

VAPOR-LIQUID EQUILIBRIA OF 2-PROPANOL--ACETONE SYSTEM

By

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TABLE OF CONTENTS

	Page
SUMMARY	1
INTRODUCTION.	2
STATEMENT OF PROBLEM.	4
EXPERIMENTAL METHOD	5
METHOD OF CALCULATION AND RESULTS	7
NOMENCLATURE.	10
DISCUSSION OF RESULTS	11
BIBLIOGRAPHY.	12
APPENDIX.	13

LIST OF TABLES

Table

1. Vapor-Liquid Equilibria at 1.0 atmosphere Pressure.	14
2. Vapor-Liquid Equilibria at 2.5 atmosphere Pressure.	15
3. Vapor-Liquid Equilibria at 4.0 atmosphere Pressure.	16

LIST OF ILLUSTRATIONS

Graph

1. Ml. of Sodium Hydroxide versus Mol Fraction Acetone	17
2. Van Laar Equation and Experimental Data at 1.0 Atmosphere Pressure.	18
3. Margules Equations and Experimental Data at 1.0 Atmosphere Pressure.	19
4. Van Laar Equations and Experimental Data at 2.5 Atmosphere Pressure.	20
5. Margules Equations and Experimental Data at 2.5 Atmosphere Pressure.	21

Graph	Page
6. Van Laar Equations and Experimental Data at 4.0 Atmosphere Pressure.	22
7. Margules Equations and Experimental Data at 4.0 Atmosphere pressure.	23
8. Logarithm of Activity Coefficients versus Mol Fraction of Acetone in Liquid	24
 Figure	
1. Equilibrium Still.	25

SAMPLE CALCULATIONS

Method of Least Squares for Evaluating Van Laar Constants.	26
Method of Least Squares for Evaluating Margules Constants.	28

SUMMARY

The vapor-liquid equilibria for the system 2-propanol--acetone was determined by the continuous distillation method at 1.0, 2.5 and 4.0 atmospheres pressure. These experimental data were used to evaluate the constants in the Van Laar and Margules equations. Corresponding values of vapor-liquid compositions were calculated to determine the internal thermodynamic agreement of the experimental data. The Van Laar equation gave the closer agreement over the entire range of composition.

INTRODUCTION

The separation of a mixture of volatile liquids depends upon the difference in composition of the liquid and the vapor evolved. All fractional distillation calculations are dependent upon a knowledge of this relationship. Robinson and Gilliland (7) have described experimental methods for the determination of vapor-liquid equilibria.

In the bomb method the sample is placed in a closed vessel which in turn is placed in a constant temperature bath and agitated until equilibrium conditions are attained. Samples of the liquid and vapor are withdrawn through suitable connections and analyzed. This procedure is repeated for various compositions.

The dynamic distillation method involves the distilling of successive small portions of the liquid and the condensate is analyzed. The quantity distilled is plotted versus the composition and the curve extrapolated to zero quantity distilled. This extrapolated value is taken as the composition of the vapor in equilibrium with the original liquid.

The dew and boiling point method requires the observation of dew and boiling point of a mixture of known composition. From a cross plot of pressure versus temperature phase equilibrium conditions are obtained.

The dynamic flow method is accomplished by passing the vapor through a series of vessels which contain liquid of suitable composition. The vapor evolved from the last vessel is assumed to be in equilibrium with the liquid.

In the continuous distillation method the vapors are condensed and returned to the still by means of static liquid head of condensate. The

process is continued until a steady state exists for a given time and equilibrium is assumed. The condensate and still liquid are sampled and analyzed. This process has been adapted for a variety of liquids and operating conditions. A modification of the Othmer and Morley (4) high pressure still was used in this work.

Acetone and 2-propanol were selected as components to be studied since they are readily converted from one to the other and a mixture of these is often encountered in the petrochemical industry. Previous works reported by Lecat (2) indicate this system to be non-azeotropic. Parks and Chaffee (6) studied this system at 25°C. and reported the partial pressure of acetone in the vapor exceed that calculated by Raoult and Dalton's laws and concluded the system was non-ideal.

STATEMENT OF PROBLEM

This investigation was undertaken to determine the vapor-liquid equilibria of 2-propanol--acetone system at 1.0, 2.5 and 4.0 atmospheres of pressure and to check the agreement with the Van Laar and Margules equations.

EXPERIMENTAL METHOD

The materials used were Merck reagent grade acetone and Bakers analyzed 2-propanol. These materials were dried with drierite and then distilled. The portions of acetone boiling at 56°-57°C. @ 760 mm. and 2-propanol boiling at 82°-83°C. @ 760 mm. were collected for use.

The still used was a modification of the still used by Othmer and Morley (4) and Wilson and Simons (10) and is shown in Figure 1. The still and condensate receiver were constructed so that the volume of the still would be large in comparison to the volume of the condensate receiver so that equilibrium could be obtained more readily.

A 700 ml. charge of approximate composition was prepared and added to the still through valve No. 1. Sufficient water-pumped nitrogen from a cylinder was admitted through the drier (packed with drierite) to thoroughly flush out the air. A pressure of 10 psi was then applied. Heat was applied to the still by means of a powerstat and electric heater. As the pressure increased, excess pressure was released through valve No. 1, until the nitrogen in the still was replaced with 2-propanol--acetone. Further required decreases in pressure were made through valves No. 6 and 7. The pressure was observed by a 0-45 psi Reid vapor pressure gauge subdivided into 0.2 psi and previously checked for accuracy. The temperature of the still was observed with an A. S. T. M. thermometer which was previously calibrated.

After distillation had begun the rate was estimated by counting the drops of condensate as they fell from the drip tip. With slight adjustments of the applied heat the pressure could be controlled over a small

range. After a steady temperature and pressure had been maintained for two hours valves 3 and 4 were closed and the heater shut off. A sample of still liquid was withdrawn through valve No. 2, with sufficient cooling being provided to prevent flashing. The condensate sample was withdrawn through valve No. 5. Wilson and Simons (10) recommend suddenly increasing the pressure 10 psi by the addition of nitrogen to stop the boiling while samples were withdrawn, however this increase in pressure upon the saturated vapor that exists in the still above the liquid would cause considerable condensation and may affect the composition of the still-liquid. By closing valves 3 and 4 and turning off the heater and withdrawing the still sample as rapidly as possible conditions were not disturbed too drastically since only 25 ml. of liquid were withdrawn from a total volume of 1600 ml.

One ml. aliquot of the sample was analyzed by reacting the acetone with hydroxylamine hydrochloride and titrating the liberated hydrochloric acid with sodium hydroxide using bromophenol indicator. The mol fraction of acetone was read from graph No. 1 which was prepared by titrating 1 ml. of synthetic samples.

METHOD OF CALCULATION AND RESULTS

Vapor pressure data for 2-propanol in the range from 1 to 4 atmospheres was reported by Wilson and Simons (10) and for pressures less than 1 atmosphere as reported by Parks and Barton (5).

Similar data for acetone was reported in "Handbook of Chemistry and Physics" (1).

Activity coefficients were calculated by:

$$\gamma_1 = \frac{P y_1}{p_1^{\circ} x_1}$$

$$\gamma_2 = \frac{P y_2}{p_2^{\circ} x_2}$$

The Van Laar equation was rearranged from: (8)

$$\ln \gamma_1 = \frac{B/T}{\left(1 + A \frac{x_1}{x_2}\right)^2}$$

$$\frac{1}{(T \ln \gamma_1)^{1/2}} = \frac{1}{B^{1/2}} + \frac{A}{B^{1/2}} \frac{x_1}{x_2}$$

This is in the form of a straight line, ($y = a + bx$) with:

$$y = \frac{1}{(T \ln \gamma_1)^{1/2}}, \quad a = \frac{1}{B^{1/2}}, \quad \text{and}$$

$$b = \frac{A}{B^{1/2}}$$

From the experimental data $\frac{1}{(T \ln \gamma_1)^{1/2}}$ and $\frac{x_1}{x_2}$ were calculated for

each set of points. The constants a and b were evaluated by the method of least squares (9) and A and B calculated from $B = \frac{1}{a^2}$ and $A = b \times B^{1/2}$.

From: $\ln \gamma_1 = \frac{B/T}{\left(1 + A \frac{x_1}{x_2}\right)^2}$ the values of $\ln \gamma_1$ were calculated for a

given temperature and $\ln \gamma_2 = A \frac{(x_1)^2}{(x_2)} \ln \gamma_1$

From: $y_1 = \frac{\gamma_1 x_1 p_1^s}{P}$ and $y_2 = \frac{\gamma_2 x_2 p_2^s}{P}$ corresponding values of y_1 and y_2 were obtained.

The proper temperature was selected so that $y_1 + y_2 = 1.00$.

The Margules equations were rearranged as follows:

$$\ln \gamma_1 = bx_2^2 + cx_2^3 \quad (1)$$

$$\frac{\ln \gamma_1}{x_2^2} = b + cx_2 \quad (2)$$

$$\ln \gamma_2 = bx_1^2 + 3/2 cx_1^2 - cx_1^3 \quad (3)$$

$$\frac{\ln \gamma_2}{x_1^2} = b + 3/2 c - cx_1 \quad (4)$$

$$x_1 = 1 - x_2 \quad (5)$$

$$\begin{aligned} \frac{\ln \gamma_2}{x_1^2} &= b + 1/2 c + cx_2 \\ &= b + c(0.5 + x_2) \end{aligned} \quad (6)$$

Equations No. 2 and 6 are in form of $y = a + b_1x$ with $y = \frac{\ln \gamma_1}{x_2^2}$ and $\frac{\ln \gamma_2}{x_1^2}$

and $x = x_2$ and $(.5 + x_2)$, and $a = b$ and $b_1 = c$.

Values of $\frac{\ln \gamma_1}{x_2^2}$ and $\frac{\ln \gamma_2}{x_1^2}$ were calculated for each experimental determination and value of the constants in the equations determined by the method of least squares (9). The experimental data, calculated values of vapor

liquid compositions by Van Laar and Margules equations and Van Laar and Margules constants are given in Tables No. 1, 2 and 3 for 1.0, 2.5 and 4.0 atmospheres pressure, respectively, and are shown by graph No. 2 through 7. Graph No. 8 shows the logarithm of activity coefficients versus mol fraction of acetone in the liquid.

NOMENCLATURE

A, B	Van Laar Constants
a, b	Geometric Constants
b, c	Margules Constants
P	Total Pressure
p°	Vapor Pressure of Pure Component
S	Sum of
T	Degrees Kelvin
x	Mol Fraction in Liquid
y	Mol Fraction in Vapor
γ	Activity Coefficient

Subscripts:

- 1 Component No. 1 or acetone
- 2 Component No. 2 or 2-propanol

DISCUSSION OF RESULTS

The Van Laar and Margules equations both were in good agreement with the experimental data at the mid-point of the composition range. The deviation was greater at the extremities which is probably due to error in the analytical determinations or equilibrium not having been attained. Accurate data are more difficult to obtain in this range since a small error in determining activity coefficients near unity will result in a larger error in the logarithm of the activity coefficient.

In the continuous distillation method there are several sources of error. There is a question whether vapor rising from a boiling liquid is in equilibrium with the residue liquid. Equilibrium would be favored by a slow distillation rate. Temperature measurements are sometimes affected by superheating of vapor. Pressure may not be constant throughout the apparatus. The condensate returning to the still may be vaporized before reaching the still and not be in equilibrium with the still liquid. At high pressures the pressurizing gas may be soluble in the condensate to the extent to affect the equilibrium.

The continuous distillation method has been widely used due to its simplicity and ease of operation.

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APPENDIX

TABLE NO. 1
Vapor-Liquid Equilibria at
1.0 Atmosphere Pressure

ml. of NaOH for 1 ml. Aliquot		Experimental				Van Laar Equations				Margules Equations			
x_1	y_1	x_1	y_1	γ_1	T°F.	x_1	y_1	γ_1	T°F.	x_1	y_1	γ_1	T°F.
42.4	47.5	.821	.923	1.05	136°	.821	.902	1.025	136°	.821	.908	1.03	136°
35.7	44.0	.694	.855	1.071	140	.694	.842	1.07	140	.694	.853	1.08	140
36.0	39.6	.504	.760	1.185	146	.504	.746	1.20	145	.504	.755	1.19	146
22.2	37.0	.430	.719	1.27	148	.430	.704	1.26	147	.430	.708	1.24	148
15.5	31.9	.301	.620	1.422	153	.301	.605	1.402	153	.301	.600	1.33	155
11.2	26.8	.218	.520	1.522	158	.218	.515	1.515	158	.218	.500	1.386	161
7.4	20.6	.141	.400	1.645	164	.141	.403	1.638	164	.141	.370	1.435	167
4.5	14.5	.087	.282	1.69	170	.087	.290	1.735	170	.087	.252	1.46	172
.75	4.0	.0145	.077	2.47	177	.0145	.06	1.88	178	.0145	.045	1.475	180

$$A = 0.95$$

$$B = 228$$

$$b = 1.01$$

$$c = -.61$$

TABLE NO. 2
 Vapor-Liquid Equilibria at
 2.5 Atmosphere Pressure

ml. of NaOH
 for 1 ml.
 Aliquot

		<u>Experimental</u>				<u>Van Laar Equations</u>				<u>Margules Equations</u>			
<u>x₁</u>	<u>y₁</u>	<u>x₁</u>	<u>y₁</u>	<u>Y₁</u>	<u>T°F.</u>	<u>x₁</u>	<u>y₁</u>	<u>Y₁</u>	<u>T°F.</u>	<u>x₁</u>	<u>y₁</u>	<u>Y₁</u>	<u>T°F.</u>
45.5	47.5	.882	.923	1.025	188°	.882	.905	1.01	187°	.882	.907	1.011	188°
38.0	42.9	.739	.831	1.061	190	.739	.815	1.05	190	.739	.823	1.061	189
29.6	37.6	.575	.731	1.149	193	.575	.722	1.135	193	.575	.735	1.15	193
29.0	37.4	.564	.725	1.145	194	.564	.716	1.145	193	.564	.730	1.15	194
20.0	31.2	.388	.606	1.275	200	.388	.606	1.255	199	.388	.606	1.28	199
14.5	26.0	.281	.505	1.375	204	.281	.508	1.40	203	.281	.502	1.35	205
9.6	20.4	.186	.396	1.51	209	.186	.396	1.52	209	.186	.382	1.412	210
6.3	15.1	.122	.293	1.605	213	.122	.298	1.615	213	.122	.271	1.45	214
1.4	4.6	.027	.089	1.985	220	.027	.081	1.76	222	.027	.070	1.49	223

A = 0.892

B = 225

b = 1.055

c = -.655

TABLE NO. 3
Vapor-Liquid Equilibria at
4.0 Atmosphere Pressure

ml. of NaOH for 1 ml. Aliquot		Experimental				Van Laar Equations				Margules Equations			
x_1	y_1	x_1	y_1	δ_1	T°F.	x_1	y_1	δ_1	T°F.	x_1	y_1	γ_1	T°F.
45.1	47.4	.875	.920	1.025	219°	.875	.901	1.01	219°	.875	.892	1.01	218°
39.3	42.7	.762	.830	1.025	222	.762	.830	1.03	222	.762	.823	1.041	220
31.8	38.1	.616	.740	1.102	224	.616	.725	1.08	224	.616	.734	1.1	224
30.8	37.8	.596	.733	1.112	225	.596	.714	1.088	225	.596	.722	1.11	224
22.0	31.0	.426	.601	1.215	229	.426	.585	1.17	230	.426	.594	1.195	229
17.2	26.4	.334	.511	1.25	233	.334	.502	1.225	233	.334	.508	1.241	234
11.5	19.3	.222	.385	1.32	238	.222	.379	1.305	238	.222	.369	1.25	239
7.4	14.0	.141	.271	1.405	242	.141	.268	1.38	242	.141	.261	1.32	243
1.8	4.0	.035	.078	1.505	248	.035	.078	1.46	249	.035	.07	1.34	250

$$A = 0.84$$

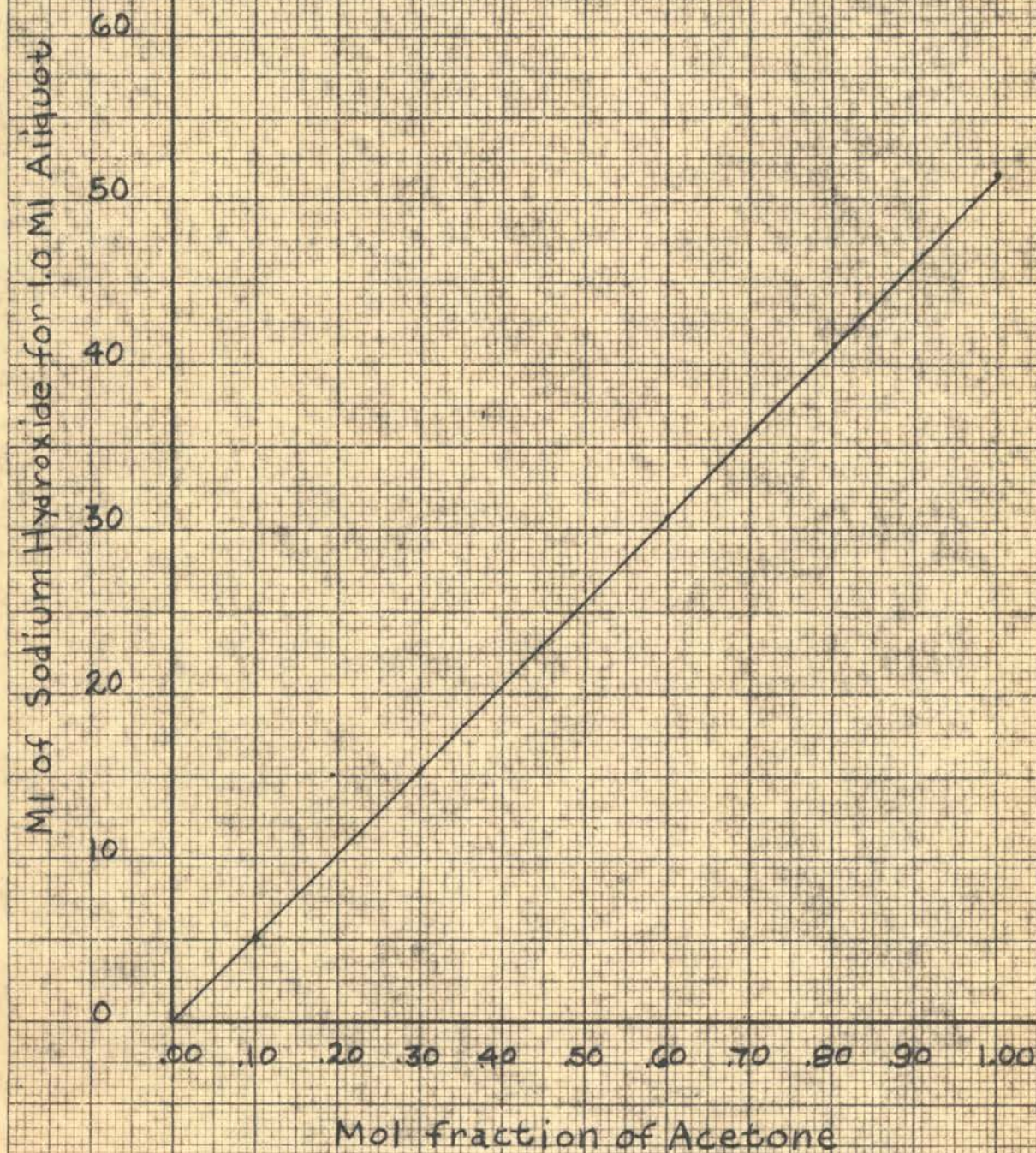
$$B = 158.5$$

$$b = .871$$

$$c = -.578$$

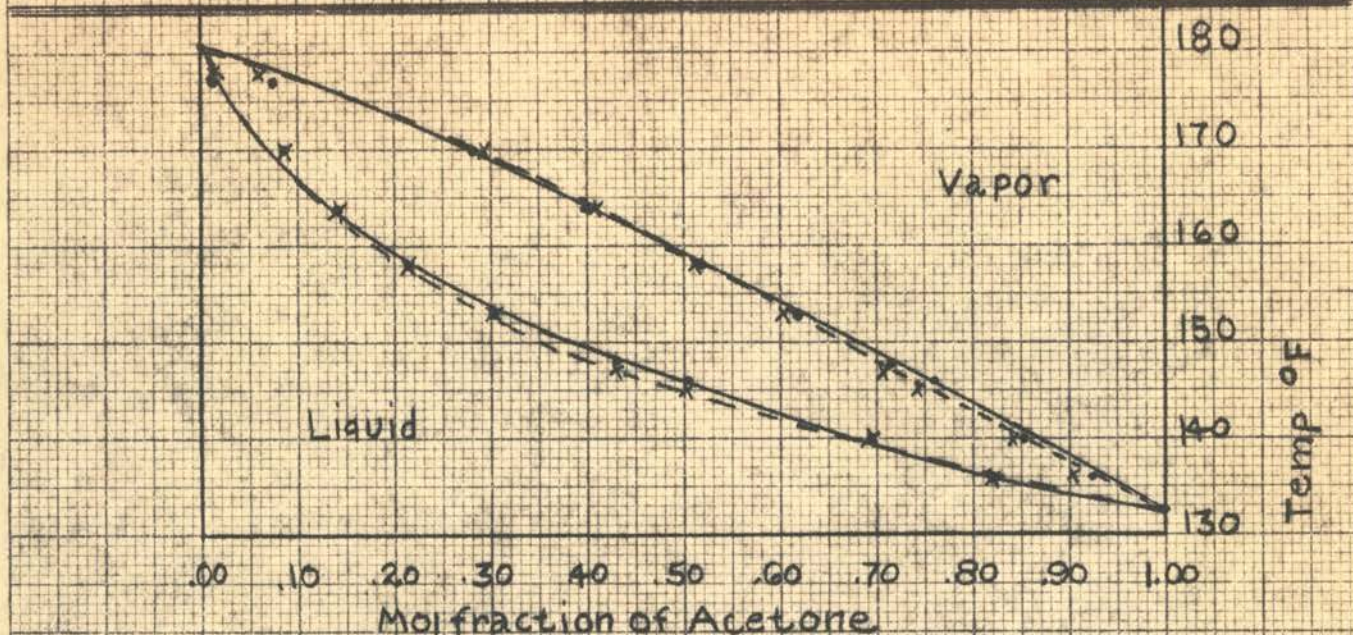
GRAPH No. 1

Sodium Hydroxide Equivalent to Acetone
in 1.0 ml Synthetic Sample

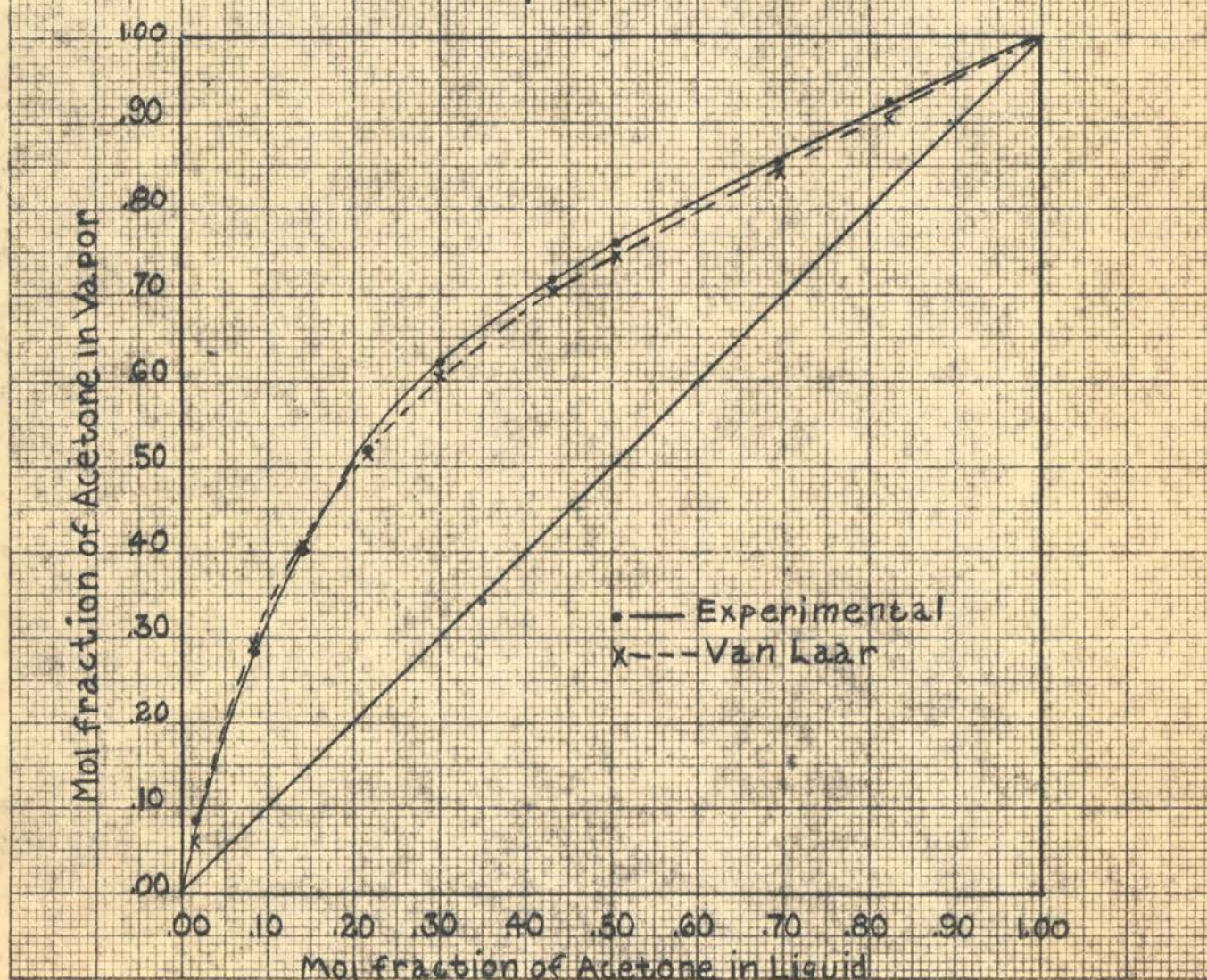


GRAPH No. 2

Van Laar Equation & Experimental Data

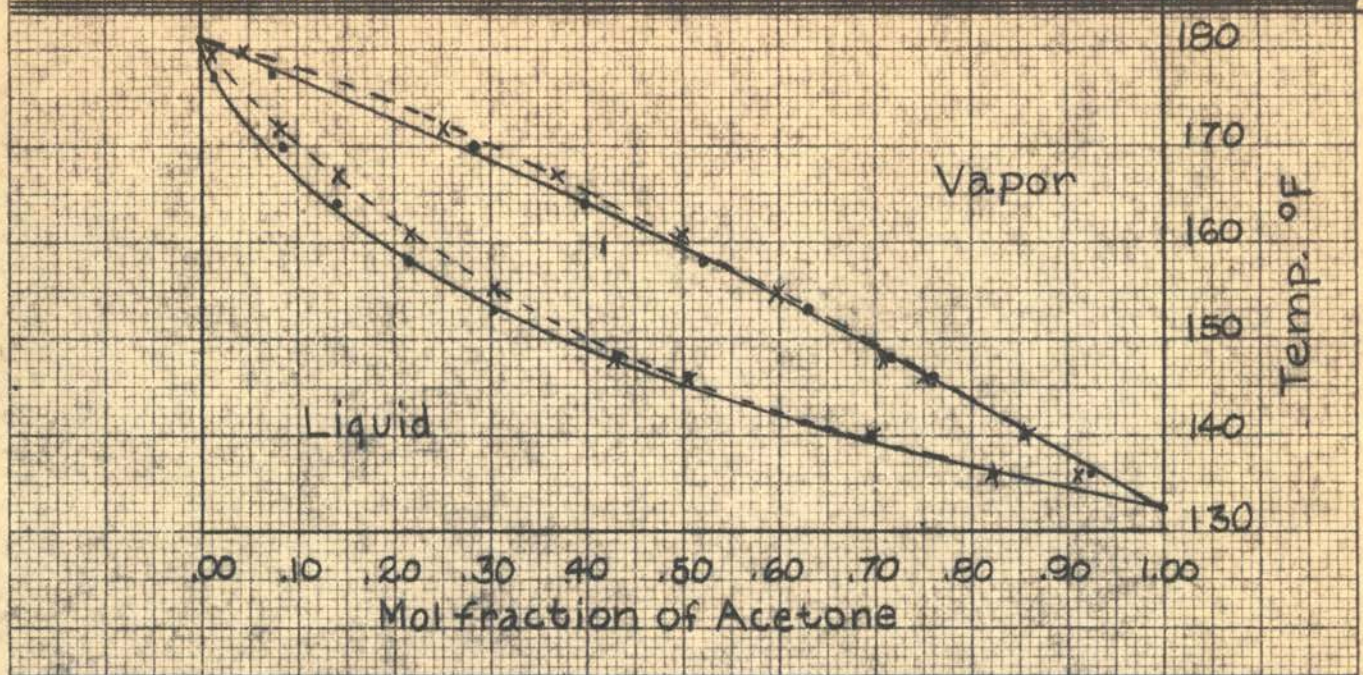


1 Atmosphere Pressure

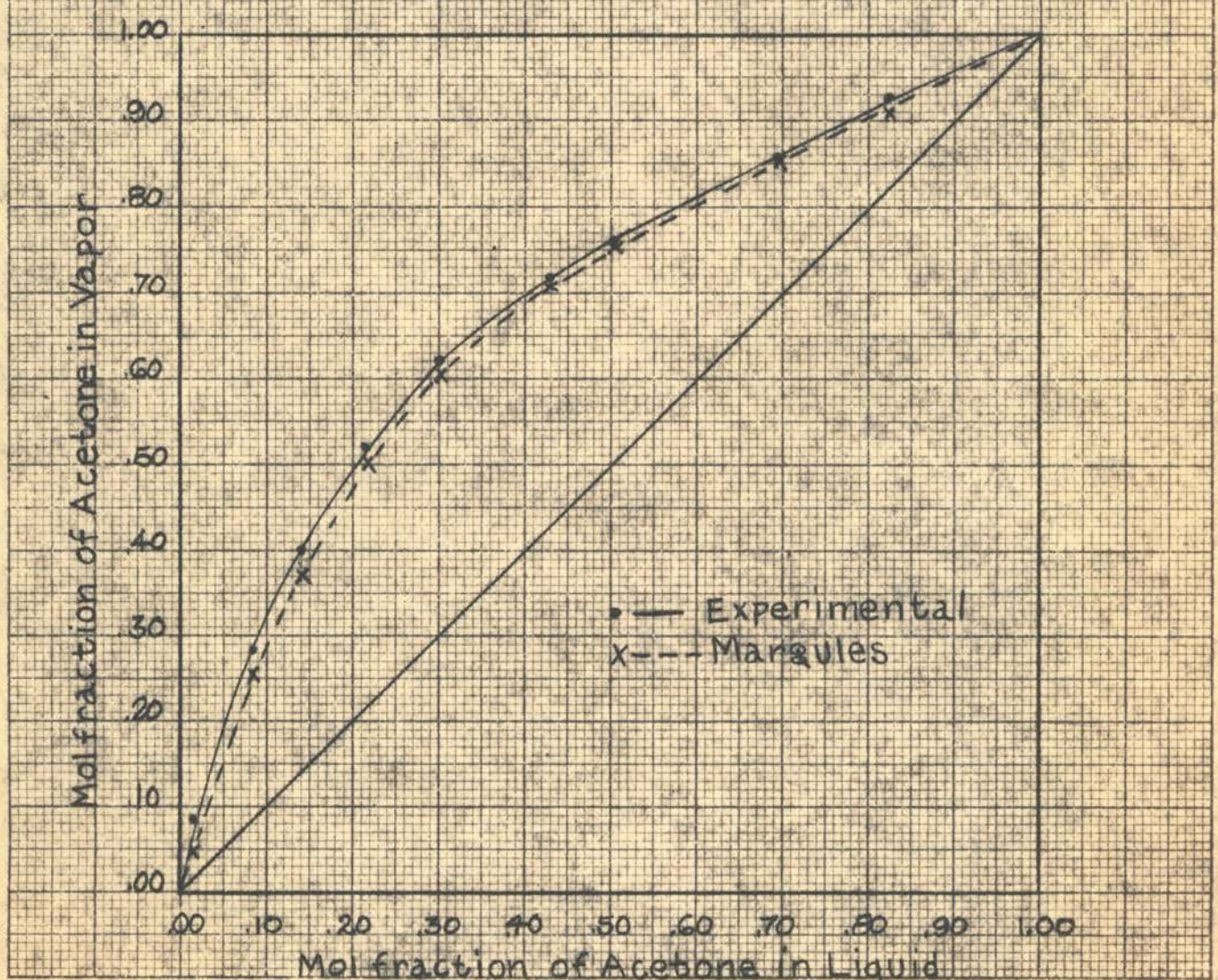


GRAPH No. 3

Margules Equations & Experimental Data

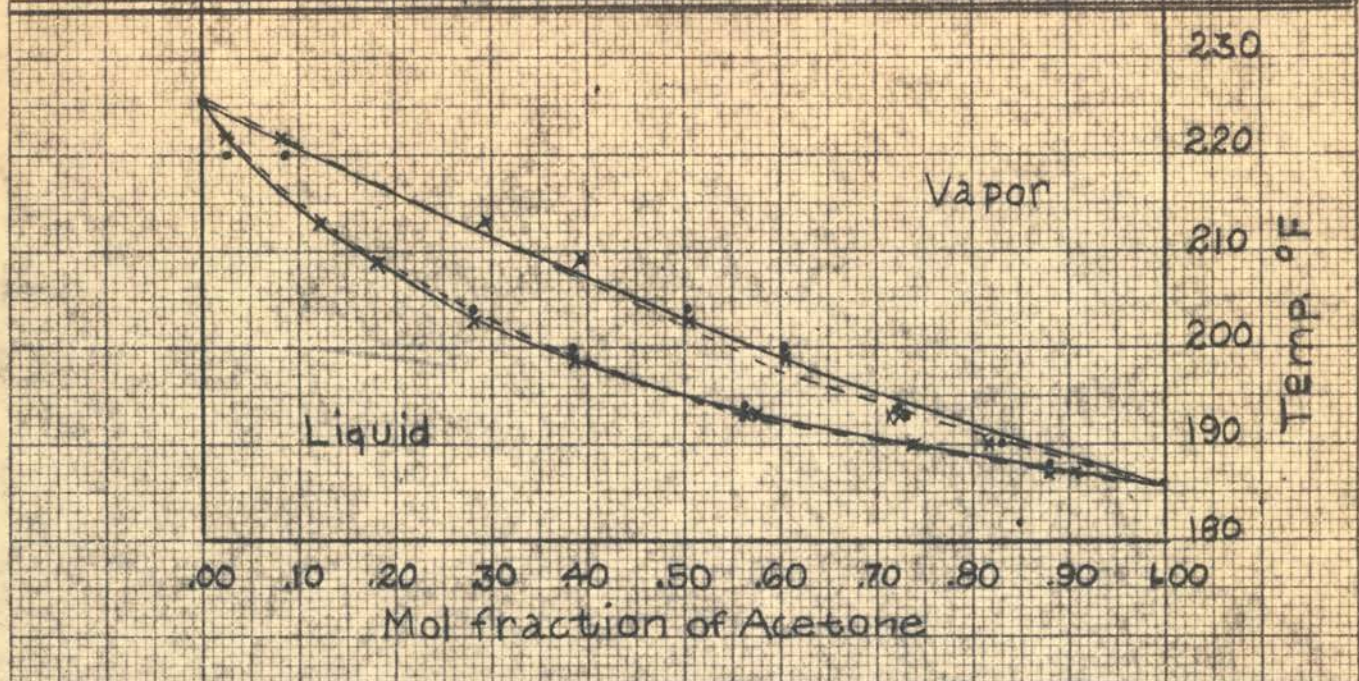


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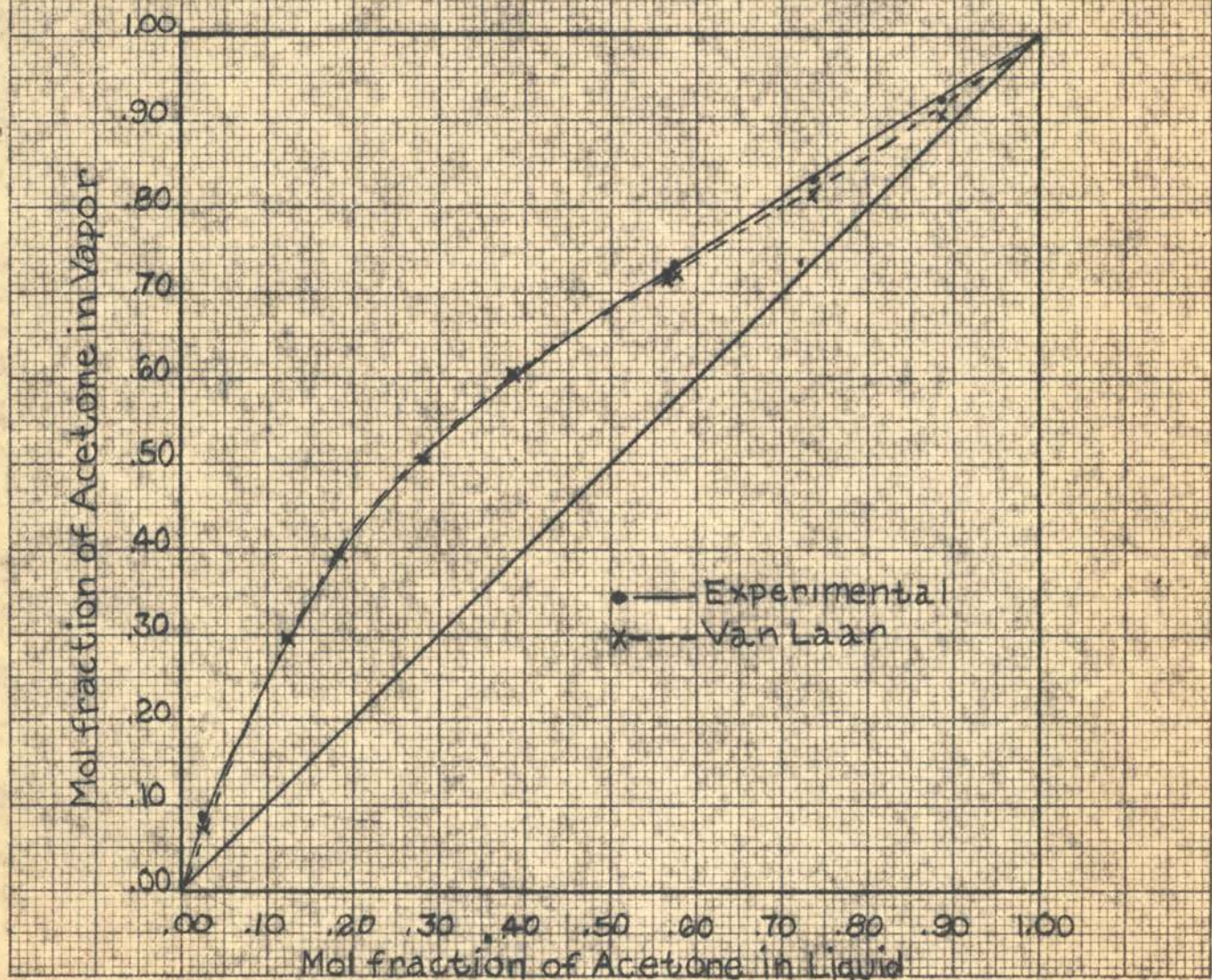


GRAPH No. 4

Van Laar Equation & Experimental Data

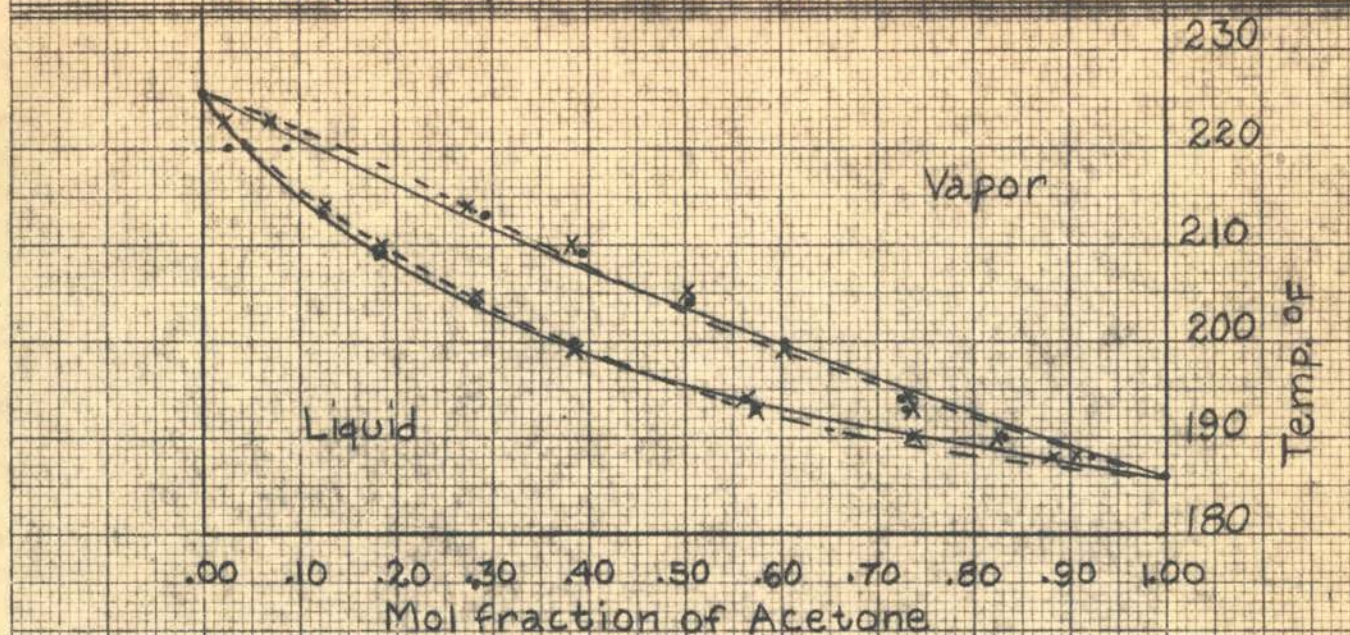


2.5 Atmospheres Pressure

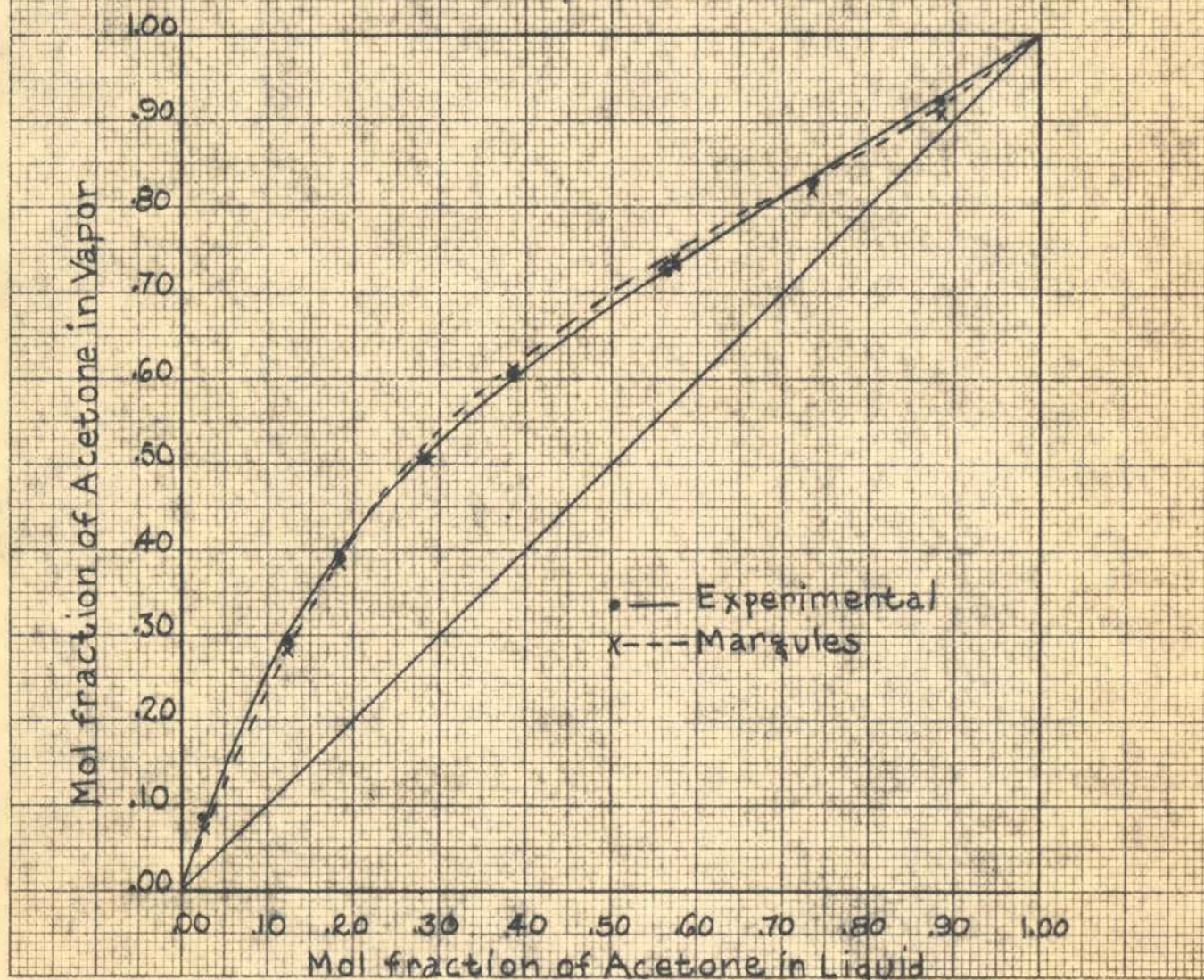


GRAPH No. 5

Margules Equations & Experimental Data

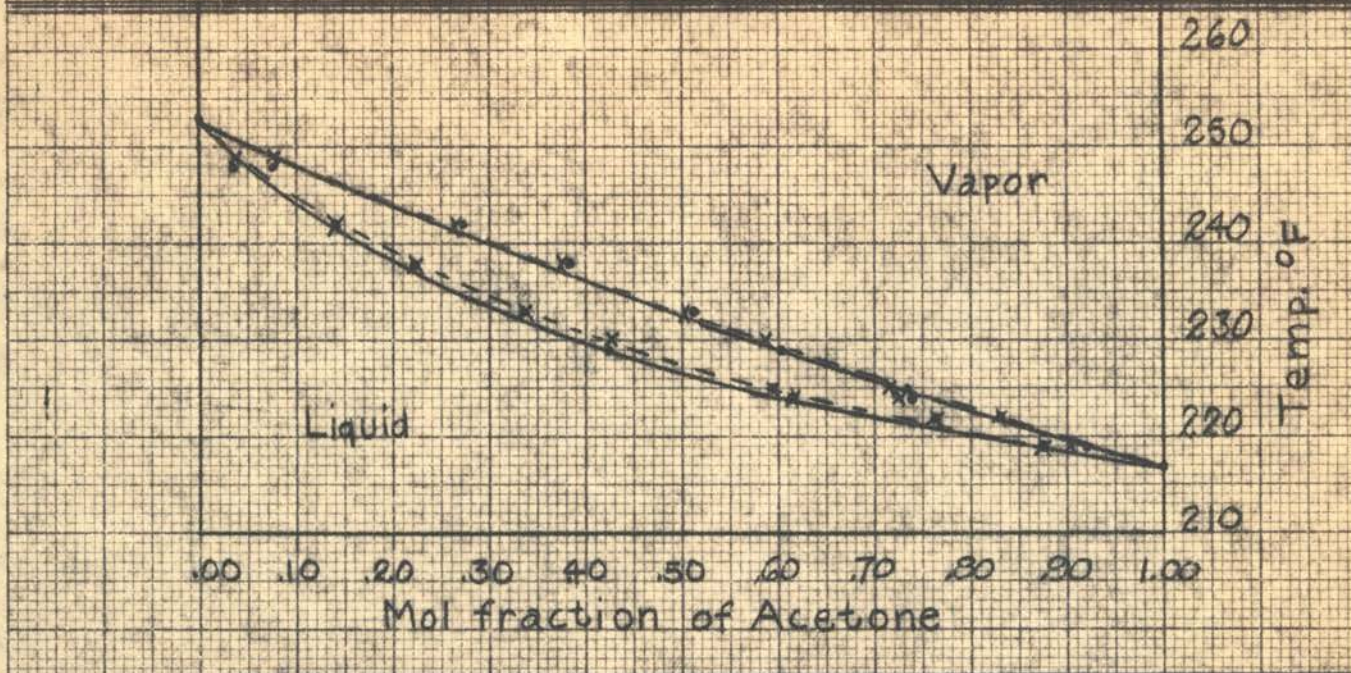


2.5 Atmospheres Pressure

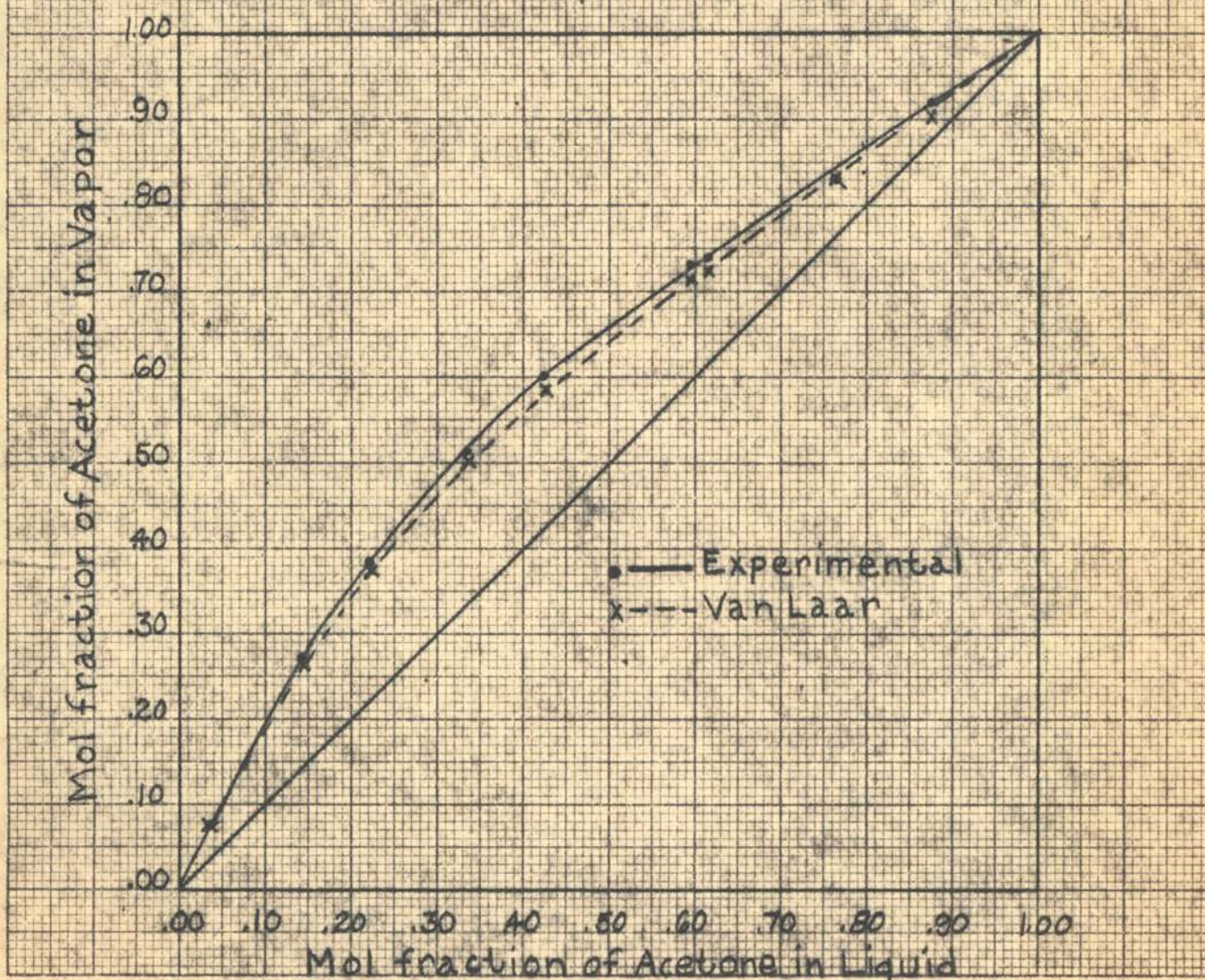


GRAPH No. 6

Van Laar Equation & Experimental Data

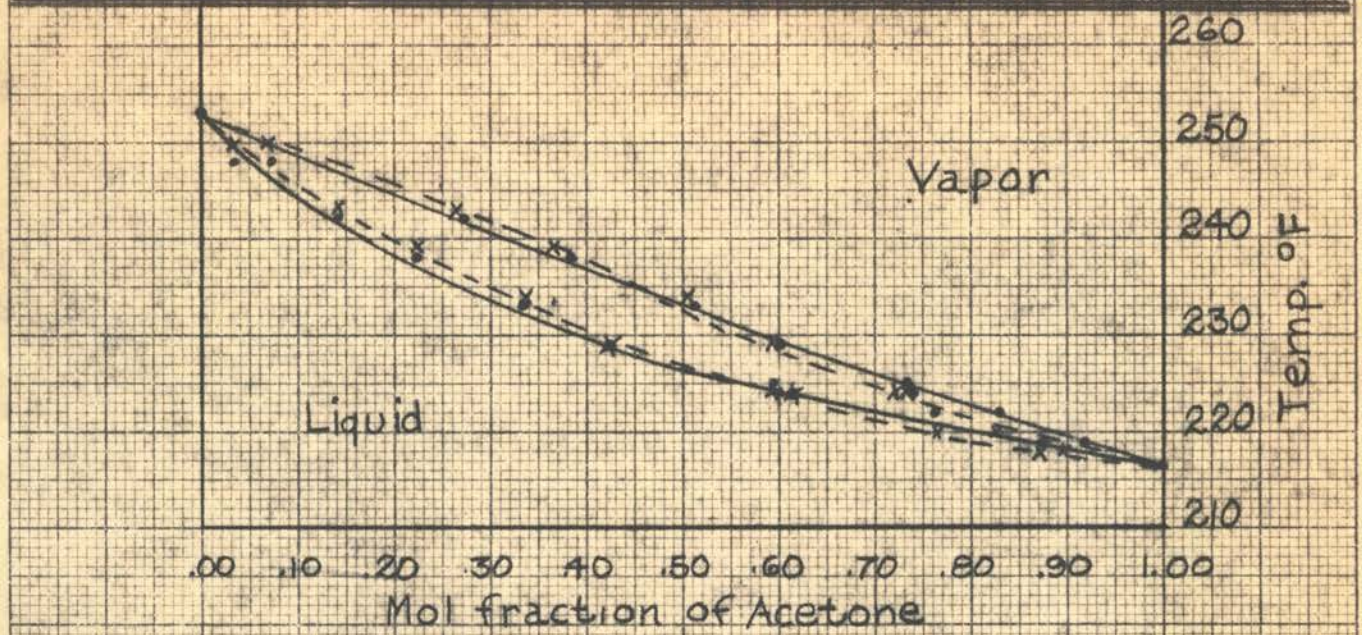


4.0 Atmospheres Pressure

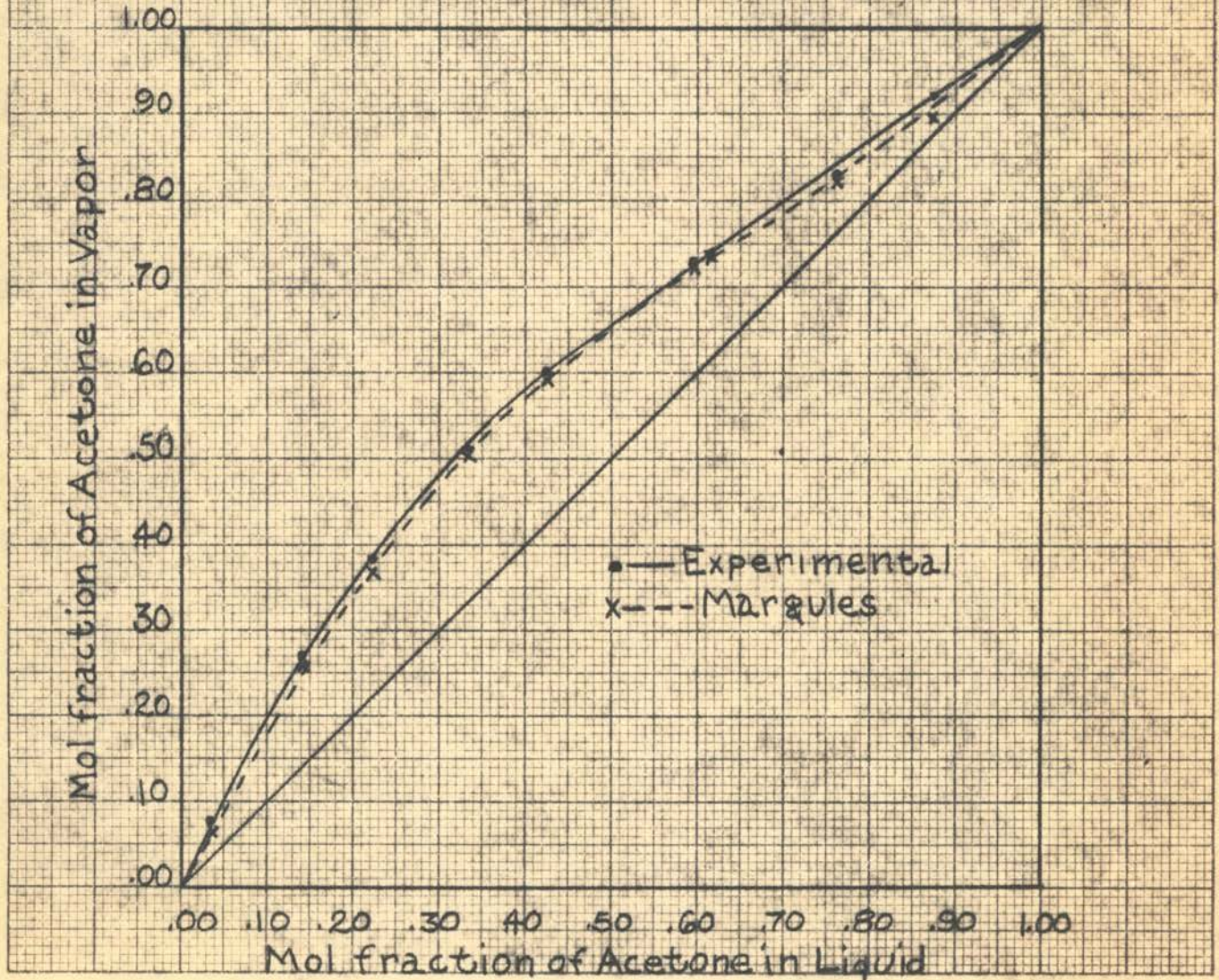


GRAPH No. 7

Margules Equations & Experimental Data



4.0 Atmospheres Pressure



GRAPH No. 8

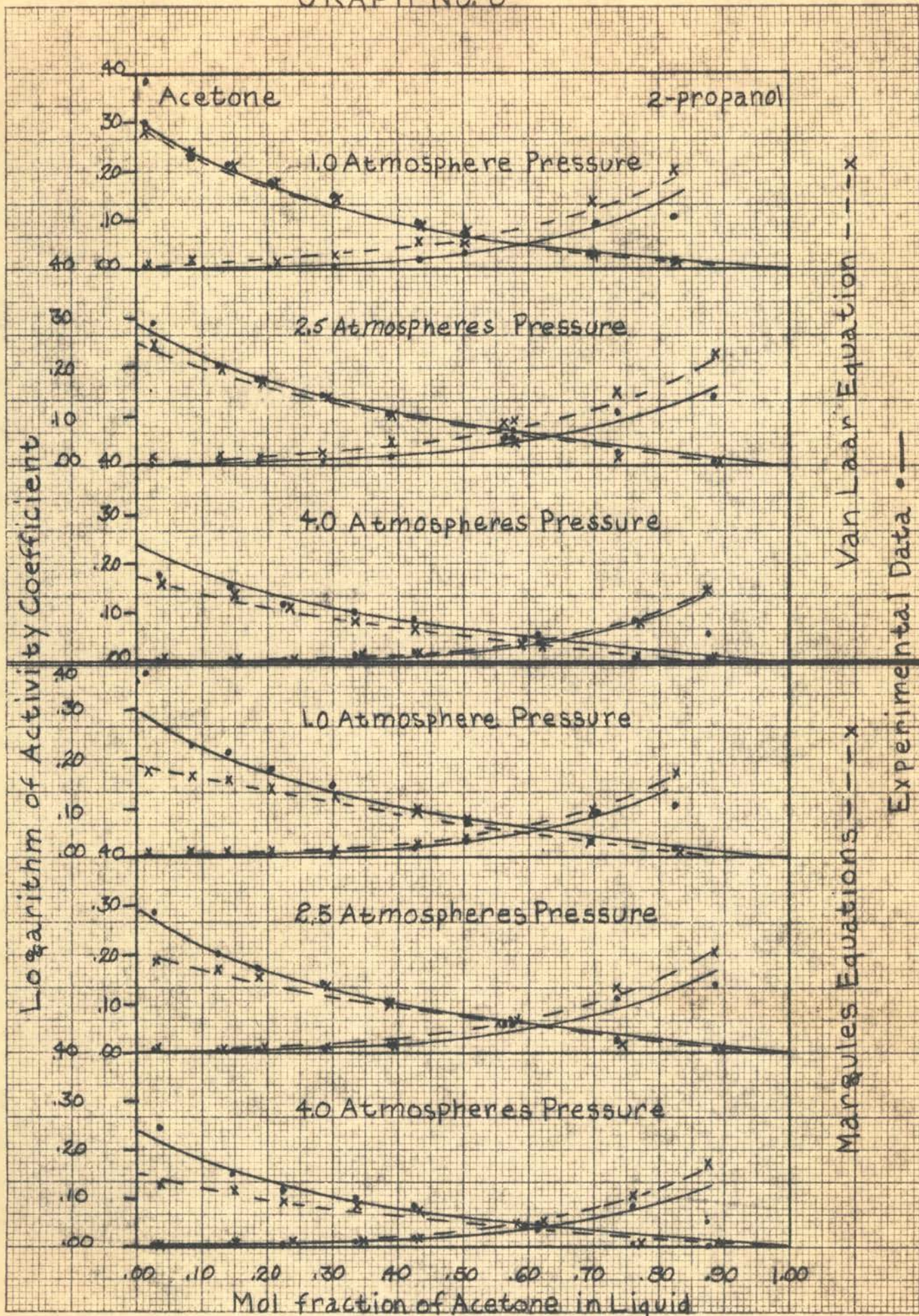
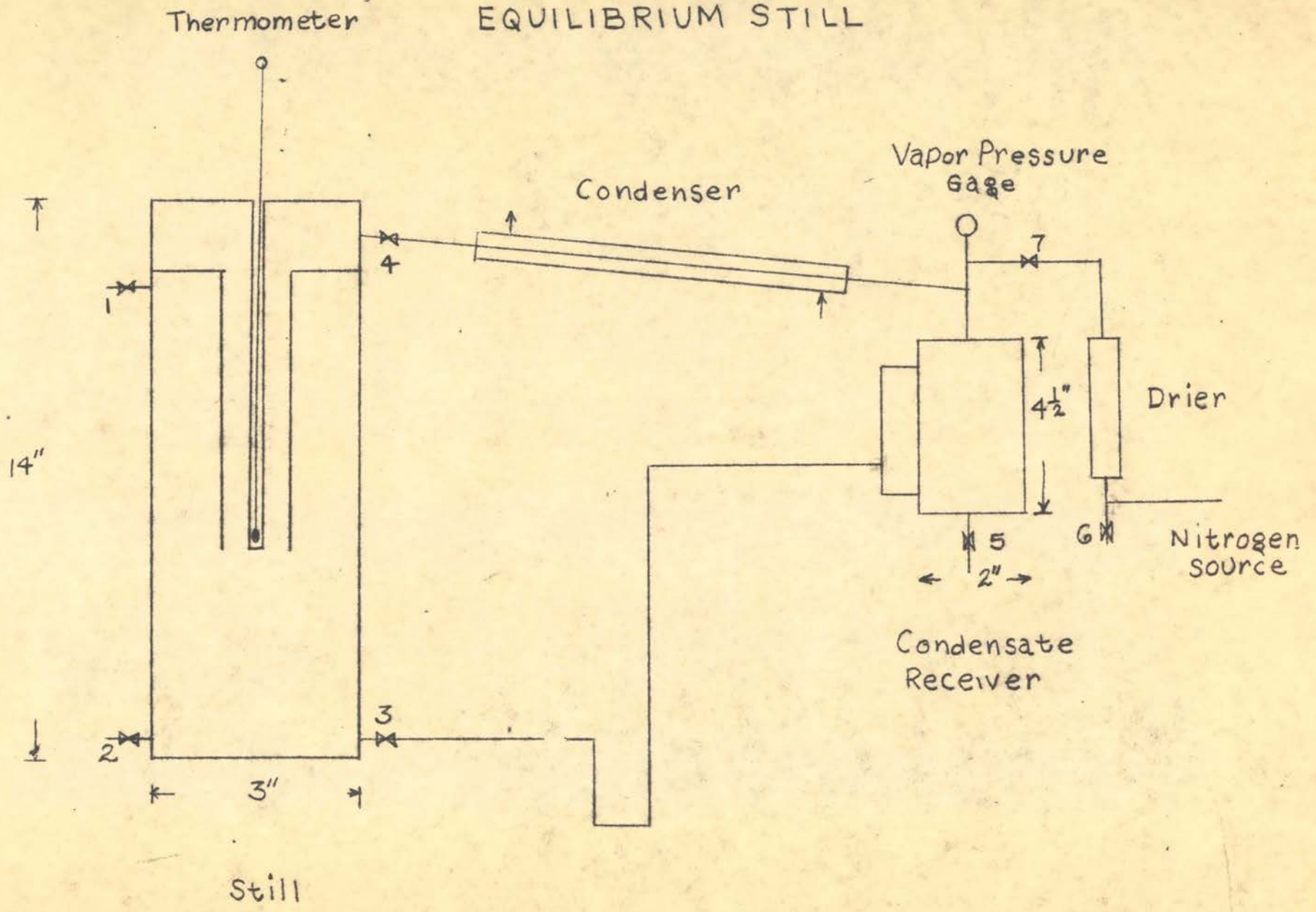


FIGURE No 1
EQUILIBRIUM STILL



Method of Least Squares for Evaluating
Van Laar Constants at 2.5 Atmospheres Pressure

x_1	y_1	T ^o P.	p_1^o	$\gamma_1 = \frac{f y_1}{P_1^o x_1}$	$\frac{1}{\sqrt{T} \ln \delta_1}$	$\frac{x_1}{x_2}$
.882	.923	188 ^o	2.55	1.025	.332	7.47
.739	.831	190	2.65	1.061	.215	2.83
.575	.731	193	2.77	1.149	.1415	1.355
.564	.725	194	2.81	1.145	.1425	1.295
.388	.606	200	3.07	1.275	.106	.634
.281	.505	204	3.27	1.375	.0925	.391
.186	.396	209	3.53	1.51	.0811	.229
.122	.293	213	3.75	1.605	.0765	.139
.027	.089	220	4.15	1.985	.0524	.0278

From the equation for a straight line, $y = a + bx$, a and b are constants for the equation so that the sum of the squares of the differences of the experimental values of y and values of y calculated from $a + bx$ will be a minimum, i.e.,

$$S = (y_1 - a - bx_1)^2 + (y_2 - a - bx_2)^2$$

$$\frac{\partial S}{\partial a} = 0 \text{ and } \frac{\partial S}{\partial b} = 0$$

this reduces to:

$$-S(y) + aS(1) + bS(x) = 0$$

$$-S(xy) + aS(x) + bS(x^2) = 0$$

$$\text{let } y = \frac{1}{\sqrt{1 + \ln \gamma_1}} \text{ and } x = \frac{x_1}{x_2}$$

x^2	x	y	xy
1.84	1.35	.141	.192
1.69	1.30	.142	.186
.40	.63	.106	.068
.15	.39	.093	.037
.05	.25	.081	.019
<u>.02</u>	<u>.14</u>	<u>.077</u>	<u>.011</u>
4.15	4.04	.640	.513

$$-.64 + 6a + 4.04b = 0$$

$$-.513 + a4.04 + 4.15b = 0$$

$$a = .0666$$

$$b = .0595$$

$$B^{1/2} = \frac{1}{a} = 15$$

$$B = 225$$

$$A = b \times B^{1/2} = .0595 \times 15 = .892$$

$$\ln \gamma_1 = \frac{\frac{225}{T}}{\left(1 + .892 \frac{x_1}{x_2}\right)^2}$$

Method of Least Squares for Evaluating
 Margules Constants at 2.5 Atmospheres Pressure

x_1	y_1	x_2	y_2	T°F.	P_2^*	$\gamma_2 = \frac{Py_2}{x_2 P_2^*}$	$\frac{\ln \gamma_2}{x_1^2}$	$\frac{\ln \gamma_1}{x_2^2}$	$x_2 + .5$
.862	1.025	.118	.077	188°	910mm	1.362	.395	1.81	.618
.739	1.061	.261	.169	190	950	1.295	.472	.876	.761
.525	1.149	.425	.269	193	1010	1.19	.519	.763	.925
.564	1.145	.436	.275	194	1030	1.165	.478	.715	.936
.388	1.275	.612	.394	200	1180	1.035	.229	.650	1.112
.281	1.375	.719	.495	204	1260	1.04	.67	.611	1.219
.186	1.51	.814	.604	209	1400	1.009	.1995	.633	1.314
.122	1.605	.878	.707	213	1500	1.02	.1235	.610	1.378
.027	1.985	.973	.911	220	1780	1.00	-0-	.720	1.473

let $y = \frac{\ln X}{x^2}$ and $x = x_2$ and $x_2 + .5$

x^2	x	y	xy
.063	.261	.676	.228
.181	.425	.763	.325
.190	.436	.715	.312
.375	.612	.650	.398
.519	.719	.611	.439
.661	.814	.622	.505
.771	.878	.610	.535
.580	.761	.472	.360
.858	.925	.519	.480
.880	.936	.478	.447
1.24	1.112	.229	.255
1.73	1.314	.1995	.262
<u>2.18</u>	<u>1.473</u>	<u>-.0</u>	<u>-.0</u>
10.233	10.666	6.745	4.546

From method of least squares:

$$-6.74 + 13a + b \cdot 10.67 = 0$$

$$-4.55 + 10.67 a + b \cdot 10.23 = 0$$

$$a = 1.055$$

$$b = -.655$$

$$\text{slope} = c = -.655$$

$$\text{intercept} = b = 1.055$$

$$\ln X_1 = bx_2^2 + cx_2^3 = x_2^2 (1.055 - .655 x_2)$$

$$\ln X_2 = bx_2^2 + \frac{3}{2} cx_2^2 - cx_2^3 = x_2^2 (.073 + .655x_2)$$

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