

A STUDY OF THE DENSITIES OF MORPHOLINE
AND MORPHOLINE-WATER MIXTURES

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AND MORPHOLINE-WATER MIXTURES

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INTRODUCTION

Morpholine, tetrahydro 1,4-oxazine, was first prepared by Knorr (1) in 1839. Up until the last few years morpholine had not been produced commercially and had remained a relatively rare substance. Recently there has been an increased interest with widening commercial uses for morpholine, ethylene diamine and other aliphatic amines.

Morpholine is an outstanding substance in its remarkable power as a solvent. A. L. Wilson (2) has stated in regard to morpholine that

"With the possible exception of some of its derivatives, there seems to be no other compound that is miscible with or solvent for, such a wide variety of organic materials. The effect of the heterocyclic ring is perhaps shown in the progressive increase in solvent action in the series, benzene, pyridine, dioxane, morpholine."

Morpholine is a colorless, mobile liquid with a sharp, ammoniacal odor. It boils at 128.9°C (3), has the freezing point of -4.9 ± 0.1 (3), and has the vapor pressure of 13 mm. at 20°C (2). It is soluble in water in all proportions but is relatively insoluble in concentrated potassium hydroxide solutions. It absorbs moisture and carbon dioxide readily from the air to form a soluble carbamate which undergoes dissociation upon heating to 95° or 100°C .

Morpholine may be referred to as a cyclic ether amine and has the usual properties of a secondary amine. It combines with acidic compounds to form salts. It may be recovered from weak acids, such as oleic acid and phenol, by boiling the water solutions. It is separated from its combination with other acids by reaction with strong bases. It cannot

be salted out of solutions by means of salts, but it is salted out by sodium or potassium hydroxide. It forms soaps just as other amines do and these have proved good for the formation of stable oil in water emulsions and film forming emulsions. It is desirable that film forming emulsions, as used for polishes, lacquers, paints, asphalt, paper coating, and leather dressing, should not be re-emulsifiable upon drying. In the preparation of these emulsions morpholine can be heated with little loss at temperatures up to 90°C, yet upon standing morpholine gradually volatilizes to leave water resistant films.

Morpholine seems to hold promise in various extractions such as the recovery or removal of alkaloids, phenols and sulfur compounds. Morpholine has been found useful in textile chemistry as a good solvent for dyes, printing pastes and such materials. It can be used as a color remover for gasoline and lubricating oils. Certain of its derivatives are being investigated with regard to their medicinal possibilities.

Contemporary with the accelerating commercial interest in morpholine, research workers have been engaged in determining its physical constants. In 1935 A. L. Wilson (2) and in 1937 Demser and Demser (3) published some physical constants for pure morpholine. In 1940 Friedman, Bernard, Doe and Fox (4) contributed values for the specific gravities, indices of refraction, viscosities, and surface tensions of morpholine-water solutions at 20°C.

It seemed of interest to investigate further the densities of morpholine and of mixtures of water and morpholine. This thesis reports

determinations of density values for morpholine and morpholine-water solutions at 25°C, 30°C, and 35°C, together with values of specific volumes, partial molar volumes, and coefficients of expansion calculated from the densities.

MATERIAL AND APPARATUS

The morpholine used for the density determinations was obtained by purifying the commercial product, using essentially the method recommended by Demer and Demer (3) and Friedman, Barnard, Doe, and Fox (4). Commercial morpholine was distilled and the fraction boiling below 127°C was discarded. The middle fraction boiling between 127° and 128°C was collected and refluxed over metallic sodium for four or five hours. Subsequent distillation gave a constant boiling product. Since morpholine readily absorbs moisture and carbon dioxide, care was taken at all times to exclude these substances.

Diagrams of the refluxing and distilling set-ups will be found in plate I. They were designed to exclude from the preparation carbon dioxide and moisture and other foreign materials that would result from contact with rubber or cork. Ground glass connections were used throughout. The side arm of the distilling flask was extended in length to act as an air condenser. Attached to the side arm was a soda-lime tube. In the refluxing operation the reaction flask was fitted with an inner reflux type of condenser. For the distillation set-up a thermometer tube was substituted for the reflux condenser.

Morpholine was recovered from the morpholine-water solutions by adding sufficient potassium hydroxide and allowing the system to come to equilibrium, following the method of Demer and Demer (3). The potassium hydroxide layer was then drawn off. Altho no exact

analysis was attempted, there was evidence that this method of extraction involved a relatively large loss of morpholine to the potassium hydroxide-water layer. Wilson (2) states that a ninety-five per cent aqueous solution of morpholine is in equilibrium with a fifty per cent solution of sodium hydroxide. The extracted morpholine began to boil at approximately 118°C which would correspond to ninety-five per cent by weight of morpholine. It seems therefore that the equilibrium condition in morpholine-water-potassium hydroxide mixtures is much the same as with morpholine-water-sodium hydroxide mixtures. As previously, the early fraction was rejected until 127°C was reached. The residue was saved and purified as before. The later density determinations were made upon the recovered morpholine.

The water used in the calibration of the pycnometers and in the preparation of solutions was freshly redistilled and boiled just before using to remove carbon dioxide and other impurities.

The constant temperatures, 25°C , 30°C , and 35°C , were maintained by water baths. The thermometers on which the temperatures were read were checked against a Bureau of Standards thermometer and found to be accurate. The temperatures were constant to $\pm 0.1^{\circ}\text{C}$.

The pycnometers were glass stoppered vessels with long graduated necks, calibrated to read to one-one thousandth of a cubic centimeter, by estimation. A sketch of the type pycnometer used will be found in plate II.

PROCEDURE

To calibrate the pycnometers, each was weighed filled with air and then weighed when filled with a quantity of water such that the meniscus fell upon the graduated part of the neck at the temperatures, 25°C, 30°C, and 35°C. The pycnometers were placed successively in the constant temperature baths at these temperatures and the volumes were read. The whole procedure was then repeated. From the known densities of water at these temperatures, the true volumes corresponding to the volume readings were calculated. The volume held by a pycnometer, corresponding to any reading, was the volume held by that pycnometer when filled to the uppermost mark minus that reading, so equations to be used with the different pycnometers were readily developed. The calibration had given six values of volume and the corresponding readings for each pycnometer. Inserting these in the equation

$$V = C - R$$

where V is the calculated volume, C the volume when filled to the upper mark and R the reading, the value for C was found. The equations for the four pycnometers are as follows:

$$\text{Pycnometer I} \quad V = 52.816 - R$$

$$\text{Pycnometer II} \quad V = 59.895 - R$$

$$\text{Pycnometer III} \quad V = 56.512 - R$$

$$\text{Pycnometer IV} \quad V = 57.649 - R$$

$$\text{Pycnometer IV} \quad V = 57.720 - R$$

(After breakage and repair)

These equations were used in calculating all volumes during the determinations.

A reduced pressure or suction method was used to fill the pycnometers. A sketch of the equipment set-up will be found in plate II. The pycnometer to be filled was fitted to the flask containing the preparation by means of a side armed connecting unit from which extended a tube reaching to the bottom of the flask. The side arm was connected by rubber tubing to a protecting suction flask which in turn was linked by tubing to a vacuum pump. The whole system was evacuated until the liquid boiled for a short time. Upon releasing the tubing from the side arm of the unit, the air admitted at atmospheric pressure forced the liquid into the pycnometer. This evacuation served to reduce the quantity of air dissolved in the liquid.

Obviously, the evaporation of the liquid caused by the low pressure tended to introduce an error into the determinations. However, the time required for filling the pycnometers was brief. It is to be noted that the density values obtained from the four pycnometers show no variation according to order of filling, as should have been the case if the solutions were concentrated when filled there. It is believed, therefore, that no error in density was produced in this method of filling.

Morpholine was freshly purified for each determination. The fraction to be used was distilled directly into the flask in which the solution was prepared and from which the pycnometers were filled. The solutions were made up in per cents by weight. The flask

containing morpholine was weighed and then reweighed upon the addition of the desired quantity of water. Since heat was evolved upon the addition of water, the flasks were cooled to room temperature before the second weighing.

In most cases four determinations were made upon each solution. The pycnometers filled with the prepared solution were carefully weighed. They were then placed in the water baths, one after another, and allowed to remain in each until the volume readings had become constant. From the volume readings the true volumes were calculated and calculation of the densities followed.

DISCUSSION

Table I gives the experimental data for the densities of morpholine and morpholine-water mixtures at 25°, 30°, and 35°C. Included in the table are the densities of water for these temperatures as taken from Dorsey (6).

Table II gives a summary of the density data. Included are the densities of morpholine-water solutions at 20°C as determined by Friedman, Barnard, Doe, and Fox (4) and the density of pure morpholine at 20°C, given by Demer and Demer (3).

Table III gives the specific volumes calculated from the densities as given in table II. The concentrations of morpholine are expressed (a) in per cents by weight and (b) in mol fractions. The moles of water per mol of morpholine are given to show more specifically the effect of adding water to morpholine.

Table IV gives the calculated partial molar volumes of morpholine and of water in the solutions at 25°, 30°, and 35°C for various concentrations of morpholine. Table V gives calculated coefficients of expansions at 30°C for morpholine and various morpholine-water mixtures.

The following graphs have been included.

I. Densities of morpholine and morpholine-water mixtures at 20°, 25°, 30° and 35°C vs. per cents by weight of morpholine. The 20° values for morpholine-water solutions are taken from Friedman, Barnard, Doe, and Fox, and the 20° value for morpholine from Demer and Demer (3).

- II. Specific volumes of morpholine and morpholine-water mixtures at 20°, 25°, 30°, and 35°C vs. per cents morpholine by weight. The 20° values were calculated from density values given in the literature as cited previously.
- III. Densities vs. mol fractions of morpholine.
- IV. Specific volumes vs. mol fractions of morpholine.
- V. Densities vs. mols of water per mol of morpholine.
- VI. Densities vs. temperature for morpholine and various per cent solutions of morpholine in water.
- VII. Specific volumes at 25°C vs. mol fractions of morpholine and showing the graphical means by which the partial molar volumes were calculated.

TABLE I

DENSITY DETERMINATIONS OF MORPHOLINE
AND MORPHOLINE-WATER MIXTURES

	25°/4°C	30°/4°C	35°/4°C
PURE H ₂ O (From Dorsey (6))	.99708	.99568	.99406
% MORPHOLINE			
14.17%	1.0064	1.0046	1.0025
	1.0065	1.0046	1.0026
	1.0066	1.0047	1.0027
	1.0067	1.0048	1.0027
Average	<u>1.0065</u>	<u>1.0047</u>	<u>1.0026</u>
21.97%	1.0127	1.0105	1.0083
	1.0128	1.0105	1.0083
	1.0128	1.0105	1.0083
	1.0127	1.0105	1.0082
Average	<u>1.0128</u>	<u>1.0105</u>	<u>1.0083</u>
26.72%	1.0165	1.0140	1.0116
	1.0165	1.0141	1.0116
	1.0164	1.0140	1.0115
	1.0165	1.0140	1.0115
Average	<u>1.0165</u>	<u>1.0140</u>	<u>1.0116</u>

TABLE I
-continued-

% MORPHOLINE	25°/4°C	30°/4°C	35°/4°C
35.55%	1.0237	1.0210	1.0180
	1.0238	1.0211	1.0181
	1.0239	1.0211	1.0180
	1.0236	1.0209	1.0179
	Average	<u>1.0238</u>	<u>1.0210</u>
47.35%	1.0324	1.0290	1.0255
	1.0324	1.0290	1.0256
	1.0325	1.0290	1.0256
	1.0325	1.0290	1.0254
	Average	<u>1.0325</u>	<u>1.0290</u>
53.07%	1.0356	1.0318	1.0281
	1.0356	1.0316	1.0281
	1.0354	1.0316	1.0278
	Average	<u>1.0355</u>	<u>1.0317</u>
57.05%	1.0368	1.0330	1.0291
	1.0369	1.0331	1.0292
	1.0369	1.0330	1.0291
	1.0368	1.0329	1.0290
	Average	<u>1.0369</u>	<u>1.0330</u>

TABLE I
-continued-

% MORPHINE	25°/4°C	30°/4°C	35°/4°C
72.67%	1.0353	1.0310	1.0267
	1.0353	1.0310	1.0265
	1.0353	1.0309	1.0266
	1.0350	1.0303	1.0258
	Average	<u>1.0352</u>	<u>1.0308</u>
84.03%	1.0249	1.0203	1.0158
	1.0249	1.0204	1.0159
	1.0249	1.0203	1.0157
	1.0230	1.0204	1.0158
	Average	<u>1.0249</u>	<u>1.0204</u>
89.85%	1.0164	1.0118	1.0073
	1.0164	1.0119	1.0073
		1.0119	
	Average	<u>1.0164</u>	<u>1.0119</u>
100.00%	.9947	.9897	.9849
	.9946	.9896	.9849
		.9896	.9851
		.9898	
	Average	<u>.9947</u>	<u>.9897</u>

TABLE II
 SUMMARY OF DENSITIES OF MORPHOLINE AND
 MORPHOLINE-WATER MIXTURES

PER CENT MORPHOLINE	MOL. FRAC- TIONS OF MORPHOLINE	MOLS H ₂ O PER 1 MOL MORPHOLINE	DENSITIES ¹			
			20°C ²	25°C/4°C	30°C/4°C	35°C/4°C
0.00 (100% H ₂ O)	.0000	∞	.99825	.99708	.99568	.99406
14.17	.0330	29.2787	1.0097	1.0066	1.0047	1.0026
21.97	.0560	17.1666	1.0164	1.0126	1.0108	1.0083
26.72	.0701	13.2560	1.0205	1.0165	1.0140	1.0116
35.55	.1024	8.7628	1.0277	1.0238	1.0210	1.0180
47.36	.1569	5.3723	1.0365	1.0325	1.0290	1.0255
53.07	.1836	4.2743	1.0402	1.0355	1.0317	1.0280
57.05	.2156	3.6390	1.0421	1.0369	1.0330	1.0291
72.67	.3549	1.8176	1.0412	1.0352	1.0308	1.0264
84.05	.5216	.9172	1.0311	1.0249	1.0204	1.0158
89.65	.6418	.5580	1.0223	1.0164	1.0119	1.0073
100.00	1.0000	.0000	.9994 ³	.9947	.9897	.9850

1--Densities for H₂O were taken from Dorsey (6).

2--Densities for morpholine-water mixtures at 20°C were estimated by graphical means for the particular percentages given from the values determined by Friedam, Barnard, Doe, and Fox (4).

3--Density of morpholine, determined by Demer and Demer (3).

TABLE III
 SPECIFIC VOLUMES FOR MORPHOLINE AND
 MORPHOLINE-WATER MIXTURES

PER CENT MORPHOLINE	MOL FRAC- TIONS OF MORPHOLINE	MOLS H ₂ O PER 1 MOL MORPHOLINE	20° C	SPECIFIC VOLUMES		
				25°/4°C	30°/4°C	35°/4°C
0.00 (100% H ₂ O)	.0000	∞	1.0018	1.0029	1.0044	1.0060
14.17	.0550	29.2767	.9904	.9934	.9953	.9974
21.97	.0880	17.1666	.9839	.9873	.9896	.9918
26.72	.0901	13.2360	.9799	.9838	.9862	.9885
35.55	.1024	8.7623	.9730	.9768	.9794	.9823
47.36	.1569	5.3725	.9648	.9685	.9718	.9751
53.07	.1696	4.2743	.9614	.9637	.9693	.9728
57.05	.2156	3.5390	.9596	.9644	.9661	.9717
72.67	.3549	1.8175	.9604	.9660	.9701	.9743
84.05	.5216	.9172	.9698	.9757	.9800	.9844
89.65	.6418	.5560	.9782	.9839	.9882	.9928
100.00	1.0000		1.0006	1.0063	1.0104	1.0152

TABLE IV

PARTIAL MOLAR VOLUMES

MOL FRACTIONS OF MORPHOLINE	PARTIAL MOLAR VOLUMES OF MORPHOLINE			
	20°C	25°/4°C	30°/4°C	35°/4°C
0.0	39.70	63.08	64.58	65.61
.2	73.55	80.13	80.79	81.44
.4	86.86	87.32	87.60	88.00
.6	87.13	87.63	87.99	88.40
.8	87.13	87.63	87.99	88.40
1.0	87.13	87.63	87.99	88.40

MOL FRACTIONS OF WATER	PARTIAL MOLAR VOLUMES OF WATER			
	20°C	25°/4°C	30°/4°C	35°/4°C
1.0	18.05	18.07	18.10	18.12
.8	17.36	17.39	17.64	17.69
.6	16.91	17.04	17.11	17.19
.4	16.86	16.97	17.05	17.12
.2	16.86	16.97	17.05	17.12
0.0	16.86	16.97	17.05	17.12

TABLE V

COEFFICIENTS OF EXPANSION FOR MORPHOLINE
AND MORPHOLINE-WATER MIXTURES AT 30°C

PER CENT MORPHOLINE	COEFFICIENTS OF EXPANSION
14.17	.0004018
21.97	.0004547
26.72	.0004766
35.55	.0005615
47.36	.0006894
53.07	.0007325
57.05	.0007541
72.67	.0008555
84.05	.0008876
89.65	.0009004
100.00	.0008805

Figure 1. Set-Up Used for Reducing Morpholine

(For distilling morpholine, a similar set-up
was used, but a thermometer was substituted
for the inner reflux condenser.)

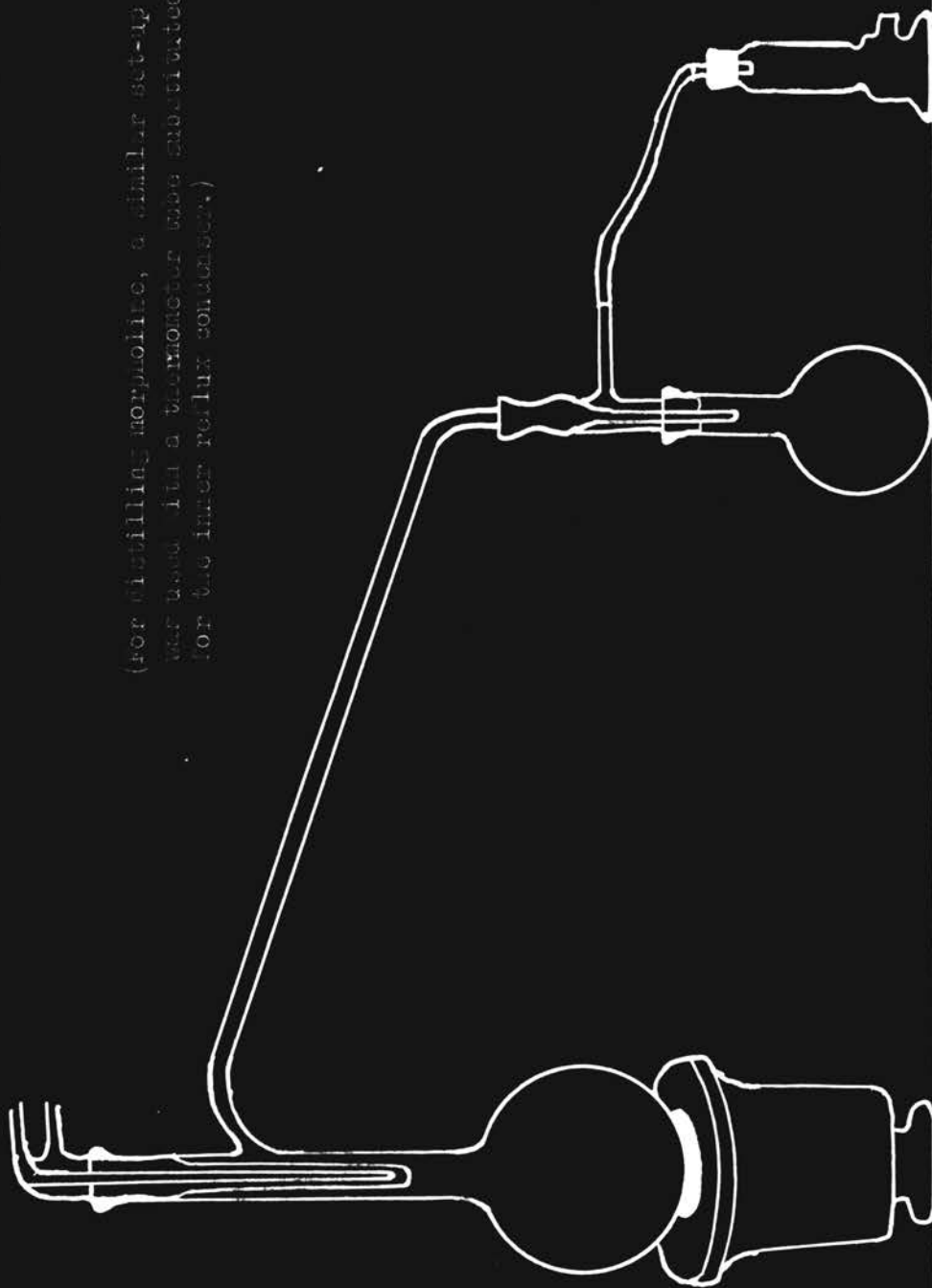
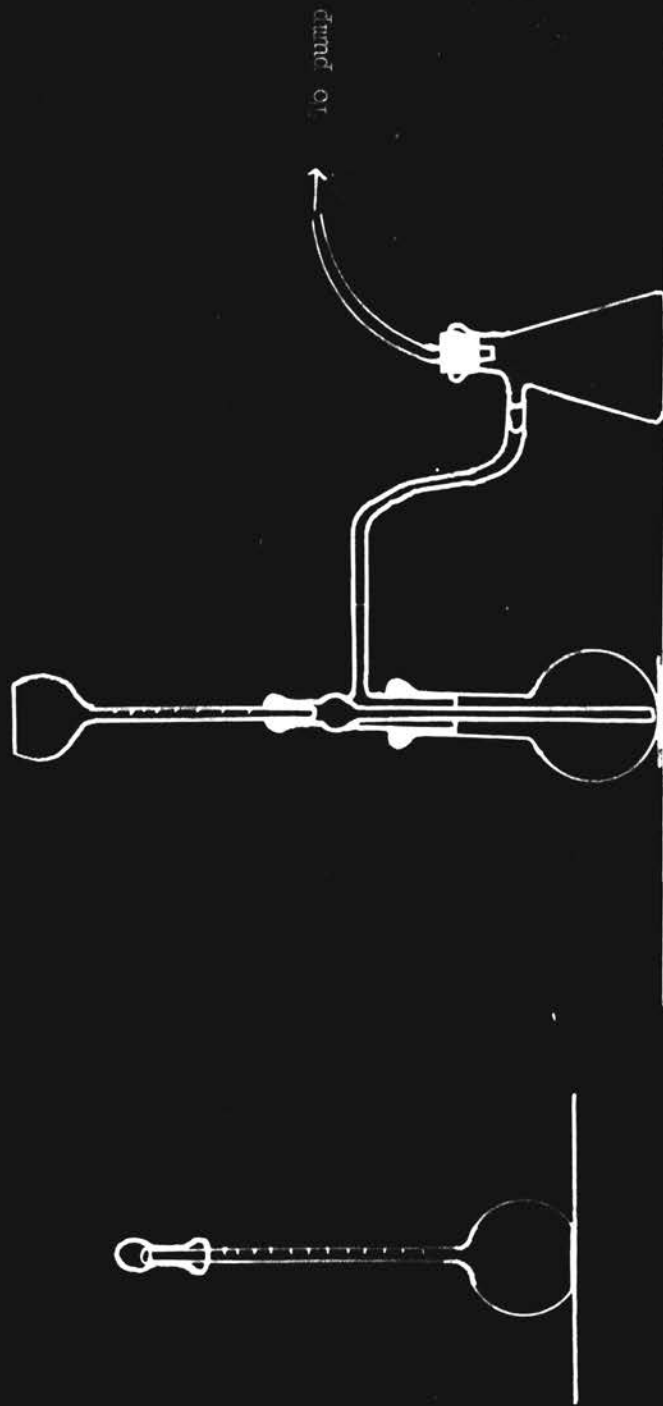


Plate II. Pyrometer and Method of Filling
Pyrometer



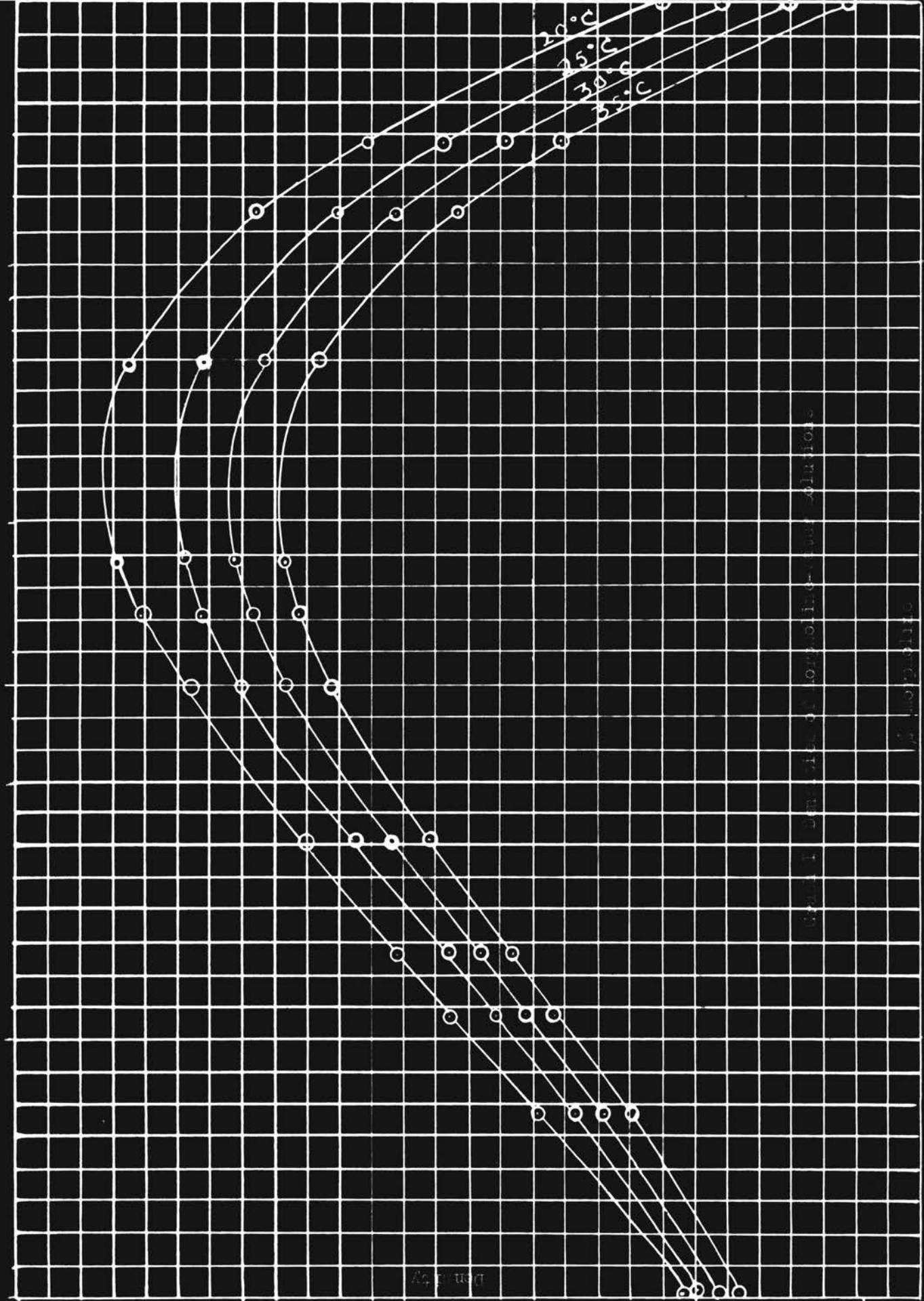
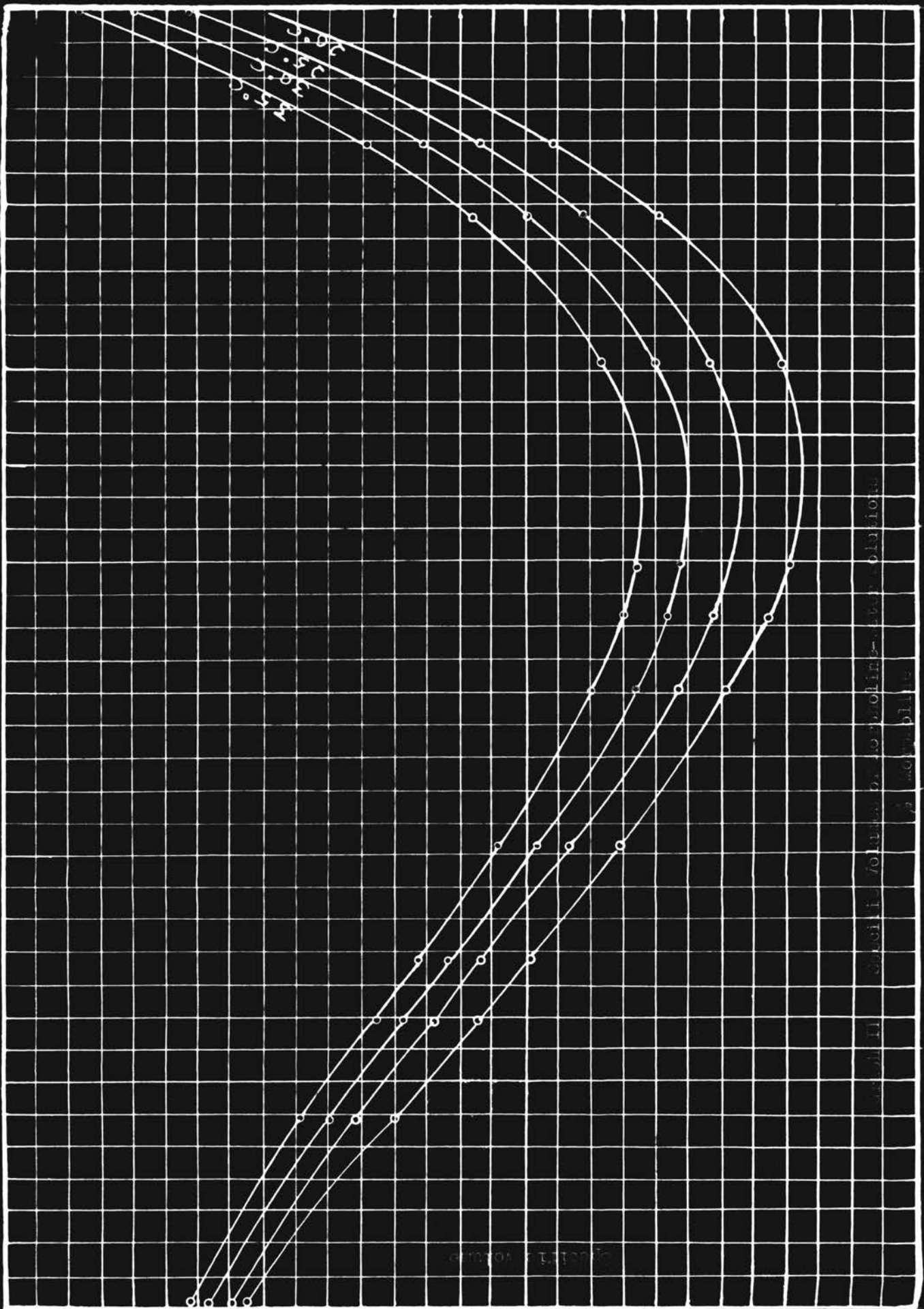


Chart I. Density of Morpholine-Toluol Solutions

M. Moravcsik

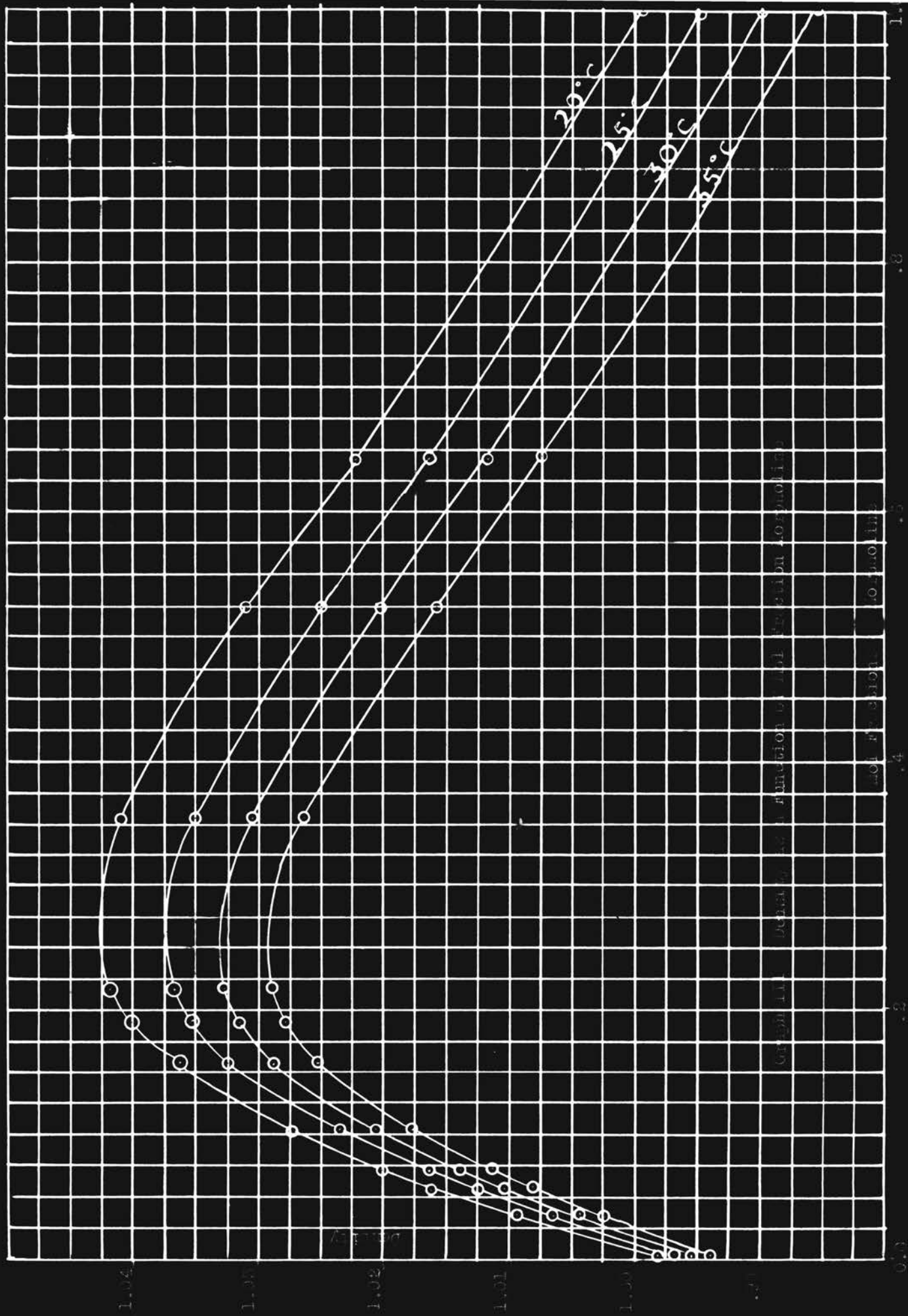


100
30
60
40
30

Graph III. Specific Volume of Acetone-ethyl Alcohol Solutions
at 20°C. (Specific Volume)

Specific Volume

1.01
1.00



Graph III. Refractive Index as a function of mol fraction for alcohol.

MOL FRACTION

1.0
.8
.6
.4
.2
0.0

1.04

1.03

1.02

1.01

1.00

1.00

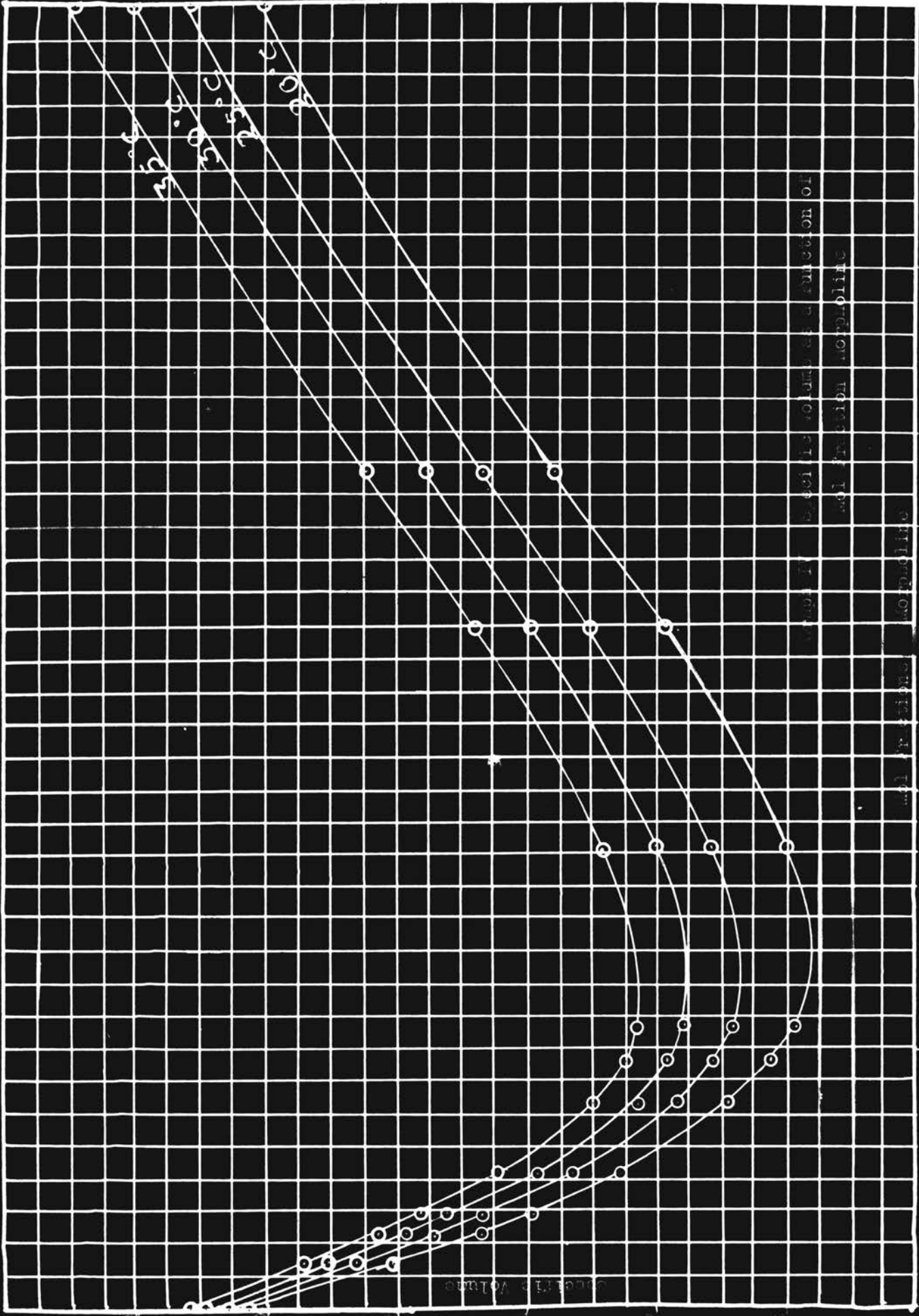
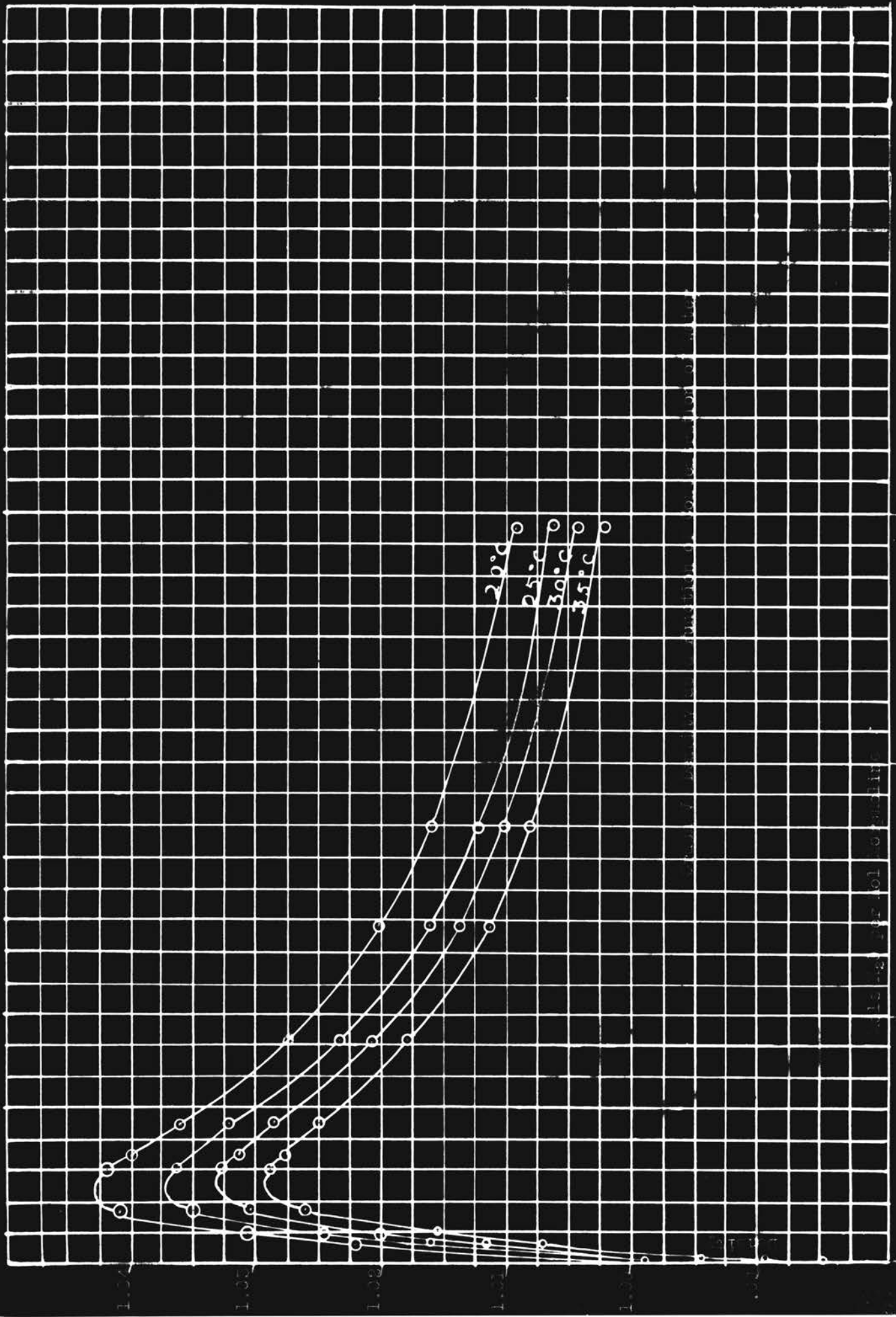
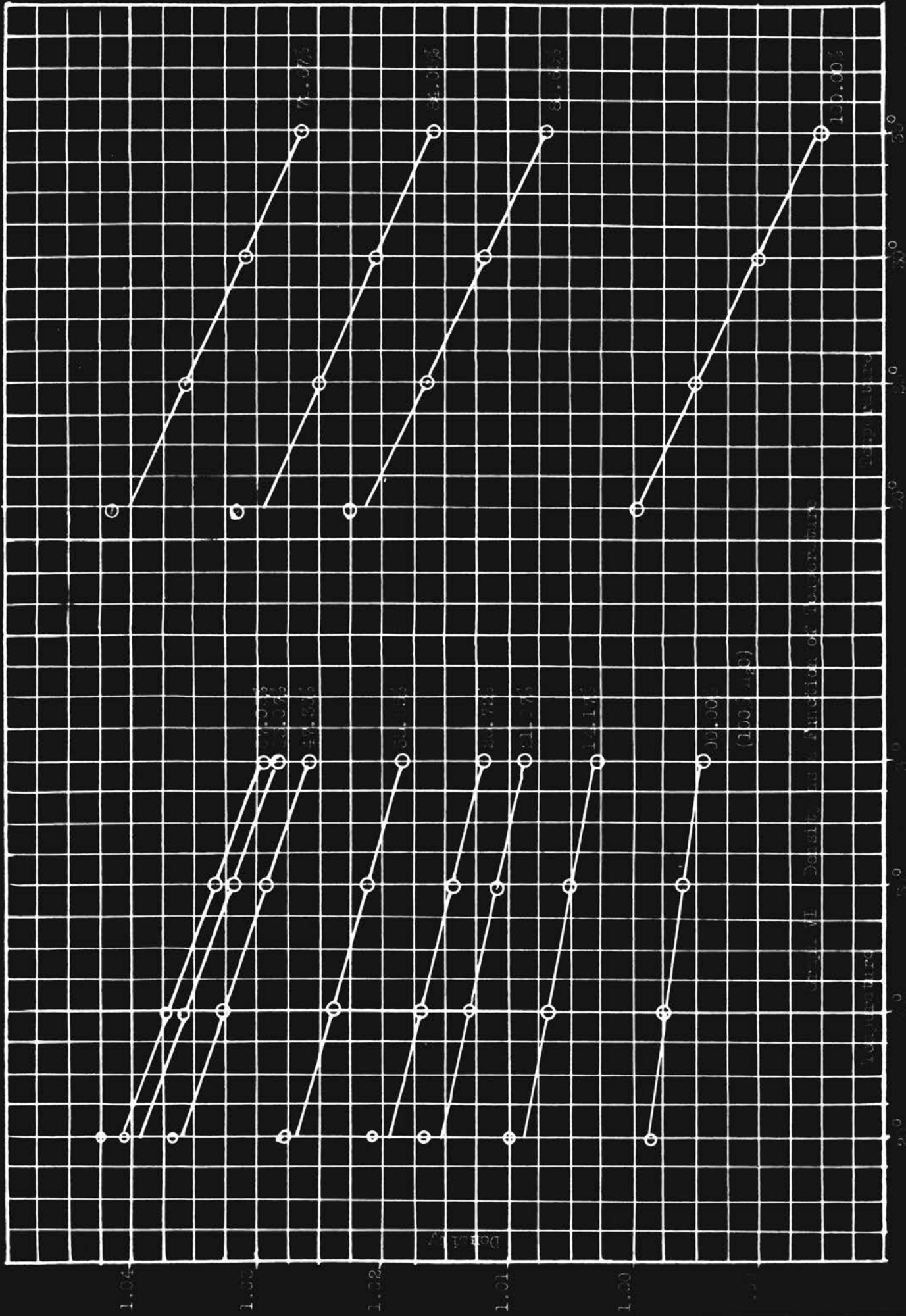


Fig. 1. Specific Volume as a Function of Temperature for Polystyrene and Triphenyl Acetylene



10-10-1917 for 10-10-1917

10 20 30



Dr. J. H. Daniels, Jr., Director of Laboratory

(100-149)

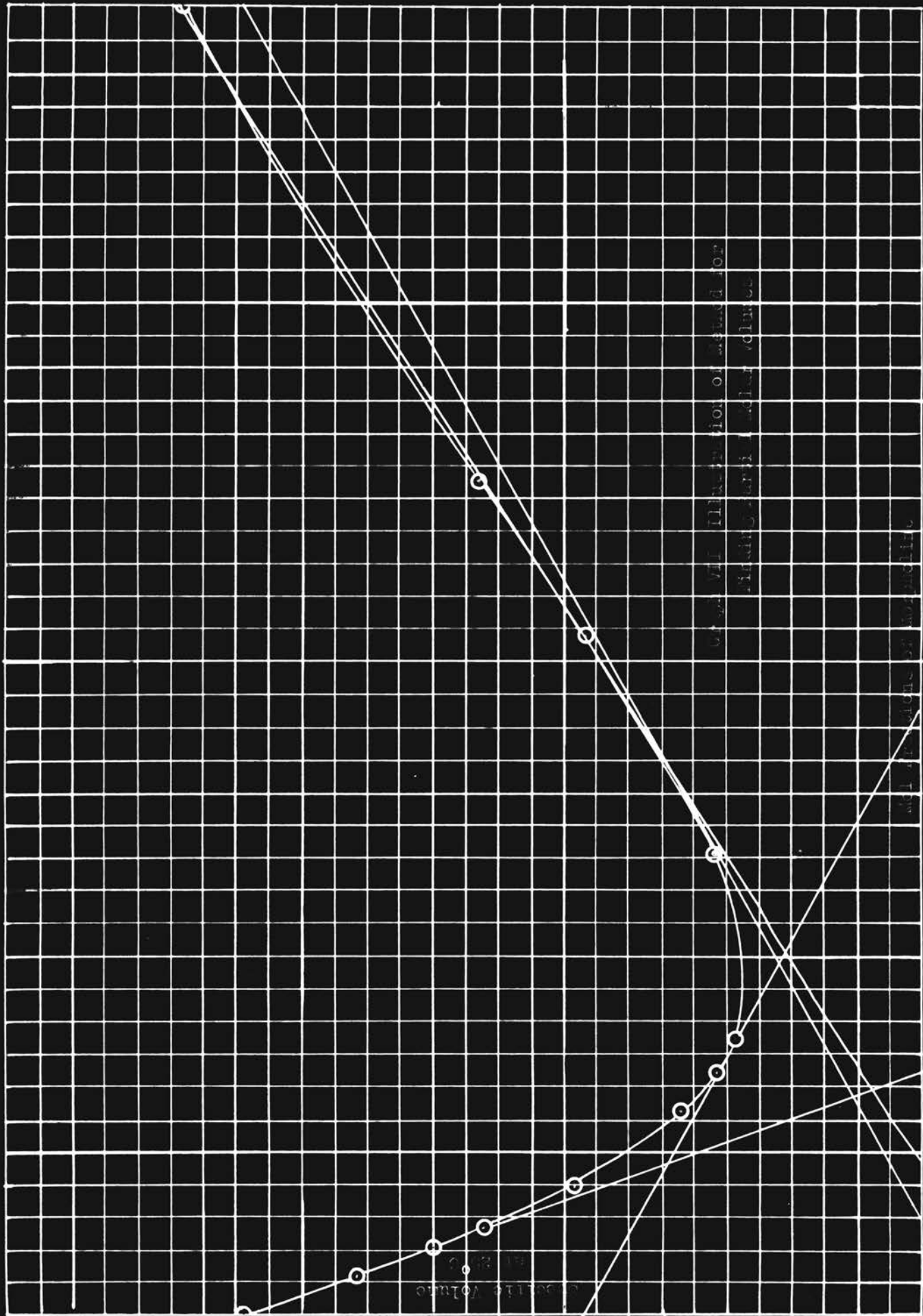


Fig. 1. Illustration of method for finding β and γ values.

Temperature in degrees Celsius

Relative Volume

1.01

1.00

The only literature found on the densities of morpholine-water mixtures is the before mentioned reference by Friedman, Barnard, Doe, and Fox (4) in which the specific gravities at 20°C are given. It would be assumed that these values are referred to water at 4°C as standard, since this is the usual procedure. This seems probable too, since the authors include with their results the specific gravity as given by Beilstein, namely, 1.0007, and this specific gravity is definitely referred to the density of water at 4°C as standard.

In graph VI the densities of solutions of the different concentrations are plotted against temperature. The values determined for 25°, 30°, and 35°C fall upon a straight line for each concentration. These lines when extrapolated to 20° fall uniformly below the Friedman, Barnard, Doe, and Fox (4) values for the same per cent solutions.

In recalculating these values to 4°C by multiplying by the ratio of the density at 20° C to density at 4°C, that is by .9982/1.0000 (6), the points fall on the straight lines passing thru the determined values. It is impossible to tell from the article by Friedman, Barnard, Doe, and Fox (4) whether this correction should be applied or not. That being the case, we have based our calculations of the coefficient of expansion upon our experimental results alone.

The most recently determined value for the density of pure morpholine at 20°C is by Denner and Denner (3) and the values for 25°, 30°, and 35°C, determined in this thesis, fall upon a straight line with the Denner and Denner value.

The maximum density or minimum volume for morpholine-water solutions occurs in approximately a 66 per cent or .25 mol fraction morpholine solution in which the ratio of water to morpholine is approximately 3.0 to 1. It is interesting to note that Barnard, Friedman, Doe, and Fox found the maximum viscosity and maximum heating effect to occur in approximately a 70 per cent morpholine solution for which the ratio of water to morpholine is almost exactly 2 to 1, corresponding to the formula $C_4H_9ON \cdot 2H_2O$.

The partial molar volumes of morpholine and of water, that is, the volumes which one mol of each actually occupies in the solution, were found for various mol fraction concentrations by using the graphical method illustrated in graph VII. The specific volumes of the solution were graphed against mol fractions. Since the mixtures were binary systems, the ordinate of 0.0 mol fraction of morpholine was the ordinate of 1.0 mol fraction of water, and vice versa. Tangents were drawn to the curve at the points corresponding to 0.0, 0.2, 0.4, 0.6, 0.8 and 1.0 mol fraction morpholine or 1.0, 0.8, 0.6, 0.4, 0.2 and 0.0 mol fraction water. The tangent lines were extended to the limits of the graph. The reading of specific volume at the intersection of the tangent line with the ordinate of 1.0 mol fraction morpholine gave the partial specific volume of morpholine for the mol fraction at which the tangent line was drawn. Likewise, the reading of specific volume for the intersection of the same tangent line with the ordinate of 1.0 mol fraction of water gave the partial specific volume of water for the mol fraction concentration at which the tangent line was drawn.

Upon consideration of graph VII, it will be noted that the tangents of greatest slope do not cut both the 0.0 and 1.0 mol fraction ordinates because of the limitations of the graph. Where this was the case, the readings for the tangent line intersection with the one ordinate and the intersection with the abscissa were taken. By the geometric method of similar triangles, a proportion was set up so that the point of intersection upon the other ordinate could be calculated.

The partial molar volumes were found from the partial specific volumes by multiplying each by its molecular weight. Both the partial molar volumes of water and of morpholine show a decline with decreasing concentrations of each.

The coefficients of expansion at 30°C for various per cent solutions were calculated from the specific volumes at 25° and 35°C. This method was suitable since a graph of specific volumes against temperature showed the expansion to follow a straight line curve. The expression used was

$$\delta = \frac{1}{V} \left(\frac{\partial V}{\partial t} \right)_P \quad \text{where}$$

δ = coefficient of expansion at 30°C.

V = the mean between the specific volumes at 25°C and 35°C.

∂V = difference between the specific volumes at 35°C and 25°C.

∂t = difference in temperature corresponding to the volume intervals chosen; 10.

It will be noted that the coefficient of expansion shows a constant and gradual increase with increasing concentrations to about ninety per cent, whence it falls slightly to the coefficient of expansion of pure morpholine.

SUMMARY

The densities of morpholine and morpholine-water mixtures have been experimentally determined for the temperatures 25°, 30°, and 35°C, and the specific volumes and partial molar volumes have been calculated for these temperatures, together with the coefficients of expansion at 30°C.

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BIOGRAPHY

Alice Frances Buse was born in Cohoes, New York, March 1915. She attended the public schools of that city, graduating from Cohoes High School in 1933. In September of the same year she entered Russell Sage College, Troy, New York and graduated from that institution with a Bachelor of Arts degree in June 1937.

For three academic years she was employed by Russell Sage College as assistant in the chemistry department. During the summer of 1939 she attended the graduate school of New York State College for Teachers, Albany, New York. The following summer she was engaged in chemical work for the Hi-Pressure Oil Company of Cohoes, New York.

In September 1940, she received from Oklahoma Agricultural and Mechanical College a half-time assistantship in the chemistry department and enrolled in the graduate school of that college. She will complete the requirements for the degree of Master of Science in August 1941.

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