 1 gram of cement and introduce slowly into the pyknometer to prevent air bubbles being entrapped. Fill the pyknometer to the mark with brombenzene. Be careful that no cement gets into the ground joints. Note the temperature and weigh. Calculate the density of the cement from the following equation to three figures.

$$\rho_1 = \frac{1.0000}{V - (W - W_o - 1.0000)\frac{1}{D}}$$

where V = volume of pyknometer

W = weight of pyknometer, brombenzene and cement

W_o = weight of pyknometer

D = density of brombenzene, obtained from table below

Temperature, °C	Density of brombenzene, g/ml.
20	1.495
21	1.494
22	1.492
23	1.491
24	1.490
25	1.488
26	1.487
27	1.486
28	1.484
29	1.483
30	1.482

8. Calculation of time of settling.- From Stokes' law $T = \frac{3\,h\,\mu\,\times 10^7}{(\rho_1 - \rho_2)\,g\,d^2}$ the time of settling for the different sizes of particles are calculated.

T = time in minutes

h = depth of settling in cm.

μ = viscosity of kerosene in millipoises

ρ_1 = density of cement, gm/ml.

ρ_2 = density of kerosene, gm/ml.

g = acceleration of gravity = 980 cm/sec^2

d = diameter of particle in microns

Since h, g, and d do not vary with temperature, they may be all collected as has been done in the table below:

Diameter of Particle	$\frac{3h}{gd^2} \times 10^7$
70.3 microns	0.0929
65	0.1087
60	0.1276
55	0.1518
50	0.1837
45	0.2268
40	0.2871
35	0.3750
30	0.5103
25	0.6428
20	0.7653
15	0.8969
10	1.0193
7.5	1.1479

Calculate the time of settling for the 14 sizes of particles over the range of temperature of the laboratory and over the range of cement densities by multiplying the values of μ and $(\rho_1 - \rho_2)$ obtained in (6) by the figures in the right-hand column. The results should be plotted on cross-section paper of 20 lines to the inch. See Figure I, Part III. If the temperature range is not more than 8 degrees, only two points are necessary with a straight line connecting them. Repeat for cement densities of 3.15, 3.17, 3.19 and 3.21, which should cover the range satisfactorily, although it may be necessary to add to this. Plot time in minutes and seconds as ordinates and degrees centigrade as abscissae. Label each line with the corresponding particle size.

9. The 200-mesh determination is that given in the Standards of the American Society for Testing Materials, 1933, Part II, p. 12.

10. Use of Potentiometer.- The potentiometer circuit consists of a storage battery, two dry cells, a potentiometer, a variable resistance, a switch, a key, and a galvanometer; the connections are shown in the diagram. Set the potentiometer at 1.5000 volts, throw the double

pole switch on the side marked "standard cell" and adjust the variable resistance until the galvanometer shows no deflection when the key is pressed. Then throw the switch to the other side, marked "E M F" and adjust the dials of the potentiometer until the galvanometer shows no deflection when the key is pressed. The reading of the dials now shows the voltage drop across the lamp, and the latter should be kept adjusted to this value. Check the potentiometer current every 15 minutes by going through the first operation with the switch on "standard cell".

IV. Test Procedures

1. Make 200-mesh determination, discard the oversize, mix the throughput as described in III(1), and place in a desiccator until ready for use.

2. Turn on lamp and adjust intensity to 20.0 microamperes, the colored filter being in the path, the tank removed, and the shunt switch open. Standardize the current through the potentiometer.

3. Weigh out the sample, stir it, and fill the tank almost to 335 ml.

4. After 15 minutes have elapsed from the time the lamp was turned on, adjust the intensity to 20.0 as above, check the potentiometer current, and measure the drop across the lamp. Record this value.

5. Place the tank in the turbidimeter, raise the shelf to the 7.5 level and fill the tank to 335 ml. Lower the shelf to the 30-60 level, remove the tank, take its temperature, place the stirring rod in the tank, and replace the tank in the turbidimeter, connecting up the stirrer.

6. Add 0.1° to the temperature as read and from the graphs of the settling time write down the times for this temperature on the report sheet.

7. Swing the hinge out. Give 10 short rapid strokes, with the stirrer at the bottom of the tank, and remove the colored filter, followed by 20 strokes of the stirrer between the two stops. On the twentieth stroke, swing the hinge aside, hang the stirrer on it and start the stop-watch. Hold stirrer against the tank wall for several seconds to drain.

8. Be sure the tank is in position against the stops and that all liquid has drained off the stirrer.

9. Close the door of the turbidimeter, check the voltage on the lamp with the potentiometer, and place the stop-watch on the top of the microammeter.

10. Read the microammeter at the times given on the report sheet for 70.3, 65, 60, 55, 50, 45, 40, 35 and 30. After obtaining the reading at 30, immediately raise the shelf to the 25 mark on the pointer scale and take a reading at the time for 25. Raise shelf and obtain readings in a like manner at the remaining marks of 20, 15, 10 and 7.5.

11. Place filter back in path of light beam, remove tank and pour contents into filter in funnel. Label the beaker under this funnel to correspond with the run number.

12. After five minutes check the light through the filter to see that it reads 20.0. Note:- there will be opportunity during the run to check the lamp with the potentiometer and make adjustments if necessary.

13. After a series of runs is finished determine the values of I_o, using the final reading of the potentiometer to hold the intensity constant.

14. Calculate the specific surface and size distribution of the cement in the following manner: (See Sample Report Sheet on next page)

1	2	3	4	5	6	7	8	9	10	11	12	13
TURBIDIMETER NO. 15 RUN NO. DATE	PARTICLE DIAM. MICRONS	TIME OF SETTLING MIN. and SEC.	I MICROAMMETER READING	LOG OF MICRO-AMMETER READING	LOG I_o/I	Δ LOG I_o/I	d AV. DIAM FOR Δ	d Δ LOG I_o/I	TOTAL SURFACE Sq. cm./gm.	INDIVIDUAL SURFACE %	Δ DIAM. MICRONS	% PER MICRON
CEMENT	70.3						67.7				5.3	
% PASSING NO. 200 / 325 SIEVE	65						62.5				5.0	
	60						57.5				5.0	
WT. OF SAMPLE	55						52.5				5.0	
	50						47.5				5.0	
TEMP. C°	45						42.5				5.0	
I_0	40						37.5				5.0	
LOG I_0	35						32.5				5.0	
	30						27.5				5.0	
ρ_1	25						22.5				5.0	
	20						17.5				5.0	
$c = \frac{60000}{\rho_1 \Sigma \text{COL. } 9}$	15						12.5				5.0	
$k = \frac{\rho_1 c}{600}$	10						8.75				2.50	
	7.5						3.75				7.5	

Fill in all the data of columns 1 (except the values of c and k), 2, 3, and 4, before and during the run. Fill in column 5 with the 5-place logarithms of the values of column 4. In column 6 put the difference between the log of I_o and the logs of $I_{70.3}$ and $I_{7.5}$ in those two rows, respectively. In column 7 beginning at the bottom with the 7.5 row fill in opposite 7.5, log I_o - log $I_{7.5}$. In the 10 row log $I_{7.5}$ - log I_{10}, in the 15 row log I_{10} - log I_{15}, and so on up to the 70.3 row, which will be log I_{65} - log $I_{70.3}$. Next multiply the numbers in column 7 by the corresponding ones in column 8 writing the products in column 9. Add column 9 and solve for c, as indicated in column 1. Multiply this value of c by (log I_o - log $I_{70.3}$) and place the product in column 10 in the 70.3 row. This is the specific surface of the sample. Solve for k as indicated in column 1 and multiply this value by the values in column 9 placing the products in column 11 in the corresponding rows. Divide the values in column 11 by the numbers in column 12, placing the quotients in column 13 in the corresponding rows. Plot a percent per micron curve for the sample using column 2 as abscissae and column 13 as ordinates. Representative percent per micron curves are shown in Part III.

NOTES

(1) Either the filter or the tank containing a suspension of cement must be in the path of the light beam at all times, or the shunt switch must be closed. If not, the full strength of the light beam will drive the pointer off the microammeter scale and possibly injure it. This is important.

(2) Keep microammeter level when in use.

(3) Turn knob from "Transit" position and set needle at zero by means of adjustment knob prior to test.

(4) At end of test return knob to "Transit" position before moving instrument.

(5) Avoid use of microammeter in proximity to wires carrying heavy electrical currents.

(6) For the lamp, R_2 is the fine adjustment of R_1.

(7) Keep the colored filter clean and free from dust.

(8) Always have the colored filter firmly against its stops, any changes in its position will change the reading.

(9) With the shunt switch closed the microammeter reads 1/3 of the total current; therefore, multiply all microammeter readings for I_o by three.

(10) Check the viscosity and density of the kerosene at least once a month.

(11) Keep stoppers in the kerosene bottles when not in use.

(12) All electrical connections must be either mechanically tight or soldered.

(13) The potentiometer readings for the intensity through the colored filter will change with time as the lamp battery becomes discharged. The last reading recorded is the correct one for controlling lamp intensity for the next run or I_o measurements.

(14) To reclaim the used kerosene put one teaspoonful of the filter charcoal in the 1-gallon bottle when it is full. Shake it up and filter through the Büchner funnel, fitted with a filter paper held down by the lead ring into the bottle containing the $CaCl_2$. From this it may be replaced in the other 5-gallon bottle when necessary. This is done by filtering it through the 7-inch funnel fitted with the 15-inch filter paper. The enlarged end of the syphon remains in the liquid of the latter bottle.

PART II

Criticism
of
Wagner's Method of Operation

The method of notation refers to that in the booklet, "Turbidimeter Instructions" attached as Appendix I.

I. List of Parts of the Apparatus.

Certain parts have been eliminated and others have been replaced as will be seen by referring to Part I, Section 1. The reasons for these changes will be pointed out in the course of the discussion.

II. Instructions for Installation.

Sections (8) and (9) have been eliminated. Section (10) has been supplemented. Additional sections have been added. The reasons for these changes will be pointed out in the course of the discussion.

III. General Operating Instructions.

1. Size of Sample.- The choice of sample weight cannot arbitrarily be fixed. Although the suggested method of Wagner will do for most cases, it was found that for the very fine cement, No. A (see Part III, Report sheets 9 and 10) insufficient light passed the suspension to give as accurate readings on the microammeter as would be obtainable with a sample smaller than 0.3 gram.

2. Preparation of suspension.- For very fine cements six drops of oleic acid may not be sufficient to prevent flocculation. It was found (Appendix II; Figure I) that when flocculation occurred a fine mirror-like film of the finest cement particles formed on the walls of the glass tank, and that the flocculation and the attendant film could be eliminated by adding another drop or two of oleic acid.

The method of mixing by means of the rotating brush was found to be superior to other methods tried and always gave consistent results.

3. Agitation of tank to bring about a uniformity of suspension before placing in path of light beam.- The method of oscillation of the tank with the hand over the top was rejected for the following reasons:

(a) It raises the temperature of the tank about 0.6°C, and thereby changes the rate of settling by an unknown amount.

(b) It affords opportunity for loss of kerosene and cement, which changes height of settling and cement concentration.

(c) It introduces many small air bubbles into the suspension. The effect of these in slowing the rate of settling is shown in Figure II, Appendix II.

(d) It introduces errors by the jarring of the tank while it is being placed in position in the turbidimeter, after settling has begun.

4. Adjustment of the intensity of the light, I_o, to 100 microamperes,- The method of using the results of an extrapolation to obtain I_o, and the use of the intensity of the light through the colored filter as a <u>permanent</u> reference standard is objected to for the following reasons:

(a) The extrapolation by drawing a straight line on semi-log paper through the two experimentally determined points is inaccurate. First because the relationship between concentration and intensity of transmitted light is not an exponential function for suspensions of this type; see Channon, Renwick, and Storr, Proc.Roy.Soc. (London) <u>94</u>, 222 (1917); and Figure III, Appendix II. Secondly because there may be experimental errors in obtaining the two points, and no check is available. Thirdly because the extrapolated value cannot be accurately read on such small scale paper as that supplied, see Figure IX, Appendix II, in which the directions of Wagner have been followed.

(b) The reading through the filter is not a permanent standard for the value of I_0 because the latter varies from one suspension to another even under the most carefully controlled conditions. See the values of I_0 measured in the turbidimeter analyses which are given in Part III.

5. Calibration of burette scale.- The burette as a timing device is rejected in its entirety for the following reasons:

(a) It does not give the correct time of settling, as calculated by Stokes' Law for the size of particle indicated.

(b) It does not give the proper correction of this time for variations of temperature. See Figures V and VI, Appendix II.

6. Calibration of the No. 325 sieve.-

7. The 325 sieve determination.- The use of the 325 sieve has been omitted and replaced by a No. 200 sieve, for the following reasons:

(a) The No. 325 sieve opening as given by the American Society of Testing Materials "Standards" 1933, Part II, p. 12 is 44 microns. Knapp found that this allows a particle of 53.5 microns to just pass through because of the square shape of the openings (see Knapp, Thesis for the Degree of Doctor of Philosophy, California Institute of Technology 1929). In these microscopic studies Knapp also found that the ratio of the actual size of a cement particle to the size given by Stokes Law is 1.28. Hence the diameter of a particle just passing the No. 325 sieve appears in the turbidimeter as 41.8 microns instead of 60, and affects all the calculations accordingly. Since it is possible that particles larger than 41.8 microns play a part in the action of cement, it was decided to replace the No. 325 sieve by the No. 200, which according to Knapp passes a particle of 70.3 microns diameter as measured in the turbidimeter.

(b) It has been found necessary to pass the cement sample to be tested through a No. 200 sieve before running it in the turbidimeter, because cement upon standing forms aggregates of the cement crystals. These aggregates are quite stable to mechanical action, but fairly readily break up in water, which the true cement crystals do not do. A series of microscopic studies was made on the effect of screening on these aggregates. It was found that the No. 325 sieve broke them up completely, and the No. 200 sieve broke them almost completely. It was therefore decided to incorporate a 200-mesh determination (dry) in the turbidimeter procedure, using the cement which passed the sieve as the source of the turbidimeter sample. In this procedure only the true cement crystals themselves are measured; and, while the fraction remaining on the sieve is rejected, it is usually small, and the particles composing it are so large that their chemical action as cement is unimportant.

(c) The No. 325 determination is inaccurate unless the sample to be run is first put through a No. 100 sieve. See Figure VII, Appendix II.

IV. Test Procedure

Sections 1 thru 8 have already been covered.

(9) The error resulting from variation in the density of the cement being tested, which was pointed out under Figure V, Appendix II, appears also in the equation for specific surface. It comes in as the constant, 38, so any error in density creates the same percentage error in the specific surface.

The method of calculating the specific surface of a cement from the same plant, by using the ratio:

$$\frac{S(\text{unknown sample})}{S(\text{known sample})} = \frac{(2 - \log I_{60})(\text{unknown sample})}{(2 - \log I_{60})(\text{known sample})}$$

is anaccurate. This inaccuracy is shown in Figure VIII, Appendix II, in which the specific surfaces of two cements from the same plant and of identical chemical composition have been measured in the turbidimeter and also calculated from the above ratio.

V. Notes

(2) In addition to the treatment with charcoal, it is recommended that the kerosene be dried with anhydrous calcium chloride. The necessity of this treatment with charcoal and $CaCl_2$ has been pointed out under Figure I, Appendix II.

(5) This fatigue phenomenon in the photocell varies with different cells. In the Photronic cell sent with the apparatus it required one-fifth of a minute per microampere change in current for the output of the cell to become constant. Since the rates of change in output of the photocell as the cement settled during a run did not exceed this value, no error resulted in the use of this particular cell. It is necessary to specify, however, that the fatigue effect be no greater than this. In all the measurements of Appendix II this fatigue factor has been eliminated by allowing sufficient time for the photocell to reach equilibrium. For further information on this subject see Weston Circular CD-10 entitled "Technical Data - Weston Photronic Cell."

(11) Since the intensity of the lamp is continually changing, it was found advisable to use a potentiometer to read the voltage drop across the lamp. By holding this voltage constant to 0.0005 volts, the light intensity as measured by the microammeter will be held at the same reading over a period of an hour or more. The use of the potentiometer allows the adjustment to be made at any time during a run. All the measurements in this report made with the turbidimeter employed the potentiometer. A diagram of connections is given in Part I.

PART III

Discussion of the results of operation of the Turbidimeter by the method of Part I and by Wagner's method given in the booklet "Turbidimeter Instructions" attached to Part II. These results are compared to those measured for the same cements by the Microneter, the most accurate instrument yet designed for measuring specific surface and particle size distribution. A discussion of the errors in the Turbidimeter is given.

Microneter Analysis Sheet

Sample No. A

Microneter No. 6178

100% of Sample in Microneter

Sample Density 3.22

J	K	N	S
Equivalent Stokes Law Diameter	Cumulative % of -200 Mesh	Individual % of -200 Mesh	Individual surface
Microns	Intercept of Tangent	ΔK	From N and Tables
		41.5	147,200
7.4	58.5	5.5	11,710
9.2	53.0	11.0	16,890
13.9	42.0	9.0	9,840
18.5	33.0	7.5	6,370
23.2	25.5	6.5	4,510
27.8	19.0	9.5	5,190
37.1	9.5	2.5	1,060
46.3	7.0	3.0	1,040
55.7	4.0	4.0	1,125
70.3	0	0	

Surface for 100% -200 mesh 204,935

Specific surface 2,049

Micrometer Analysis Sheet

Cement No. B

Sample density 3.19

97.5% of Sample in Micrometer

J	K	N	S
Equivalent Stokes Law Diameter	Cumulative % of -200 Mesh	Individual % of -200 Mesh	Individual surface
Microns	Intercept of Tangent	Δ K	From N and Tables
		28.0	100,200
7.4	72.7	4.0	8,500
9.3	68.8	12.5	19,200
14.0	56.6	10.5	11,550
18.5	46.4	8.0	6,860
23.2	38.6	6.0	4,200
27.8	32.7	11.0	6,050
37.1	22.0	9.0	3,850
46.4	13.2	8.0	2,800
55.7	5.4	3.0	850
70.3	0	0	

Surface for 100%	-200 Mesh	164,110
Total Sample surface as	-200 Mesh	160,007
" " " "	+200 Mesh	160,535

Specific Surface 1605

Microneter Analysis Sheet

Sample No. C

Microneter No. 6151

90% of Sample in Microneter

Sample Density 3.22

J	K	N	S
Equivalent Stokes Law Diameter	Cumulative % of -200 Mesh	Individual % of -200 Mesh	Individual surface
Microns	Intercept of Tangent	Δ K	From N and Tables
		24.0	85,100
7.4	76.0	4.0	8,520
9.2	72.0	10.0	15,320
13.9	62.0	8.5	9,300
18.5	53.5	7.5	6,380
23.2	46.0	6.0	4,160
27.8	40.0	12.5	6,820
37.1	27.5	9.5	4,040
46.3	18.0	10.0	3,470
55.7	8.0	8.0	2,250
70.3	0	0	

Surface for 100% -200 Mesh	145,360
Total Sample surface as -200 Mesh	130,824
" " " " +200 Mesh	2 ,090
Total Sample Surface	132,914
Specific Surface	1329

Microneter Analysis Sheet

Sample No. D

Microneter No. 6195

86% of Sample in Microneter

Sample Density 3.18

J Equivalent Stokes Law Diameter Microns	K Cumulative % of -200 Mesh Intercept of Tangent	N Individual % of -200 Mesh Δ K	S Individual surface From N and Tables
		16.0	56,900
7.5	84.0	3.5	7,460
9.3	80.5	9.5	14,580
14.1	71.0	9.0	9,850
18.7	62.0	9.0	7,670
23.4	53.0	8.0	5,570
28.1	45.0	14.5	7,930
37.4	30.5	11.5	4,900
46.7	19.0	13.0	4,520
56.2	6.0	6.0	1,700
70.3	0	0	

Surface for 100% -200 Mesh	121,080
Total Sample Surface as -200 Mesh	104,129
" " " " +200 Mesh	2,970
Total Sample Surface	107,099
Specific Surface	1,071

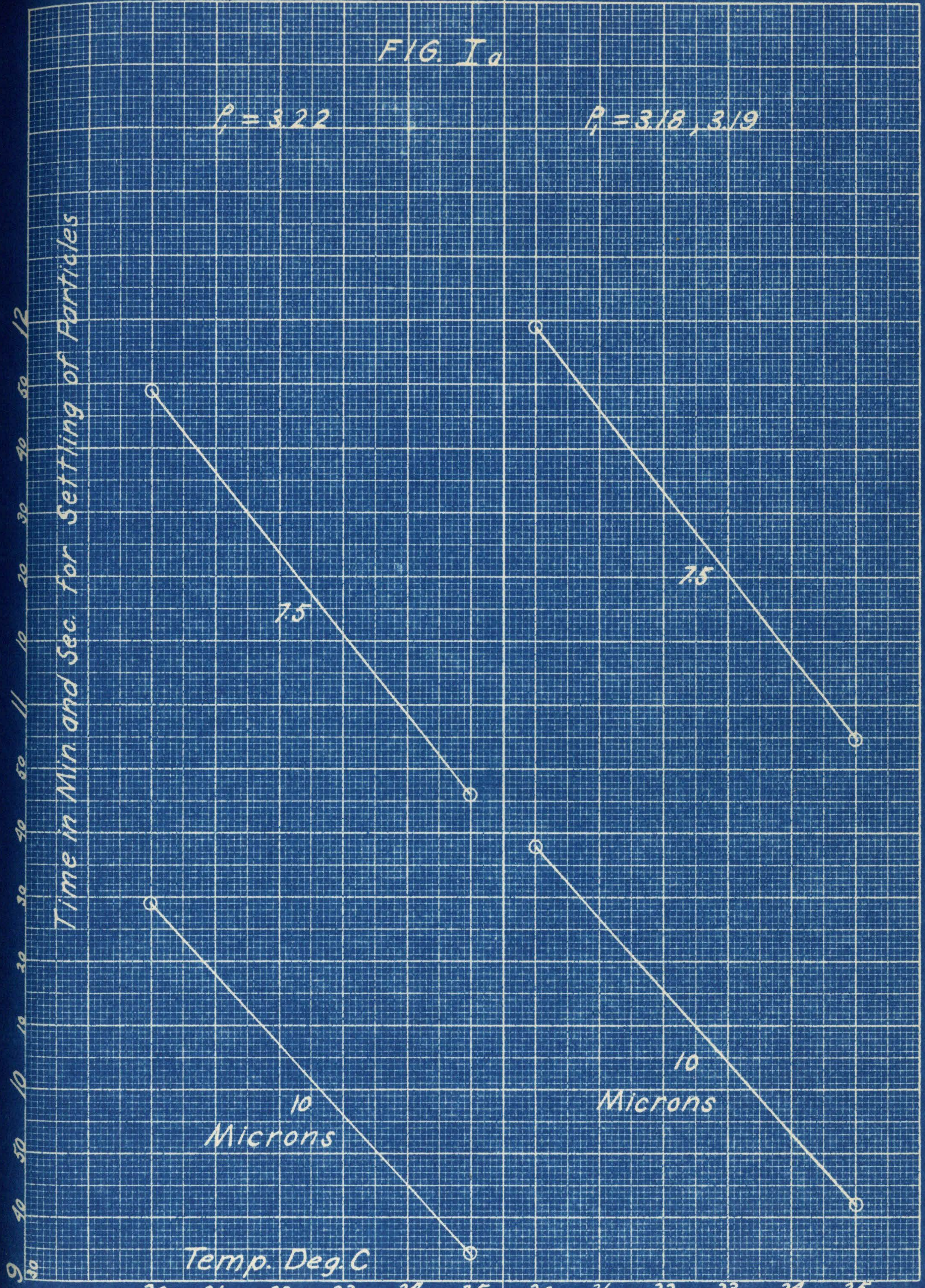
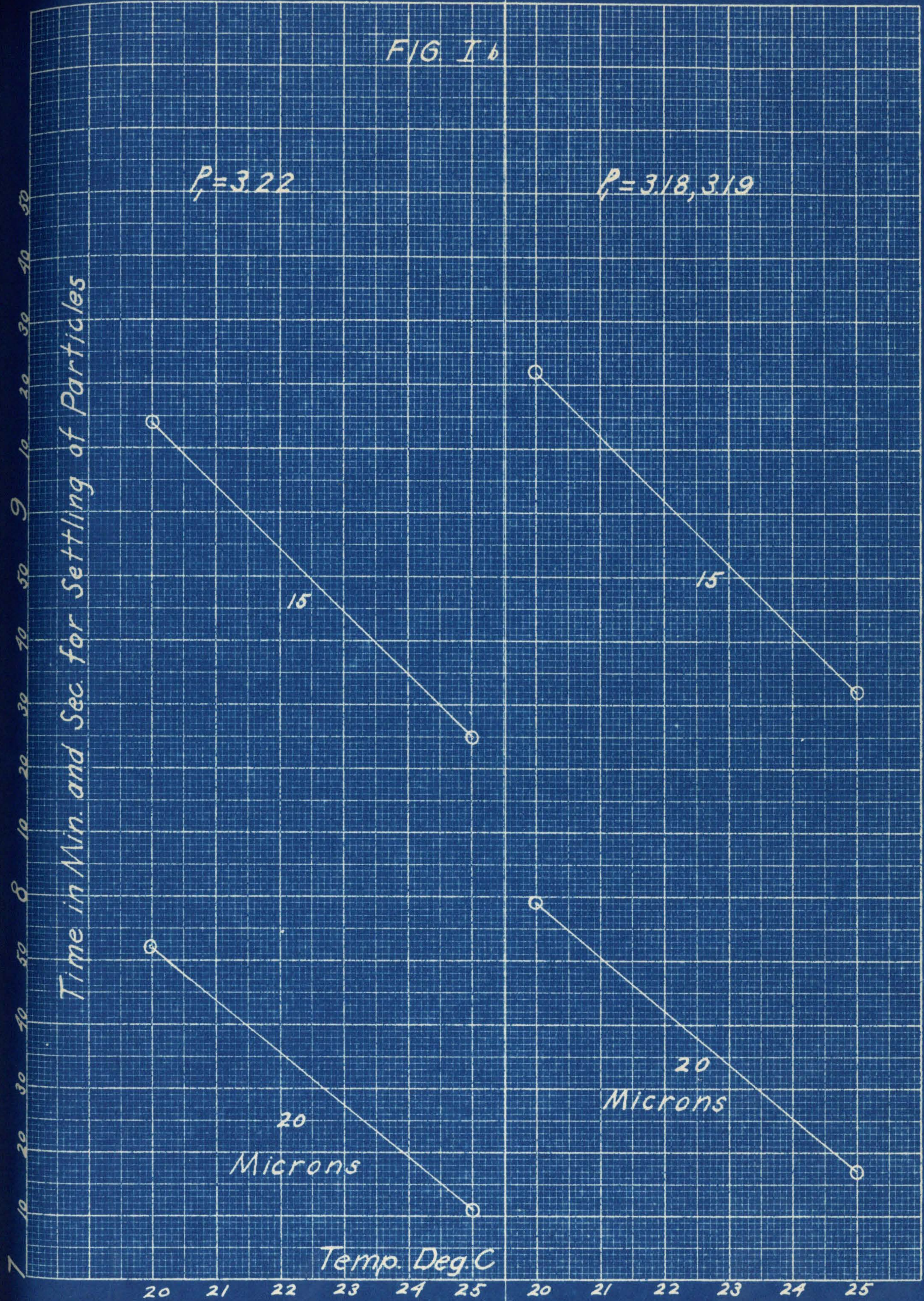


FIG. Ia

$\rho_1 = 3.22$
$\rho_1 = 3.18, 3.19$
Time in Min. and Sec. for Settling of Particles
12
50
40
30
20
10
11
50
40
30
20
10
10
50
40
9 30
7.5
7.5
10 Microns
10 Microns
Temp. Deg. C
20
21
22
23
24
25
20
21
22
23
24
25

FIG. I b



$\rho_1 = 3.22$
$\rho_1 = 3.18, 3.19$
Time in Min. and Sec. for Settling of Particles
50
40
30
20
10
9
50
40
30
20
10
8
50
40
30
20
10
7
15
15
20
Microns
20
Microns
Temp. Deg. C
20
21
22
23
24
25
20
21
22
23
24
25

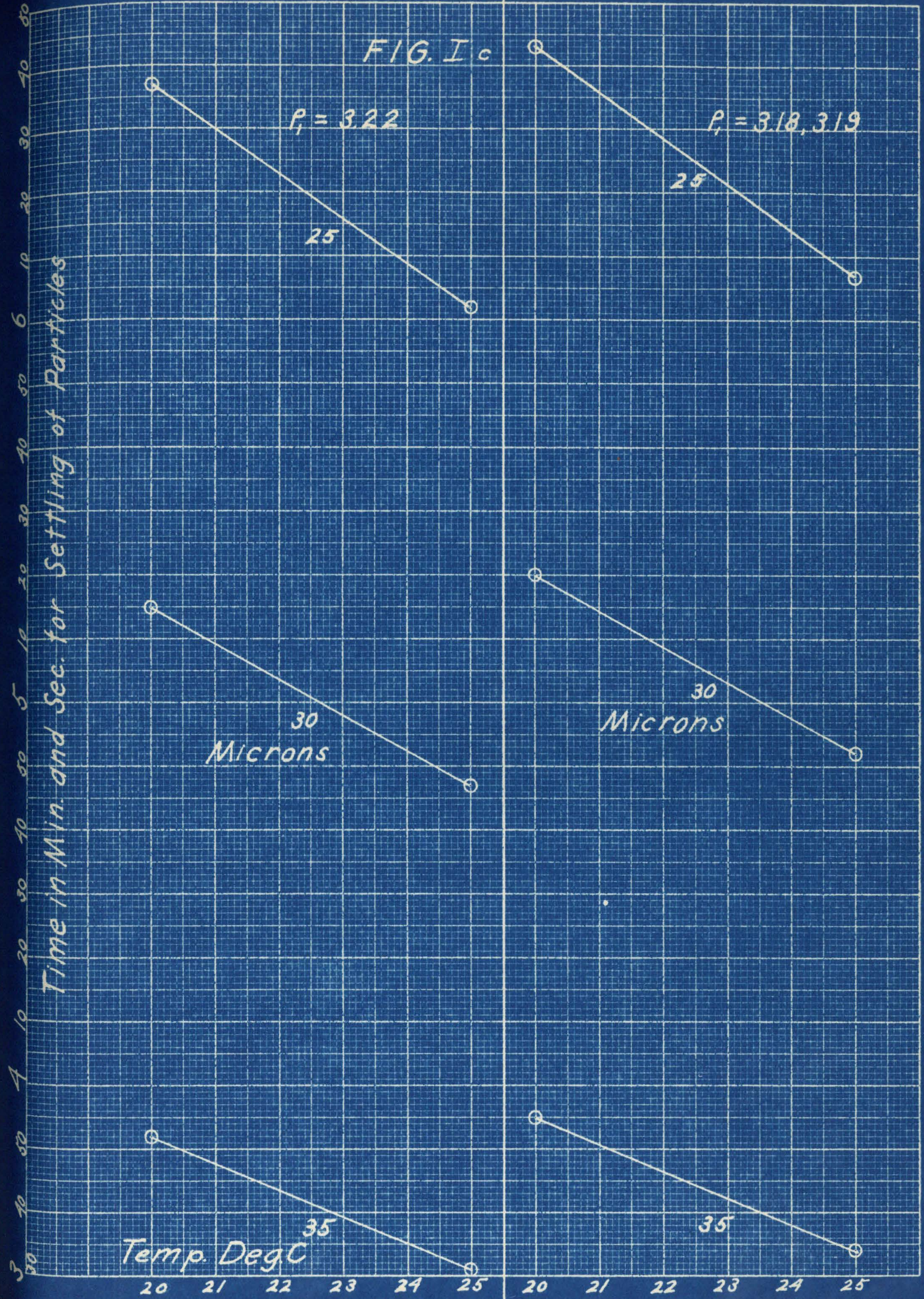
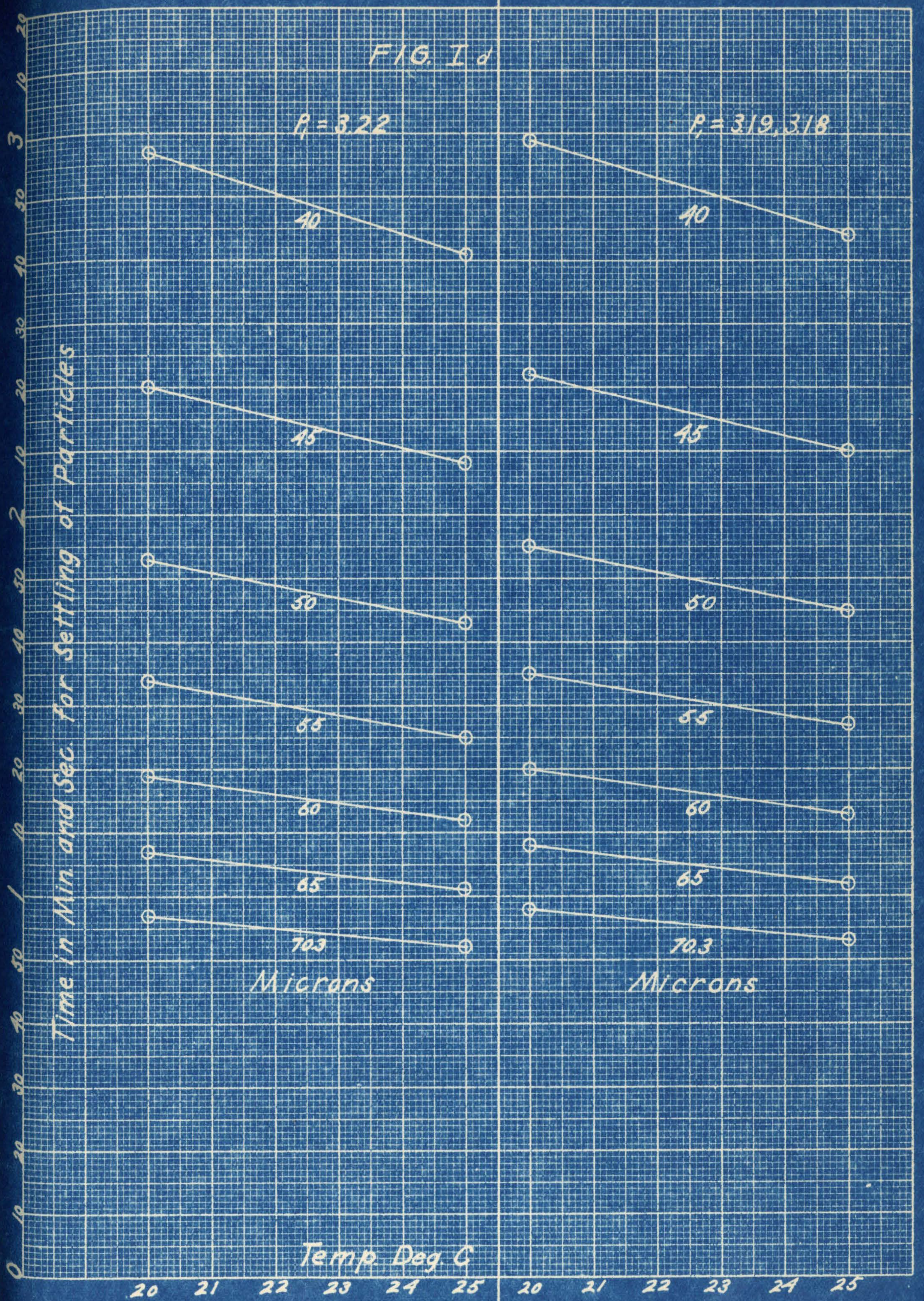


FIG. I c

$P_1 = 3.22$
$P_1 = 3.18, 3.19$
25
25
30
Microns
30
Microns
35
35
Time in Min. and Sec. for Settling of Particles
Temp. Deg C
50
40
30
20
10
6
50
40
30
20
10
5
50
40
30
20
10
4
50
40
30
3
20
21
22
23
24
25
20
21
22
23
24
25

FIG. I d


Time in Min. and Sec. for Settling of Particles
20
10
3
50
40
30
20
10
2
50
40
30
20
10
1
50
40
30
20
10
0
$\rho_1 = 3.22$
40
45
50
55
60
65
70.3
Microns
$\rho_1 = 3.19, 3.18$
40
45
50
55
60
65
70.3
Microns
Temp. Deg. C
20
21
22
23
24
25
20
21
22
23
24
25

1	2	3	4	5	6	7	8	9	10	11	12	13
TURBIDIMETER NO. 15 RUN NO. 1 DATE 12/11/33	PARTICLE DIAM. MICRONS	TIME OF SETTLING MIN. and SEC.	I MICROAMMETER READING	LOG OF MICRO-AMMETER READING	LOG I_o/I	Δ LOG I_o/I	d AV. DIAM FOR Δ	d Δ LOG I_o/I	TOTAL SURFACE Sq. cm./gm.	INDIVIDUAL SURFACE %	Δ DIAM. MICRONS	% PER MICRON
CEMENT A	70.3	0:55	31.8	1.50243	0.56090	0.00136	67.7	0.0921	2972	2.62	5.3	0.494
% PASSING NO. 200 SIEVE 100 ~~325~~	65	1:04	31.9	1.50379		0	62.5	0	c=5298	0	5.0	0
	60	1:16	31.9	1.50379	0.55954	0	57.5	0	3044	0	5.0	0
WT. OF SAMPLE 0.1100	55	1:30	31.9	1.50379		0.00136	52.5	0.0714	c=5441	2.03	5.0	0.406
	50	1:49	32.0	1.50515		0.00271	47.5	0.1287		3.66	5.0	0.732
TEMP. C° 22.2	45	2:15	32.2	1.50786		0.00134	42.5	0.0570		1.62	5.0	0.324
I_0 115.7	40	2:50	32.3	1.50920		0.00535	37.5	0.2006		5.71	5.0	1.142
	35	3:43	32.7	1.51455		0.00396	32.5	0.1287		3.66	5.0	0.732
LOG I_0 2.06333	30	5:03	33.0	1.51851		0.00524	27.5	0.1441		4.10	5.0	0.820
	25	6:22	33.4	1.52375		0.00645	22.5	0.1451		4.13	5.0	0.826
ρ_1 3.22	20	7:34	33.9	1.53020		0.01511	17.5	0.2644		7.52	5.0	1.504
$c = \frac{60000}{\rho_1 \Sigma \text{COL. 9}}$	15	8:52	35.1	1.54531		0.02523	12.5	0.3154		8.97	5.0	1.794
	10	10:05	37.2	1.57054		0.02385	8.75	0.2087		5.94	2.50	2.376
$k = \frac{\rho_1 c}{600}$ 28.42	7.5	11:21	39.3	1.59439		0.46894	3.75	1.7585		50.03	7.5	6.771
								3.5147				

1	2	3	4	5	6	7	8	9	10	11	12	13
TURBIDIMETER NO. 15 RUN NO. 1 Error Sheet DATE	PARTICLE DIAM. MICRONS	TIME OF SETTLING MIN. and SEC.	I MICROAMMETER READING	LOG OF MICRO-AMMETER READING	LOG I_o/I	Δ LOG I_o/I	d AV. DIAM FOR Δ	d Δ LOG I_o/I	TOTAL SURFACE Sq. cm./gm.	INDIVIDUAL SURFACE %	Δ DIAM. MICRONS	% PER MICRON
CEMENT A	70.3		± 0.05	±0.00068	±0.00101	±0.00096	67.7	±0.0650	±101		5.3	±0.349
% PASSING NO. 200 SIEVE 325	65		"	68			62.5		179		5.0	
	60		"	6 8			57.5				5.0	
WT. OF SAMPLE	55		"	68		96	52.5	502			5.0	286
	50		"	68		95	47.5	451			5.0	258
TEMP. C°	45		"	67		95	42.5	404			5.0	230
I_0 ± 0.2	40		"	67		94	37.5	352			5.0	204
	35		"	66		93	32.5	302			5.0	173
LOG I_0 ± 0.00075	30		"	66		93	27.5	256			5.0	148
ρ ± 0.005	25		"	65		91	22.5	205			5.0	119
	20		"	64		89	17.5	156			5.0	102
$c = \frac{60000}{\rho \ \Sigma \text{ COL. } 9}$	15		"	62		86	12.5	108			5.0	086
	10		"	59		81	8.75	71			2.50	114
$k = \frac{\rho c}{600}$ ±0.96	7.5		"	55		93	3.75	0.0035			7.5	225
								0.1186				

1

TURBIDIMETER NO.

RUN No. 1'

DATE 12/11/33

CEMENT A

% PASSING No. 200 ~~325~~ SIEVE 100

WT. OF SAMPLE 0.1500

TEMP. DEG. C 22.0

I_0 115.7

LOG I_0 2.06333

ρ_1 3.22

$c = \dfrac{60000}{\rho_1 \Sigma \text{ COL. } 9}$

$k = \dfrac{\rho_1 c}{600} = 21.10$

2	4	10	13
PARTICLE DIAM. MICRONS	MICROAMMETER READING	TOTAL SURFACE sq. cm./gm.	% PER MICRON
70.3	21.o	2913	0
65	21.0		0
60	21.0	2913	o.498
55	21.1	c=3931	0.456
50	21.2		0.410
45	21.3		0.364
40	21.4		1.274
35	21.8		0.814
30	22.1		1.128
25	22.6		1.080
20	23.2		1.356
15	24.2		1.730
10	26.1		2.256
7.5	28.0		6.504

1	2	4	10	13
TURBIDIMETER NO. RUN No. 2	PARTICLE DIAM. MICRONS	MICROAMMETER READING	TOTAL SURFACE sq.cm./gm.	% PER MICRON
DATE 12/11/33	70.3	31.9	2944	0
CEMENT A	65	31.9	c=5305	0.484
% PASSING No. 200 ~~325~~ SIEVE 100	60	32.0	3009	0
WT. OF SAMPLE 0.1100	55	32.0	c=5435	0.406
TEMP. DEG. C 22.5	50	32.1		0.728
I_0 114.5	45	32.3		0.648
LOG I_0 2.05881	40	32.5		0.852
ρ_1 3.22	35	32.8		0.976
$c = \frac{60000}{\rho_1 \Sigma \text{COL. } 9}$	30	33.2		0.814
$k = \frac{\rho_1 c}{600} = 28.46$	25	33.6		0.988
	20	34.2		1.492
	15	35.4		1.534
	10	37.2		2.268
	7.5	39.2		6.629

1
TURBIDIMETER NO. 15
RUN No. 3
DATE 12/12/33
CEMENT D
% PASSING No. 200 ~~[illegible]~~ SIEVE 86.0
WT. OF SAMPLE 0.5000
TEMP. DEG. C 22.0
I_0 112.4
LOG I_0 2.05077
ρ_1 3.18
$c = \frac{60000}{\rho_1 \Sigma \text{ COL. } 9}$
$k = \frac{\rho_1 c}{600} = 9.79$

2	4	10	13
PARTICLE DIAM. MICRONS	MICROAMMETER READING	TOTAL SURFACE sq. cm./gm.	% PER MICRON
70.3	18.5	1447	1.160
65	18.9	c=1846	0.836
60	19.2	1581	1.256
55	19.7	c=2060	0.896
50	20.1		1.186
45	20.7		1.200
40	21.4		1.312
35	22.3		1.448
30	23.5		1.448
25	25.0		1.540
20	27.1		1.708
15	30.4		1.796
10	36.0		1.788
7.5	40.6		2.162

1
TURBIDIMETER NO. 15
RUN No. 4
DATE 12/12/33
CEMENT D
% PASSING No. 200 ~~325~~ SIEVE 86.0
WT. OF SAMPLE 0.5000
TEMP. DEG. C 21.6
I_0 109.8
LOG I_0 2.04060
ρ_1 3.18
$c = \dfrac{60000}{\rho_1 \Sigma \text{COL. } 9}$
$k = \dfrac{\rho_1 c}{600} = 9.90$

2	4	10	13
PARTICLE DIAM. MICRONS	MICROAMMETER READING	TOTAL SURFACE sq. cm./gm.	% PER MICRON
70.3	18.6	1440	1.168
65	19.0	c=1867	1.120
60	19.4	1594	1.010
55	19.8	c=2115	0.902
50	20.2		1.392
45	20.9		1.034
40	21.5		1.322
35	22.4		1.340
30	23.5		1.462
25	25.0		1.560
20	27.1		1.776
15	30.5		1.692
10	35.7		1.860
7.5	40.4		2.148

1	2	4	10	13
TURBIDIMETER NO. 15 RUN No. 5	PARTICLE DIAM. MICRONS	MICROAMMETER READING	TOTAL SURFACE sq. cm./gm.	% PER MICRON
DATE 12/16/33	70.3	15.8	1748	1.160
	65	16.1	c = 2064	0.742
CEMENT C	60	16.3	1907	1.010
% PASSING No. 200 ~~325~~ SIEVE 90.0	55	16.6	c = 2289	0.906
	50	16.9		1.070
WT. OF SAMPLE 0.4000	45	17.3		0.934
	40	17.7		1.204
TEMP. DEG. C 20.8	35	18.3		1.338
	30	19.1		1.218
I_0 111.0	25	20.0		1.262
LOG I_0 2.04532	20	21.2		1.806
ρ_1 3.22	15	23.6		1.530
	10	26.8		2.180
$c = \dfrac{60000}{\rho_1 \Sigma \text{COL. } 9}$	7.5	30.5		3.108
$k = \dfrac{\rho_1 c}{600} = 11.07$				

1
TURBIDIMETER NO. 15
RUN No. 6
DATE 12/16/33
CEMENT C
% PASSING No. 200 ~~325~~ SIEVE 90.0
WT. OF SAMPLE 0.4000
TEMP. DEG. C 20.9
I_0 109.2
LOG I_0 2.03822
ρ_1 3.22
$c = \dfrac{60000}{\rho_1 \Sigma \text{COL. } 9}$
$k = \dfrac{\rho_1 c}{600} = 11.37$

2	4	10	13
PARTICLE DIAM. MICRONS	MICROAMMETER READING	TOTAL SURFACE sq. cm./gm.	% PER MICRON
70.3	15.6	1798	0.806
65	15.8	c= 2120	1.162
60	16.1	1960	0.702
55	16.3	c = 2358	0.946
50	16.6		1.118
45	17.0		0.978
40	17.4		1.050
35	17.9		1.404
30	18.7		1.138
25	19.5		1.220
20	20.6		1.754
15	22.8		1.764
10	26.3		2.160
7.5	29.8		3.210

1
TURBIDIMETER NO. 15
RUN No. 7
DATE 12/18/33
CEMENT B
% PASSING No. 200 ~~325~~ SIEVE 97.5
WT. OF SAMPLE 0.3000
TEMP. DEG. C 21.2
I_0 106.1
LOG I_0 2.02572
ρ_1 3.19
$c = \dfrac{60000}{\rho_1 \Sigma \text{ COL. } 9}$
$k = \dfrac{\rho_1 c}{600} = 12.18$

2	4	10	13
PARTICLE DIAM. MICRONS	MICROAMMETER READING	TOTAL SURFACE sq. cm./gm.	% PER MICRON
70.3	11.9	2178	0.566
65	12.0	c = 2292	0.452
60	12.1	2293	0.500
55	12.2	c = 2432	0.904
50	12.4		0.404
45	12.5		1.068
40	12.8		0.920
35	13.1		1.290
30	13.6		1.258
25	14.2		1.306
20	15.0		1.878
15	161		1.858
10	19.1		2.276
7.5	21.6		4.213

1

TURBIDIMETER NO. 15

RUN No. 8

DATE 12/18/33

CEMENT B

% PASSING No. 200 ~~325~~ SIEVE 97.5

WT. OF SAMPLE 0.3000

TEMP. DEG. C 21.5

I_0 106.6

LOG I_0 2.02776

ρ_1 3.19

$c = \dfrac{60000}{\rho_1 \Sigma \text{ COL. } 9}$

$k = \dfrac{\rho_1 c}{600} = 12.14$

2	4	10	13
PARTICLE DIAM. MICRONS	MICROAMMETER READING	TOTAL SURFACE sq. cm./gm.	% PER MICRON
70.3	12.1	2158	0.555
65	12.2	c = 2284	0.540
60	12.3	2271	0.490
55	12.4	c = 2421	0.888
50	12.6		0.790
45	12.8		1.040
40	13.1		0.896
35	13.4		1.008
30	13.8		1.234
25	14.4		1.438
20	15.3		1.838
15	16.9		1.888
10	19.5		2.228
7.5	22.0		4.164

1	2	3	4	5	6	7	8	9	10	11	12	13
TURBIDIMETER NO. 15 RUN NO. 9 DATE 12/19/33	PARTICLE DIAM. MICRONS	TIME OF SETTLING MIN. and SEC.	I MICROAMMETER READING	LOG OF MICRO-AMMETER READING	LOG I_o/I	Δ LOG I_o/I	d AV. DIAM FOR Δ	d Δ LOG I_o/I	TOTAL SURFACE Sq. cm./gm.	INDIVIDUAL SURFACE %	Δ DIAM. MICRONS	% PER MICRON
CEMENT A	~~70.3~~						67.7				5.3	
% PASSING NO. ~~200~~ 325 SIEVE 95.5	~~65~~			1.50000			62.5				5.0	
	60		6.1	0.78533	1.21467	0	57.5	0	3100	0	5.0	0
WT. OF SAMPLE 0.3000	55		6.1	0.78533	[illegible]	0.00706	52.5	0.3707	c = 2559	4.98	5.0	0.996
	50		6.2	0.79239		0	47.5	0		0	5.0	0
TEMP. C°	45		6.2	0.79239		0.00695	42.5	0.2954		3.97	5.0	0.794
I_0 100	40		6.3	0.79934		0.00684	37.5	0.2565		3.44	5.0	0.688
	35		6.4	0.80618		0.00675	32.5	0.2187		2.94	5.0	0.588
LOG I_0 2.00000	30		6.5	0.81291		0.01316	27.5	0.3619		4.86	5.0	0.972
	25		6.7	0.82607		0.01273	22.5	0.2876		3.86	5.0	0.772
ρ 3.15	20		6.9	0.83885		0.02447	17.5	0.4282		5.75	5.0	1.150
$c = \frac{60000}{\rho \; \Sigma \text{ COL. 9}}$	15		7.3	0.86332		0.05049	12.5	0.6311		8.48	5.0	1.696
	10		8.2	0.91381		0.04998	8.75	0.4373		5.87	2.50	2.348
$k = \frac{\rho c}{600}$ = 13.43	7.5		9.2	0.96379		1.03621	3.75	3.8858		52.19	7.5	6.958
				1.42213								

1	2	3	4	5	6	7	8	9	10	11	12	13
TURBIDIMETER NO. 15 RUN NO. 9 Error Sheet DATE	PARTICLE DIAM. MICRONS	TIME OF SETTLING MIN. and SEC.	I MICROAMMETER READING	LOG OF MICRO-AMMETER READING	LOG I_o/I	Δ LOG I_o/I	d AV. DIAM FOR Δ	d Δ LOG I_o/I	TOTAL SURFACE Sq.cm./gm.	INDIVIDUAL SURFACE %	Δ DIAM. MICRONS	% PER MICRON
CEMENT A	~~70.5~~						67.7				5.3	
% PASSING NO. ~~300~~ SIEVE 325 ± 0.8	~~65~~			±0.01303			62.5				5.0	
	60		± 0.05	356	±0.01774		57.5		± 116		5.0	
WT. OF SAMPLE	55		"	356		±0.00499	52.5		± 89		5.0	±0.797
	50		"	350			47.5				5.0	
TEMP. C°	45		"	350		492	42.5				5.0	.656
I_0 ± 4	40		"	345		484	37.5				5.0	.568
	35		"	340		476	32.5				5.0	.485
LOG I_0 ±0.01738	30		"	334		465	27.5				5.0	.367
	25		"	324		451	22.5				5.0	.292
ρ ± 0.04	20		"	314		432	17.5				5.0	.209
$c = \frac{60000}{\rho \cdot \Sigma \text{ COL. } 9}$	15		"	297		397	12.5				5.0	.089
	10		"	264		354	8.75				2.50	.194
$k = \frac{\rho \cdot c}{600}$ ± 0.57	7.5		"	236		1890	3.75				7.5	.321
				4434								

1

TURBIDIMETER NO. 15

RUN No. 10

DATE 12/19/33

CEMENT A

% PASSING No. ~~300~~ 325 SIEVE 95.5

WT. OF SAMPLE 0.3000

TEMP. DEG. C

I_0 100

LOG I_0 2.00000

ρ_1 3.15

$c = \frac{60000}{\rho_1 \Sigma \text{ COL. } 9}$

$k = \frac{\rho_1 c}{600} = 13.36$

2	4	10	13
PARTICLE DIAM. MICRONS	MICROAMMETER READING	TOTAL SURFACE sq. cm./gm.	% PER MICRON
70.3			
65			
60	6.0	3101	0
55	6.0	c = 2544	0
50	6.0		0.912
45	6.1		0.802
40	6.2		0
35	6.2		1.198
30	6.4		0.982
25	6.6		0.780
20	6.8		1.160
15	7.2		1.886
10	8.2		2.336
7.5	9.2		6.92

1	2	4	10	13
TURBIDIMETER NO. 15	PARTICLE DIAM. MICRONS	MICROAMMETER READING	TOTAL SURFACE sq. cm./gm.	% PER MICRON
RUN No. 11	70.3			
DATE 12/19/33	65			
CEMENT B	60	11.9	2228	1.030
% PASSING No. ~~200~~ 325 SIEVE 86.4	55	12.1	c = 2354	0.464
	50	12.2		0.416
WT. OF SAMPLE 0.3000	45	12.3		0.736
	40	12.5		0.954
TEMP. DEG. C	35	12.8		0.808
	30	13.1		1.106
I_0 100	25	13.6		0.372
LOG I_0 2.00000	20	14.1		1.534
ρ_1 3.15	15	15.3		1.804
	10	17.5		2.508
$c = \frac{60000}{\rho_1 \Sigma \text{ COL. } 9}$	7.5	20.0		4.320
$k = \frac{\rho_1 c}{600} = 12.36$				

1

TURBIDIMETER NO. 15

RUN No. 12

DATE 12/20/33

CEMENT B

% PASSING No. ~~200~~ 325 SIEVE 86.4

WT. OF SAMPLE 0.3000

TEMP. DEG. C

I_0 100

LOG I_0 2.00000

ρ_1 3.15

$c = \dfrac{60000}{\rho_1 \Sigma \text{ COL. } 9}$

$k = \dfrac{\rho_1 c}{600} = 12.32$

2	4	10	13
PARTICLE DIAM. MICRONS	MICROAMMETER READING	TOTAL SURFACE sq. cm./gm.	% PER MICRON
70.3			
65			
60	11.5	2199	0.532
55	11.6	c = 2348	0.482
50	11.7		0.862
45	11.9		0.758
40	12.1		0.658
35	12.3		0.838
30	12.6		1.146
25	13.1		0.902
20	13.6		1.584
15	14.8		1.854
10	17.0		2.568
7.5	19.5		4.37

1
TURBIDIMETER NO. 15
RUN No. 13
DATE 12/20/33
CEMENT C
% PASSING No. ~~200~~ 325 SIEVE 78.4
WT. OF SAMPLE 0.4000
TEMP. DEG. C
I_0 100
LOG I_0 2.00000
ρ_1 3.15
$c = \dfrac{60000}{\rho_1 \Sigma \text{COL. } 9}$
$k = \dfrac{\rho_1 c}{600} = 10.17$

2	4	10	13
PARTICLE DIAM. MICRONS	MICROAMMETER READING	TOTAL SURFACE sq.cm./gm.	% PER MICRON
70.3			
65			
60	15.1	1587	1.328
55	15.5	c = 1939	0.594
50	15.7		1.056
45	16.1		0.920
40	16.5		0.990
35	17.0		0.996
30	17.6		0.948
25	18.3		1.058
20	19.3		1.156
15	20.8		1.394
10	23.6		2.080
7.5	27.0		2.89

1

TURBIDIMETER NO. 15

RUN No.14

DATE 12/21/33

CEMENT C

% PASSING No. ~~200~~ 325 SIEVE 78.4

WT. OF SAMPLE 0.4000

TEMP. DEG. C

I_0 100

LOG I_0 2.00000

ρ_1 3.15

$$c = \frac{60000}{\rho_1 \Sigma \text{ COL. } 9}$$

$$k = \frac{\rho_1 c}{600} = 10.37$$

2	4	10	13
PARTICLE DIAM. MICRONS	MICROAMMETER READING	TOTAL SURFACE sq. cm./gm.	% PER MICRON
70.3			
65			
60	15.4	1600	0.900
55	15.7	c = 1975	0.896
50	16.0		0.796
45	16.3		0.928
40	16.7		0.996
35	17.2		1.004
30	17.8		1.090
25	18.6		0.854
20	19.4		1.174
15	20.9		1.698
10	24.3		2.236
7.5	28.0		2.866

1

TURBIDIMETER NO. 15

RUN No. 15

DATE 12/21/33

CEMENT D

% PASSING No. ~~200~~ 325 SIEVE 69.7

WT. OF SAMPLE 0.5000

TEMP. DEG. C

I_0 100

LOG I_0 2.00000

ρ_1 3.15

$c = \dfrac{60000}{\rho_1 \Sigma \text{ COL. } 9}$

$k = \dfrac{\rho_1 c}{600} = 8.43$

2	4	10	13
PARTICLE DIAM. MICRONS	MICROAMMETER READING	TOTAL SURFACE sq. cm./gm.	% PER MICRON
70.3			
65			
60	18.3	1180	0.910
55	18.7	c = 1605	0.814
50	19.1		0.900
45	19.6		1.092
40	20.3		0.930
35	21.0		0.998
30	21.9		1.162
25	23.2		1.098
20	24.8		1.042
15	26.9		1.446
10	31.5		1.816
7.5	36.3		1.854

1

TURBIDIMETER NO. 15

RUN No. 16

DATE 12/21/33

CEMENT D

% PASSING No. ~~200~~ 325 SIEVE 69.7

WT. OF SAMPLE 0.5000

TEMP. DEG. C

I_0 100

LOG I_0 2.00000

ρ_1 3.15

$c = \frac{60000}{\rho_1 \Sigma \text{ COL. } 9}$

$k = \frac{\rho_1 c}{600} = 8.30$

2	4	10	13
PARTICLE DIAM. MICRONS	MICROAMMETER READING	TOTAL SURFACE sq. cm./gm.	% PER MICRON
70.3			
65			
60	18.3	1162	1.118
55	18.8	c = 1580	0.796
50	19.2		0.880
45	19.7		0.918
40	20.3		0.916
35	21.0		1.090
30	22.0		1.138
25	23.3		1.012
20	24.8		1.166
15	27.2		1.464
10	32.0		1.728
7.5	36.7		1.806

FIG. II

Showing Relations of Specific Surfaces as Measured by the Microneter, the Turbidimeter (Recommended Method), and Wagner's Method

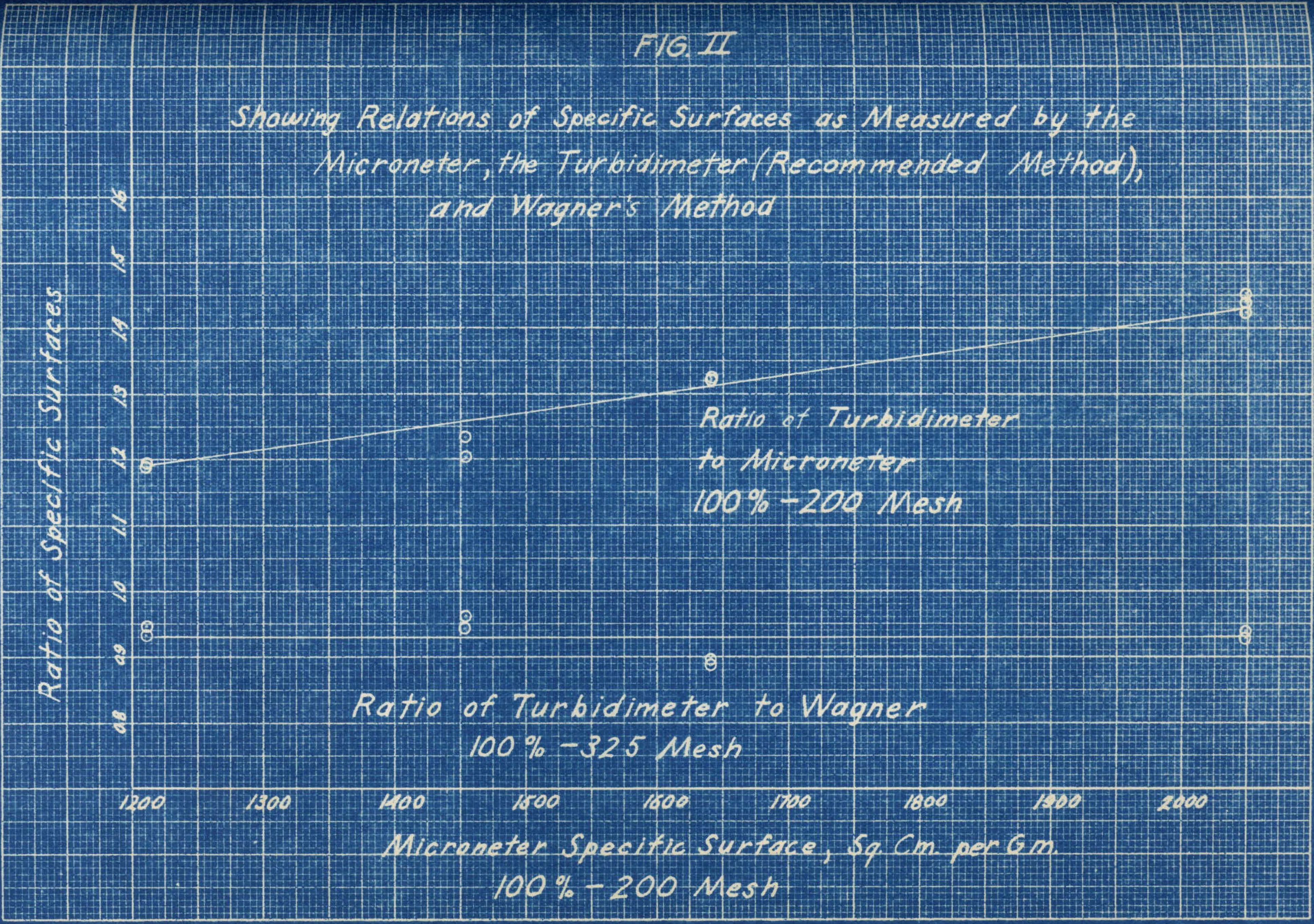


FIG. III a

Percent per Micron Plots

Cement A

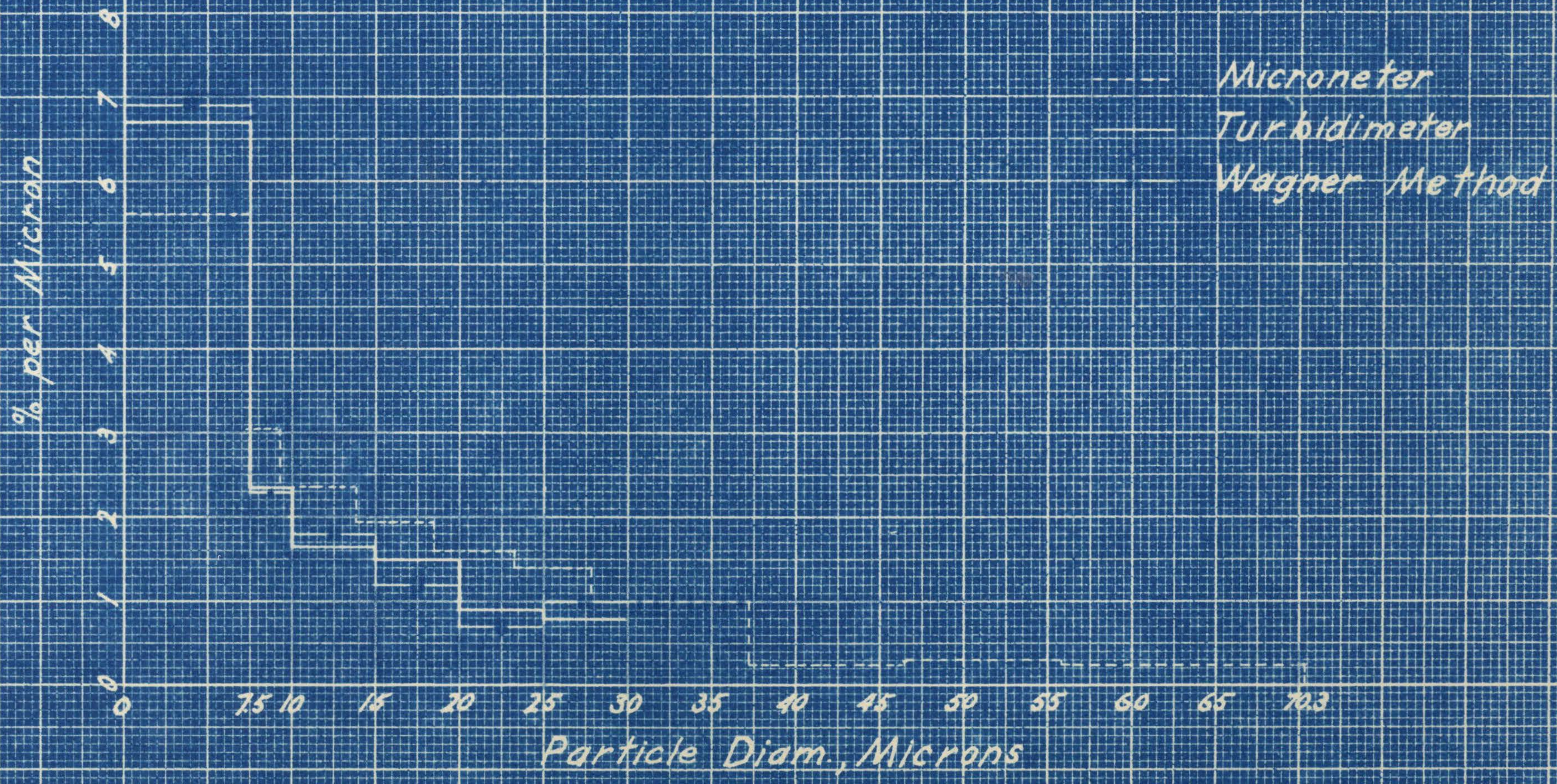


FIG. III b

Cement B

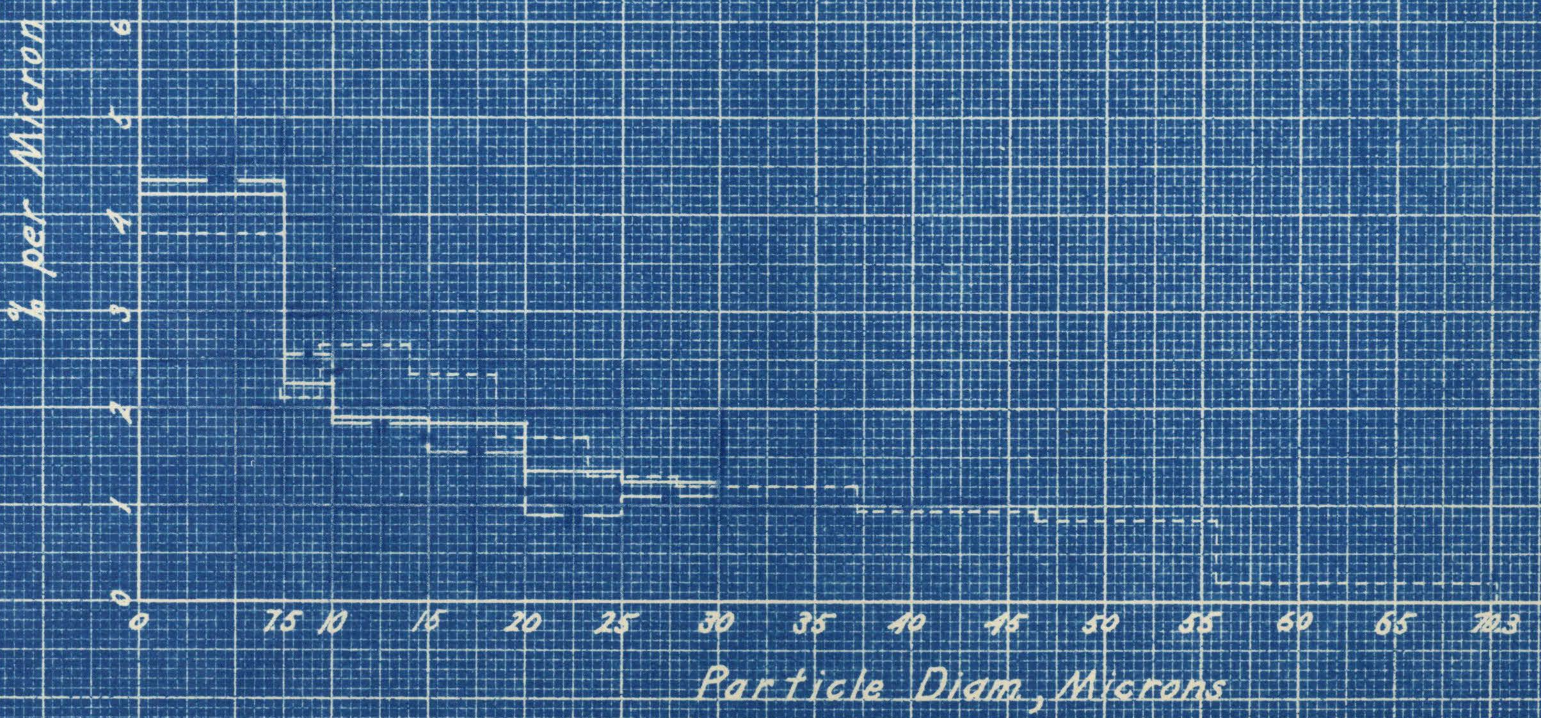


FIG. III c

Cement C

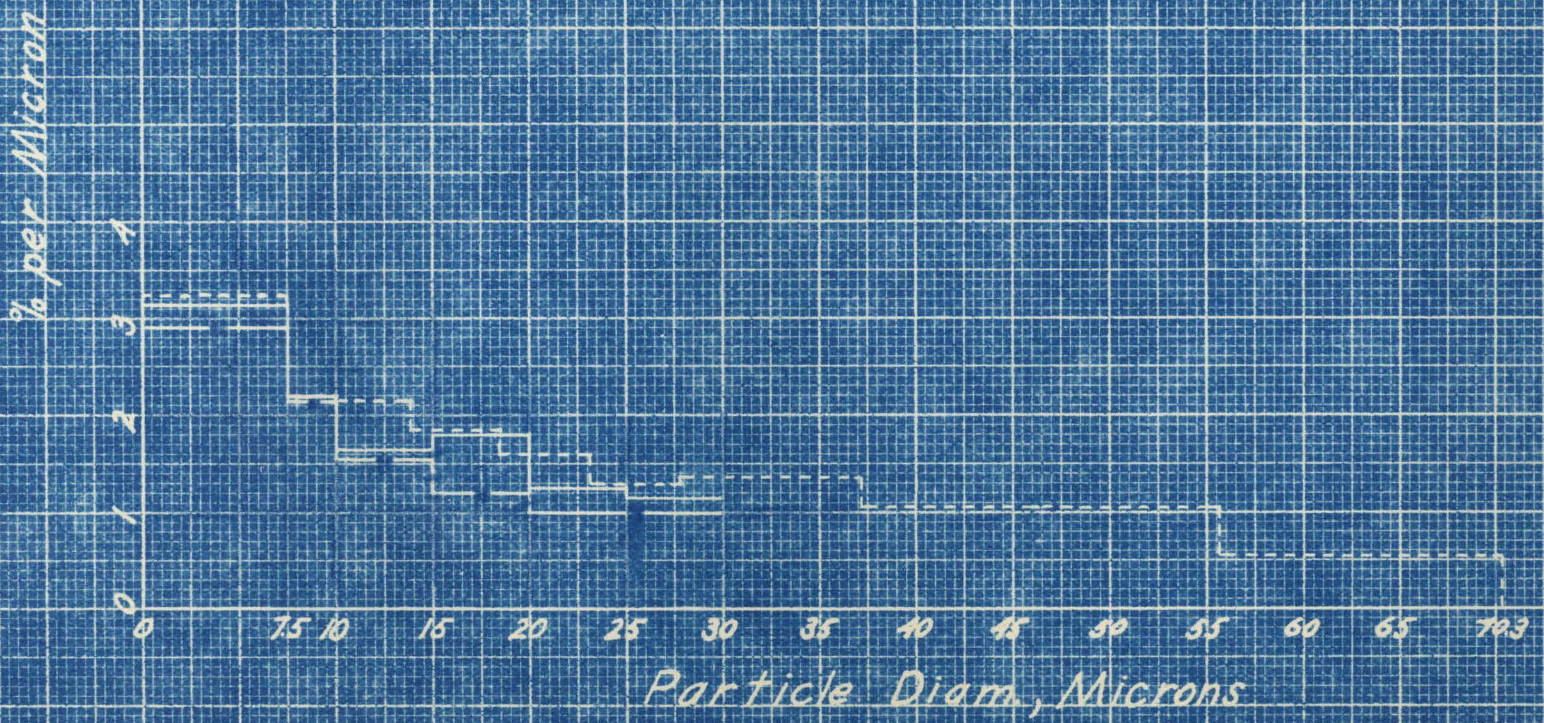
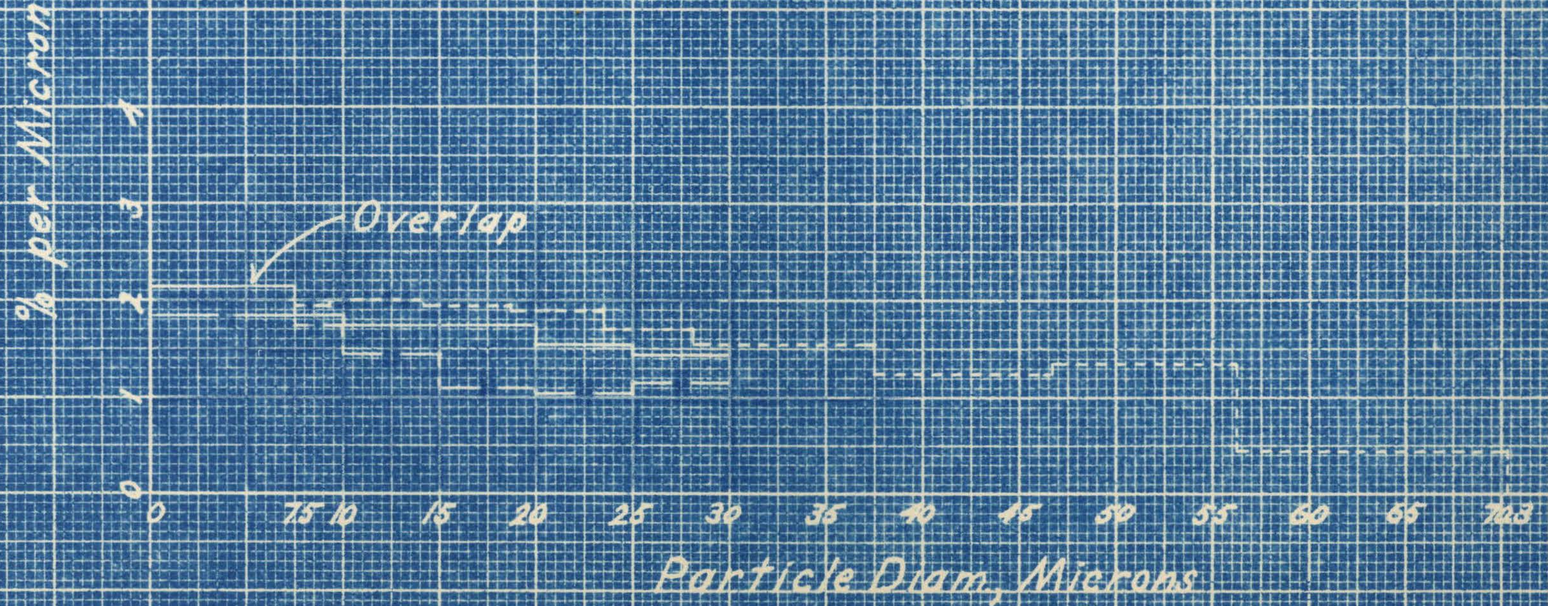


FIG. III d

Cement D



The four cements tested, Nos. A, B, C, and D, were supplied by the Riverside Cement Company of Riverside, California, together with an analysis of each made on the microneter; these analyses are reproduced above. A description of the microneter is given by Knapp, the inventor, in his Thesis for the Degree of Doctor of Philosophy, California Institute of Technology, 1929.

Eight pairs of duplicate runs were made on these cements using the turbidimeter. The results are given on the report sheets attached above. Runs 1 - 8 were made using the turbidimeter procedure given in Part I (Figure I shows the timing data used). Runs 9 - 16 were done by Wagner's method, given in the booklet "Turbidimeter Instructions" attached to Part II.

The complete calculations are shown for Runs 1 and 9 only; the other sheets give all the essential data used for their calculation, but are condensed for simplicity of reproduction in this paper.

The calculations for Runs 1 - 8 are made as in Part I; the calculations for Runs 9 - 16 are made as Wagner recommends in his article in the Proceedings of the A.S.T.M., except that the value of "C" listed by us is Wagner's "C" divided by the sample weight. For simplicity all the results are given on the same report form. The two methods of calculation differ only in arrangement of procedure and not in theory. The method of calculation given in Part I is recommended, because all the results may be obtained in a continuous operation.

The error sheets shown for Runs 1 and 9 give the approximate maximum experimental errors to be expected from the two methods of operating the turbidimeter. With careful operation these errors may be reduced, as has been illustrated by the results of other turbidimeter operators.

These error sheets do not show systematic errors resulting from defects in construction or theory of the turbidimeter.

Many of these systematic errors have been eliminated from the turbidimeter procedure, as explained in Part II. However, others remain and suggestions for their elimination are set down in Part V.

Figure II shows a comparison of the values of specific surface as obtained by the two turbidimeter procedures to those given by the microneter. Because the microneter analyses are started at 70.3 microns they cannot be directly compared to the Wagner method results which begin at 60. To overcome this difficulty, the analyses made by the method of Part I have been calculated for both 70.3 and 60 microns, so that these may be compared to each of the others. The results of these calculations are listed in the report sheets of Runs 1 - 8 in column 10 in their respective rows. The ratios of the values in the 70.3 row of Runs 1 - 8 to those given by the microneter (listed in the microneter analysis sheets as "surface for 100% -200 mesh") are plotted in the upper curve of Fig.II against the microneter specific surface. The lower curve was obtained by plotting the ratio of the values in the 60 row (column 10) of Runs 1 - 8 (multiplied by the fraction of sample passing the No. 325 sieve) to the value of the specific surface of Runs 1 - 9, versus the same abscissae.

The lower curve shows that the ratio of the specific surfaces given by the two turbidimeter procedures is a constant for the four different cements studied and that the specific surface given by the Wagner method is larger than that given by the method of Part I.

The upper curve shows that the specific surface given by the method of Part I is larger than that given by the microneter; and, furthermore, that this discrepancy increases the higher the specific surface of the cement being measured. The fact that this ratio is greater than one is partially accountable to the difference in methods of calculation of the turbidimeter and microneter. In the former the specific surface of the finest particles, the 0 - 7.5 micron fraction, is calculated on the assumption that the average diameter for the fraction is 3.75 microns. In the latter a value somewhat larger than this is used, experiment having shown this to be closer to the truth. The fact that the ratio is greater, the greater the specific surface is accountable to a fundamental error in the turbidimeter calculations: that the relation between the intensity of the transmitted light and the concentration of the suspension is not an exponential function, as is assumed for purposes of calculation (see Part II). Reference to the specific surface results of Runs 1, 1', and 2 will show that for Run 1' the specific surface is smaller than for the others. This is explainable on the above basis, because the size of sample in Run 1' was larger than for Runs 1 and 2. The same cement was used in all three cases.

Figure III shows the comparison of particle size distribution results for the three methods. These are reported as "Percent per Micron" plots, because this manner of representation gives the best picture of size distribution and the relative importance of the various fractions. The ordinates are the individual percentages (of the total sample) for each increment of particle diameter, divided by the increment. The abscissae are the diameters. Hence the area under the plot to the left of any abscissa gives the percentage of the sample smaller than the diameter indicated by the abscissa; the relative heights of the ordinates indicate the relative importance of each size of particle in making up the total sample. These curves are plotted from the average of each pair of turbidimeter runs.

The solid line shows the results of the method of Part I (listed in the plots as "Turbidimeter"). The broken line shows the results of the Wagner method of operation. The dotted line is the microneter. The plots for the turbidimeter are not carried above 30 microns because the errors in the per cent per micron figures above 30 are so large (see error sheets 1 and 9).

An examination of the analysis sheets of Runs 1 - 16 and Figures II and III shows that the method of Part I gives values of specific surface and particle size distribution more closely approaching the "actual" or microneter values, than does the Wagner method. There is little to choose between the accuracy for specific surface of the two turbidimeter methods; but, although time did not allow sufficient analyses to be made to prove it, it is confidently believed that the method of Part I will show consistently better accuracy than the Wagner method, and that with careful work a maximum deviation of specific surface in duplicate runs of ± 1% may reasonably be expected.

PART IV

GENERAL CONCLUSIONS

The method given in Part I will give values of specific surface of cement samples to a reproducibility of ± 1%. It will give a fairly good particle size distribution analysis for particles from 0 to 30 microns in diameter.

The method given by Wagner in Appendix I will not give as accurate results as that of Part I.

The results of the method of Part I, while accurate relatively, deviate considerably from what are believed to more closely approximate the absolute values. Furthermore, the deviation is not in the form of a constant ratio to the absolute.

The size of sample used in the turbidimeter is too small to give high accuracy of representation of the lot from which it is taken.

PART V

Recommendations for Research on the Wagner Turbidimeter

I. Find the true relation between intensity of light transmitted through a cement suspension and the concentration of the suspension. (See Channon, Renwick and Storr, Proc.Roy.Soc. (London) 94, 222 (1917).

II. Design the turbidimeter so that the size of sample is larger, preferably 50 grams.

III. Use the same size sample for all cements (this can be accomplished by varying I_o).

IV. Increase the accuracy of the microammeter reading.

V. Use a photoelectric cell with less fatigue characteristics than the "Photronic" type.

VI. Decrease the vertical width of the light beam to give a more accurate value for the concentration at any given depth in the suspension.

VII. Make the intensity of the light beam more uniform over the cross-section of the beak.

VIII Place the colored filter so that light will not be reflected from it up into the suspension.

IX. Use a petroleum fraction of more uniform composition than commercial kerosene. Use a more viscous liquid so that eddy currents from stirring will more quickly die out.

X. Use a larger value than 3.75 as the average diameter of the 0 - 7.5 micron fraction.

XI. Study the effect of variations of optical properties of various cements upon the turbidimeter determinations, as compared to results obtained by methods which are independent of optical properties.

ACKNOWLEDGEMENTS

Thanks are due the Metropolitan Water District of Southern California for furnishing the turbidimeter and the funds to cover necessary expenses other than those covered by the California Institute.

Thanks are due the Riverside Cement Company of Riverside, California, for supplying the cement samples used and their microneter analyses; the California Portland Cement Company of Colton, California, for discussing their investigations of turbidimeter methods; Dr. Robert T. Knapp of the California Institute of Technology for valuable advice; and Mr. C.D.Cooksey for making the photographs of the Wagner drawings.

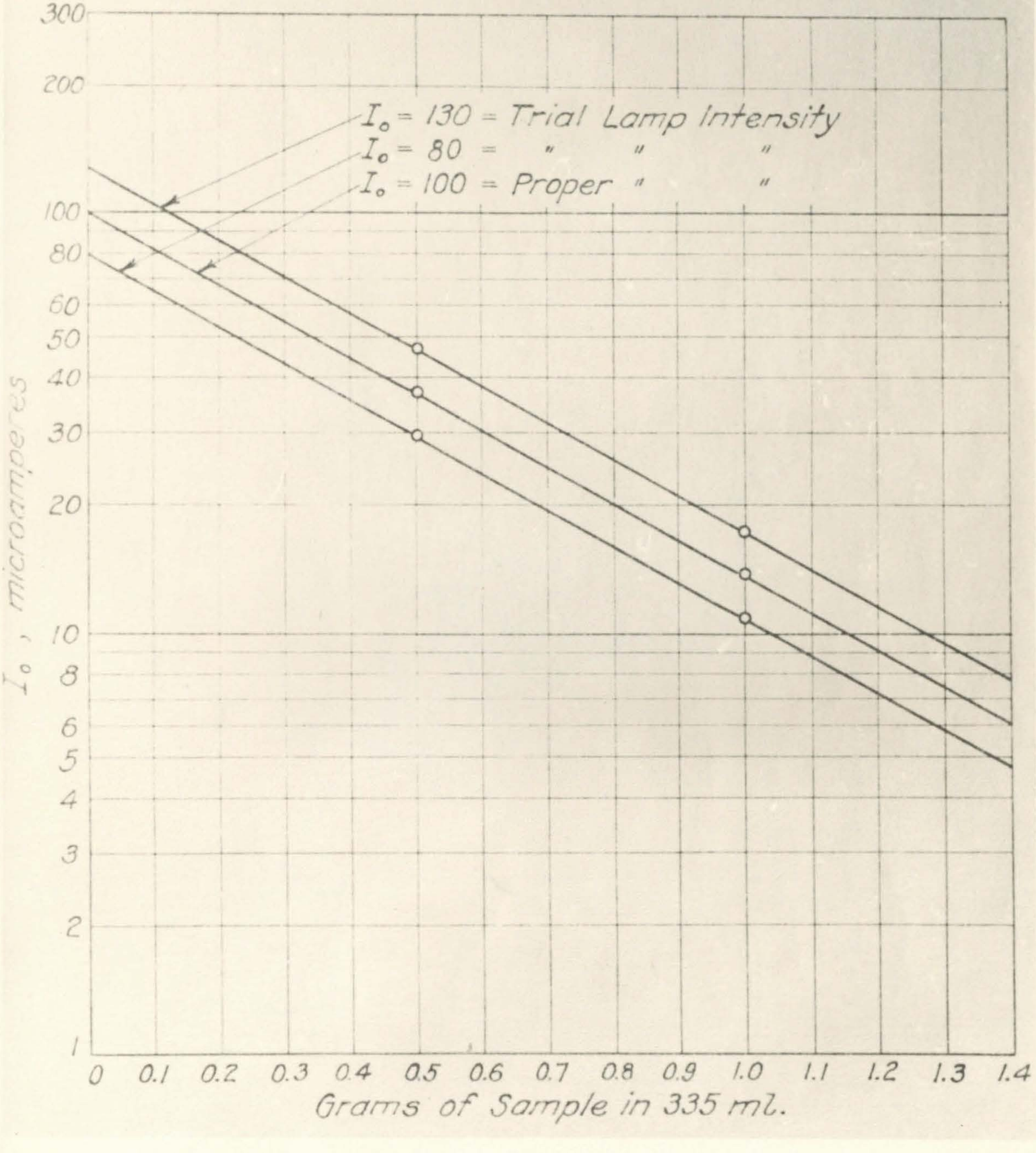
The work was done under the supervision of Professor William N. Lacey, to whom acknowledgement is hereby made for his valuable direction.

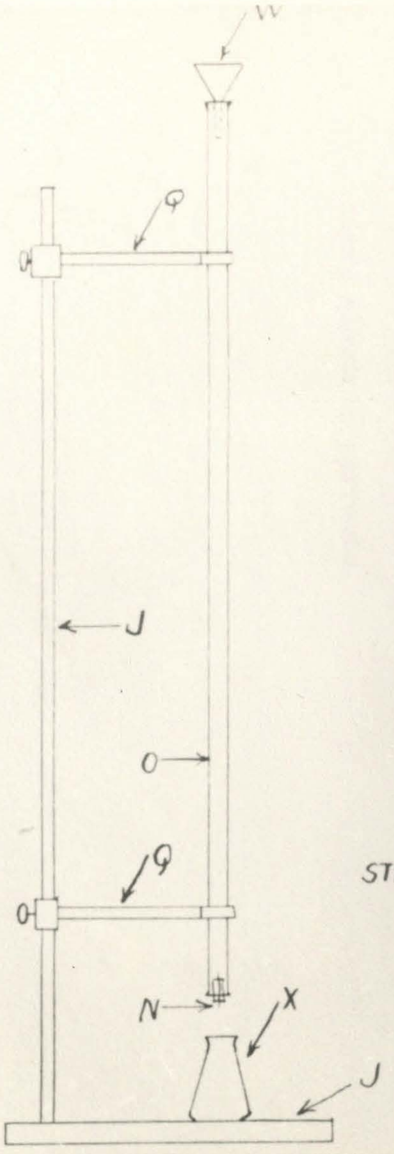
APPENDIX I

"Turbidimeter Instructions"

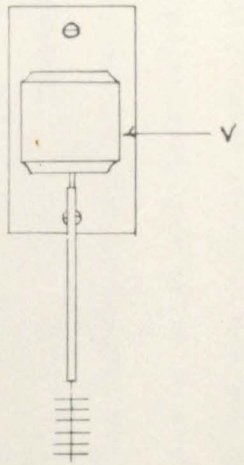
issued by

Cement Reference Laboratory

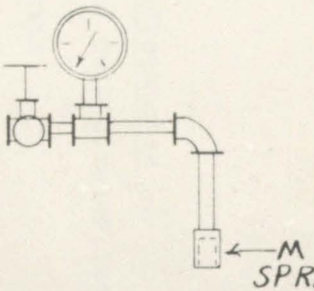

I_o = 130 = Trial Lamp Intensity
I_o = 80 = " " "
I_o = 100 = Proper " "
300
200
100
80
60
50
40
30
20
10
8
6
5
4
3
2
1
I_o , microamperes
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1.0
1.1
1.2
1.3
1.4
Grams of Sample in 335 ml.



TIMING BURETTE



STIRRING APPARATUS



SPRAY NOZZLE

Turbidimeter Instructions

Use of Microammeter

(1) Read Note 1 on page 9 of Turbidimeter Instructions before using microammeter.

(2) Keep microammeter level when in use.

(3) Turn knob from Transit position and set needle at zero by means of adjustment knob prior to test.

(4) At end of test return knob to Transit position before moving instrument.

(5) Avoid use of microammeter in proximity of wires carrying heave electric currents.

I. List of Parts of the Apparatus

(Letters refer to the drawing)

3 - Supporting brackets (A)
1 - Turbidimeter cabinet (B)
1 - Base for tank (D)
1 - Ring stand (J)
1 - Microammeter (K)
1 - Water spray (M)
1 - Capillary tube (N)
1 - Burette (O)
1 - Photoelectric cell (P)
2 - Burette clamps (Q)
1 - Sedimentation tank (T)
1 - Stirring motor (V)
1 - Funnel (W)
2 - Receiving flasks (X) not furnished
1 - Automobile storage battery, not furnished
1 - No. 325 sieve
1 - Bottle oleic acid with dropping stopper
1 - Can filter charcoal
1 - Bottle standard sample
6 - Sheets semi-log paper

II. Instructions for Installation

(Numerals in parentheses ( ) refer to notes at end of Sec. IV)

1. Attach the supporting brackets (A-see drawing) for the cabinet (B) and base (D) for the tank (T) to a masonry or concrete wall, column or pier. Attach tank bracket 3/4 inch lower than cabinet brackets. Level and mount cabinet and tank base on the brackets.
2. Adjust height of tank base (D) so that the surface of the liquid in the tank (containing 335 ml.) is 1 3/8 inches above the top of the shelf (E), when the pointer (F) is at the 0.0 mark on the scale (G).
3. Lower shelf until the pointer is at the 30-60 position on the scale and adjust position of tank base so that the tank has equal clearance from all sides of the slot (H) in the shelf.
4. Adjust leveling screws (I) until tank has equal clearance from sides of shelf slot with shelf at 0.0 level.
5. Connect lamp leads to storage battery.
6. Connect leads from photocell (P) to microammeter (K) and unclamp microammeter needle. (Warning: The microammeter is an extremely sensitive and fragile instrument and must be handled with great care. Do not overload it(1).)
7. Mount stirring motor (V) and connect to 100 volts a.c. or d.c.
8. Attach spray nozzle (M) to water tap.
9. Fit capillary tube (N) into lower end of burette (O) and mount burette on ring stand (J) with clamps (Q) so that bottom of capillary tube is 6 inches above base of stand. Place funnel (W) containing absorbent cotton mat in top of burette. Place receiving flask (X) below burette.
10. Set aside at least 20 gallons of clear kerosene in a sutiable container with draw-off tap.(2)

III. General Operating Instructions

1. Size of sample.- Use 0.3 g. for a very fine cement (more than 95 per cent passing a No. 200 sieve), 0.4 for a medium cement (85-95%) and 0.5 for a coarse cement (less than 85 per cent passing the 200 sieve).(3)

2. Preparation of suspension.- Place sample in test tube with 10-15 ml. of kerosene and 5 drops of oleic acid. Stir mixture for one minute with rotating brush. Transfer to tank using fresh kerosene to wash cement from bottom of tube, finally washing tube and brush with kerosene containing one drop of oleic acid. Add all washings to settling tank and adjust volume of suspension to 335 ml.

3. Agitation of tank to bring about a uniformity of suspension before placing in path of light beam.- Cover tank with the palm of the hand and oscillate tank 180° around a horizontal axis at the rate of about one inversion per second for one minute. Place tank in an upright position and wipe away any kerosene that drains down outside of tank.

4. Adjustment of the intensity of the light, I_o, to 100 microamperes.- With the filter (Y) in the path of the light beam,(1) but with the tank removed, adjust rheostats (R_1) and (R_2) (4) until microammeter reads 20 microamperes.(5) Prepare a suspension containing 0.5 g. of the standard turbidity sample, oscillate tank one minute and place in position(6) in the turbidimeter, the scale pointer being at the 30-60 position. Read the microammeter at exactly one minute from end of oscillating period. Prepare a second suspention using 1 g. of the same standard sample and following the same procedure, obtain a second microammeter reading. Plot readings as ordinates on semi-log paper against weight of sample as abscissa as shown in Fig. 2. Draw

a straight line through the two points. If the intersection of line with axis of zero weight of sample is other than 100, readjust intensity of lamp through filter and repeat the process until this value is obtained. When the desired intensity of 100 microamperes is found, the reading through the filter but with the tank removed becomes a permanent reference value of the intensity of the light and the lamp must be adjusted to give this value for each analysis.(7)

5. Calibration of burette scale.- Note temperature of kerosene, adjust intensity of lamp as explained in the preceding paragraph, place scale pointer at 30-60 position, oscillate and place in position a suspension containing one gram of the standard turbidity sample. Note elapsed time, t, in seconds, from end of oscillating period until microammeter reads value specified for standard sample (23.4 microamperes for Turbidity Standard No. 2). Repeat twice using the average of the three values of t, multiply by values of h/d^2, Table I, to obtain values of $T (T = th/d^2)$, and fill in fourth column of the table (compute T in minutes and seconds to an accuracy of one second).

Fill burette with kerosene of same temperature as noted above. Start a timing clock at the instant kerosene in burette drains past the zero line and mark on strip of paper behind burette the levels reached by the draining kerosene in the time intervals, T in minutes, calculated as described. Write at these marks the corresponding particle diameters from Table 1.(8)

6. Calibration of the No. 325 sieve.- Adjust pressure on spray nozzle to 10 lbs. per sq.in. and wash one gram of standard sieve sample for one minute. Dry sieve and residue in oven or over hot plate,(9)

TABLE I. Values of "h" and "d" to be used for calibration of the apparatus.

Particle diameter d	Depth of suspension h	h/d^2	Settling period equals th/d^2 T
microns	centimeters		minutes
60	15	0.00417	This column is to be filled in by operator after the interval, t, has been determined.
55	15	.00496	
50	15	.00600	
45	15	.00741	
40	15	.00938	
35	15	.01225	
30	15	.01667	
25	13.1	.0210	
20	10.0	.0250	
15	6.6	.0293	
10	3.3	.0333	
7.5	2.1	.0375	

brush residue from sieve, weigh on an analytical balance, and calculate per cent passing. The difference between value obtained in this manner and value given for standard sample is the sieve correction.

7. The 325 sieve determination.- Use the same procedure for a cement to be tested as that described for calibration of the sieve. Add or subtract the sieve correction from the actual per cent passing to obtain the corrected value, r, of the per cent passing the 325 sieve.(10)

IV. Test Procedure.

1. Place pointer scale at 30-60 position.
2. Adjust intensity of lamp through filter, but with tank removed, to proper value (see Sec. III, paragraph 4).
3. Prepare suspension of sample to be tested (see Sec. III, paragraph 2).
4. Fill burette with kerosene. Immediately commence oscillation of tank and continue until kerosene in burette drains to zero line. Cease oscillation, wipe away any kerosene which drains down outside of tank and place the tank in position in path of light beam.
5. Remove filter and close cabinet door.
6. Read microammeter at instants kerosene in burette drains past marks 60, 55, 50, 45, 40, 35 and 30. After obtaining the reading at 30 immediately raise the shelf to the 25 mark on the pointer scale and read the microammeter as the kerosene drains past the corresponding mark on the burette. Raise shelf and obtain readings in a like manner at the remaining marks of 20, 15, 10 and 7.5.
7. Place filter back in path of light beam, remove tank, and check intensity of lamp. (11)

8. Make a 325 sieve determination on another sample of the same cement, the per cent passing being denoted as r.

9. Calculate the specific surface of the sample from the equation

$$S = \frac{38\ r\ (2 - \log I_{60})}{1.5+0.75 \log I_{7.5} + \log I_{10} + \log I_{15} \ldots \text{etc.} + \log I_{55} - 11.5 \log I_{60}}$$

where

S = specific surface of sample, sq.cm. per gram.

r = per cent by weight of sample passing 325 sieve.

$I_{7.5}$, I_{10}, I_{15}, etc. = microammeter readings which correspond to particle sizes of 7.5, 10, 15, etc. microns

Having determined the specific surface of a sample of cement from a given plant, the specific surface of subsequent samples from the same plant can be quickly obtained by observing only the first turbidity reading, I_{60}, of the subsequent suspensions and calculating their specific surface from the ratio:

$$\frac{S \text{ (unknown sample)}}{S \text{(known sample)}} = \frac{2 - \log I_{60} \text{ (unknown sample)}}{2 - \log I_{60} \text{(known sample)}} \quad (12)$$

Notes

(1) Either the filter or the tank containing a suspension of cement or both must be in the path of the light beam at all times. If not, the full strength of the light beam will drive the pointer off the microammeter scale and possibly injure it. This is important.

(2) The used kerosene may be reclaimed by stirring one tablespoon of filter charcoal with 5 gallons of kerosene, allowing the cement to settle, and filtering off the clear kerosene.

(3) The size of sample is selected so that the microammeter readings for the suspension will fall in the middle portion of the microammeter scale.

(4) R_2 is the fine adjustment for R_1.

(5) A few minutes are required for the photocell to reach equilibrium after the light is turned into it.

(6) The base for the tank has stops on the side and on the back so that the tank may always be placed upon it in exactly the same position.

(7) If the lamp, photocell, tank, filter or any other part of the light system is changed or moved, the value of the intensity of the beam through the filter must be redetermined.

(8) By using the calibrated burette the apparatus may be used within the normal range of room temperatures without further correction, the change in rate of flow of the kerosene from the burette automatically compensating for change in viscosity of the suspension due to temperature. The kerosene in the burette and that of the suspension should be kept the same within 1°F. This condition will ordinarily exist if the supply of kerosene is kept in the same room as the apparatus.

(9) Care should be taken not to heat the sieve hot enough to soften the solder.

(10) After four or five determinations the sieve should be dipped in 10 per cent hydrochloric acid and immediately rinsed with water. This removes particles lodged in the meshes. After 25 determinations the sieve should be recalibrated.

(11) If the intensity of the lamp has shifted more than 0.3 microamperes from the initial setting, the test must be repeated. A freshly charged storage battery should be short-circuited momentarily to reduce the voltage to constant value. If the intensity of the lamp continues to fluctuate, look for loose connections in the lamp circuit and make sure that the bulb and bulb socket are rigidly fixed in the lamp.

(12) The particle size distribution of the sample may also be calculated from the microammeter readings as obtained with this apparatus. Details of the process will appear in the Proceedings of the American Society for Testing Materials for 1933.

APPENDIX II

Detailed Report of Experimental Work

Appendix II

FIGURE I: Showing effect of method of preparation of kerosene and sample suspension.

Commercial Kerosene Used Just as Purchased				Commercial Kerosene purified with $CaCl_2$ and Charcoal			
Run	Method of Suspending	I	$\frac{I_o}{I}$	Run	Method of Suspending	I	$\frac{I_o}{I}$
1	Stirred only	33.2	3.36	9	Stirred only	34.4	3.30
2	" "	36.3	3.07	10	" "	35.9	3.16
3	2 Drops of Oleic and Stirred	32.2	3.50	11	2 Drops of Oleic and Stirred	31.9	3.49
4	2 Drops of Oleic and Stirred	33.8	3.34	12	2 Drops of Oleic and Stirred	31.9	3.49
5	Boiled and Stirred	50.8	2.20	13	Boiled and Stirred	43.3	2.61
6	" " "	42.5	2.63	14	" " "	42.2	2.68
7	2 Drops of Oleic Boiled and Stirred	27.5	4.06	15	2 Drops of Oleic Boiled and Stirred	27.9	3.99
8	2 Drops of Oleic Boiled and Stirred	29.4	3.84	16	2 Drops of Oleic Boiled and Stirred	31.2	3.57

These experiments were designed to show:

(a) The effect on flocculation of the purification of the kerosene, as carried out in Part I, Sec. II, 8.

(b) The effect of boiling the sample in the kerosene to mix it, as specified in Boulder Dam Specifications No. 591-D.

(c) The effectiveness of oleic acid in preventing flocculation.

The data of Figure I were obtained by suspending 0.0400 gram of cement No. A in 98 ml. of kerosene in a glass tank of the same dimensions as the one sent with the apparatus with the exception of height, it being only four inches high. This was used instead of the larger one because several other liquids were to be tried for flocculation, and it was not desired to subject the large tank to their action. This concentration of cement corresponds to 1.36 gm. in 335 ml. and was made purposely large to insure flocculation whenever in might occur. Two drops of oleic acid were added in eight of the runs, this concentration of oleic acid corresponding to 6.84 drops in 335 ml. Sixteen runs were made with fresh materials each time and in all of them the method of stirring with the rotating brush given in Part I, Sec. III, 2, was used to mix the cement, kerosene, and oleic acid.

In Runs 1 and 2 the cement was weighed out, placed in a test tube with 15 ml. of unpurified kerosene and stirred with the rotating brush for 1 minute. This suspension was then transferred to the tank, the test tube washed out, 15 ml. more of the kerosene added to the test tube and then stirred with the brush again. These washings were transferred to the tank and the volume was adjusted to 98 ml. The tank was placed in the turbidimeter with the shelf at the 30-60 level. In this position the center of the light beam was 0.969 in. below the surface of the liquid. The stirring rod (see drawing, Part I) was placed in the liquid and the suspension stirred for one minute. The stirring rod was then removed from the tank and a stopwatch started. The colored filter was removed from the path of the light and the cement was allowed to settle until all particles larger than ten microns in diameter had settled past the center line of the light. A reading (I) was then taken, the tank was removed, and the cement filtered from the kerosene. This kerosene was then put back in the (cleaned) tank and a reading taken for I_o with the tank in position and the colored filter removed, using the calibrated shunt on the microammeter. From this the ratio of I_o/I was calculated. The time at which the reading (I) was taken was calculated from Stokes' law for spheres ten microns in diameter and corrected in each case for the temperature of the tank, from the known viscosity and density vs. temperature relations of the kerosene.

Runs 9 and 10 were done in the same manner using the purified kerosene.

In Runs 3, 4, 11, 12, 7, 8, 15, and 16 two drops of oleic acid were placed in the kerosene in the test tube before the cement was weighed into it. Following the addition of the oleic acid, the procedure in runs 3 and 4 using unpurified kerosene, and 11 and 12 using purified kerosene was the same as for 1 and 2.

In Runs 5, 6, 7, 8, 13, 14, 15, and 16 the test tube contents were boiled for two minutes after stirring with the rotating brush; the suspension was allowed to cool and then was stirred again for one minute, after which the contents were transferred to the tank and the procedure of runs 1 and 2 followed.

An inspection of the two columns of values for I_o/I shows the effect of these procedures; the reason for recording the results as a ratio is that the value of I_o, the light transmitted through the liquid and tank with no cement in suspension, varied from one type of run to another; and in order to compare the results strictly, it was necessary to correct for this change.

Any fluctuation between the values of I_o/I was interpreted as caused by flocculation. The smaller the value of I_o/I, the greater the effect of flocculation: the value of I is larger, indicating that more light passed through the suspension; this means either that less cement was present at the level of the light, or that it existed there in larger particles which absorbed less light, or both.

It will be seen that with unpurified kerosene no pair of runs checked, indicating that flocculation may be expected unless the kerosene is thoroughly dried and cleaned.

With purified kerosene the runs in which oleic acid was used (11, 12, 15, and 16) showed less flocculation than those in which it was absent. Runs 1, 2, 5, 6, 9, 10, 13 and 14 showed a mirror-like film of the finest cement particles deposited on the walls of the tank. Although runs 15 and 16 in which the suspension was boiled showed higher values of I_0/I than runs 11 and 12, the latter gave duplicable values and the former did not. A further series of thirteen runs showed that the method of 11 and 12 gave the same value of I_0/I to 1% while the method of 15 and 16 gave an average deviation of 9% from the mean. It was therefore concluded that flocculation was not present in the method of 11 and 12, and that the effect of boiling was to introduce some other uncertainty into the procedure.

FIGURE II: Showing difference between shaking and stirring methods of suspension

Run	Method of suspension	Final Temp. of Tank °C	Initial Reading (no settling)	Time for Reading to reach 10.0 microamps. Min. and Sec.
1	Wagner	23.1	7.2	10:20
2	"	23.2	7.2	11:05
3	"	23.3	7.2	10:39
4	Stirrer	22.1	7.2	7:28
5	"	22.1	7.2	7.03
6	"	22.2	7.2	7:50
7	"	22.2	7.2	7:30
8	"	22.3	7.2	7:37

Experimental Procedure: 0.3000 gram of cement No. A was placed in 335 ml. of kerosene in the tank by the method of III (2). Runs 1, 2, and 3 were agitated by Wagner's method given in III (3) and placed in the turbidimeter with the shelf at the 30-60 level. An initial microammeter reading of the light transmitted was taken. The shelf was raised to the 7.5 level and the time from cessation of shaking until the microammeter read 10.0 was taken with a stopwatch. After the run the tank was removed, its temperature noted, and it was allowed to stand until it regained room temperature. When this was accomplished, the tank was re-agitated and the readings of Run 2 taken. Run 3 followed Run 2 in similar fashion.

Run 4 was made on a new suspension of the same weight of cement treated as in III (2) and placed in 335 ml. in the tank. The tank was placed in the turbidimeter and stirred with the stirrer (described in Part I), for one minute. The initial light intensity was then read, the stopwatch was started and the time required for the microammeter reading to reach 10.0 was taken, the shelf being at the 7.5 level, as above. After the temperature was noted, the suspension was re-stirred and Run 5 made. Runs 6, 7 and 8 followed in the same manner. All the runs were made with the light through the colored filter as an intensity standard, its value being 20.0 microamperes which was controlled by the potentiometer and checked between runs.

A comparison of the results of the last column shows that the time required for the settling of 9.2 micron particles is about three minutes longer when the suspension is shaken by Wagner's method, than when stirred with the stirrer, all other conditions being equal (the correction for temperature difference shown would increase the discrepancy slightly). That this difference in time is caused by the presence of the air-bubbles introduced in the shaking method, and not by a failure of the stirring rod to properly mix the suspension is shown by the fact that the initial intensities are the same, indicating the same distribution of cement particles in both cases.

The stirring rod has none of the disadvantages of the shaking method, which have already been mentioned. It has one disadvantage in the fact that prolonged stirring causes electrostatic effects in the suspension. These are the probable cause of the differences in times noted in Runs 4, 5, 6, 7 and 8. It has been found, subsequent to these runs, that these effects may be almost entirely eliminated by connecting the grounded wire to the stirrer and the tank base. The small hole in the top of the turbidimeter for the stirrer handle does not admit enough light to have the slightest measurable effect.

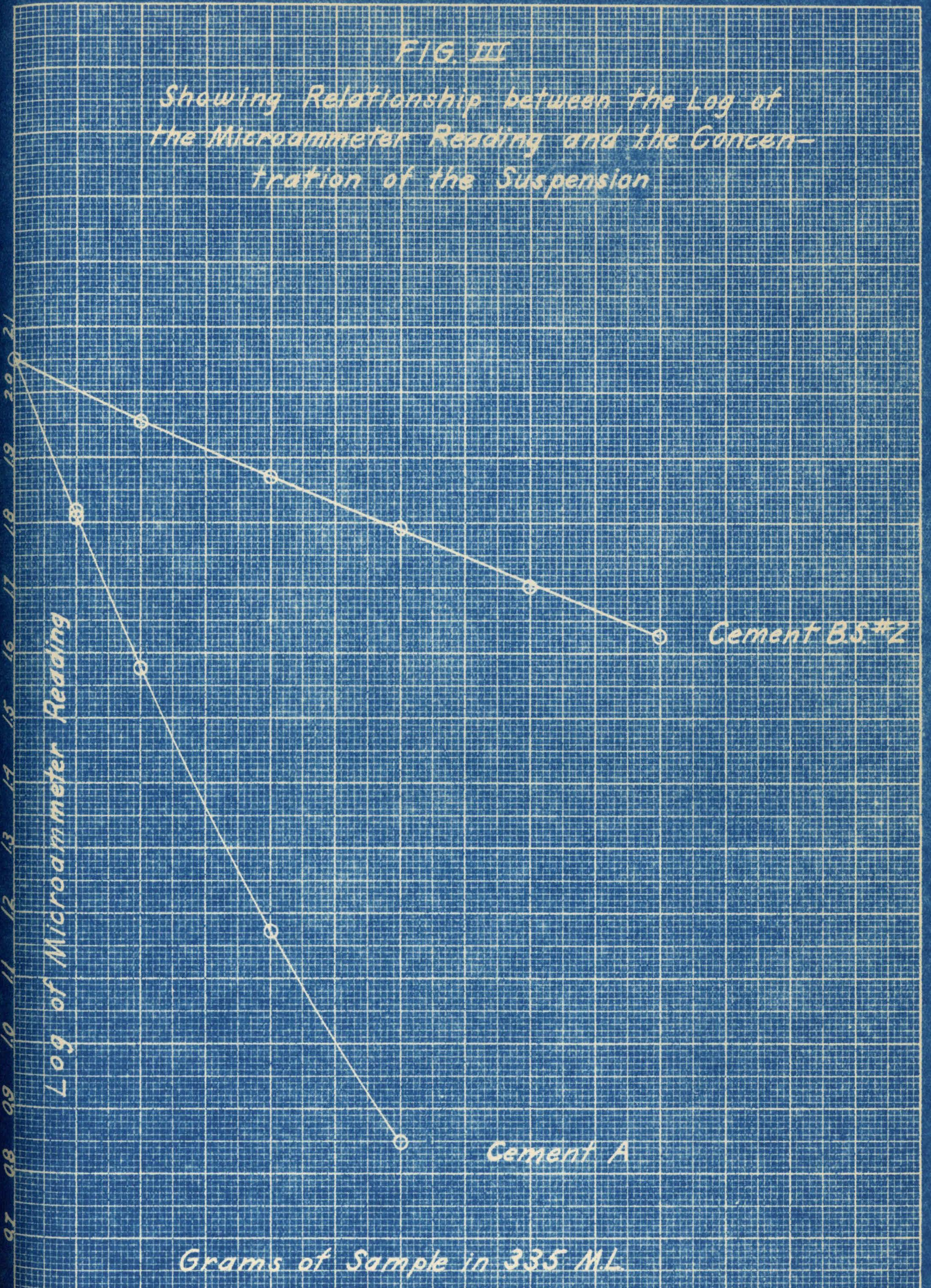


FIG. III

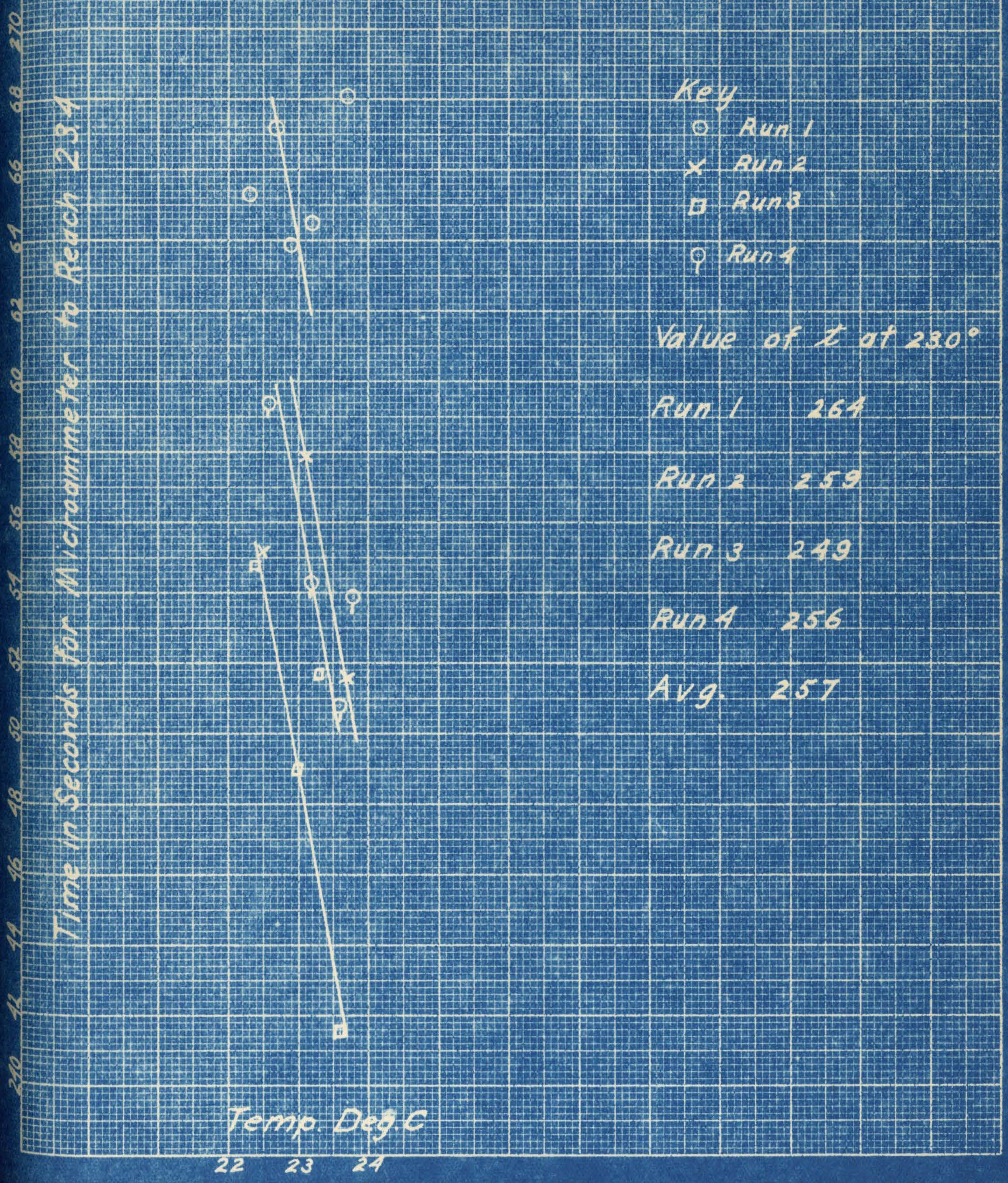
Showing Relationship between the Log of the Microammeter Reading and the Concentration of the Suspension

The data for Figure III were obtained in the following manner: Varying quantities of the cement used, No. A and B.S. No. 2, were weighed out, mixed with oleic acid and kerosene as in III(2), and suspended in 335 ml. of clean, dry kerosene by stirring with the stirrer for one minute. The stirrer was removed and at one minute from cessation of stirring a reading, I, was taken. The suspensionwas then filtered and the filtrate put back in the cleaned tank and a reading, I_o, taken. After each run the intensity of the light through the colored filter, with the tank removed, was checked, its value being held at 20.0 microamperes throughout the work by means of the potentiometer. Four pairs of duplicate runs were made on No. A, the quantities used varying from 0.0500 gram to 0.3000 gram. With the exception of the 0.05 gram runs, the results of the two runs on each concentration checked to better than 1%. Five pairs of duplicate runs were made on cement No. 2, the quantities varying from 0.1000 to 0.5000 gram, and in each case the pairs of values checked to better than 1%. In all the runs the value of I_o/I was calculated, and the value of I which is plotted was calculated by dividing 112.5 by this ratio. This was necessary because the value of I_o varied between the different runs. The value of 112.5 is the average of all the I_o's measured. Each curve, therefore, represents the true relationship between the logarithm of intensity of transmitted light, measured as microamperes, and the concentration of cement in suspension as grams in 335 ml. of kerosene. If Wagner's assumption (that this relationship is exponential) were true, a straight line drawn thru any two points and extended to zero concentration would cut the ordinate scale at 2.05115 ($\log_{10}$ 112.5). In the lower curve this is not the case, but in the upper curve this condition is very closely approximated.

Since cement No. A has a very high specific surface and cement No. 2 a very low specific surface, it may be concluded that for cements of high specific surface the turbidimeter does not give the relationship claimed for it by Wagner, whereas for those of lower specific surface the relationship is more closely followed. Since the calculations of the specific surface of a cement from turbidimeter data are based on the assumption that the above relationship is exponential, the value of the specific surface as calculated will vary the more from the true value the greater the specific surface of the cement being tested. Reference will again be made to this inherent error in the turbidimeter in a discussion of the results of actual tests on cement.

FIG. IV

Data Obtained in Calibration of Burette Scale



Value of Z at 23.0°

Run 1	264
Run 2	259
Run 3	249
Run 4	256
Avg.	257

The data shown in Figure IV were obtained by following Wagner's procedure of III, 5. It was decided to make four runs instead of three when the results of the first three appeared inconclusive. Since the method of shaking raises the temperature of the tank by an indefinite amount, each of the four samples was reshaken and results obtained at several temperatures. The temperature of the tank was taken after the run was over, it having been discovered that the final temperature of the tank did not differ by more than 0.1°C from the average temperature throughout the run. These temperatures are plotted in Figure IV as abscissae, with the elapsed time in seconds as ordinates. Four straight lines having the same slope were drawn through the four sets of values; this method of plotting is justified because the variation of time of settling over the temperature range is a straight line function of temperature; and, because the same kerosene was used, it should be the same function in all four cases. The times were read from these plots for 23.0°C and the average, 257 seconds, was taken as the correct value of t for the calibration. The burette scale was then calibrated by the directions at 23.0° to an accuracy of 0.5 second.

FIGURE V: Showing the error resulting from use of the timing burette.

Particle Diam. d Microns	Depth of Settling h cm	Time by Stokes Law Min	Time by Burette Min	% Error
60	15.0	1.234	1.072	13.1
55	15.0	1.468	1.275	"
50	15.0	1.776	1.542	"
45	15.0	2.19	1.904	"
40	15.0	2.78	2.41	"
35	15.0	3.63	3.15	"
30	15.0	4.93	4.28	"
25	13.1	6.21	5.39	"
20	10.0	7.40	6.42	"
15	6.6	8.67	7.53	"
10	3.3	9.86	8.56	"
7.5	2.1	11.11	9.64	"

Temperature = 23.0°C

Viscosity of Kerosene, μ = 23.3 millipoises

Density of Kerosene, ρ_2 = 0.81 gm/ml

Density of Cement, ρ_1 = 3.22 gm/ml

$g = 980$ cm/sec^2

Figure V shows the time for the settling of the various sizes of particles which are employed as calculated from III, Table I, for the burette; and as calculated from Stokes' law ($T = \frac{3\, h \mu \times 10^7}{(\rho_1 - \rho_2) g\, d^2}$). A cement of high density (3.22) is purposely employed in the calculation by Stokes' law to show that variation in cement density cannot be neglected. Wagner makes the assumption, for the purpose of the turbidimeter, that all cements have a density of 3.15. If this were true the per cent error listed in the last column would be reduced from 13.1 to 10.5; however, cements of higher density are frequently met with and to neglect this fact allows an avoidable error. Wagner neglects this same source of error in the equation for specific surface given in IV, 9. The times of settling listed in columns 3 and 4 of Figure V are those at 23.0°C.

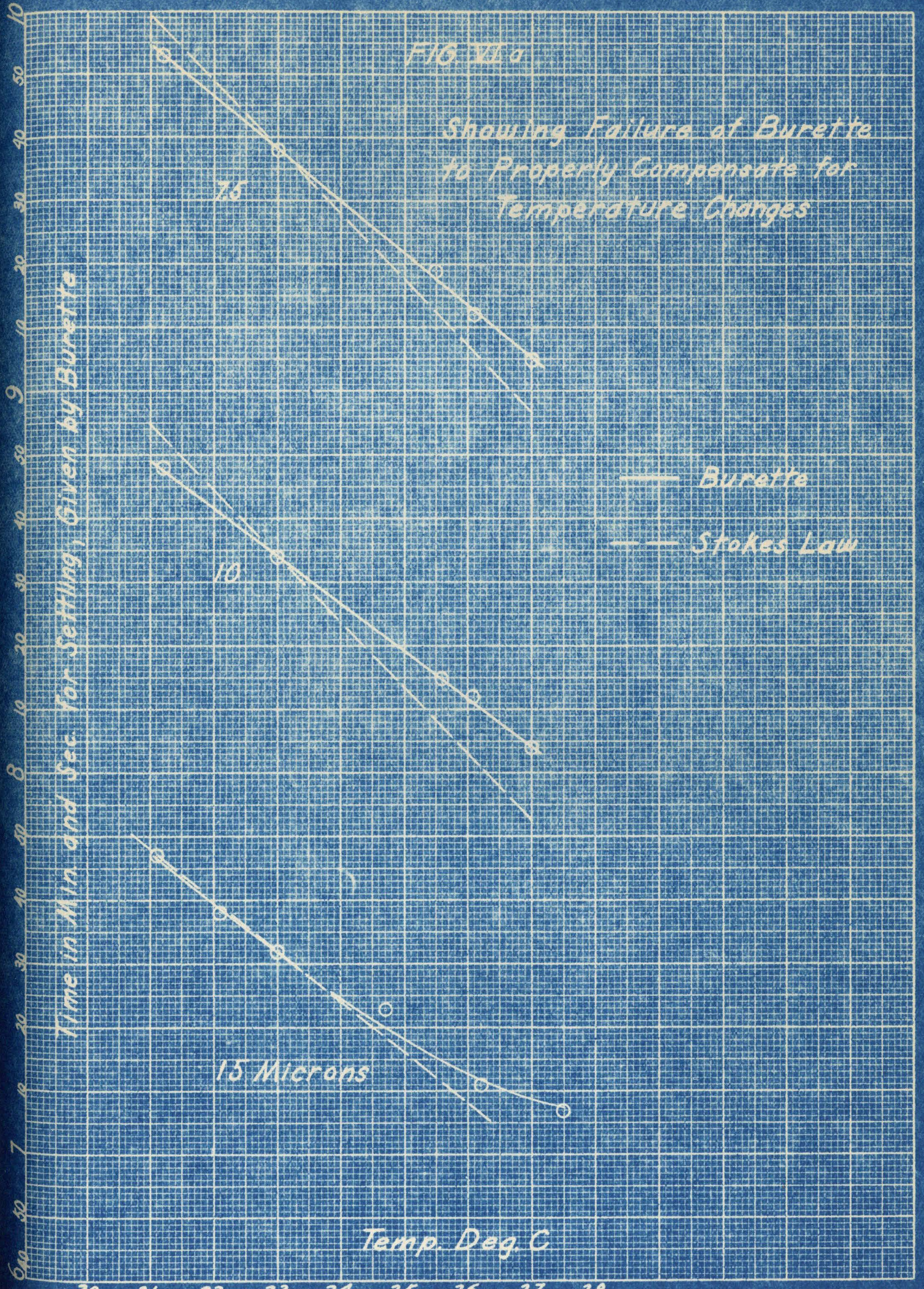


FIG. VIa

Showing Failure of Burette to Properly Compensate for Temperature Changes

FIG. VI b

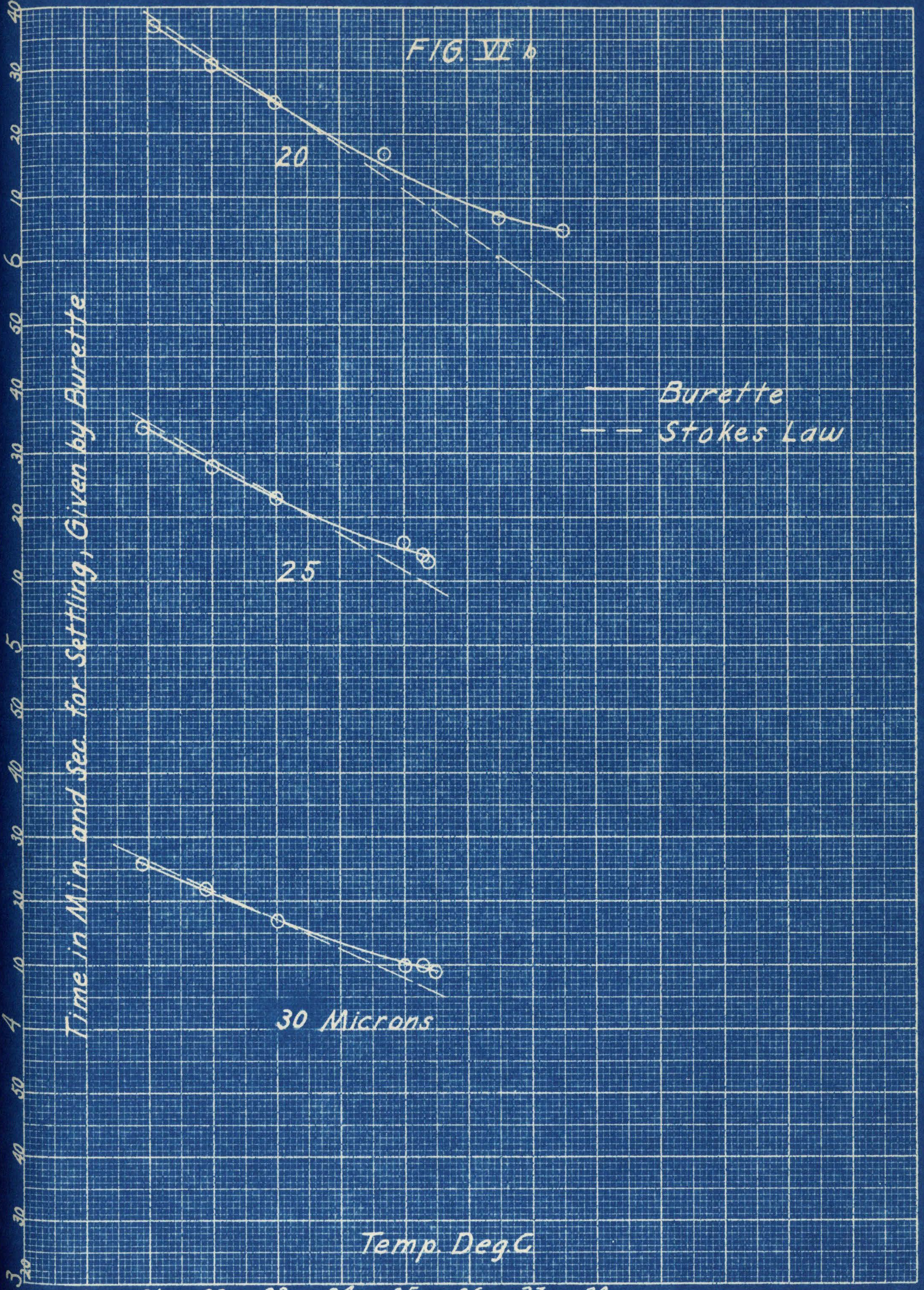
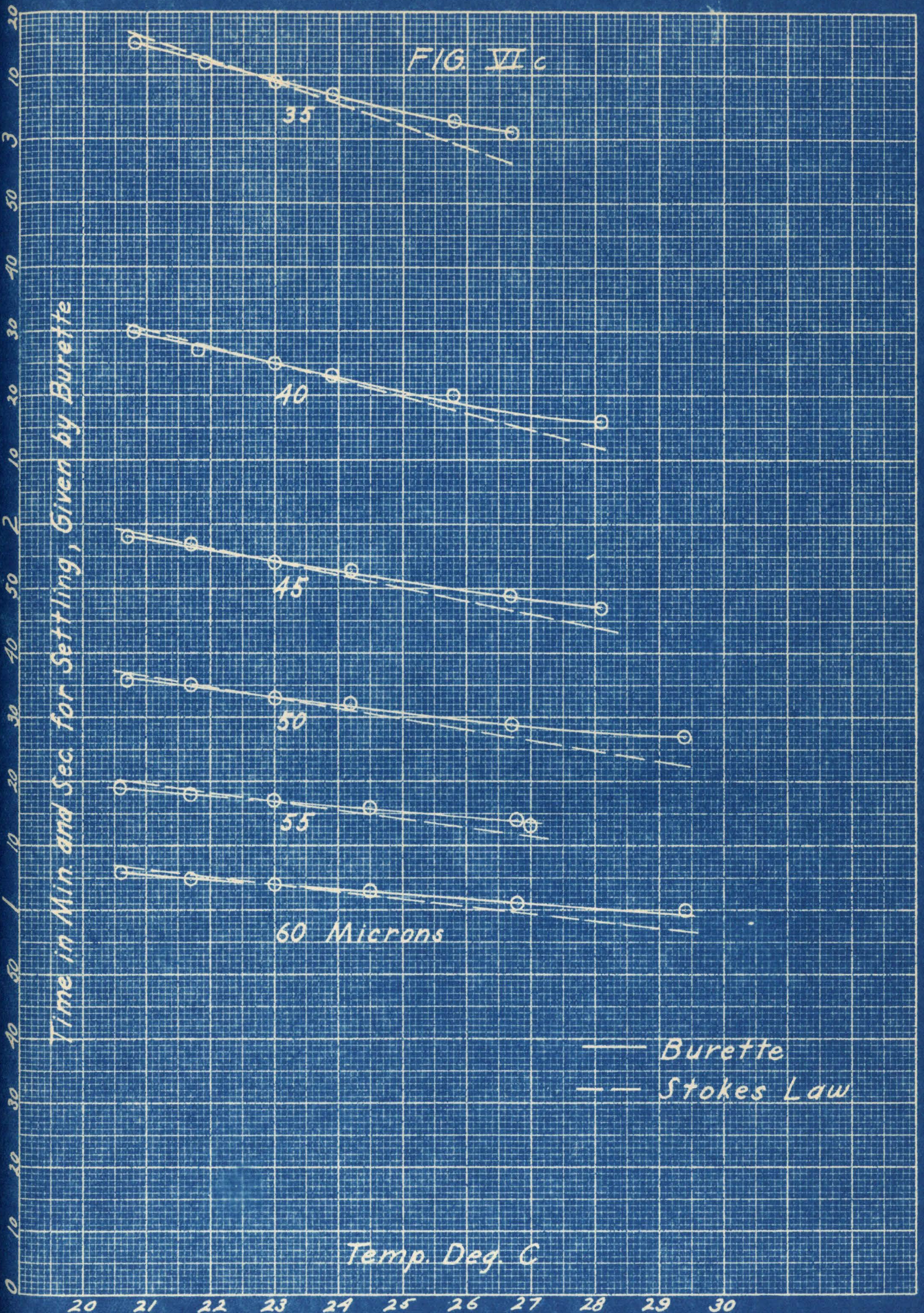

20
25
30 Microns
Burette
Stokes Law
Time in Min. and Sec. for Settling, Given by Burette
Temp. Deg. C.
40
30
20
10
6
50
40
30
20
10
5
50
40
30
20
10
4
50
40
30
20
3
20
21
22
23
24
25
26
27
28

FIG. VI c


Time in Min and Sec for Settling, Given by Burette
20
10
3
50
40
30
20
10
2
50
40
30
20
10
1
50
40
30
20
10
0
35
40
45
50
55
60 Microns
Burette
Stokes Law
Temp. Deg. C
20
21
22
23
24
25
26
27
28
29
30

In Figure VI the time of settling for the various sizes of particles as given by the burette are plotted as ordinates, with the temperatures at which these times were measured plotted as abscissae. The solid lines drawn through these points represent, therefore, the variation in the settling times with different temperatures as given by the burette. The dotted lines show the variation which the burette should give if it were to follow Stokes' law (from which the slopes of the dotted lines were calculated). The solid lines were obtained by taking the times of draining to the levels, as obtained by Wagner's method, with a stopwatch. An insulating jacket was placed on the burette to hold the temperature constant while the burette was draining. It will be seen that the burette fails to give a sufficient correction for temperature change. The position of the dotted line does not give the absolute value of the time of settling, it is merely superimposed on the burette value to show the difference in slope.

FIGURE VII: Showing the effect of passing the sample through a No. 100 sieve on the accuracy of the No. 325 sieve determination.

Cement A

Without Preliminary 100 Mesh Sieving % Passing No. 325 sieve		With Preliminary 100 Mesh Sieving % Passing No. 325 sieve	
Run 1	94.7	Run 3	99.0
Run 2	96.3	Run 4	99.0

The data in Figure VII were obtained by making No. 325 sieve determinations using the method given by Wagner in III, 7, the sieve having been calibrated as in III, 6. Four runs were made on cement No. A, two using the cement just as furnished (in a sealed container), and two using the cement which passed through a No. 100 Tyler standard sieve. Since any cement should pass completely through a No. 100 sieve, the only effect of the latter was to break up and eliminate aggregates which had formed in storage. The effect of these aggregates is clearly shown in the accuracy of the results.

FIGURE VIII: Showing the inaccuracy of computing specific surface by the ratio:

$$\frac{S(\text{unknown sample})}{S(\text{known sample})} = \frac{2 - \log I_{60}(\text{unknown sample})}{2 - \log I_{60}(\text{known sample})}$$

Run	Cement tested	S measured	S calculated
9	A	3100	
10	A	3101	3118
13	C	1587	2094
14	C	1600	2072

The run numbers are those of Part III.

The results of the third column were obtained by the regular trubidimeter procedure recommended by Wagner. The fourth column was calculated from the ratio given by Wagner, using the value 3100 of run 9 and the corresponding value of I_{60} in run 9 as the "known" values.

FIG. IX

Determination of I_0

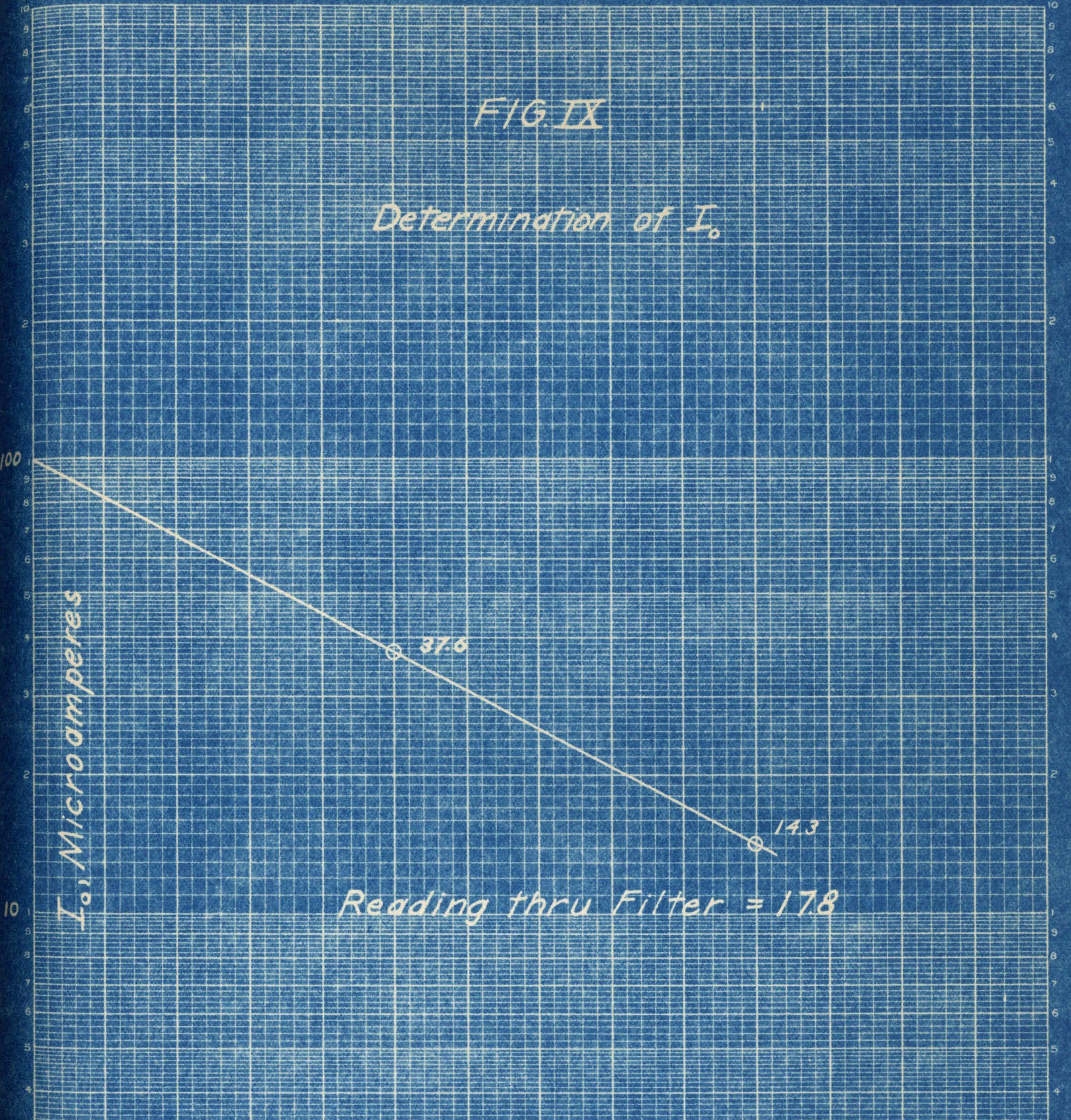


FIGURE IX: Showing the determination of I_o as recommended in III (4). This result is used in Part III.

Determination of No. 325 sieve corrections by method of III, 6

	Run 1	Run 2
Weight of Sample	1.000 gm.	1.000 gm.
Weight of Residue	0.139 gm.	0.139 gm.
Weight retained on Standard Sieve	0.135 gm	0.135 gm.
Correction to be added to throughput	0.4%	0.4%

AN INSTRUMENT FOR THE MEASUREMENT
OF THE
THERMAL CONDUCTIVITY OF WATER,
AND FOR THE
INVESTIGATION OF PHENOMENA IN HEAT TRANSFER
IN THE LIQUID AND AT THE LIQUID-SOLID BOUNDARY

INTRODUCTION

The work on the thermal conductivity of water herein described was begun in 1928 but was not continued beyond the construction stage until 1932 when the present writer was assigned to it as a research project for the degree of Master of Science. The original object of the work was to investigate the temperature coefficient of the thermal conductivity of water and aqueous solutions, in a more careful manner than had heretofore been used. The present conductivity cell was designed for preliminary work and was to be succeeded by a more elaborate one. It became apparent during the construction and installation of the cell that it was not preëminently suited to the measurement of conductivity at other than room temperature, but that the principle of the exploring thermocouple was adaptible to the investigation of the boundary phenonena at the solid-liquid interface. It was decided, therefore, to concentrate more upon the development of the latter phase and to postpone the former until some time when funds would be available to permit the construction of more elaborate equipment. Since this work was begun Bates (Ind.Eng.Chem., 25, 431 (1933)) has developed a conductivity cell which is better suited to the measurement of the temperature coefficient. Bates also employed a series of fixed thermocouples in the liquid space and obtained results similar to those found in the present work. The exploring thermocouple described herein offers a more flexible means of measuring the temperature gradient in the liquid than Bates' fixed couples, so that the results obtained with our apparatus, although supplementary to Bates' work, represent in some respects an improvement on his technique.

PART I

Design, Construction, and Operation of the Thermocouple Potentiometer.

(1) For economic reasons it was decided to build the potentiometer which was to be used in measuring the temperatures and temperature differences in the cell by means of thermocouples. The wiring diagram of the instrument is shown in Figure 1. (in envelope)

(2) The battery circuit consists of a group of variable series resistances for adjustment of the battery current, fixed resistances, and a measuring bridge of nine coils and a slide wire.

The total fixed resistance of the battery circuit is 2635 ohms. The total variable resistance of the battery circuit is 1900 ohms, and is designed to give a minimum variation over the range, 0 - 1900 ohms, of 0.01 ohm. The instrument may therefore be operated by any source having a voltage of 1.8 to 3.1 volts, the battery current being 6.838×10^{-4} amperes. The resistance of each of the nine bridge coils and the slide-wire is 1.4624 ohms. Across the measuring bridge is a shunt controlled by a switch (marked "Range") which changes the total voltage drop across the bridge from 0.01 volts to 0.005 volts or to 0.0005 volts, enabling the instrument to measure from 0.000011 to 0.01 volts by 1×10^{-6} volt steps, or from 0.0000055 to 0.005 volts by 5×10^{-7} volt steps, or from 0.00000055 to 0.0005 volts by 5×10^{-8} volt steps. In Figure 1 two range switches are shown, they are both controlled by the same handle and work together automatically. The shunt resistances and the bridge circuit resistances are so designed that the total current being drawn from the battery is the same for all three settings of the range switch.

The battery current is adjusted by balancing the drop through a portion of the circuit against the known E M F of a Weston calomel cell. The voltage drop across two resistances is measured, one resistance being fixed and another variable with respect to the standard cell circuit. The former resistance is 1486.5 ohms, the latter is 4.4 ohms and is constructed from a potentiometer of 357.6 ohms resistance with a shunt across it of 4.454 ohms. The potentiometer is of the ordinary sliding blade type. This design permits the use of any standard cell having an E M F of from 1.0179 to 1.0189 volts. Since the E M F of the cell is known, the small potentiometer may be set so that the total resistance across which the drop is being measured yields the correct current in the battery circuit.

The actual measuring circuit consists of a series of nine coils and a slidewire placed (mechanically) parallel to the bridge of the battery circuit. Both these sets of coils and slidewires are constructed of No. 18 (B+S) "Advance" wire. This alloy (55% Cu, 45% Ni) is used because it has a low temperature coefficient of resistance and good keeping qualities. The battery circuit bridge is continuous, the nine coils and the slidewire being wound of one piece. The measuring circuit wire is continuous through the coils but is broken between the first coil and the slidewire. The principle of the double bridge circuit is to maintain a constant resistance in the measuring circuit. This is done by means of two sliders which are made of "advance" ribbon and which make contact between the two parallel wires. One slider is on the coil side of the break in the measuring circuit and the other on the slidewire side. Since each set of coils and each unit length of the slidewire has the same resistance as the complementary section of the parallel wire, the setting of the sliders has no effect on the resistance of the measuring circuit, while the voltage impressed on the latter may be varied at will. The thermocouples are placed in series in this measuring circuit, two switches permitting several thermocouples to be used in various combinations. The galvanometer is also in series in this circuit. It is provided with five series resistances, a "short", a critical damping resistance, and a key. The series resistances range from, 0 to 36,000 ohms. The short is connected to a switch which is thrown when the galvanometer is not in use. The critical damping resistance is so connected that it is automatically thrown in when the key is released and out when it is pressed, or it may be permanently thrown in by means of a switch.

A double-throw switch places the galvanometer in the standard cell circuit so that the battery current may be checked at any time without making any other adjustments.

A double-throw switch marked "X" and "ELIM" (for "elimination" of parasitic E M F's) permits the measuring circuit to be checked for parasitic E M F's. When the switch is thrown to "ELIM" the battery current through the bridge is broken and shunted through a resistance, r_1. This has the same resistance as the alternate path through the bridge so that the battery current is not changed and there is no need of readjusting it after using "ELIM". At the same time the thermocouples are cut out of the measuring circuit and a resistance, r_2, equal to them is inserted. The parasitic E M F is then measured by the galvanometer deflection, which may be calibrated, and the correction applied to the reading made in the "X" position. Two assumptions must be made in order to justify this connection: one, that there are no parasitic E M F's in the thermocouple circuit which has been cut out; and the other that no parasitic E M F's have been introduced in the measuring circuit by means of the new connections made. The former assumption is correct if the thermocouple circuit consists of continuous wires with no joints other than the temperature junctions; the latter has been shown to be correct by permitting the instrument to remain untouched over a period of time (24 hours), no switches or contacts being moved, when no deflection of the galvanometer is shown; hence any parasitic E M F is caused by friction in the switches or contacts.

All the switch parts and binding posts, coils and leads in the actual measuring circuit where parasitic E M F's are important are constructed of copper, all flat parts from the same sheet and round parts from the same bar. All joints and wire connections were made by joining the parts mechanically in a firm manner, the touching surfaces being scraped bright, the joints were then soldered to insure the mechanical connections holding. Where the "advance" parts of the circuit join the copper, i.e., the two larger galvanometer series resistances, the battery and measuring bridge resistances, and the ELIM circuit, the junctions were all made in the above fashion and placed in a small Dewar flask, which was then filled with paraffine. In Figure 1 this Dewar is shown as two for convenience. In effect, therefore, the circuit is continuously copper from binding post to binding post.

In order that the calibrated bridge of the battery circuit may be used as a standard, four connections are made to it and lead to binding posts marked "SLIDE WIRE"; "END", "UPPER", "LOWER", "END". These have nothing to do with the use of the instrument as a potentiometer.

The I^2R loss in no part of the instrument is sufficient to cause any change of resistance in any of the circuits.

(3) The instrument was calibrated with a Wolff potentiometer. This was done by connecting the Wolff to the X_1X_2 binding posts, closing the X_1X_2 switches, closing the two double-pole switches marked "X", cutting out all the galvanometer series resistance, closing the galvanometer shunt switch, and pressing the key (see Figure 1). When a current was passed through the battery circuit a voltage was impressed on the X_1X_2 terminals, its magnitude depending on the setting of the two bridge sliders. The upper slider was set at the point "0" on the brass drum and the lower slider was moved to approximately the center of the exposed portion of the wire between the first and second coils, the voltage drop was read to an accuracy of better than one part in ten thousand and a mark "1000" made on the brass ring. The slider was then moved to the exposed portion between the second and third coils and adjusted until the voltage drop was twice the first value and a mark "2000" made. This was continued until the nine coils (9 times the first value) had been calibrated, each with an accuracy as above. The slider was then moved back to the first exposed portion, i.e., between "0" and "1000". The voltage drop across this interval was found to be eleven one-thousandths of the drop from "0" to "1000" so the setting was labelled "11"; it is the smallest value which the instrument will read.

The slide wire was then calibrated by setting the lower slider at the 1000 mark and moving the upper slider, increasing the voltage drop by five one-thousandths of the "0" - "1000" drop at a time and marking the brass drum "5", "10", "15", -- "1050". Each one of these increments was then subdivided into five equal parts by linear measurement. It was found unnecessary to calibrate closer than every five since the wire is so uniform in diameter and composition. Throughout the calibration the voltage drop from "0" - "1000" was used as a standard and the current through the bridge regulated to keep it constant. The accuracy of calibration is such that the error between any two readings is less than one of the unit divisions on the drum. The reading glasses are so set that they correspond in position to the instrument used in marking the lines on the drum.

With the lower slider on "9000" and the upper slider on "1000" the current through the bridge was adjusted until the drop across these resistances was exactly 0.010000 volts, the range switch being on "10". The Wolff potentiometer was then connected to the terminals marked "Std.Cell", the double-pole switch thrown to "Std.Cell", the remainder of the circuit being as above. The dial marked "Std.Cell" was then adjusted to the values 1.0179 - 1.0189 volts as read on the Wolff and these settings marked on it.

The instrument is somewhat specialized in that it was designed particularly for thermocouple work, although it may be used for measureing any E M F not to exceed 0.01 volts. In addition it has a built-in critical damping resistance of 14 ohms for use with a particular galvanometer (Leeds and Northrup, high sensitivity, copper suspension, No. 106065).

(4) To use the potentiometer: Connect a single lead storage cell (preferably new) to the terminals marked "Bat." A freshly charged cell should first be shorted for about ten seconds to bring it down to the flat portion of its discharge curve. Connect a Weston standard calomel cell to the terminals marked "Std.Cell", and set the dial marked "Std.Cell" to the voltage of the cell. Connect the galvanometer to the terminals marked "Galv". Connect the thermocouple leads to the terminals marked

"X_1, X_2, X_3, X_4". The hot copper-constantan junction is positive. X_1 and X_4 are the positive terminals, X_2 and X_3 the negative. Being sure the connections have the proper polarity, throw on the switch marked "Bat", throw the double-pole switch to "Std.Cell" and the other double-pole switch to "X". Set the single-pole switch marked "CDRX" and "Short" so it does not make contact in either position. Set the galvanometer series resistance switch on "36,000". Set the range switch on the desired range. Set all the battery series resitance switches on the "On" position, except the two knobs marked "17" and "0.75". Set the "17" on the "In" position and the 0.75 on the middle of its range. Press the key very lightly (it should only touch for an instant) and note the direction of deflection of the galvanometer. Throw the switch marked "945" to the "Off" position, this throws 945 ohms into the battery circuit; press the key and note the deflection. If it has reversed, throw the switch back to "On". Next try the "475" switch and so on down the line in succession, leaving "On" those switches which reverse the galvanometer. Next turn the "17" knob until the galvanometer shows no deflection. Move the Series Galv.Resis. switch to "3600" and continue to adjust the "17" dial to no deflection. Move the Series Galv.Resis. switch to "360" and adjust the "0.75" dial to zero deflection. Repeat this on the "0" position and the battery current is standardized.

To read the thermocouple E M F: set the series galvanometer resistance on "36,000" and throw the double-pole switch from "Std.Cell" to "X". Set the drum slider on "0" and the lower slider on "11". The sliders must always be lifted from the wires when they are moved, otherwise the wires will become worn and parasitic E M F's will be generated by friction. The brass knob on the right side of the instrument sets the lower slider. The brass knob on the drum handle lifts the drum slider when turned. The lower slider is lifted by pulling it away by hand. When the sliders are set press the key lightly and note the direction of deflection. Set the lower slider on the "1000" position and note the direction of deflection. Continue to do this until the galvanometer reverses. Then set the lower slider on the next lower setting and move the drum slider until no deflection occurs. Move the series galvanometer resistance switch to "3600" and move the drum slider to no deflection. Continue until the galvanometer shows no deflection on the "0" setting of the series galvanometer resistance. The unknown E M F is then balanced and is read by adding the lower slider reading to the upper slider reading. The result is in microvolts (or millivolts x 10^{-3}) and must be multiplied by a factor depending on the setting of the "Range" switch. If the setting is "10" multiply by 1, if "5" multiply by 0.5, if "0.5" multiply by 0.05. In addition to this value the ELIM correction must be added or subtracted. This connection is found by throwing the double-pole switch from "X" to "ELIM" after the reading has been made as above. Note the zero position of the galvanometer light when the key is pressed half-way (enough to release the CDRX but not enough to make contact below). Then press the key until it touches the lower contact and note the number of units of deflection. To calibrate these units set the range switch on "o.5", connect the binding posts X_1 and X_2 with a piece of <u>copper</u> wire having a resistance of 80.0 ohms, throw the H and C switches to "X_1" and "X_2", set the lower slider on "11" and the upper slider on "0". Note the zero point as above and press the key and record the deflection. Then set the upper slider on "100" and record the deflection. After each of these two readings throw the switch to "ELIM" and record these two values. If the latter are not the same repeat the test until they are; it is best to make this calibration after the instrument has stood idle for some hours. Subtract the "0" deflection from the "100" deflection, divide the difference into 100 and multiply by 1, 0.1, or 0.05, depending on whether this factor is to be used on the 0.5, 5, or 10 range, respectively. This elim factor is then multiplied by the deflection obtained on the ELIM setting. To find whether to add or subtract this correction: note the direction of deflection obtained when adding a small E M F to the "X" reading by turning the drum counter-clockwise. If the ELIM deflection is in the same direction add it and vice versa.

If the method of galvanometer deflections is to be used to measure E M F differences on "X", the ELIM system may not be used unless the resistance of the thermocouple from X_1 to X_2 posts is equal to r_2, 80.0 ohms.

The battery current may be checked at any time merely by throwing the double-pole switch to "Std.Cell." The 0.75 knob is usually sufficient to make any necessary adjustment.

When changing from one range setting to another it is necessary to readjust the battery current, since it was not possible in the construction of the instrument to exactly balance the resistance to give the same total battery circuit resistance and keep the range factors as they are. The adjustments to be made are small.

Should the slidewire become tarnished and the slider fail to make contact, rub it gently with the finest grade of "crocus" paper.

The switch blades and contacts should be kept lubricated with a dry paste made of graphite and cup-grease.

There is no need to turn off the battery current since it is so small. It may be left running continuausly until the battery is dischafged.

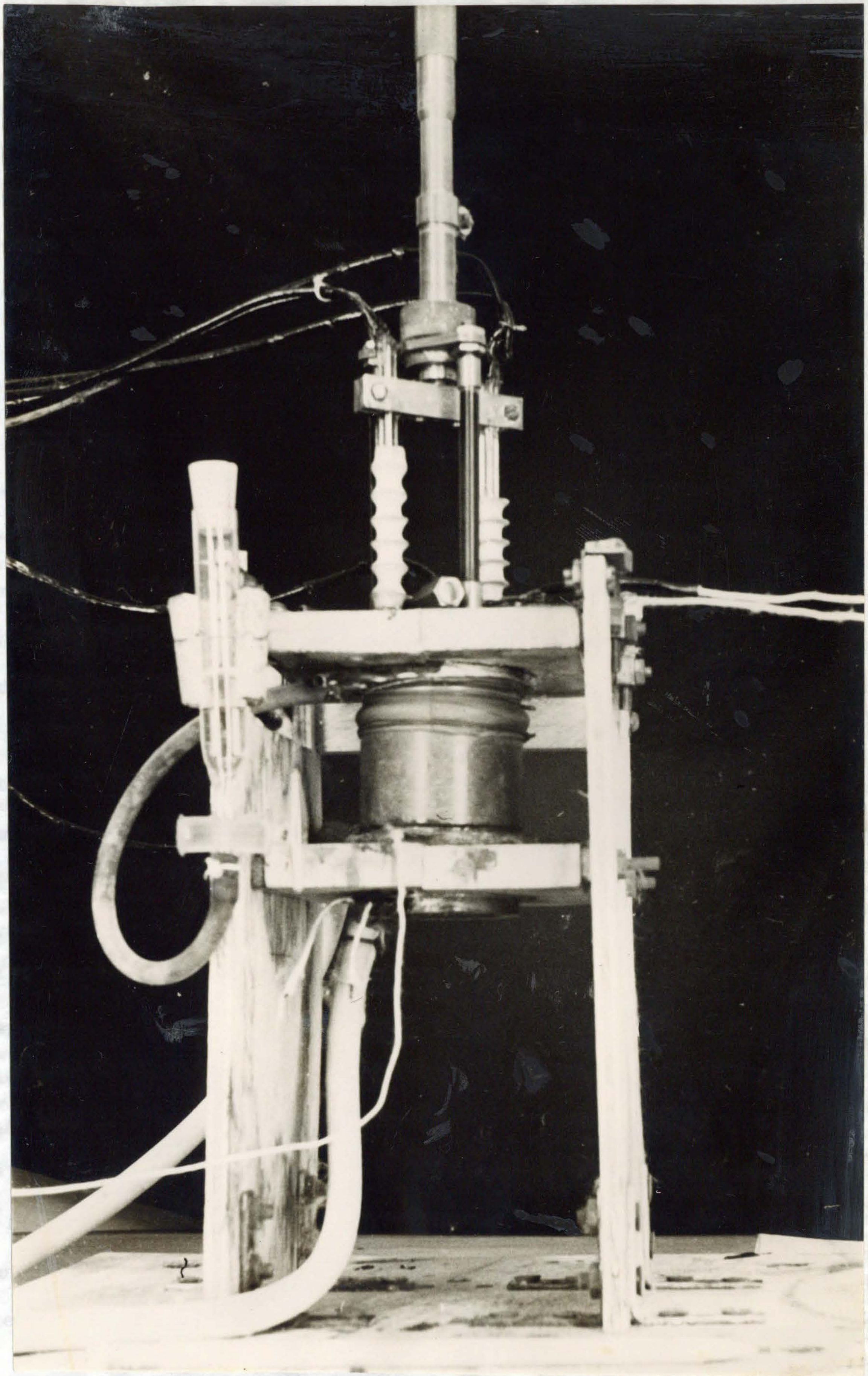
Keep the galvanometer shorted when not in use.

PART II

Design and Construction of the Thermal Conductivity Cell and Auxiliary Apparatus

(1) The conductivity cell (Figures 2 and 3) (Figure 2 is in envelope at end) is constructed from two cylindrical copper blocks two inches in diameter. These blocks are mounted coaxially, one above the other, by means of soft wood clamps which are in turn supported by a wooden framework. The lower block and mounting are rigidly fixed in position; the upper block and clamp are suspended from the wooden framework by means of four bolts which enable the upper block to be moved up and down, thereby varying the distance between the two blocks. The lower face of the upper block and the upper face of the lower block are turned flat and polished. Connecting the two blocks is a hollow bakelite cylinder 2.008 inches inside diameter and 2.079 inches outside diameter. This is fastened to the lower block by means of Westinghouse amber varnish which is baked to give a hard water-tight seal. The bakelite sleeve is attached to the upper block by means of a rubber sleeve which is held to the bakelite and the copper by twisted wires. The space inside the bakelite and between the two blocks is filled with distilled water, previously boiled to remove air. Both the upper and lower blocks have heating units of No. 18 constantan wire of approximately one ohm resistance. These units are located in wells bored in the blocks and are held in place with plaster of Paris. Each block has four turns of 1/8 inch i.d. brass tubing wound around the outside and held to the block by solder. Thus heat may be introduced to either block electrically and removed from the other by pumping water through the coil. In the center of each of the blocks, 1/8 inch from the polished face, is

Fig. 3



located a copper-"advance" thermocouple of No. 36 wire. These thermocouples were separately calibrated and are insulated from the blocks.

Attached to the upper face of the upper block is a tripod constructed of bakelite rods and holding a machinist's micrometer head. This micrometer operates a push rod, held against it with a spring, on which is mounted a brass carriage which holds two glass capillary tubes. The capillary tubes fit loosely into two holes running vertically through the upper block and opening into the space between the two blocks. Through the capillaries and fitting tightly in them are run two lengths of No. 24 (0.02 inch diameter) stainless steel spring wire. These wires extend about 1 inch beyond the lower end of the glass and are pointed on their lower ends. This whole mounting allows the steel wires to be pushed up or down between the two blocks when the micrometer head is turned. The needles are adjusted and trimmed with a fine file until their ends are the same distance from the lower surface of the upper block. This is accomplished by drawing the needles up into the block and then turning them down until they touch a flat metal plate held on the surface of the block; the contact is registered by a small lamp in series with the block, a dry cell, and the needle, and is accurate to 0.0002 inch. The micrometer reading for this zero point of the needles is recorded. The lower block is mounted and leveled with a spirit level. The upper block is adjusted and leveled with a spirit level and the micrometer head is turned until the needles both touch the lower face at the same time. This is registered with the simple electric circuit shown in Figure 4 (a): the single-throw switch is closed and the double-throw switch is on "a", the micrometer is turned until the light flashes, and by opening the single-throw switch the needle touching the lower face may be determined. The suspension bolts

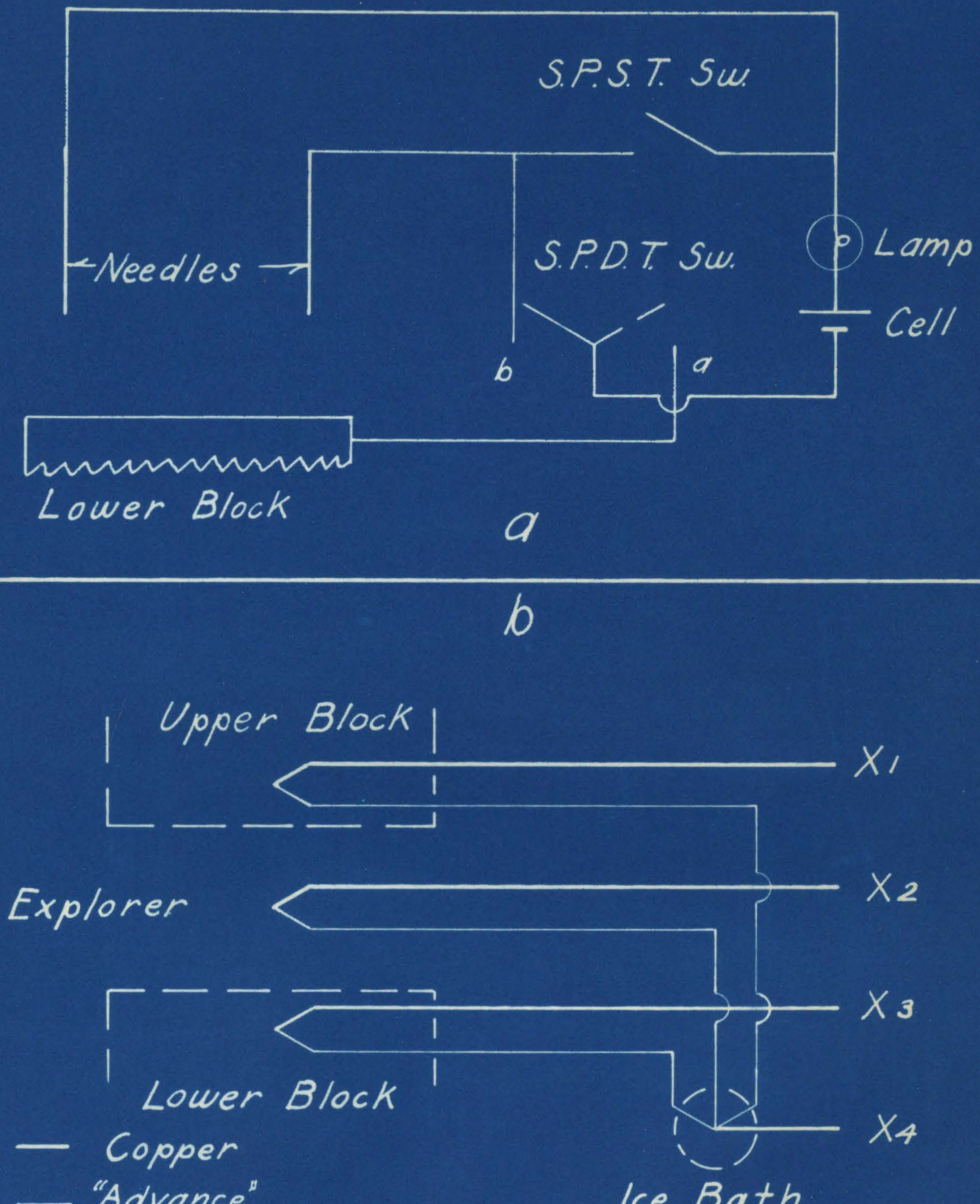


FIG. IV

Circuits for Needles and Thermocouples

are then adjusted to raise this needle and the test is continued until both needles touch at once. This is indicated by the light flashing when the single-throw switch is open and the double-throw switch is on "b". The micrometer reading in this position subtracted from the above "zero" reading gives the distance between the blocks to 0.0005 inch. In addition the micrometer enables the faces to be set parallel with a greater accuracy than the spirit level, and it tells the position of the exploring thermocouple (see below) with respect to the block faces.

Fastened across between the two needles is a thermocouple constructed of No. 40 (0.029 inches diameter) "advance" and copper wire. The junction of the two metals is butt ended and the two are joined with silver solder. The thermocouple is straight for 1 1/4 inches and the joint does not exceed the wire in diameter.

Much difficulty was experienced in constructing these couples before a suitable method was devised so a brief description of it is given below:

The stock wires are first stretched to straighten them and a three-inch section of each cut from the stock wire with a sharp razor blade on a smooth steel surface. Each section is inserted in a separate glass capillary tube of slightly larger bore, with about one-quarter inch extending on the end where the joint is to be made. A small quantity of silver solder is fused with a little borax in a porcelain crucible. The tips of the wires are then dipped into this and "tinned." The borax will attack the wire so the dipping must be rapid. A slight amount of extra solder should be picked up on one tip in order to make a strong joint. The capillaries with the wires in them are then inserted in holes drilled to fit them in two brass blocks. The blocks are located one inch apart and the holes in them are concentric. Set-screws in the blocks enable the glass tubes to be clamped in position. A flat coil of No. 28 chromel wire about 1/4 inch in diameter is mounted midway between the two blocks and at such a height that the two ends of the small wires meet just above it without touching it. Covering the space between the two blocks is a semicircular piece of 2-inch or larger glass tubing which is sealed to exclude air. Entrance is made through one of the blocks for the wires to the chromel coil and for a tube connected to a hydrogen tank. The hydrogen is allowed to pass for ten minutes, meanwhile the two ends to be soldered are adjusted to touch concentrically above the chromel coil. A 40-power binocular microscope is necessary to align the wires properly. Turn the hydrogen stream to a very small flow and throw the switch connecting the chromel coil with a 110-volt line, a 250-watt load being in series with it. A screen should be put over the glass cover of the apparatus to prevent damage in case of explosion. The chromel coil will melt the solder and if the two ends are in contact it will flow and form the joint. This should be watched with the microscope and the heater turned off when done. The thermocouple is then removed by pulling it out from one end. Any borax left in it may be dissolved off with dilute HCl. The resulting joint is stronger than the copper wire. In the case of the present work it was necessary to use silver solder because lead solder was too weak; the latter may be used, however, in this method of manufacture.

The resulting thermocouple is mounted under tension between the two steel needles as above. It is held in place on two bakelite insulators which are 3/32 inch cubes and have holes drilled in them for the needles. At right angles to the needle a hole is drilled in one of the insulators, and in the other a groove is milled with its vertical axis parallel to the needle. The thermocouple is mounted by inserting it through the hole in the one insulator and fastening it to this insulator with DeKhotinsky wax. The other end is then grasped and the thermocouple pulled against the spring of the needle and laid in the groove of the other insulator, where it is fastened with the wax. The micrometer head is then unscrewed and the thermocouple moved up to the face of the upper block. By using a light it is possible to watch the thermocouple and its shadow on the face of the block approach each other. The thermocouple may be adjusted on the groove side by melting the wax with a piece of heavy copper wire stuck in a cork. Adjustment is made in this fashion until the thermocouple is parallel to the face of the block; i.e., when the wire and its shadow meet simultaneously throughout its length. The micrometer reading in this position is recorded. The reading at any point in the liquid subtracted from this value gives the distance of the explorer from the upper block face.

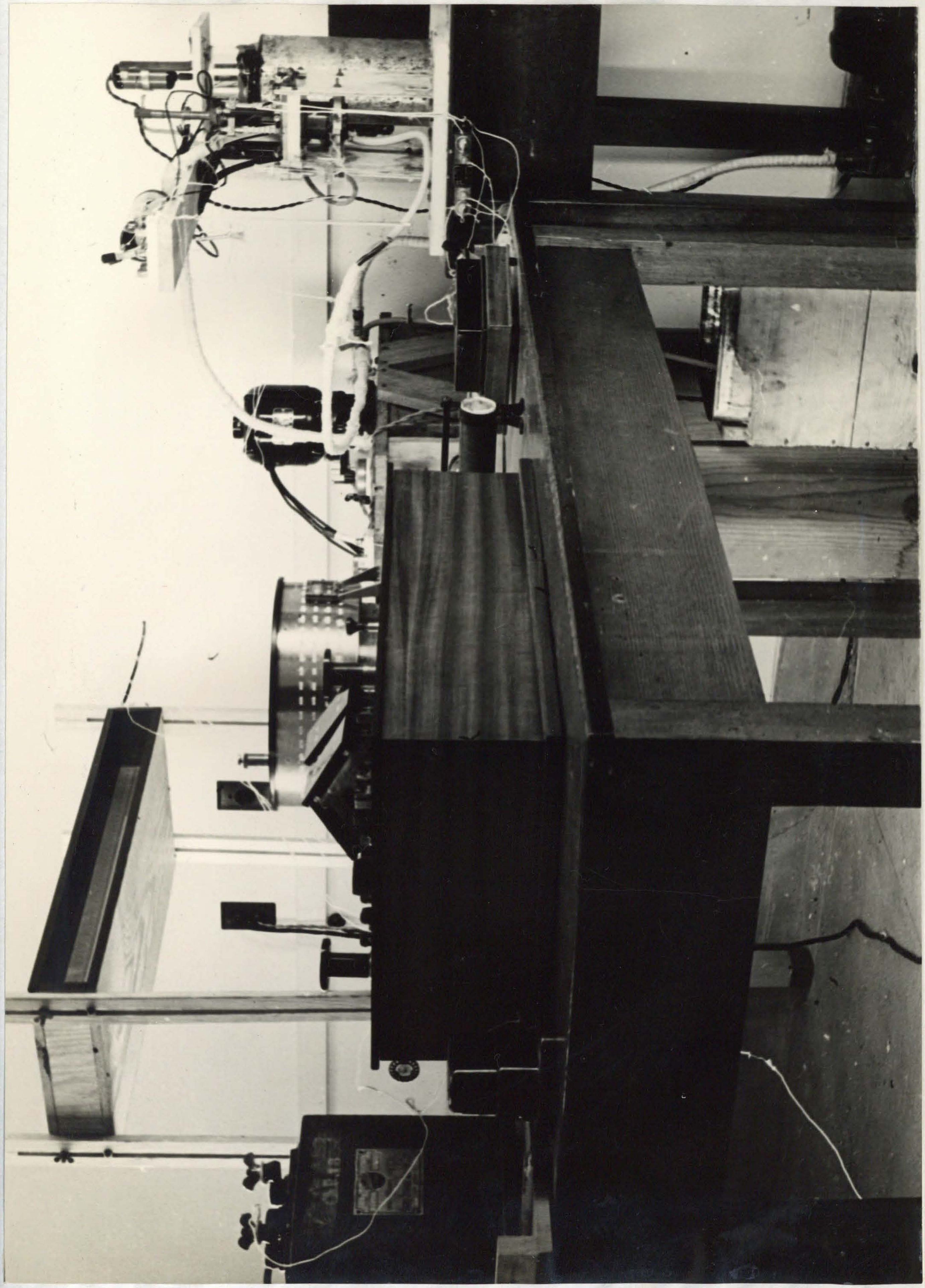
The thermocouple ends are soldered with soft solder to wires varnished to the outside of the glass rods and leading to the cold junction. Wire from the same spool as that in the thermocouple itself is used.

To compensate for the piston action of the glass rods, a small reservoir constructed of the lower end of a burette is connected to the liquid space of the cell. This reservoir is separately attached to the wooden frame and is connected with rubber tubing.

To minimize evaporation from the cell, collapsible rubber sleeves are attached to the upper block and fit snugly about the glass rods. They are built on the sylphon principle, but are not attached to the rods at the top because the exposed portion of the glass rods is too short, for the amount of vertical movement involved, to permit the rubber to take all the compression. In a future design this should be taken into account and the part of the glass rods projecting above the upper block made about an inch longer so that a tight rubber seal of this design can be used.

(2) The parts are assembled as follows: The lower block with the bakelite sleeve varnished in place is mounted in the wooden framework, leveled with a small spirit level and clamped in place. The rubber sleeve is wired on to the bakelite sleeve and folded down over the latter. The glass reservoir is attached to the upper block with five inches of rubber tubing. The rubber sylphon sleeves are put on the glass capillary rods and wired on to the copper sleeves designed for them. The needles are adjusted to proper length as above, and the thermocouple is installed as above, being placed as near the ends of the needles as possible. The upper block assembly is then dipped into a 400-ml. beaker filled with distilled water, the glass reservoir is filled with distilled water and the stopcock on it opened and the water allowed to run out through the block until the reservoir is almost empty, when the stopcock is closed while the water is flowing. This procedure removes air from the passages in the block. The exploring thermocouple must always be flush against the block surface when liquid comes in contact with it, otherwise it will be bent by surface tension. The bakelite chamber is next filled to the brim with distilled water and the upper block lowered into it. The lowering

Fig 5



must be done at an angle so that no air will be entrapped on the face of the lower block. After the upper block has been secured in place, the glass reservoir is mounted on the wooden frame-work and filled. The stopcock is opened and water allowed to overflow between the upper block and the bakelite. The rubber sleeve is brought up and wired onto the upper block while the water is flowing, to prevent air entrapment. Once the upper block face is in contact with the water, the upper block must not be raised or air will be drawn in. When the rubber sleeve has been fastened tightly, the upper block is leveled by means of a spirit level and the needles, as above. The reservoir is adjusted so the water level is about ½ inch below the upper block's upper surface when the thermocouple is at "zero"; this prevents possible leakage from the rubber sylphon connections. To test for other leaks, the stopcock on the reservoir is closed and the apparatus left overnight; the water level will fall when it is reopened if there is leakage. The heating coil and cooling water connections are next made. The whole assembly is then enclosed in four wooden walls one foot square and 14 inches high. The box thus formed is filled with kapok until only the scale of the micrometer heat is exposed. Figure 5 shows the complete assembly before being packed in kapok.

Figure IV (b) shows the thermocouple circuits used.

(3) The auxiliary equipment consists of a thermostat to provide cooling water; a precision voltmeter and ammeter to read the heat input to the cell; an Edison battery to supply the latter, with a rheostat to control it; and a Dewar flask with a small motor-driven stirrer for the cold junction of the thermocouple circuit.

The cooling-water thermostat is made of a metal water-tank enclosed in two inches of cork insulation. In the tank are a stirrer, a 60-Watt light bulb, a small centrifugal pump, a coil of copper tubing and a glass mercury regulator. The pump circulates the water from the tank through the cooling coil mounted on the thermal conductivity cell block. The stirrer and pump are driven by a 1/8 H P motor mounted on top of the thermostat. The water after passing through the coil on the block is returned to the tank. The heat it brings back is removed from the tank by a flow of ice-water through the copper coil in the tank. This ice-water comes from a five-gallon can and is pumped by means of a small gear pump and motor to operate it. This latter unit is separate from the thermostat. The mercury regulator operates a relay which controls a 110-volt circuit in which the 60-watt bulb is connected. A pilot light on top of the thermostat shows when the bulb is on. The instrument is so adjusted that the ice-water removes slightly more heat than the water in the tank receives from the heat cell and the surroundings, when the 60-watt bulb will operate intermittently to make up the difference. The temperature of the heat cell block varies only 0.01°C about its mean as a result of this intermittent operation.

PART III

Experimental Procedure and Results

After the cell was set up, connected, and packed in insulation, the ice-water pump was started and the thermostat motor started. The temperature of the thermostat water was set at the desired value by means of the mercury regulator, the distance between the block faces was measured, and the input to the hot block of the cell adjusted so that the temperature of the block was approximately room temperature. It was found that at least four hours were required for the apparatus to reach equilibrium. This was ascertained by keeping the heat input constant and measuring the temperature of the hot block. This temperature required at least four hours to become constant, and when it had remained so for 1/2 hour it was assumed that equilibrium was reached. The cold block temperature was automatically held constant throughout the run by the thermostat. The temperatures of the hot and cold blocks were then read. The temperature difference between the upper block and the exploring thermocouple was read for successive positions of the latter. Beginning with the explorer touching the upper surface, readings were taken at increments of 0.001 inch out to 0.010 inch from the surface, after which they were taken at 0.050 inch intervals until the space between the two blocks was covered. The final reading was of necessity about 0.10 inch from the lower face because the needles project that far beyond the exploring thermocouple and prevent the latter being moved any closer. The upper and lower block temperatures were checked at the middle of the run and at the end.

Preliminary measurements indicated that the amount of heat carried by the steel needles did not affect the readings. This is further borne out by calculations from the known conductivity of steel and water, which show that the error from this source is 0.2% in the conductivity of the water when the needles are carrying heat; this error is considerably less than others found in the apparatus.

The first set of measurements was made passing the heat upward through the water. No data could be obtained because the effect of the eddy currents was so great as to make reading the lower block temperature and the explorer temperature impossible. It was noted that the eddy currents affected the explorer even when the latter was in contact with the upper block, indicating that the thermocouple read the temperature of the liquid about it rather than that of the face with which it was in contact. This is, of course, explainable on the basis of the fact that the area of contact between the round wire and the flat surface is very small. It is interesting to note that the eddy currents caused continuous violent fluctuations of temperature down to at least 0.0029 inch from the block surface; this is difficult of explanation on the basis of the classical film theory of heat conduction in moving fluids.

The connections were next changed so that the direction of flow of heat was from top to bottom, and no further evidence of eddy currents was noted. This checks the observations of Bates (loc.cit.). Five runs were made using the exploring thermocouple, as above. When the sixth run was started it was discovered that one of the glass rods had cracked from internal stress. The apparatus was then rebuilt and a sixth run taken, the results of which checked the first five; so it was concluded that the break in the glass occurred after the fifth run was completed. At some time <u>after</u> the sixth run was completed the new exploring thermocouple parted under its tension; and, since two weeks are required to install another, it was decided to abandon the measurements with the exploring thermocouple and devote the remaining time to the measurement of conductivities. The results of these six runs are plotted in Figures VI and VII (Figure VI is in envelope at end); the abscissae are the distance in inches from the surface of the upper block to the top of the exploring thermocouple wire, the ordinates are the temperature, °C, as read with the explorer at the position given by the abscissa. Figure VI shows the readings obtained at the 0.05 inch increments. Figure VII shows on a larger scale the readings obtained at the 0.001 inch increments and constitutes an enlargement of the upper left-hand ends of the curves of Figure VI. Run 1 is not shown on Figure VI because it was found that the upper block temperature had changed during the part of the run which would have been plotted in Figure VI. Run 2 is not shown on Figure VII for the same reason. Run 3 is only partially complete on Figure VI for the same reason. The extreme left and right end points on Figure VI are extrapolated. Table I shows a compilation of the temperature differences between these extrapolated values and the temperature of the face of the block as measured by the thermocouple set in the block and as measured (for the upper block) by the exploring thermocouple in contact with the block face.

It will be seen in Table I that the temperature of the liquid in contact with the copper face is different from the temperature of the face itself. Further, that because of the curvature of the first portion of the plot as shown in Figure VII the temperature difference for the extrapolated values is less than for the values

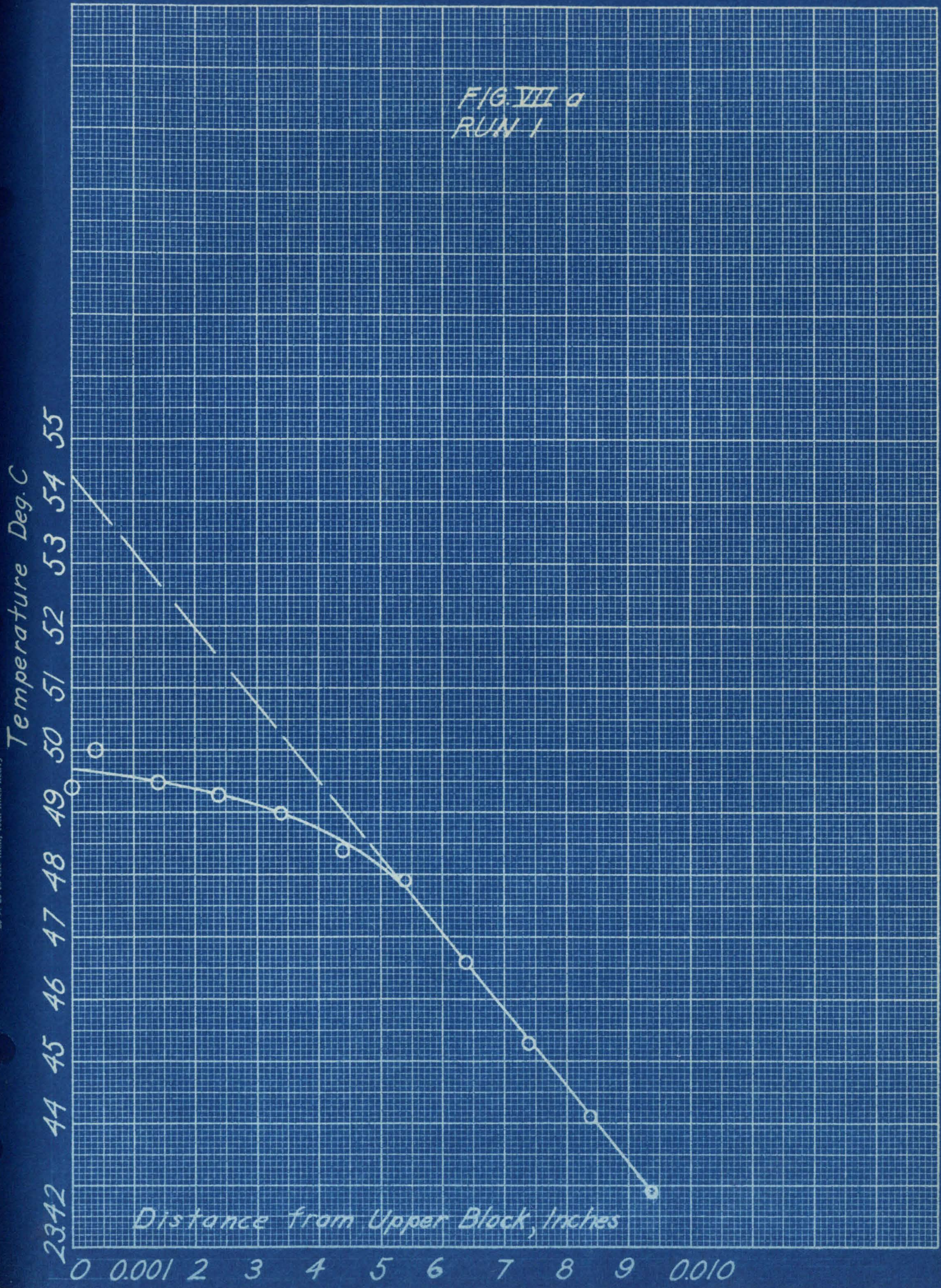
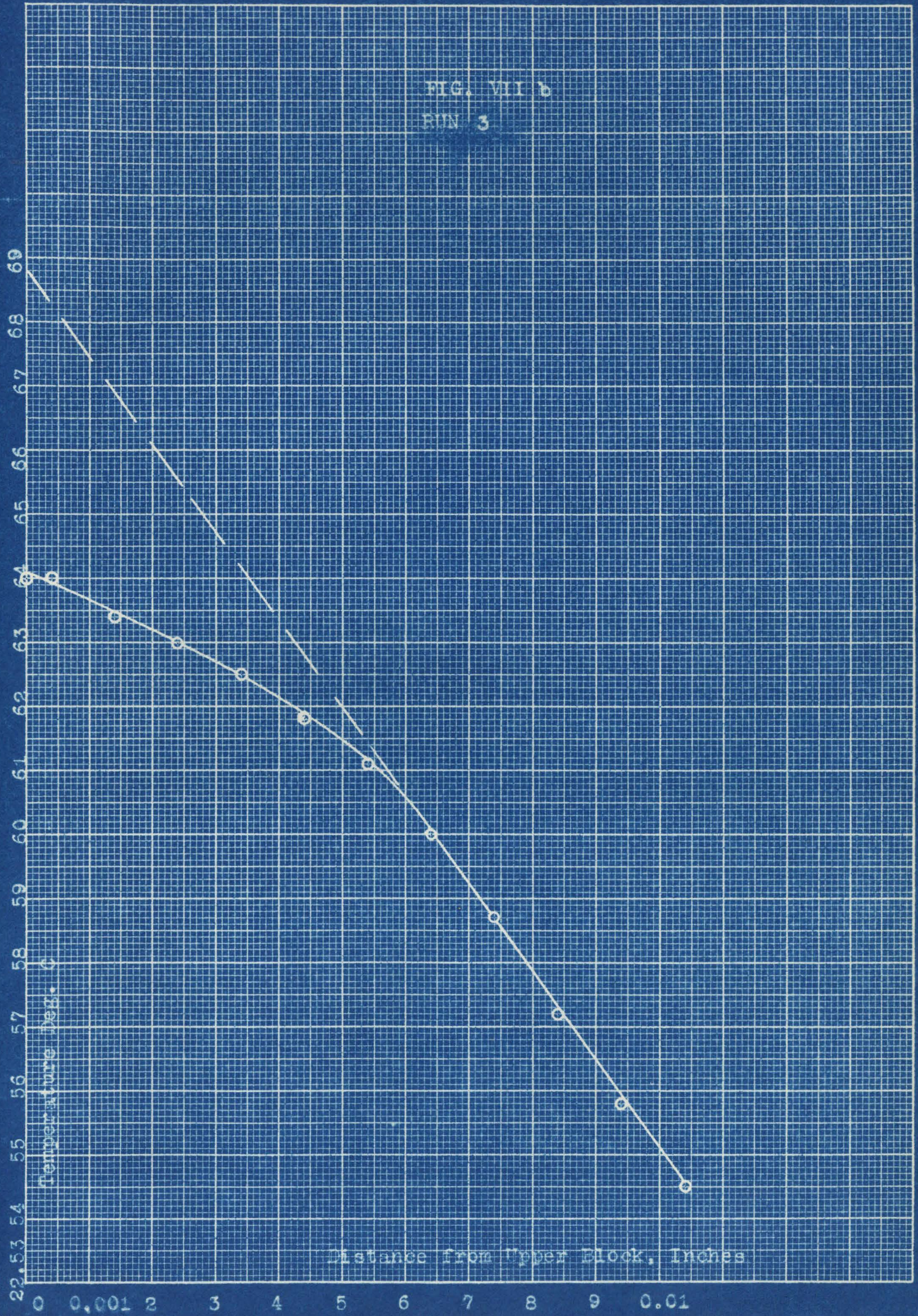


FIG. VII a
RUN 1

FIG. VII b

RUN 3



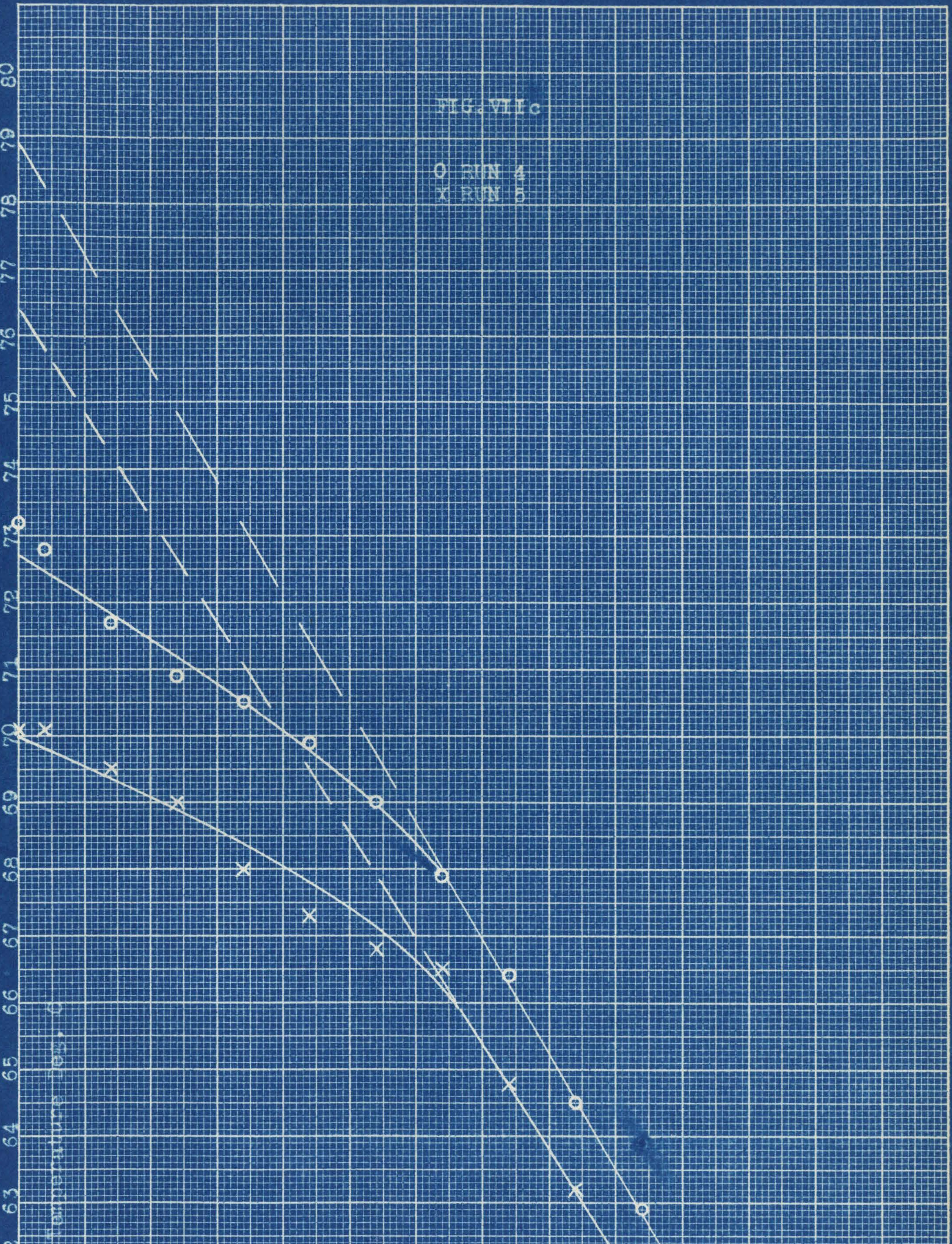


FIG. VIIc

O RUN 4
X RUN 5
Temperature Deg. C
80
79
78
77
76
75
74
73
72
71
70
69
68
67
66
65
64
63
62
61
22.6
Distance from Upper Block, Inches
0
0.001
2
3
4
5
6
7
8
9
0.010

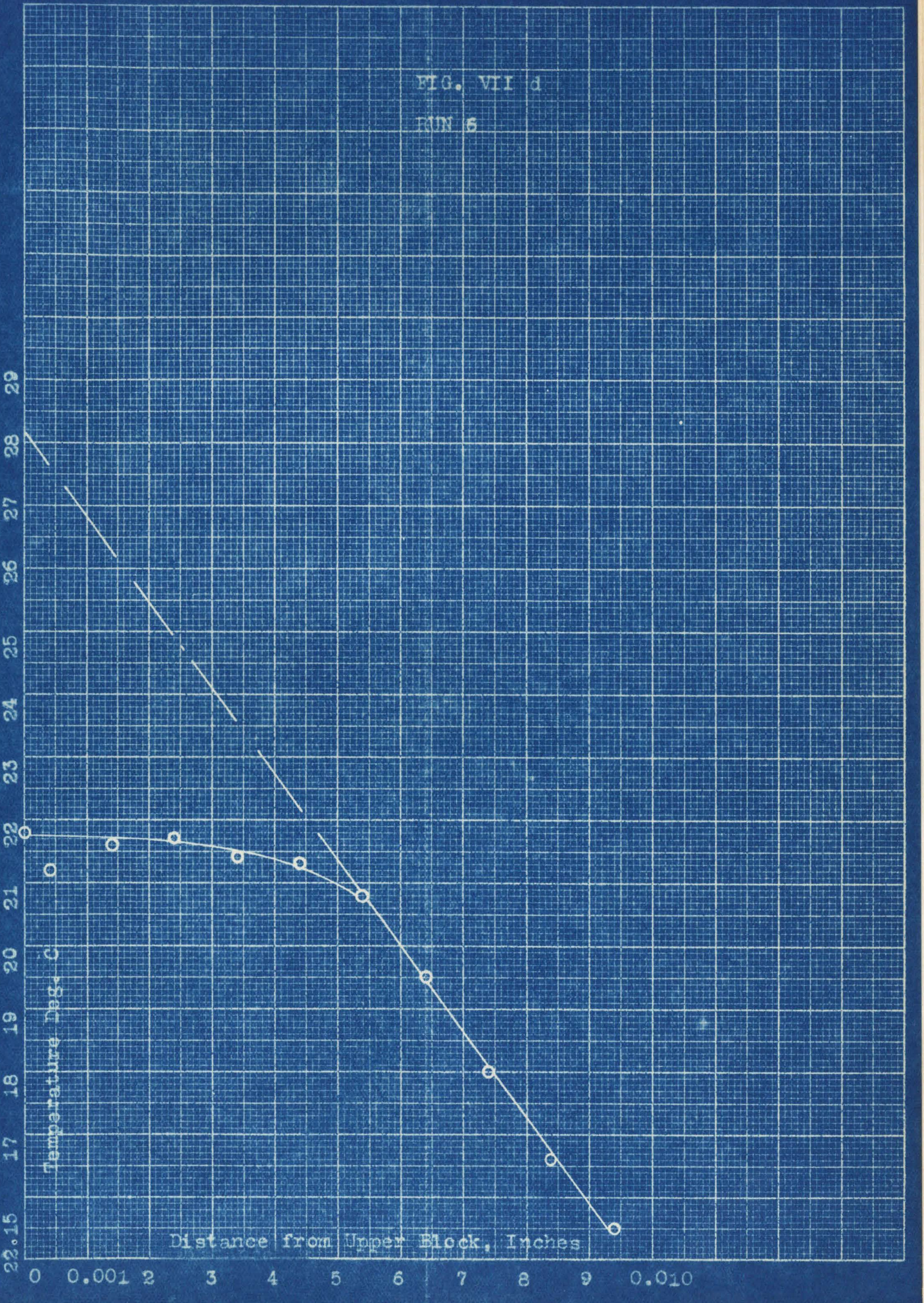


FIG. VII d

RUN 6

TABLE I

Run	Temp. of Upper Block read by thermocouple in block T_1	Temp. of Upper Block read by explorer T_2	Temp. of Upper Block by extrapolation on Figs VI and/or VII T_3	$T_1 - T_2$	$T_1 - T_3$	$T_3 - T_2$
1	23.79	23.49	23.57	0.30	0.22	0.08
2	23.08		22.75		0.33	
3	22.92	22.64	22.69	0.28	0.23	0.05
4	23.03	22.73	22.79	0.30	0.24	0.06
5	23.04	22.70	22.76	0.34	0.28	0.06
6	22.43	22.22	22.28	0.21	0.15	0.06

Run	Temp. of Lower Block read by thermocouple in block T_4	Temp. of Lower Block by extrapolation on Figure VI T_5	$T_5 - T_4$	$T_1 - T_4$
1				
2	12.32	12.65	0.33	10.76
3				
4	12.31	12.64	0.33	10.72
5	12.31	12.64	0.33	10.73
6	14.31	14.53	0.22	8.12

measured with the explorer. The phenomenon of the extrapolated curve not meeting the surface at the temperature of the surface itself was first discovered by Bates (loc.cit.) and was explained by him as being caused by a resistance to heat flow at the surface greater than the resistance of the water itself. Bates, as pointed out previously, did not have the advantage of a movable thermocouple in the liquid and so did not come closer to the surface than 0.2 inch, hence he did not obtain the curved portion within 0.006 inch of the surface, as shown in Figure VII. This curved portion has a possible explanation if it is assumed that there is a resistance to the passage of heat over a distance of molecular magnitude from the copper surface. In this case there would be a considerable temperature difference between the copper and the first layers of water molecules, and any water molecule striking the copper surface would acquire a greater kinetic energy than its neighbors. This would create a "microturbulence" in the surface region which would hold this region at a higher average temperature than it would have in the steady state existing in the body of the liquid. Hence instead of a straight-line fall in temperature from the surface, the first part is distorted and the beginning of the straight line pushed away, in effect, from the surface.

Although the exploring thermocouple could not be taken close enough to the lower face to determine the shape of the temperature-distance curve there, it is reasonable to assume that a resistance to heat flow exists at the lower face similar to that shown at the upper face. So, in order to calculate the ratio of the sum of the two surface drops to the total temperature drop, it was decided to add the same correction for curvature, $(T_3 - T_2)$ in Table I, to the lower drop,

$T_5 - T_4$. This has been done and the ratio calculated in Table II

TABLE II

Run	$\frac{(T_1 - T_2) + (T_3 - T_2) + (T_5 - T_4)}{(T_1 - T_4)}$
2	0.073
4	0.064
5	0.068
6	0.060

It will be seen in Table II that the temperature drop at the two surfaces is about $6\frac{1}{2}\%$ of the total drop from metal to metal; or in the case where heat is flowing from only one surface to the liquid, as in a boiler tube, the drop then becomes about 3% of the overall. Bates also found that the two (extrapolated) surface drops amounted to $6\frac{1}{2}\%$ of the overall difference.

After the sixth run, twelve more were made to measure the specific conductivity of the whole heat path from metal to metal. These measurements were made in exactly the same manner as the first six runs, omitting the measurements with the exploring thermocouple. Four measurements were made at each of three distances, in addition to the first six runs, between the two copper surfaces. The specific conductivity was calculated for the 18 runs from the heat transfer equation: $\frac{H}{t} = k\,A\,\frac{\Delta T}{\ell}$ or $k = \frac{H}{t}\,\frac{\ell}{A\Delta T}$, where ℓ is the distance between the two surfaces as measured with the needles, A is the cross-sectional area of the water in the bakelite sleeve, ΔT is the temperature difference of the two thermocouples buried in the upper and lower blocks, and $\frac{H}{t}$ is the heat passing through the water per unit time. This last is calculated from the readings of the voltmeter and ammeter for the input to the heating coil and is corrected for the loss in the leads from the voltmeter to the coil in the block; and for the quantity of heat passing through the bakelite, from the known conductivity of the latter by means of the above equation. In addition the specific conductivity has been calculated for runs 2-6 from the differential form of the equation: $k = \frac{dH}{dt} \frac{1}{A} \frac{d\ell}{dT}$, the value of $\frac{d\ell}{dT}$ being read from the slope of the curves in Figure VI. All these results are listed in Table III.

TABLE III

Run	Distance between blocks cm.	k_{H_2O} at 19°C from overall measurements	Weighted	k_{H_2O} at 19°C from slopes of Figure VI
1	2.06	0.0508	1	
2	2.06	0.0488	1	0.0507
3	2.06	0.0493	2	0.0509
4	2.06	0.0490	3	0.0513
5	2.06	0.0485	3	0.0501
6	1.90	0.0495	3	0.0522
7	1.63	0.0500	3	
8	1.63	0.0483	3	
9	1.63	0.0503	3	
10	1.63	0.0493	3	
11	0.856	0.0465	2	
12	0.856	0.0465	2	
13	0.856	0.0470	3	
14	0.856	0.0470	3	
15	1.24	0.0478	3	
16	1.24	0.0478	3	
17	1.24	0.0475	3	
18	1.24	0.0485	1	

Weighted average: 0.0484

Value given by I.C.T. for H_2O at 19°C; 0.0378

The units of k are: Watts per cm. per deg. C.

In Table III it will be seen that the conductivities calculated from the slope of the curves in Figure VI are from 3 to 4% greater than those calculated from overall values. This fact was pointed out by Bates, who found a difference of about 7% in the conductivities as calculated by these two methods. Such a difference is to be expected under the theory of a resistance to heat flow peculiar to the liquid-solid interface. Table III also shows that the specific conductivity as calculated from the results with this apparatus is considerably greater than that determined by previous experimenters. This illustrates the shortcomings of this design as a conductivity measuring device. It was assumed in the calculations that no heat was lost to the outside from the upper block because the latter was operated within ± 1/2 degree C of room temperature and insulated with five inches of kapok. It was also assumed that a negligible amount of heat passed from the upper to the lower block through the kapok insulation and through the wooden mounting. The latter assumption, in particular, is open to question. Any correction for this would bring the results closer to the work of others.

CONCLUSIONS

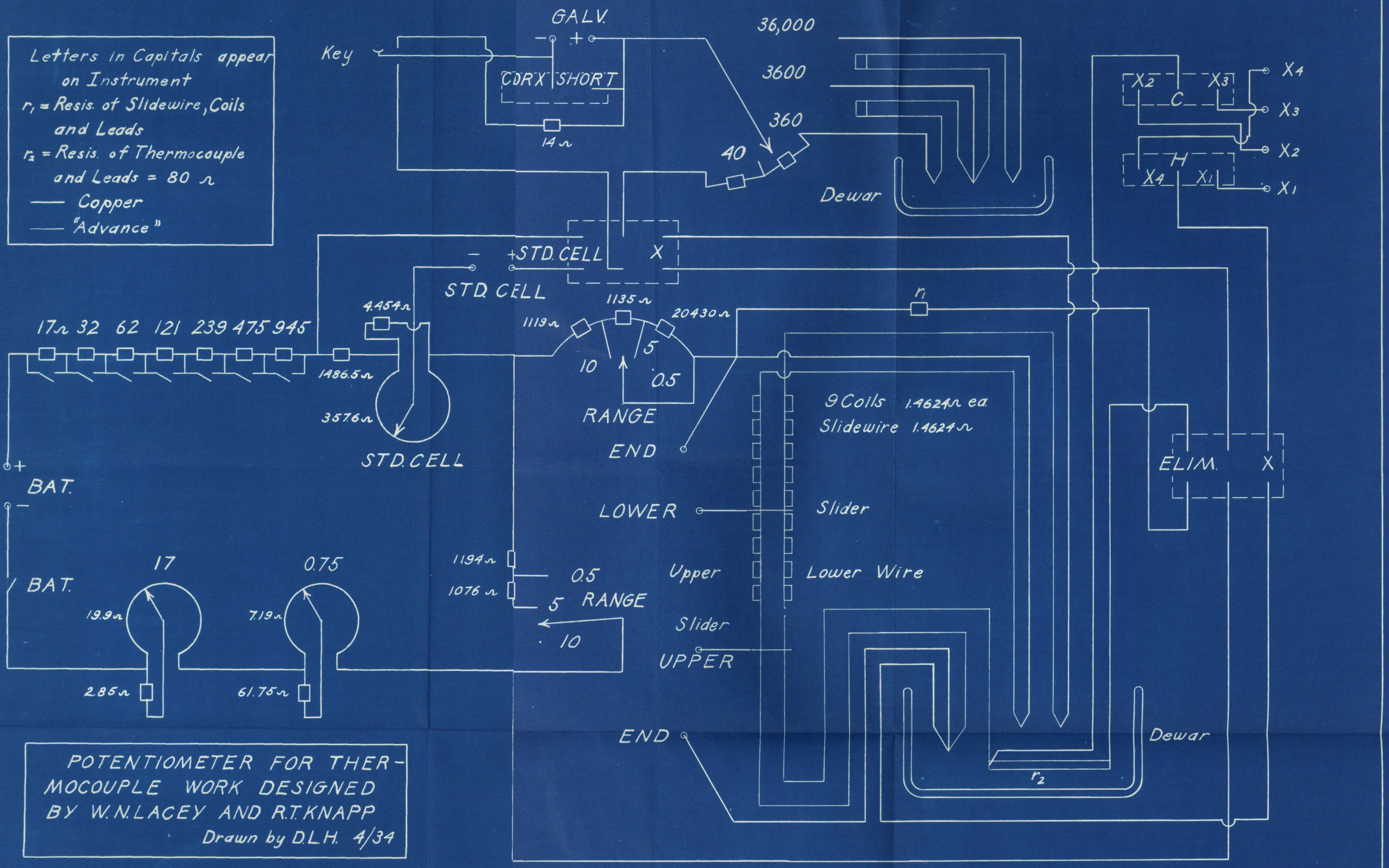
The foregoing design of thermal conductivity apparatus has shown that a sufficiently small exploring thermocouple may be constructed to show the phenomena at liquid-solid boundaries. This device may well be adapted to work on boundary temperature and boundary velocity measurements, a survey of the literature having indicated that it is perhaps the most sensitive device yet constructed which will serve this purpose.

The results of the work constitute a rough check on the work of Bates, Ind.Eng.Chem., 25, 431 (1933).

It is recommended that this design be used in conjunction with Bates' adiabatic jacket on his conductivity cell, if it is desired to continue study of the interfacial resistance to heat flow; since in its present form the apparatus is too inaccurate for careful thermal conductivity measurements.

ACKNOWLEDGEMENTS

Thanks are due Dr. R.T.Knapp for valuable advice, Mr. C.D.Cooksey for making the photographs, and Dr. William N. Lacey under whose direction the work has been carried out.



POTENTIOMETER FOR THERMOCOUPLE WORK DESIGNED
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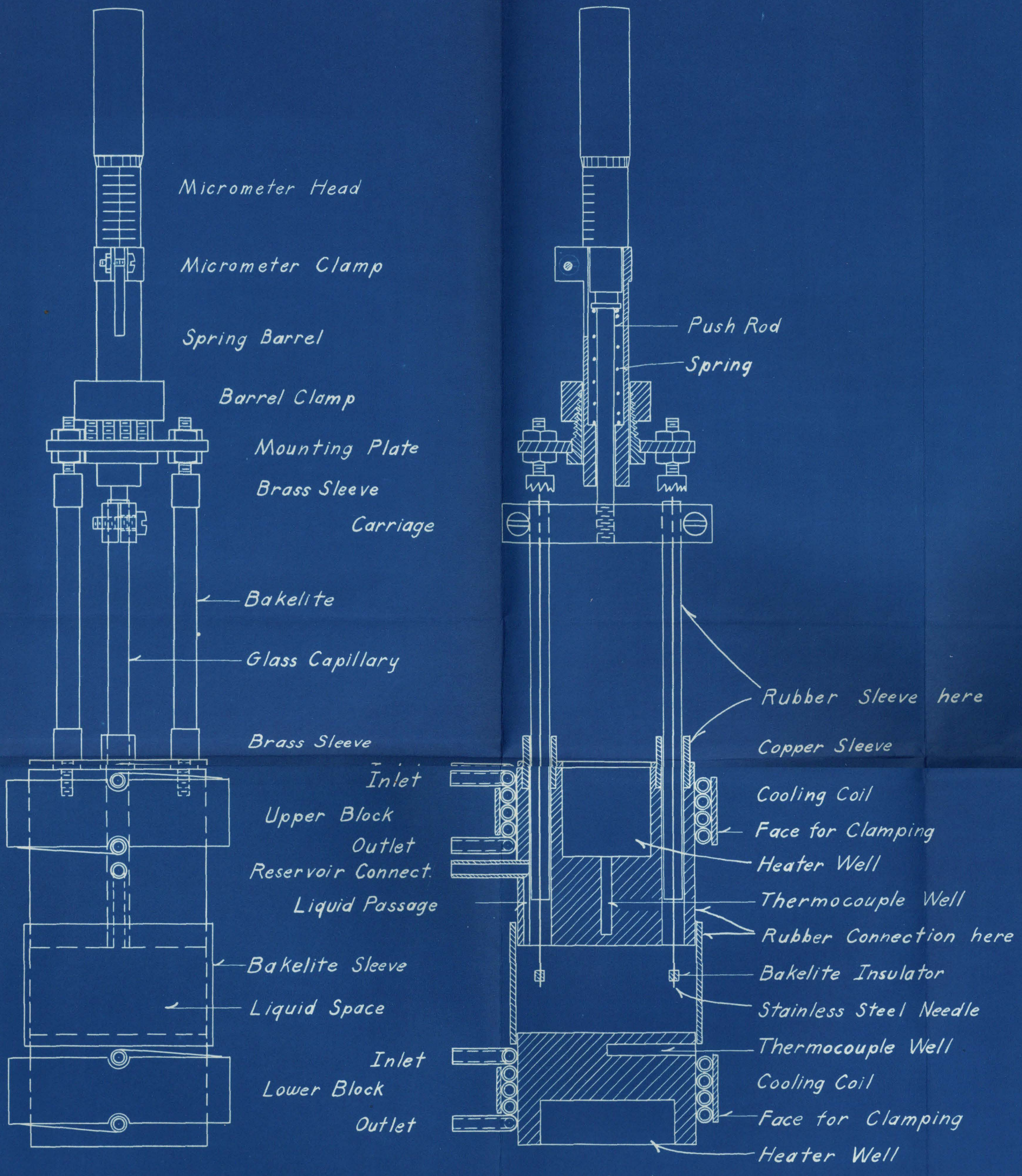
FIG. I

FIG. II



THERMAL CONDUCTIVITY CELL

Scale: 1 Inch = 1 Inch

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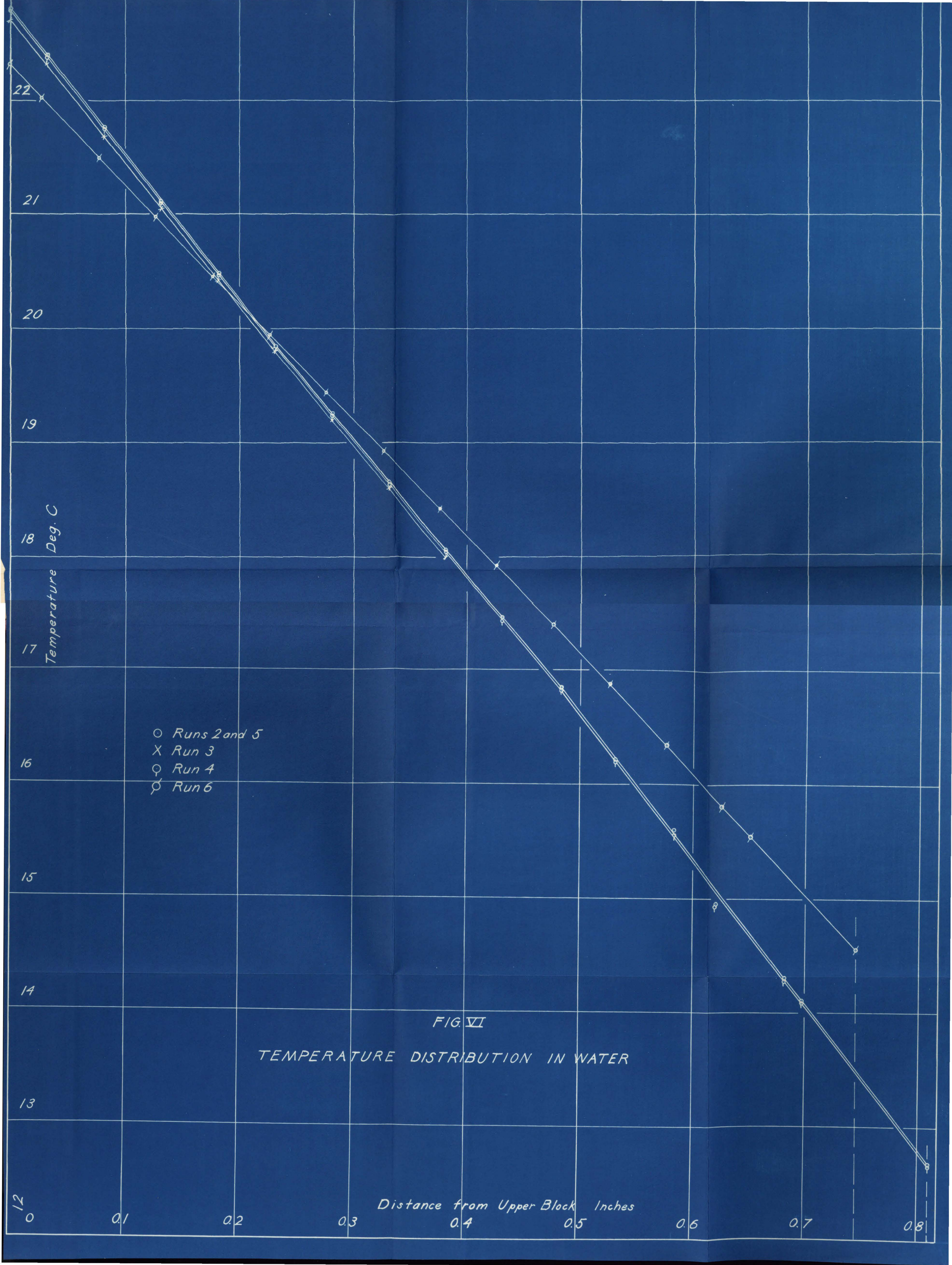

FIG. VI

TEMPERATURE DISTRIBUTION IN WATER