PLATE EFFICIENCIES OF A KOCH KASKADE FRACTIONATOR USING THE SYSTEM METHANOL-WATER

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SUMMARY

This investigation was concerned with determination of over-all plate efficiencies of a Koch KASKADE tray. Equipment used by Thompson[7] in his study of the system cyclohexane-isooctane was modified slightly for use with the system methanol-water.

The column studied was 14 inches in diameter and contained five trays. The piping was so arranged that rectifying, stripping, and total reflux operation could be investigated.

A procedure for the analysis of methanol-water solutions was developed involving the use of refractive index and density measurements. Vapor-liquid equilibrium data, as obtained from the literature[1], were revised by calculation to employ a fictitious molecular weight of 39 for methanol so that heats of vaporization of methanol and water would be equalized.

Over-All plate efficiencies were determined at superficial vapor velocities in the range of 1.2 to 9.1 feet per second, liquid loadings in the range of 0.7 to 11.2 gallons per minute per square foot of tower cross sectional area, and liquid to vapor ratios ranging from 0.28 to 4.78. These efficiencies ranged from 84% at total reflux to 25% under stripping conditions, appearing to decrease with an increase in liquid loading and increase with an increase in vapor loading. Liquid to vapor ratio appeared to have a profound effect on efficiency in that high efficiencies were obtained when L/V was 1.0, and relatively low efficiencies were obtained at L/V below and above 1.0.

INTRODUCTION

When operated under conditions of heavy liquid loads, bubble-cap columns become inefficient. The manufacturer of the Koch KASKADE tray claims to overcome this disadvantage by a radical design. KASKADE trays can be used in various installations where bubble-cap trays are inapplicable.

At the present time there are very little data in the literature on the operational characteristics of the Koch KASKADE tray[2][3][7][9]. Therefore one of the primary objectives of this investigation was to extend the existing knowledge of this tray. The classical system methanol-water was chosen as a basis for this investigation.

STATEMENT OF THE PROBLEM

The primary object of the investigation was to extend the limited knowledge on the performance of a Koch KASKADE tray in distillation. The problem resolved into the following steps:

1. The development of an accurate, rapid method of analysis for the system methanol and water.
2. The modification of existing equipment for use with this system.
3. The calibration of flow devices.
4. The determination of over-all plate efficiencies as affected by liquid to vapor ratio, liquid loading, and vapor loading.

SOURCE OF MATERIALS

1. METHANOL - The methanol used in this investigation was commercial-grade methanol. Fractionation of the stock in a five-plate laboratory still at approximately two to one reflux ratio resulted in no change in refractive index.
2. WATER - The water used in this investigation was steam condensate obtained from the steam header. Refractive index of this condensate was found to be the same as for distilled water.

PROCEDURE

Preliminary Run

The equipment as used by Thompson[7] was operated with water to determine what modifications were needed before the system methanol-water could be investigated. The high latent heat of vaporization and low specific volume of the methanol-water liquid system necessitated the revision of the flow measuring equipment and recalibration. Miscellaneous piping was revised and the pumps were repacked with a methanol resistant packing. The resulting flow diagram of the modified unit is presented in Figure 1.

Start-Up Procedure

In all cases, the column was first started up under total reflux. The following steps were taken to bring the column to steady operating conditions:

1. Water to the condenser was started (valve C-5).
2. Reboiler circulation pump was put into operation (pump No. 2).
3. The vent on the reboiler steam side (valve C-8) was opened, and the condensate line was opened to the condensate return line(valve C-10), which returned condensate to the power plant.
4. Valves C-15, C-11, and C-12 were opened. Steam was started to the reboiler and was slowly increased (Pressure Controller PC-1) until the over-head temperature indicated that vapor was entering the condenser.
5. The reflux pump (pump No. 1) was started, and valves C-1 and C-2 were opened slightly.
6. Steam was increased, and valve C-1 was adjusted to hold level in the surge drum constant.

7. Steam was adjusted by weights on PC-1 to give the desired boil-up rate.

Total Reflux Operation

To operate the column under total reflux, valves C-3 and C-7 were closed, and valve C-1 was adjusted to give a constant level in the surge drum. When the level in the surge drum remained constant for one hour without readjustment of valve C-1, the run was started. Figure 2 shows the flow of material during a total reflux run.

Stripping Operation

To operate the column as a stripper, the unit was first brought to steady operation as a total reflux run. Then valve C-1 was opened so that the rate of flow from the surge drum was greater than the flow to it from the overhead of the column. This deficit was made up by addition of material from the reboiler by opening valve C-7 and adjusting until the level in the surge drum became constant. After one hour of steady operation without readjustment of either valves C-1 or C-7, the stripping run was started. The flow of materials in a stripping run is shown in Figure 3.

Rectifying Operation

To operate the column as a rectifier, the unit was brought to steady operation as a total reflux run. Then valve C-1 was closed down so that the rate of flow leaving the surge drum was lower than flow entering it from the overhead of the column. The level in the surge drum was maintained constant by opening valve C-3 which transferred material from the surge drum to the reboiler. When the level had remained constant for one hour without readjustment of either valves C-1 or C-3, the rectifying run was started. The flow of material during a rectifying run is shown in Figure 4.

Sampling and Data Procedure

When steady operation was established, the run was started by opening valve C-9, closing valve C-10, and starting the stop watch. Temperatures, flows, and pressures were recorded. The samples were taken, starting at the top of the column and working down. The temperatures, flows, and pressures were again recorded. The run was ended by closing valve C-9, opening valve C-10, and stopping the stop watch. The weight of steam condensate collected during the run was recorded.

Sampling Technique

Liquids and vapors were sampled through sample ports equipped with a copper coil, which was immersed in a water-ice mixture to prevent excessive air stripping of the samples. The coils were flushed out with the first ten milliliters of sample. The final sample of 25 milliliters was collected in a sample vial and stoppered tightly. Sampling points in the column are shown in Figure 5.

Analytical Procedure

The large number of samples taken in this investigation necessitated the use of a rapid method of analysis. However, the method needed to be sensitive, since small errors in composition would result in large errors in plate efficiencies.

Refractive index measurements offered a rapid and sufficiently accurate method of analysis for the complete range of composition of the methanol-water system with the exception of the range 22 to

49 true mol percent methanol. This is illustrated in Figure 6. Density measurements were made when the samples fell within this range.

Refractive Index Measurement

The refractive index of a sample was measured with a dipping refractometer in a water bath maintained at 25.0° C. ± 0.1°. Air stripping of the sample usually occurred, so the sample was cooled in its sample bottle before introducing it to the refractometer cell.

The calibration curve, Figure 6, of scale reading versus composition was obtained from solutions of known composition. Since two values of composition were represented by the same scale reading, it was necessary to determine which composition was correct. This was done very easily by the addition of a drop of water to the sample and observing whether the scale reading increased or decreased.

Density Measurement

When the refractive index of a sample was such that the scale reading of the refractometer was greater than 33.0, it was returned to its sample bottle and to the cooling bath for a density measurement. The density measurements in this investigation were made with tared ten milliliter pycnometers weighed on an analytical balance. The density-composition relation presented in Figure 7 was obtained from solutions of known composition at 25° C.

RESULTS

The calibration curves for refractive index-composition and density-composition appear in Figures 6 and 7 respectively. The analysis by either measurement is sensitive to composition differences of ± 0.2 mol percent. Table I presents the analytical results of this investigation. For convenience, the table is divided into total reflux (t) runs, rectifying (r) runs, and stripping (s) runs.

Since flows were much smaller than those encountered in previous work(7), new orifice plates were cut, put in place and calibrated. Figure 8 presents the calibration curves for the tower orifices, and Figure 9 presents the calibration curve for water to the condenser.

Operational and calculated data for this investigation are presented in Table II. Over-all plate efficiencies were calculated by the McCabe-Thiele method(4) using a fictitious molecular weight of 39 for methanol to equalize the molar latent heats of vaporization of the two components. Figure 10 is a calculated curve for converting from true to fictitious mol percent. Figure 11 is the resulting vapor-liquid equilibrium curve incorporating fictitious mol percent. For purpose of comparison, the true mol percent vapor-liquid equilibrium diagram is presented in Figure 11 also(1).

Figures 12, 13, and 14 illustrate typical McCabe-Thiele plots used in calculating theoretical plate requirements for total reflux, rectifying, and stripping runs. Although pinching occurred, all trays were used for calculation of efficiency except the bottom tray. The operating lines were constructed with their calculated slope through the experimental points of passing liquid and vapor streams.

Since orifices measure flow on a volume flow basis, conversion to mol flow basis was necessary. Figure 15 is a calculated curve of pound mols per pound versus composition. Used in conjunction with Figure 16, conversion from volume flow to mol flow basis was easily accomplished. Specific heat and heats of vaporization of methanol-water solutions were obtained from Perry[5][6].

Heat balances made across the column, without correction for insulation or radiation losses, appear in Table III.

Plots of over-all plate efficiencies versus L/V, L, V, and v are presented in Figures 17, 18, 19, and 20. Although the points scatter widely, it is believed that correlation exists.

DISCUSSION

Thompson[7], in his investigation of the cyclohexane-isooctane system, found:

1. That liquid and vapor loading have about an equal effect on over-all plate efficiency and that this effect was a decreased efficiency with an increased loading,
2. That L/V has no detectable effect on efficiency, and
3. That over-all efficiency is lower with stripping operation than with total reflux operation.

In this investigation with the system methanol-water, the following conclusions can be reached:

1. The effect of an increased liquid loading is a decrease in efficiency, while the opposite is true with an increased vapor loading,
2. That L/V has a definite effect on over-all efficiency, and
3. That stripping operation is less efficient than either rectification or total reflux operation.

Although these conclusions drawn from each investigation appear to contradict one another, the following considerations should be made: that liquid loading expressed volumetrically is much greater in the cyclohexane-isooctane system than in the methanol-water system; that while expressed on a mol basis the vapor loadings are directly comparable, the corresponding liquid load is still greatly different. In other words, on a volume basis, the investigation of the methanol-water system was in a range much lower than that covered in the cyclohexane-isooctane investigation. The maximum liquid loading obtained in this investigation was 11.2 gallons per minute per square foot, which is close to the minimum liquid loading of eight gallons per minute per square foot as investigated by Thompson[7].

Comparison of the performance of the Koch KASKADE tray with that of a bubble-cap tray is difficult, in that plate efficiencies of bubble-cap trays vary considerably from investigator to investigator. However, qualitative comparison might be based on a recent investigation. Williams et al.[8] investigated plate efficiencies of a bubble-cap column ten inches in diameter and reported efficiencies of 32.5 to 58.6 % while using the methanol-water system. Vapor velocities ranged from 0.63 to 1.16 feet per second and L/V ranged from 0.63 to 2.4. Higher plate efficiencies were obtained with stripping operation than with total reflux or rectifying operation.

Comparison with the results of this investigation shows that the Koch KASKADE tray has a higher plate efficiency than a bubble-cap in rectification or total reflux operation, but is less efficient in stripping operation. The Koch KASKADE tray has a much higher capacity than the bubble-cap tray.

The heat input appeared to balance very well with the heat removal as can be seen in Table III. This would indicate that no operational upsets occurred during each run.

FUTURE WORK

This investigation was a small part of the work that could be done on the Koch KASKADE tray. The high throughput and low pressure drop characteristics are of particular interest in the field of vacuum fractionation of heat-sensitive or polymer-forming materials. Therefore, if future work is intended with this tray, it is suggested that consideration be given to the determination of over-all plate efficiencies, pressure drop, and throughputs in vacuum distillation.

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NOMENCLATURE

c_p - Specific heat of reflux, BTU/lb./°F.

c_{pm} - Heat capacity of Bottoms, BTU/lb. mol/°F.

d - Density of reflux, lb./gal.

E - Percent over-all plate efficiency, 100 (Number of actual plates/Number of theoretical plates).

L - Liquid loading, lb. mol/sec. (Fictitious).

ΔL_5 - Increased liquid loading due to sub-cooled reflux, lb. mols/sec. (Fictitious).

m - Fictitious mols/lb. reflux.

M_1 - Flow of cooling water, lb./min.

M_R - Average fictitious molecular weight of reflux, lb./lb. mol.

M_v - Average fictitious molecular weight of bottoms, lb./lb. mol.

P_1 - Pressure in tower bottoms, psig.

P_2 - Pressure of steam entering reboiler, psig.

P_3 - Pressure of source steam, psig.

P_4 - Discharge pressure of circulation pump, psig.

P_5 - Pressure in tower overhead, psig.

$P_{avg.}$ - Average tower pressure, psia = $\frac{P_1 + P_5}{2} + 14.4$.

R - Reflux flow, lb. mol/sec. (Fictitious).

R' - Reflux flow, gal./min.

S_n - Net steam consumption, lb./sec.

T_1 - Temperature of reflux stream, °F.

T_2 - Temperature of vapor leaving the tower, °F.

T_3 - Temperature of product leaving condenser, °F.

T_4 - Temperature of cooling water entering condenser, °F.

T_5 - Temperature of cooling water leaving condenser, °F.

T_6 - Temperature of tower bottoms, ^{o}F.

T7 - Temperature in throttling calorimeter.

$T_{avg.}$ - Average tower temperature, $^{o}R. = \frac{T_2 + T_6}{2} + 460.$

V - Vapor loading, lb. mol/sec. (Fictitious).

v - Superficial vapor velocity, ft./sec.

λ_s - Latent heat of vaporization of steam at P_2, BTU/lb.

λ_t - Latent heat of vaporization at top of column at T_2, BTU/lb. mol. (Fictitious).

λ_v - Latent heat of vaporization of bottoms at P_1, BTU/lb.

x_a - Composition of liquid leaving plate a, fictitious mol fraction.

x_b - Composition of liquid in reboiler, fictitious mol fraction.

x_R - Composition of liquid reflux, fictitious mol fraction.

y_a - Composition of vapor leaving plate a, fictitious mol fraction.

APPENDIX

Description of Equipment

The equipment used in this investigation is adequately described by Thompson[7]. For convenience, the description of major equipment will be repeated.

1. Column.

The column was a five-tray, Koch KASCADE type, 14 inches in diameter. Tray spacing was 24 inches and the weir length eight inches. Detailed drawing of the column and tray is presented by Thompson[7].

2. Reboiler and Condenser.

The reboiler and condenser were shell and tube type exchangers. The shell was 6-5/8 inches I.D. and seven inches O.D. There were 28 tubes of 0.50 inches I.D. and 0.84 inches O.D., 19 feet in length. The exchangers were equipped with 3-inch flanged outlets. The condenser was installed vertically and cooling water entered the tube side. The reboiler was installed at an angle of 15^{o} with the horizontal, and steam entered the shell side.

3. Pumps.

Pump No. 1. The reflux pump was an Allis-Chalmers "Electro-fugal", size 2 inch X 1-1/2 inch, type SS-DH, with a capacity of 100 GPM at 120 ft. head. The motor was a 5 hp, 3-phase induction motor.

Pump No. 2. The circulation pump was a Deming, size 2-1/2 inch X 2 inch, figure 4012, type 2A, with a capacity of

200 GPM at 60 ft. head. The motor was a 7-1/2 hp, 3-phase induction motor.

4. Pressure Regulator (Steam).

The pressure of the steam to the reboiler was regulated by a Davis counter-weighted piston, type No. 2, size 2 inches, screwed, regulator.

5. Surge Drum.

The surge drum was a sixty gallon galvanized drum with a volumetric constant of 0.695 gallons per centimeter in the range of the sight glass.

TABLE I

ANALYTICAL RESULTS

Run No.	x_b	x_1	x_2	x_3	x_4	x_5	x_R
t17	0.002	0.004	0.006	0.024	0.107	0.290	0.567
t18	0.003	0.002	0.003	0.016	0.048	0.205	0.473
t19	0.001	0.001	0.004	0.027	0.114	0.251	0.507
t20	0.002	0.074	0.070	0.147	0.310	0.414	0.627
t22	0.003	0.006	0.014	0.034	0.066	0.187	0.429
t23	——	0.004	0.007	0.024	0.114	0.316	0.577
t24	0.003	0.001	0.003	0.045	0.126	0.304	0.555
t25	——	0.055	0.054	0.155	0.300	0.395	0.628
r1	0.043	0.061	0.046	0.052	0.066	0.115	0.427
r2	0.023	0.054	0.033	0.032	0.064	0.145	0.418
r3	0.052	0.078	0.054	0.049	0.063	0.140	0.375
r4	0.037	0.055	0.041	0.051	0.066	0.118	0.404
r5	0.011	0.020	0.016	0.031	0.085	0.160	0.451
r6	0.022	0.030	0.025	0.050	0.061	0.177	0.394
r7							
r8	0.012	0.016	0.018	0.044	0.061	0.231	0.469
s1	0.076	0.170	0.219	0.244	0.223	0.269	0.255
s2	0.090	0.164	0.199	0.216	0.215	0.223	0.225
s3	0.100	0.173	0.192	0.204	0.209	0.208	0.232
s4	0.052	0.153	0.201	0.243	0.277	0.220	0.303
s5	0.118	0.197	0.222	0.107	0.259	0.315	0.262
s6	0.096	0.180	0.233	0.262	0.287	0.295	0.294
s7	0.044	0.112	0.253	0.304	0.348	0.309	0.373
s8	0.104	0.191	0.229	——	0.250	0.277	0.254
s9	0.082	0.175	0.240	0.269	0.284	0.292	0.286
s10	0.067	0.158	0.247	0.290	0.312	0.324	0.318
s11	0.041	0.415	0.255	0.320	0.370	0.417	0.431
s12	0.108	0.177	0.259	0.303	0.317	0.406	0.341
s13	0.080	0.164	0.245	0.293	0.311	0.328	0.324
s14	——	0.370	0.257	0.322	0.372	0.400	0.404
s15	0.144	0.227	0.267	0.301	0.296	0.292	0.297
s16	0.088	0.201	0.250	0.286	0.300	0.312	0.310
s17	0.140	0.216	0.240	0.255	0.247	0.249	0.261

TABLE I (Continued)

Run No.	y_1	y_2	y_3	y_4	y_5
t17	0.006	0.017	0.076	0.284	0.567
t18	0.003	0.008	0.031	0.170	0.473
t19	0.004	0.015	0.056	0.158	0.507
t20	0.072	0.120	0.313	0.392	0.627
t22	0.031	0.029	0.089	0.197	0.429
t23	0.009	0.015	0.072	0.242	0.577
t24	0.006	0.022	0.143	0.242	0.555
t25	0.068	0.176	0.295	0.471	0.628
r1	0.158	0.173	0.209	0.189	0.427
r2	0.112	0.116	0.133	0.149	0.418
r3	0.158	0.170	0.213	0.208	0.375
r4	0.146	0.154	0.187	0.170	0.404
r5	0.056	0.057	0.082	0.133	0.451
r6	0.073	0.085	0.108	0.118	0.394
r7					
r8	0.059	0.083	0.093	0.180	0.469
s1	0.317	0.384	0.332	0.580	0.612
s2	0.411	0.435	0.472	0.552	0.565
s3	0.428	0.497	0.473	0.543	0.561
s4	0.302	0.484	0.467	0.599	0.632
s5	0.240	0.301	0.394	0.378	——
s6	0.303	0.374	0.357	0.454	0.642
s7	0.310	0.384	0.362	0.471	0.687
s8	0.288	0.312	0.297	0.341	0.601
s9	0.309	0.360	0.337	0.418	0.616
s10	0.322	0.368	0.373	0.373	0.640
s11	0.337	0.426	0.533	0.611	0.654
s12	0.461	0.536	0.594	0.613	0.623
s13	0.460	0.441	0.562	0.607	0.634
s14	0.379	0.507	0.594	0.638	0.675
s15	0.472	0.555	0.592	0.608	0.628
s16	0.457	0.529	0.590	0.604	0.633
s17	0.500	0.578	0.574	0.554	0.590

TABLE II

OPERATING AND CALCULATED DATA

Run No.	P_1	P_2	P_3	P_4	P_5	S_n	T_7	λ_s
t17	2.00	10.5	140	13		0.213	300	952
t18	3.40	20.0	132	15	2.80	0.364	300	940
t19	6.50	32.0	127	22	5.30	0.471	297	927
t20	7.70	10.5	143	35	7.70	0.072	303	952
t22	1.40	10.0	151	30	1.30	0.202	304	953
t23	1.35	14.5	147	29	0.80	0.333	298	946
t24	2.85	19.5	141	31	2.00	0.379	300	940
t25	2.70	8.0	152	31	2.90	0.127	306	956
r1	4.25	18.0	131	24	3.30	0.412	298	935
r2	3.70	18.0	130	22	2.80	0.420	297	937
r3	4.60	18.0	130	24	3.50	0.431	298	935
r4	6.50	25.0	123	25	5.00	0.512	295	934
r5	6.20	28.0	124	23	5.00	0.461	296	936
r6	5.55	26.0	124	23	3.30	0.489	296	936
r7	1.25	10.0	148	25	1.00	0.300	296	953
r8	9.20	30.0	137	37	8.00	0.505	280	929
s1	8.35	8.5	151	35	7.90	0.232	304	955
s2	8.30	8.0	150	35	7.80	0.242	304	956
s3	9.25	8.0	150	35	8.70	0.223	304	956
s4	6.15	9.0	151	35	5.80	0.238	305	954
s5	9.10	12.0	144	36	8.00	0.382	303	950
s6	8.30	12.5	145	35	7.30	0.381	303	949
s7	6.45	14.0	146	34	5.50	0.367	303	947
s8	11.90	18.0	141	39	10.40	0.445	298	942
s9	10.50	18.0	141	38	9.10	0.438	299	942
s10	10.50	18.5	140	38	9.10	0.425	297	942
s11	9.80	8.0	152	39	9.70	0.107	304	956
s12	12.40	6.0	152	41	12.20	0.115	304	959
s13	.75	3.5	151	29	0.50	0.258	302	964
s14	6.50	6.5	152	34	6.40	0.155	306	958
s15	9.40	6.0	151	36	9.20	0.158	305	959
s16	7.35	6.5	151	33	7.00	0.211	304	958
s17	9.95	6.0	151	36	9.60	0.218	304	959

TABLE II (Continued)

Run No.	M_1	T_4	T_5	V	R	ΔL_5	L
t17	125	44	138	0.0117	0.0132	0.0009	0.0141
t18	258	44	126	0.0199	0.0170	0.0013	0.0183
t19	350	44	123	0.0253	0.0235	0.0014	0.0249
t20	28	44	185	0.0040	0.0029	0.0003	0.0032
t22	148	43	124	0.0111	0.0101	0.0011	0.0112
t23	321	43	103	0.0182	0.0151	0.0035	0.0183
t24	312	43	115	0.0206	0.0186	0.0010	0.0196
t25	304	42	66	0.0070	0.0052	0.0007	0.0059
r1	275	43	133	0.0212	0.0072	0.0004	0.0076
r2	287	43	128	0.0218	0.0112	0.0008	0.0120
r3	279	43	135	0.0220	0.0059	0.0003	0.0062
r4	329	43	132	0.0266	0.0114	0.0007	0.0121
r5	308	43	130	0.0249	0.0206	0.0010	0.0216
r6	329	43	129	0.0259	0.0163	0.0008	0.0171
r7	183	42	137	———	0.0157	———	———
r8	342	43	132	0.0268	0.0209	0.0014	0.0223
s1	142	43	135	0.0127	0.0327	0.0010	0.0335
s2	208	43	109	0.0133	0.0489	0.0009	0.0498
s3	208	43	110	0.0123	0.0584	0.0002	0.0586
s4	179	43	109	0.0130	0.0253	0.0009	0.0262
s5	317	43	113	0.0208	0.0730	0.0019	0.0749
s6	317	43	112	0.0208	0.0546	0.0015	0.0561
s7	342	43	105	0.0204	0.0329	0.0012	0.0341
s8	300	43	125	0.0242	0.0884	0.0009	0.0875
s9	300	43	125	0.0238	0.0635	0.0007	0.0642
s10	304	43	123	0.0231	0.0515	0.0008	0.0523
s11	50	44	175	0.0059	0.0082	0.0003	0.0085
s12	50	44	182	0.0064	0.0144	0.0004	0.0148
s13	287	44	87	0.0141	0.0230	0.0008	0.0238
s14	342	42	69	0.0085	0.0127	0.0009	0.0136
s15	296	42	74	0.0087	0.0210	0.0008	0.0218
s16	254	42	92	0.0116	0.0261	0.0011	0.0272
s17	292	42	87	0.0120	0.0506	0.0010	0.0516

TABLE II (Continued)

Run No.	T_1	T_2	T_3	T_6	λ_v	c_p	v	L/V	E
t17	101	172	93	224	963	0.76	4.78	1.00	78
t18	105	184	100	230	959	0.80	7.66	1.00	76
t19	130	190	126	231	958	0.79	8.90	1.00	76
t20	107	186	90	233	957	0.74	1.21	1.00	47
t22	67	175	59	219	966	0.82	4.66	1.00	52
t23	78	171	73	222	964	0.76	7.80	1.00	76
t24	118	175	115	226	961	0.77	8.18	1.00	84
t25	60	171	43	223	963	0.73	2.69	1.00	50
r1	122	194	118	223	963	0.82	7.86	0.36	42
r2	118	190	113	223	963	0.82	8.30	0.54	42
r3	127	197	124	221	964	0.85	8.01	0.28	32
r4	140	201	136	229	959	0.83	8.97	0.45	40
r5	142	194	137	236	954	0.81	8.50	0.86	55
r6	140	197	136	231	058	0.84	9.13	0.66	44
r7	91	188	87	215	968	0.86			
r8	130	196	134	238	954	0.80	7.85	0.82	56
s1	164	188	62	215	968	0.91	3.75	2.64	34
s2	173	191	50	215	968	0.92	3.94	3.73	25
s3	190	195	50	216	968	0.92	3.52	4.78	28
s4	151	181	52	218	967	0.89	4.24	2.00	40
s5	174	189	75	214	969	0.91	6.25	3.57	
s6	164	186	78	215	968	0.89	6.79	2.68	32
s7	145	178	76	220	965	0.85	6.60	1.66	35
s8	196	188	109	221	964	0.91	6.32	3.62	
s9	182	192	120	223	963	0.89	6.63	2.69	27
s10	175	189	117	225	962	0.88	6.44	2.26	29
s11	162	188	108	228	960	0.82	1.65	1.41	31
s12	170	197	88	222	964	0.87	1.60	2.32	25
s13	136	165	47	201	977	0.87	6.11	1.67	30
s14	125	178	43	218	967	0.83	2.71	1.56	36
s15	157	191	44	216	968	0.89	2.45	2.50	25
s16	151	185	45	214	969	0.88	3.58	2.32	29
s17	177	195	45	212	970	0.91	3.31	4.29	

TABLE III

HEAT BALANCES ACROSS THE SYSTEM

Run No.	Heat In BTU/Min	Heat Out BTU/Min	Difference BTU/Min	Percent Difference
t17	12,170	11,750	- 420	- 3.5
t18	20,530	21,160	+ 1070	+ 5.2
t19	26,200	27,650	+ 1450	+ 5.5
t20	4,110	3,950	- 160	- 3.9
t22	11,550	12,000	+ 450	+ 3.9
t23	18,900	19,260	+ 360	+ 1.9
t24	21,380	22,460	+ 1080	+ 5.1
t25	7,280	7,300	+ 20	+ 0.3
r1	23,110	24,750	+ 1640	+ 7.1
r2	23,610	24,400	+ 790	+ 1.9
r3	24,180	25,670	+ 1490	+ 6.2
r4	28,690	29,280	+ 590	+ 2.1
r5	25,890	26,800	+ 910	+ 2.4
r6	27,460	28,290	+ 830	+ 2.3
r7	17,150	17,390	+ 240	+ 1.4
r8	28,150	30,440	+ 2290	+ 8.1
s1	13,290	13,060	- 230	- 1.7
s2	13,880	13,730	- 150	- 1.1
s3	12,790	13,940	+ 1150	+ 9.0
s4	13,000	11,810	- 1190	- 9.1
s5	21,770	22,190	+ 420	+ 1.9
s6	21,690	21,870	+ 180	+ 0.8
s7	20,850	21,200	+ 350	+ 1.7
s8	25,150	24,600	- 550	- 2.2
s9	24,760	24,600	- 160	- 0.6
s10	24,020	24,320	+ 300	+ 1.3
s11	6,140	6,550	+ 410	+ 6.7
s12	6,620	6,900	+ 280	+ 4.2
s13	14,920	12,340	- 2580	-17.3
s14	8,910	9,230	+ 320	+ 3.6
s15	9,090	9,470	+ 380	+ 4.2
s16	12,130	12,700	+ 570	+ 4.7
s17	12,540	13,140	+ 600	+ 4.8

FIGURE 1

FLOW DIAGRAM OF EQUIPMENT

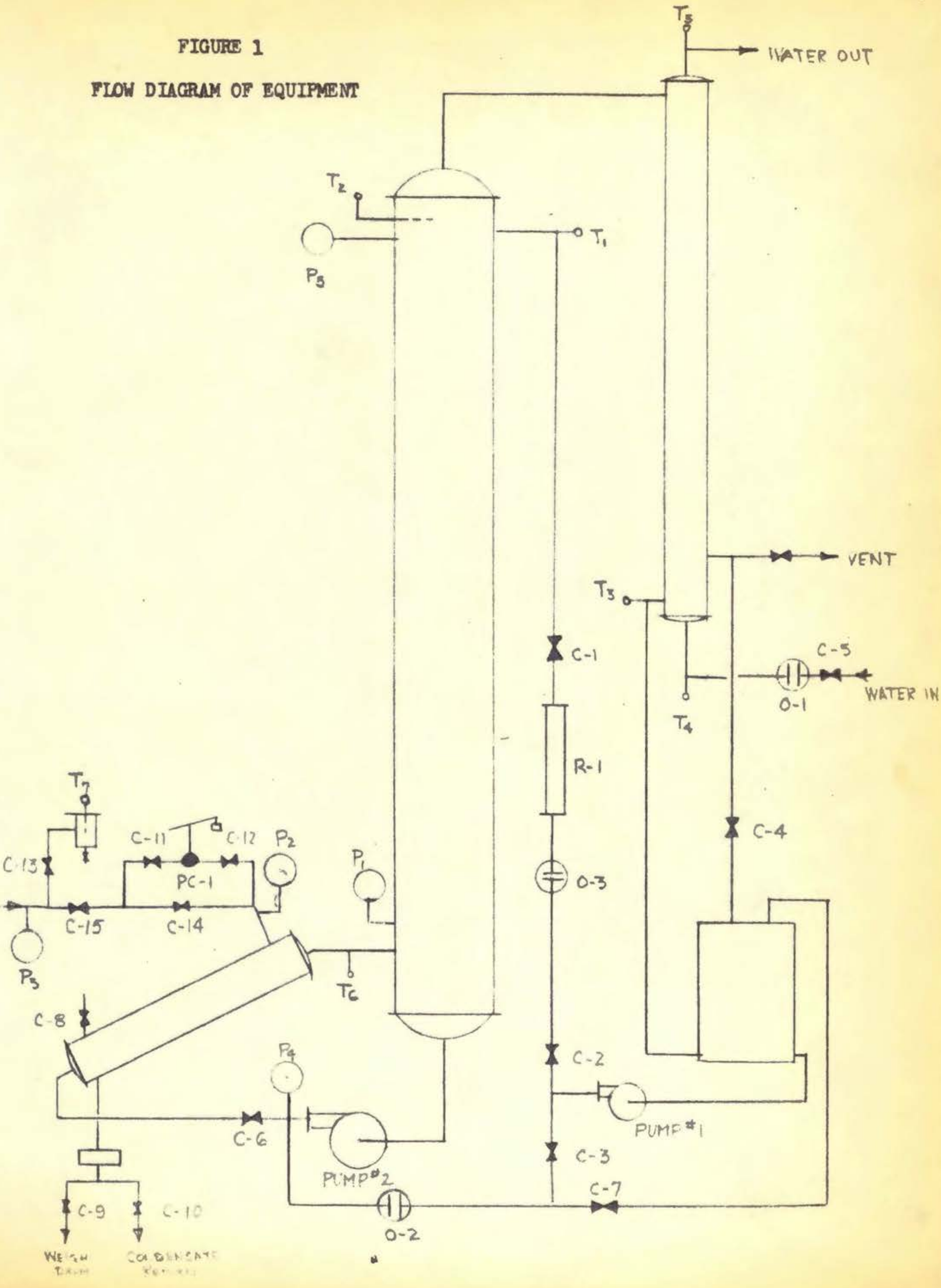


FIGURE 2

TOTAL REFLUX OPERATION

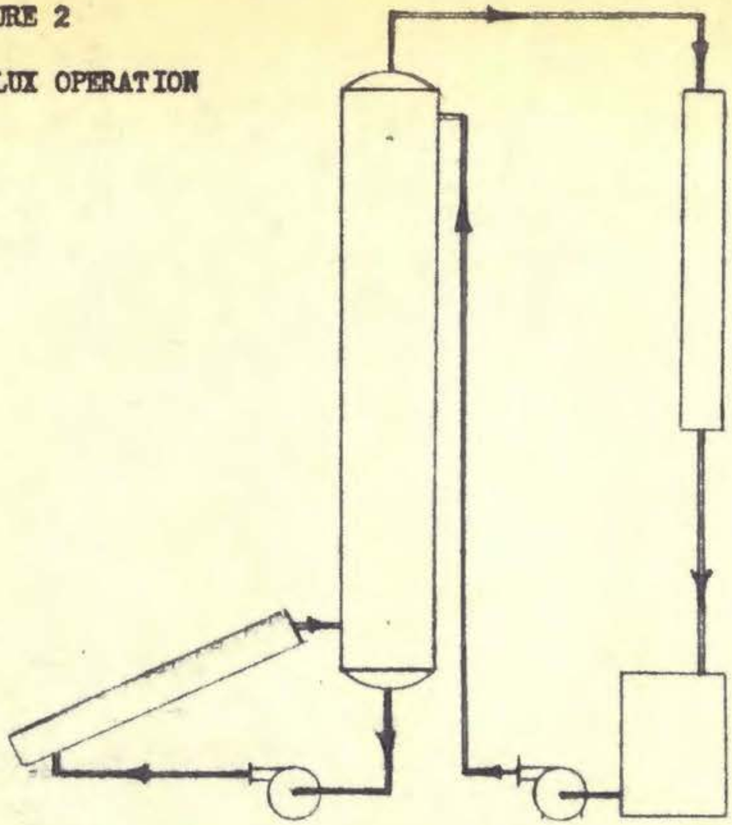


FIGURE 3

RECTIFICATION OPERATION

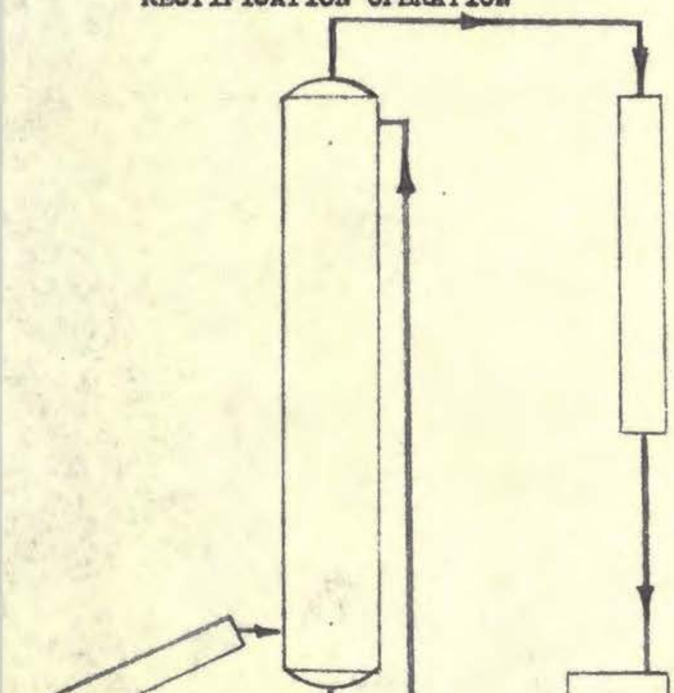


FIGURE 4

STRIPPING OPERATION

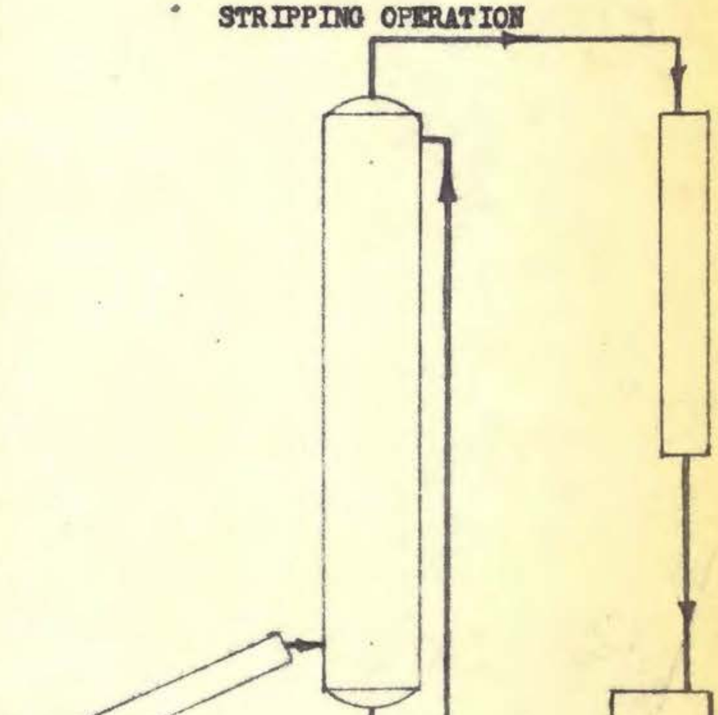
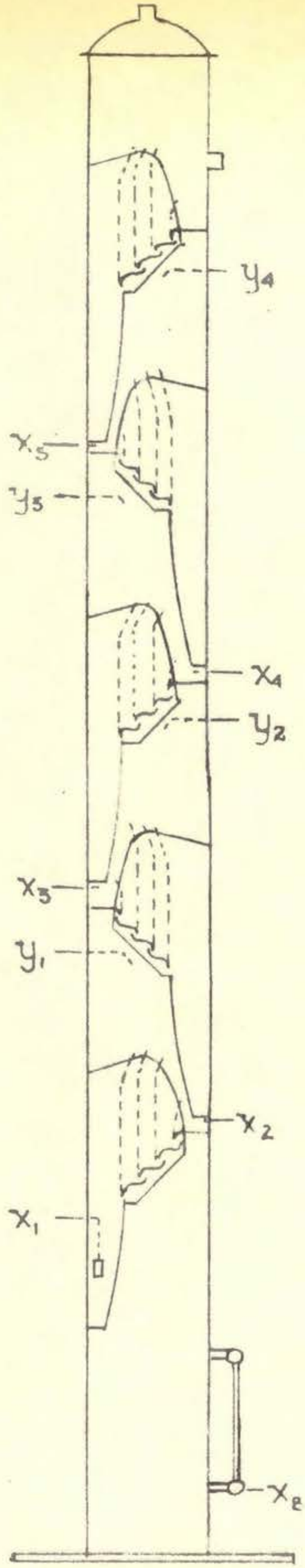


FIGURE 5

COLUMN SAMPLE POINTS



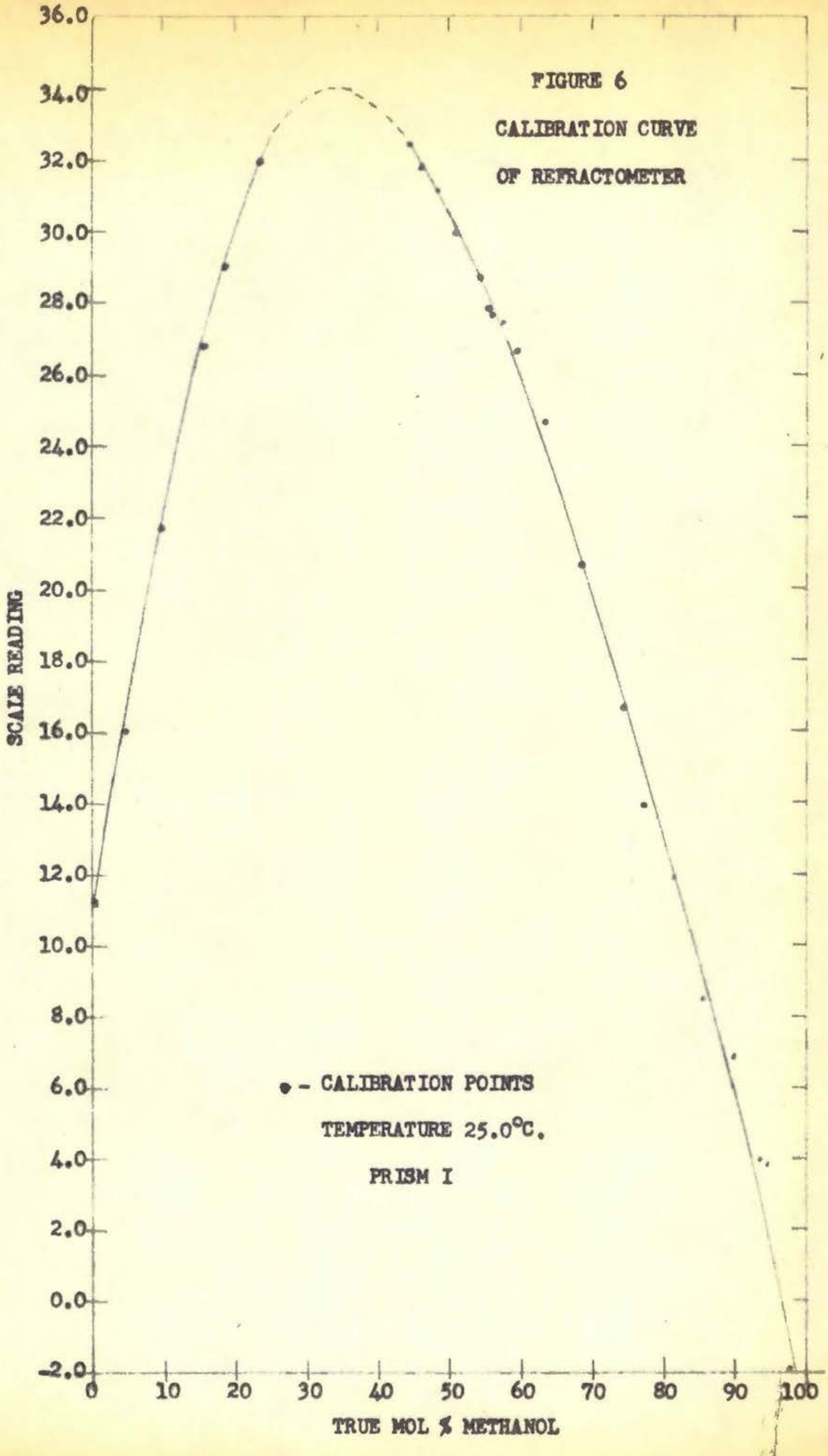
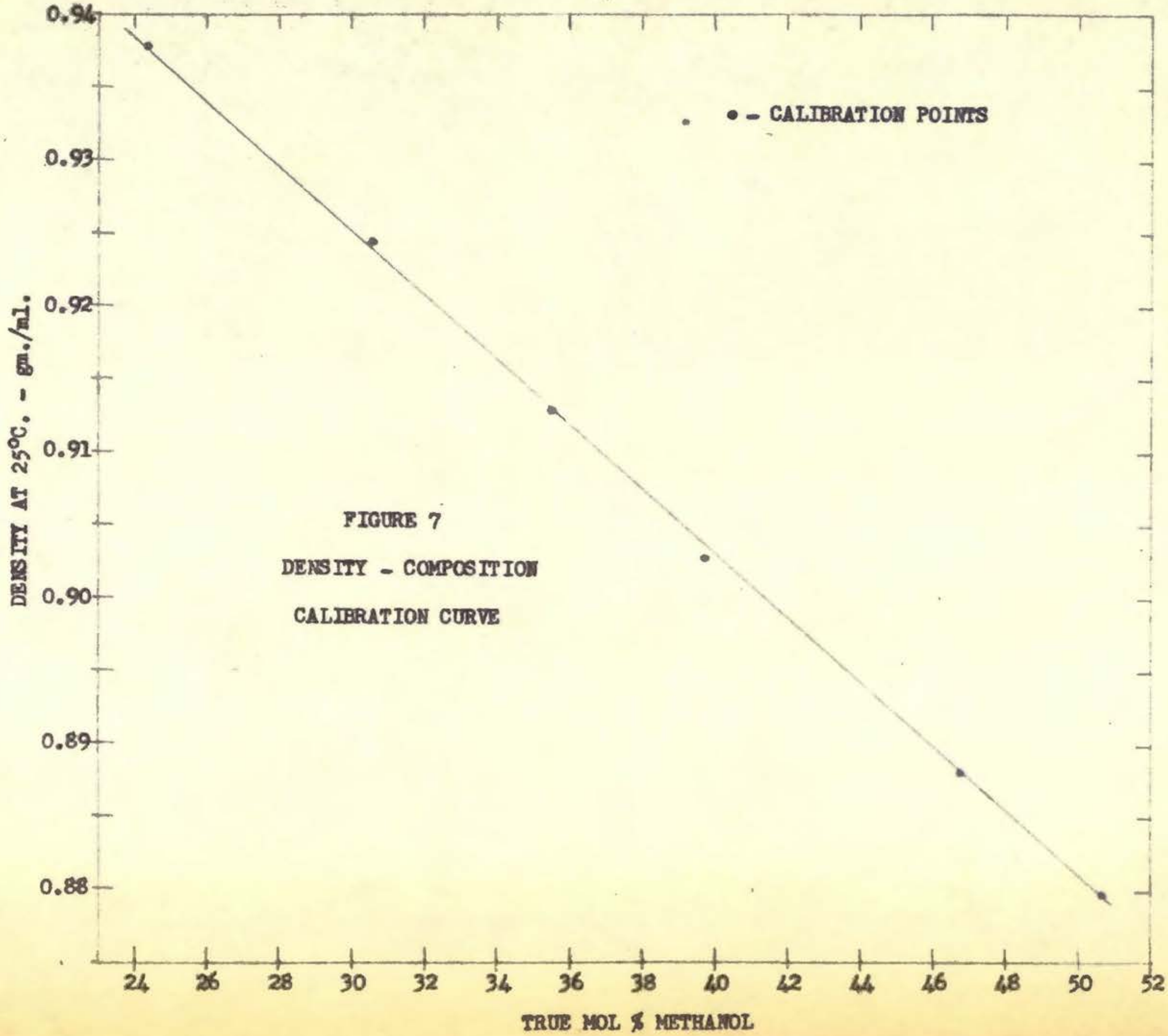


FIGURE 6
CALIBRATION CURVE
OF REFRACTOMETER
• - CALIBRATION POINTS
TEMPERATURE 25.0°C.
PRISM I
SCALE READING
TRUE MOL % METHANOL
36.0
34.0
32.0
30.0
28.0
26.0
24.0
22.0
20.0
18.0
16.0
14.0
12.0
10.0
8.0
6.0
4.0
2.0
0.0
-2.0
0
10
20
30
40
50
60
70
80
90
100


• - CALIBRATION POINTS
FIGURE 7
DENSITY - COMPOSITION
CALIBRATION CURVE
DENSITY AT 25°C. - gm./ml.
0.94
0.93
0.92
0.91
0.90
0.89
0.88
24
26
28
30
32
34
36
38
40
42
44
46
48
50
52
TRUE MOL % METHANOL

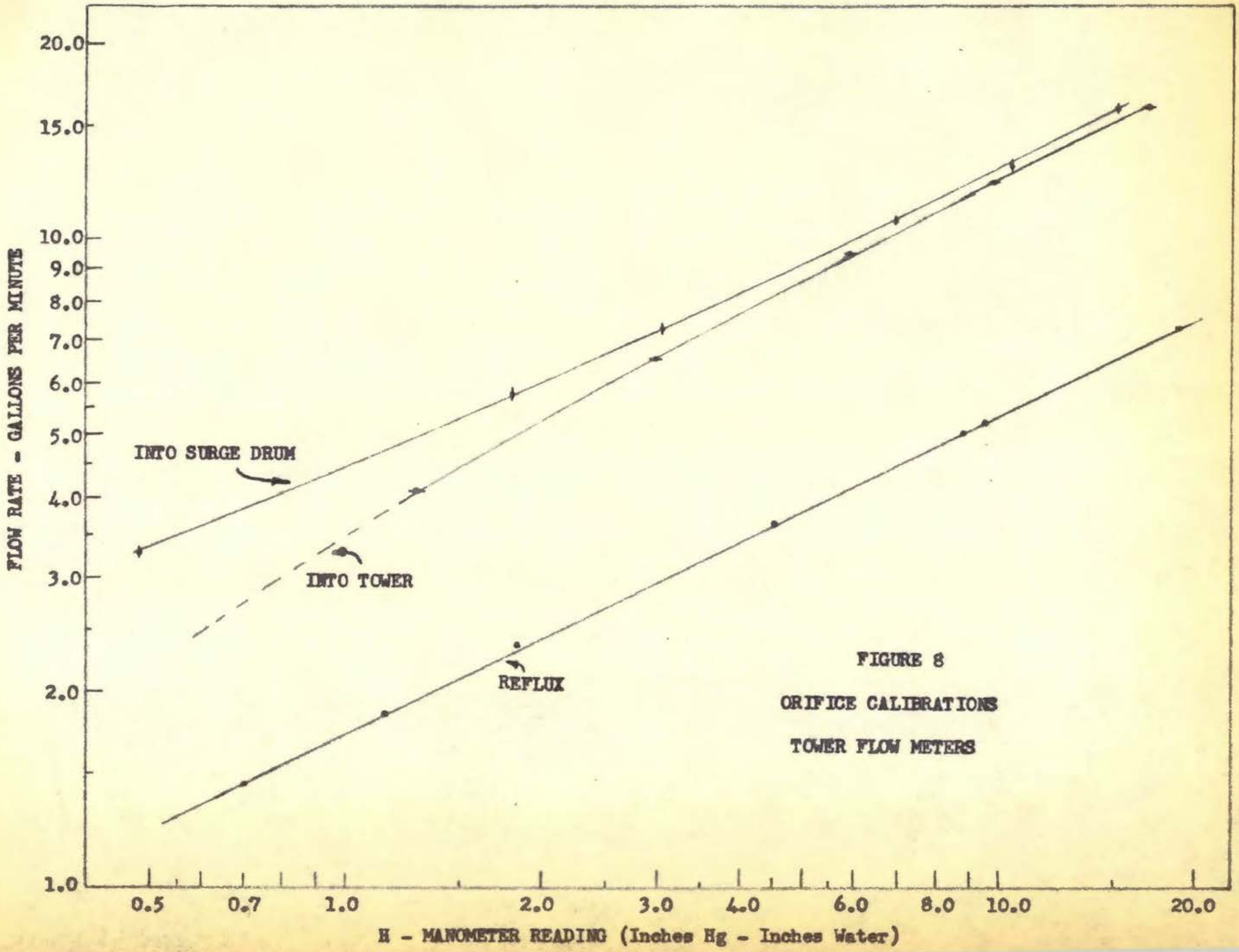


FIGURE 8

ORIFICE CALIBRATIONS

TOWER FLOW METERS

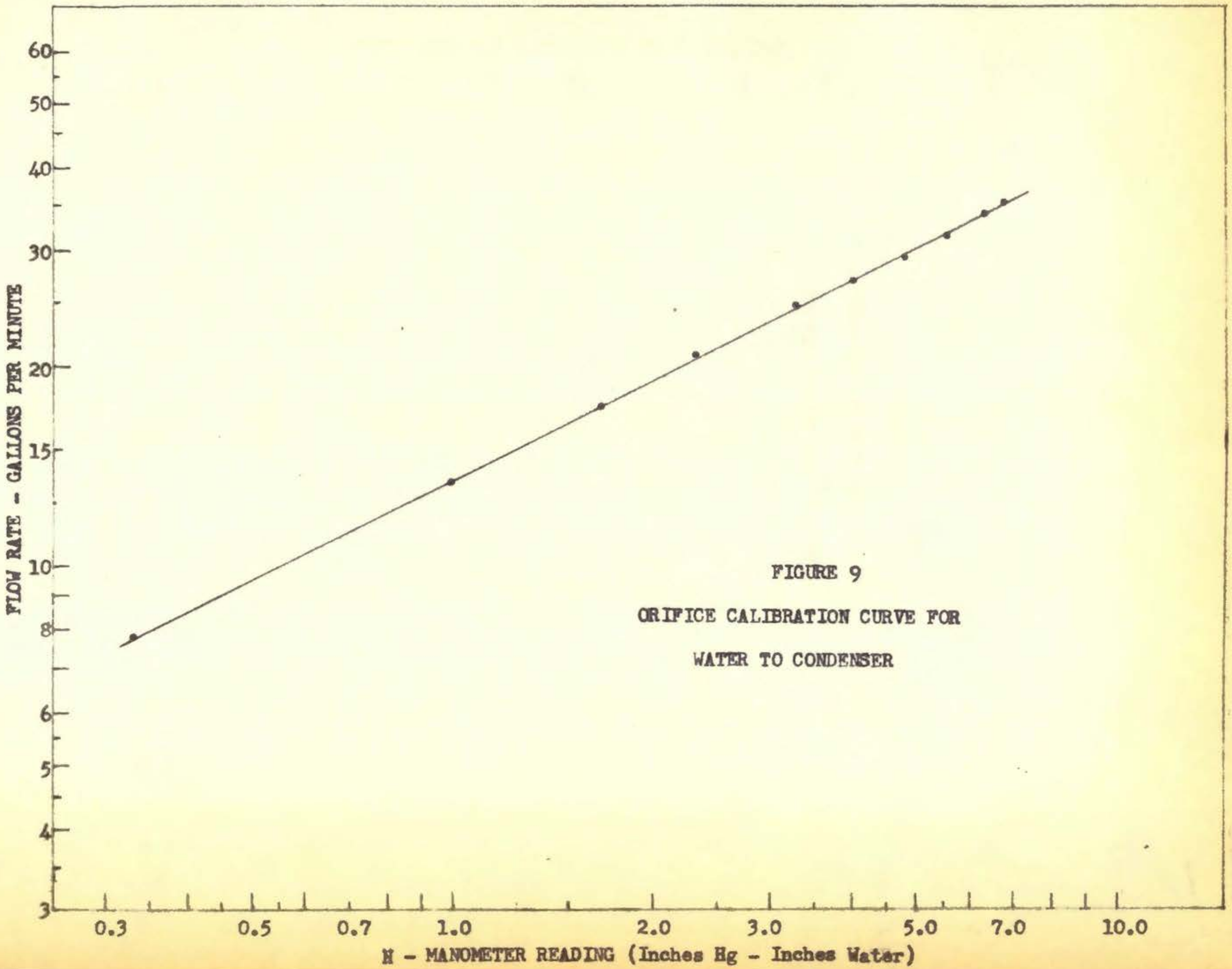


FIGURE 9

ORIFICE CALIBRATION CURVE FOR

WATER TO CONDENSER

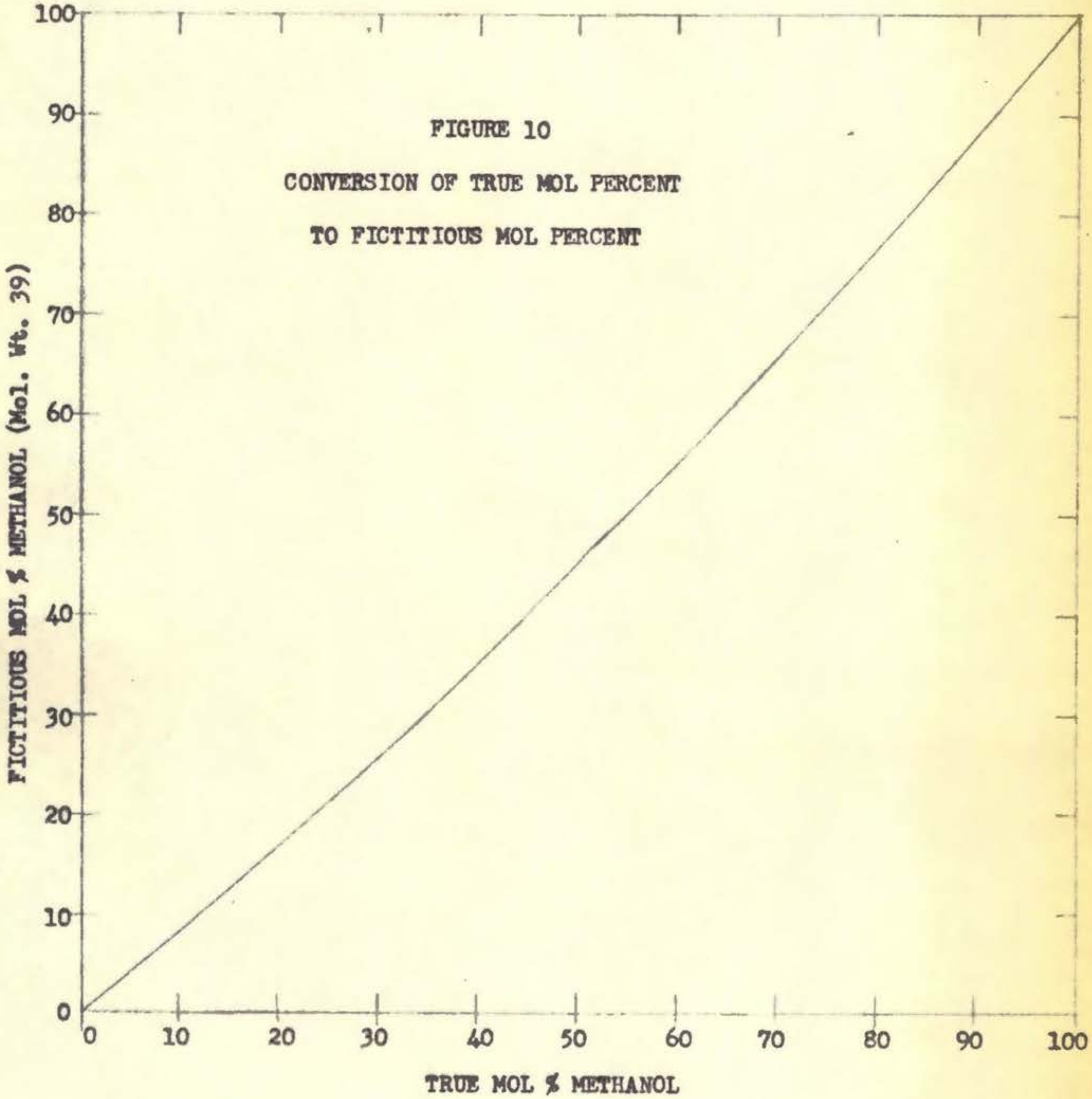


FIGURE 10
CONVERSION OF TRUE MOL PERCENT
TO FICTITIOUS MOL PERCENT
FICTITIOUS MOL % METHANOL (Mol. Wt. 39)
TRUE MOL % METHANOL
0
10
20
30
40
50
60
70
80
90
100

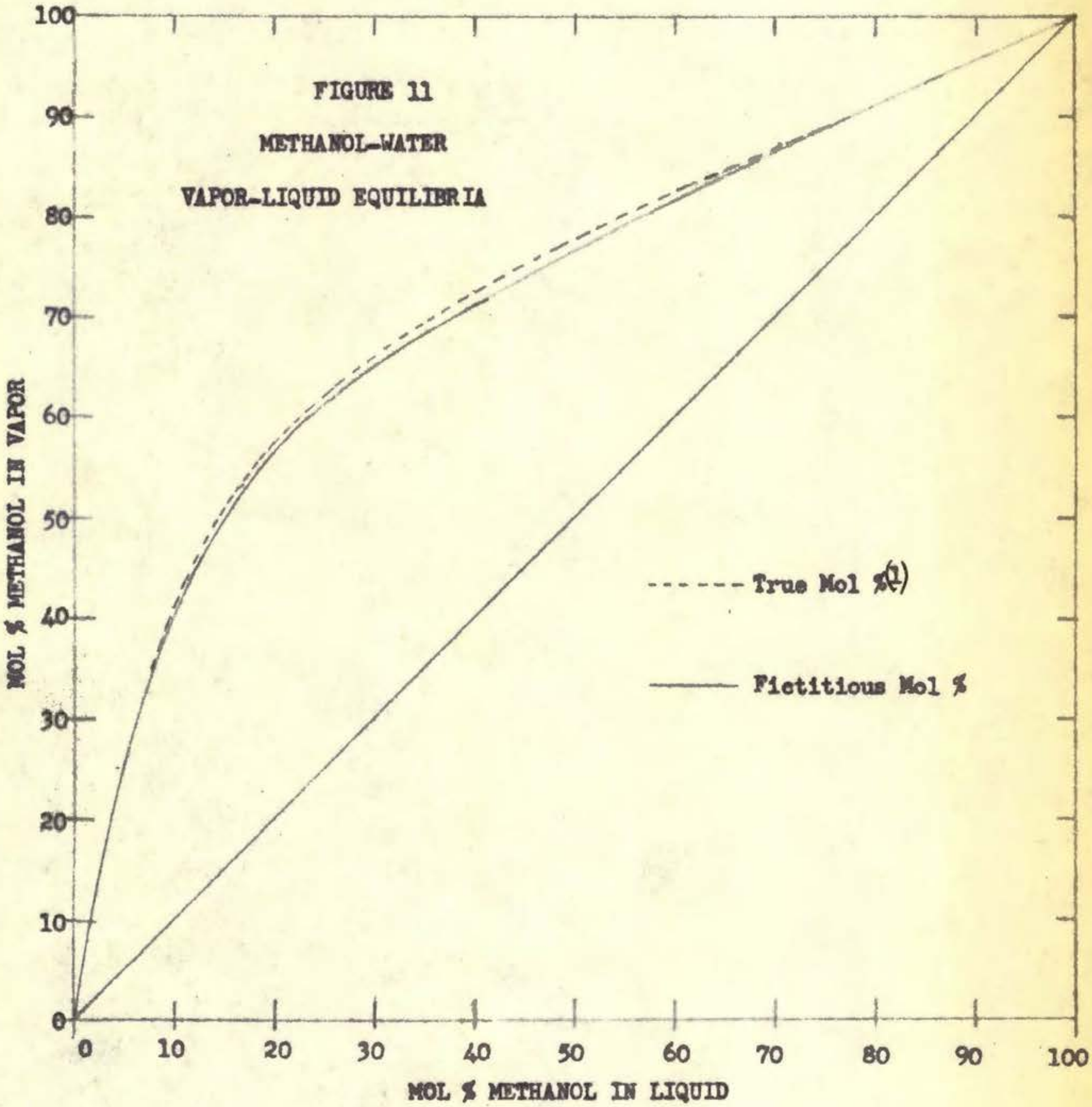


FIGURE 11
METHANOL-WATER
VAPOR-LIQUID EQUILIBRIA
True Mol %(1)
Fictitious Mol %
MOL % METHANOL IN VAPOR
MOL % METHANOL IN LIQUID
0
10
20
30
40
50
60
70
80
90
100

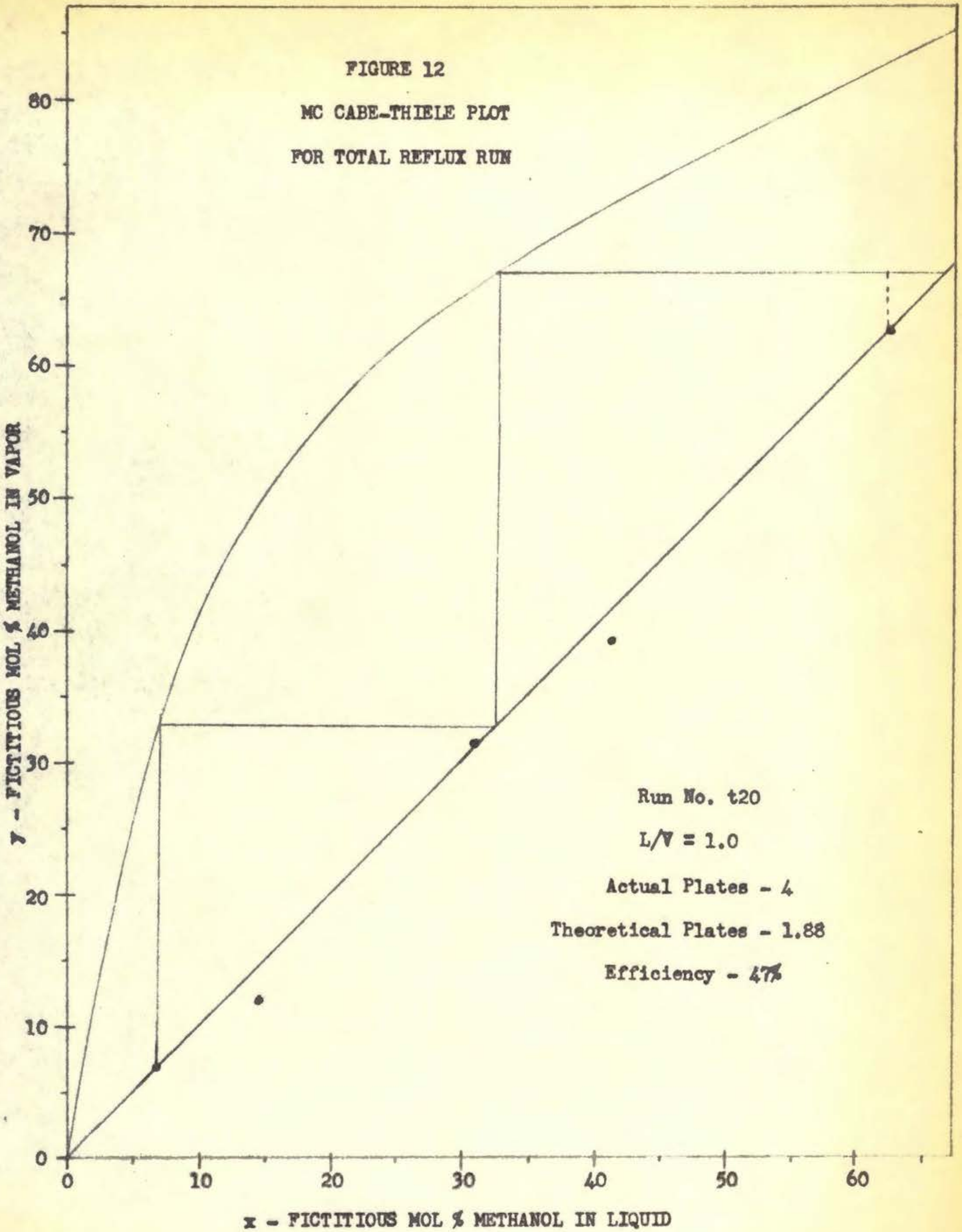


FIGURE 12
MC CABE-THIELE PLOT
FOR TOTAL REFLUX RUN
Run No. t20
L/V = 1.0
Actual Plates - 4
Theoretical Plates - 1.88
Efficiency - 47%
y - FICTITIOUS MOL % METHANOL IN VAPOR
x - FICTITIOUS MOL % METHANOL IN LIQUID
0
10
20
30
40
50
60
70
80

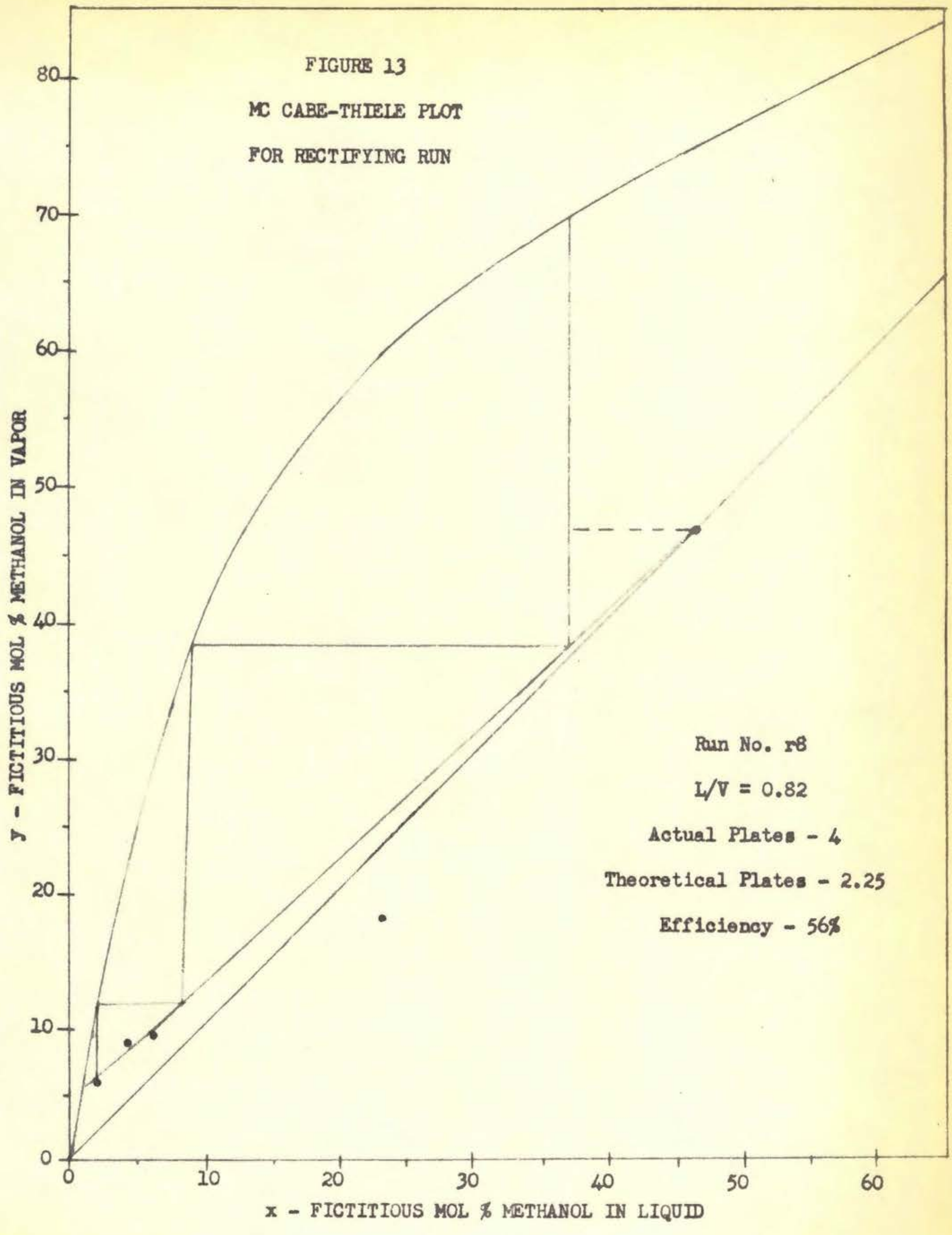


FIGURE 13
MC CABE-THIELE PLOT
FOR RECTIFYING RUN
Run No. r8
L/V = 0.82
Actual Plates - 4
Theoretical Plates - 2.25
Efficiency - 56%
y - FICTITIOUS MOL % METHANOL IN VAPOR
x - FICTITIOUS MOL % METHANOL IN LIQUID
0
10
20
30
40
50
60
70
80

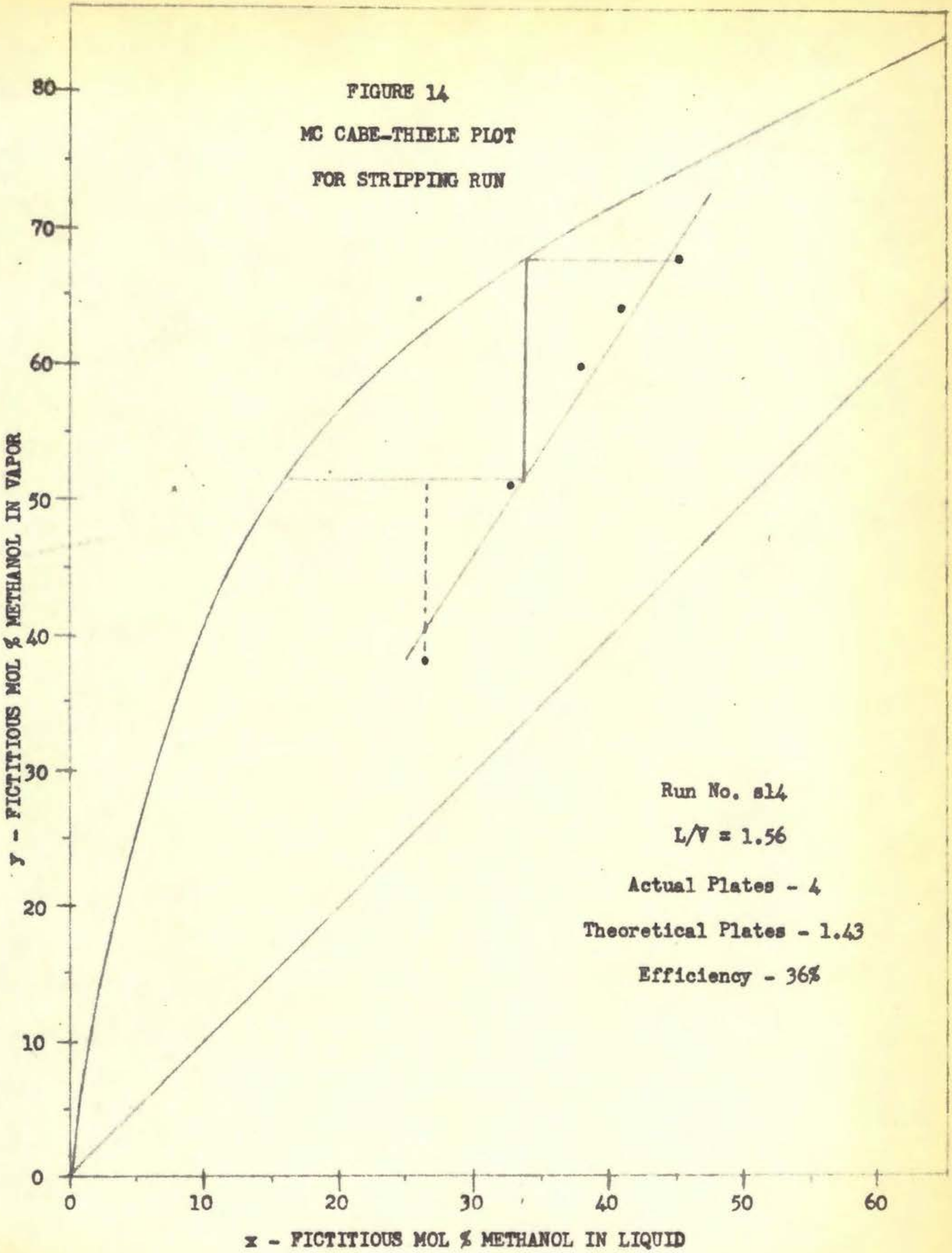


FIGURE 14
MC CABE-THIELE PLOT
FOR STRIPPING RUN
Run No. s14
L/V = 1.56
Actual Plates - 4
Theoretical Plates - 1.43
Efficiency - 36%
y - FICTITIOUS MOL % METHANOL IN VAPOR
x - FICTITIOUS MOL % METHANOL IN LIQUID
0
10
20
30
40
50
60
70
80

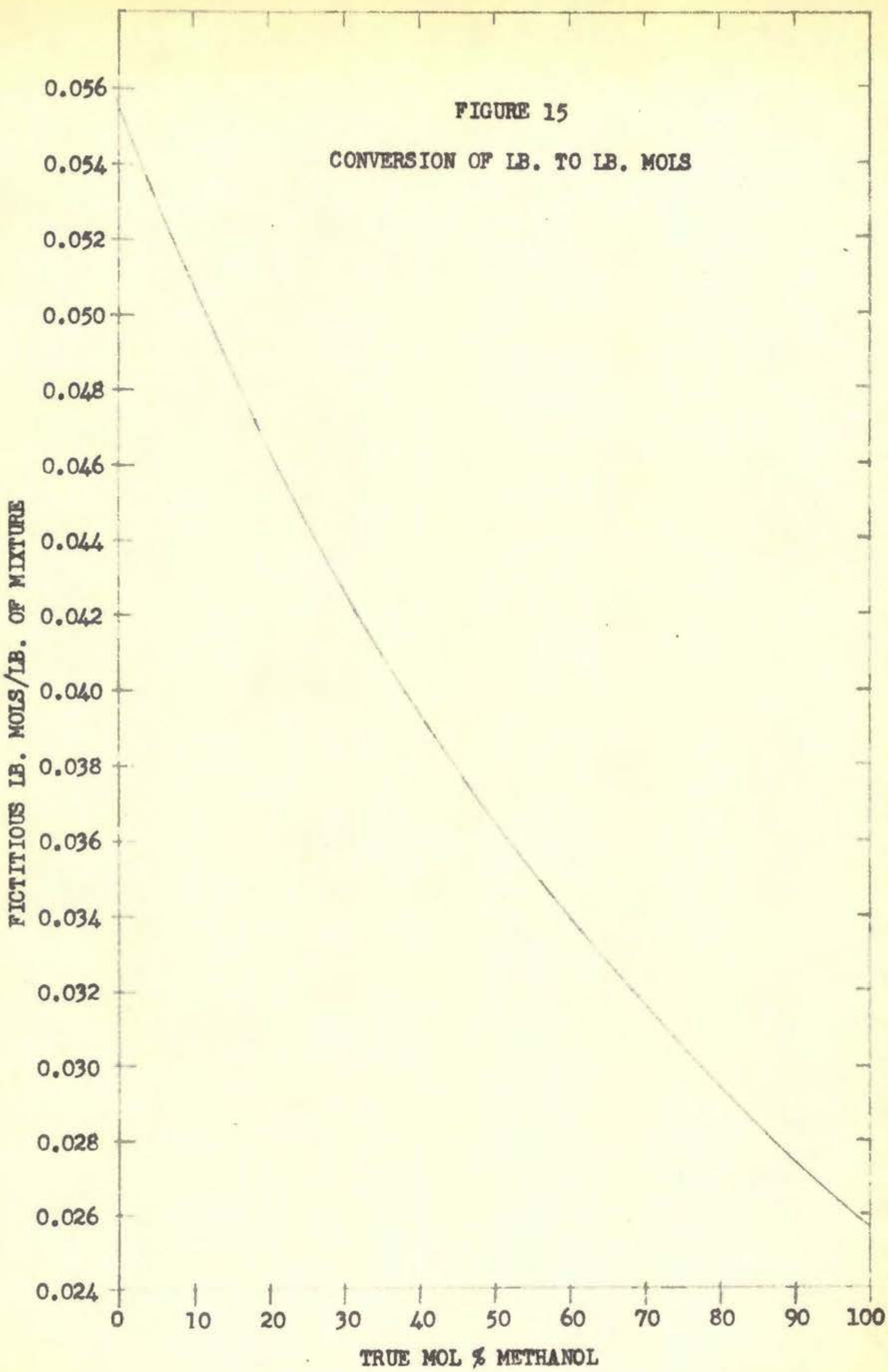


FIGURE 15
CONVERSION OF LB. TO LB. MOLS
FICTITIOUS LB. MOLS/LB. OF MIXTURE
0.056
0.054
0.052
0.050
0.048
0.046
0.044
0.042
0.040
0.038
0.036
0.034
0.032
0.030
0.028
0.026
0.024
0
10
20
30
40
50
60
70
80
90
100
TRUE MOL % METHANOL

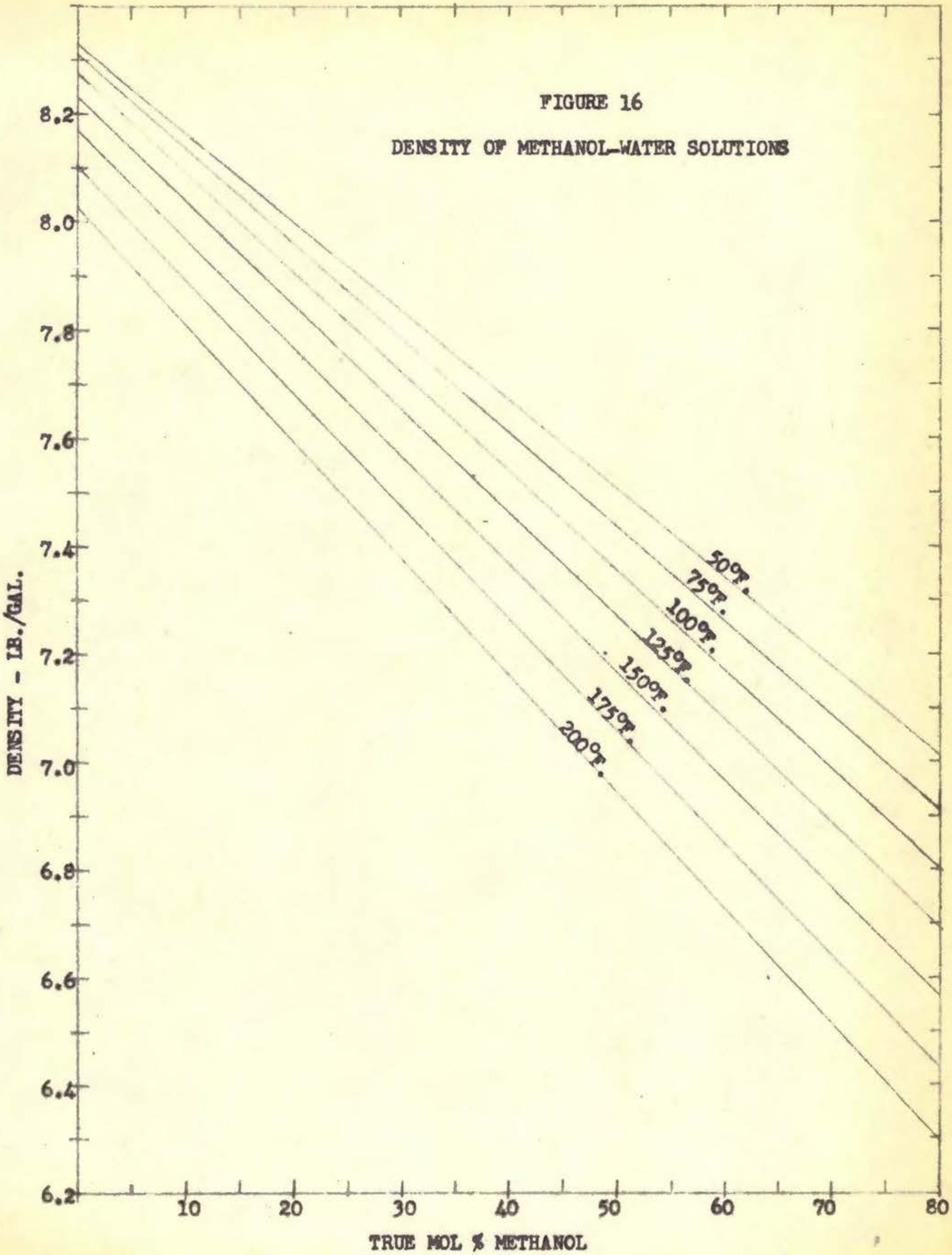


FIGURE 16
DENSITY OF METHANOL-WATER SOLUTIONS
DENSITY - LB./GAL.
8.2
8.0
7.8
7.6
7.4
7.2
7.0
6.8
6.6
6.4
6.2
10
20
30
40
50
60
70
80
TRUE MOL % METHANOL
50°F.
75°F.
100°F.
125°F.
150°F.
175°F.
200°F.

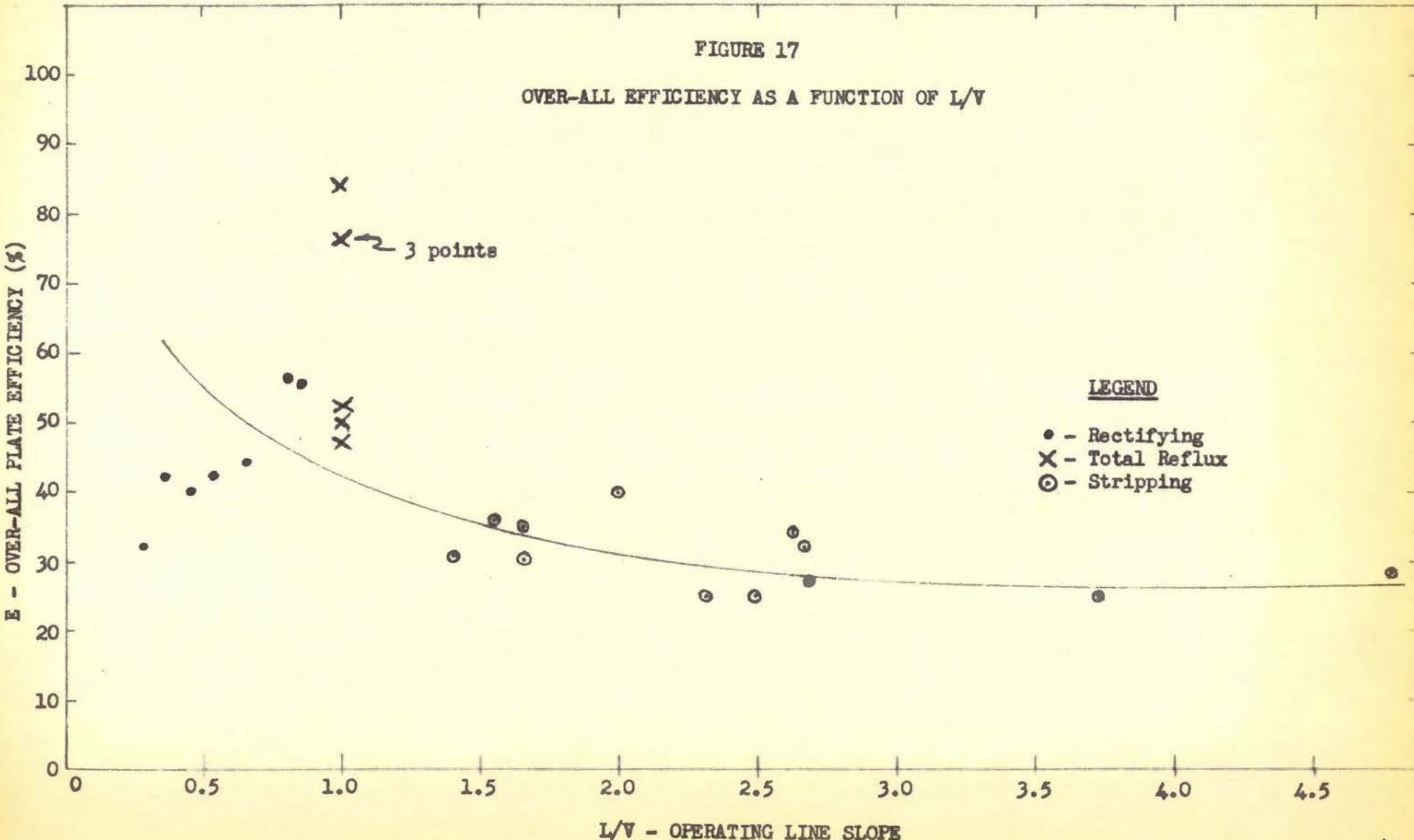


FIGURE 17

OVER-ALL EFFICIENCY AS A FUNCTION OF L/V

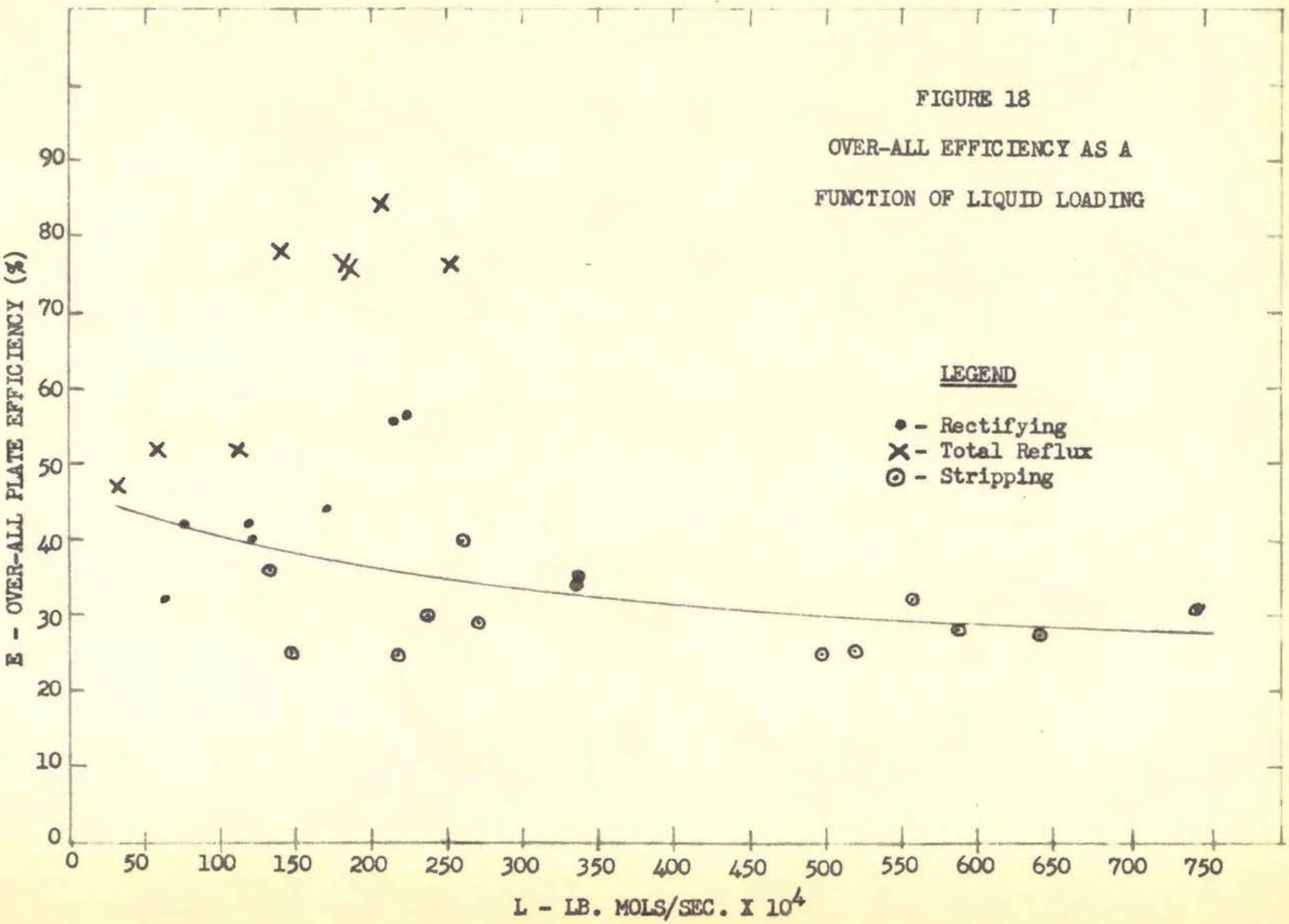


FIGURE 18

OVER-ALL EFFICIENCY AS A

FUNCTION OF LIQUID LOADING

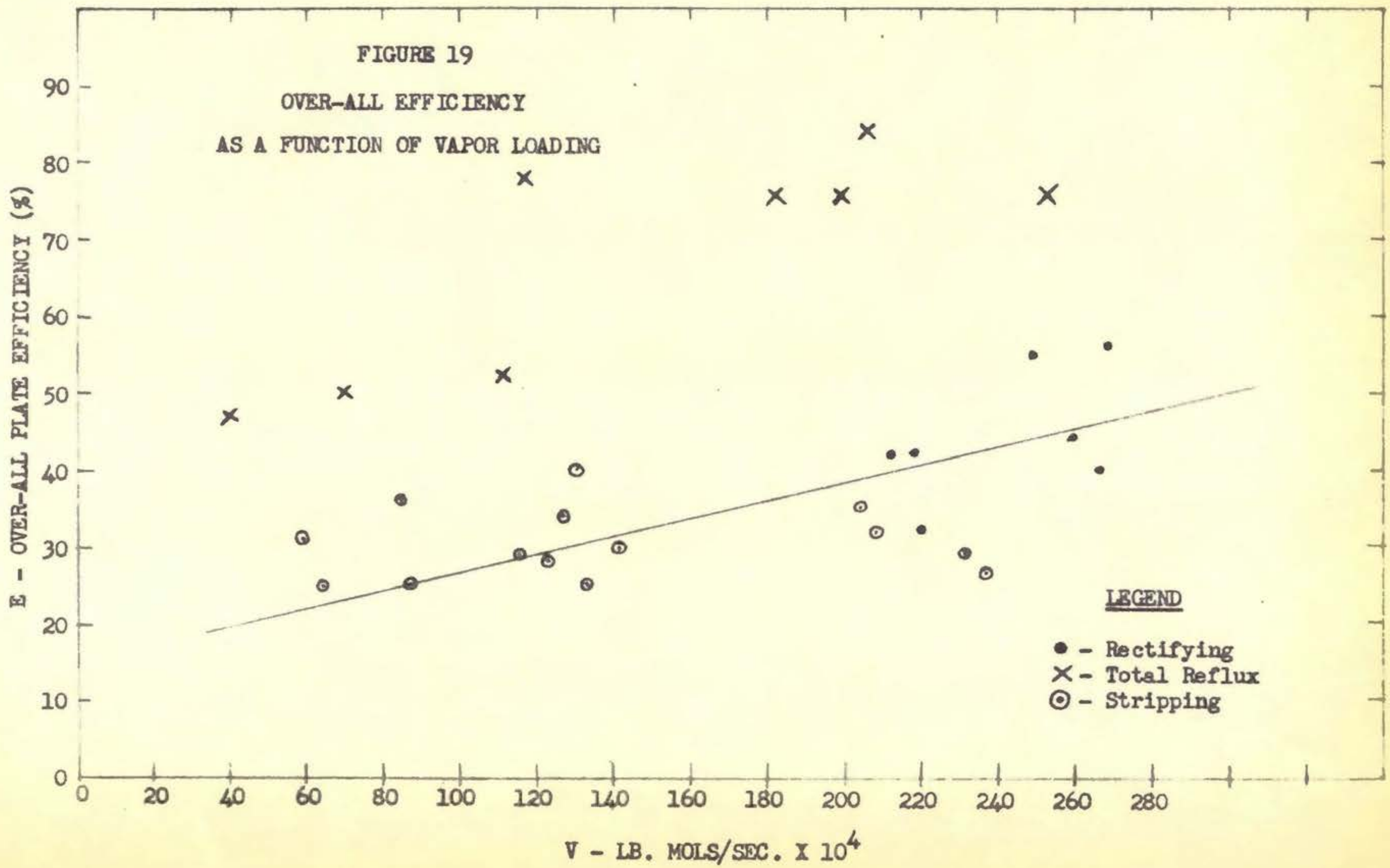


FIGURE 19
OVER-ALL EFFICIENCY
AS A FUNCTION OF VAPOR LOADING
E - OVER-ALL PLATE EFFICIENCY (%)
0
10
20
30
40
50
60
70
80
90
0
20
40
60
80
100
120
140
160
180
200
220
240
260
280
V - LB. MOLS/SEC. X 10^4
LEGEND
● - Rectifying
X - Total Reflux
⊙ - Stripping

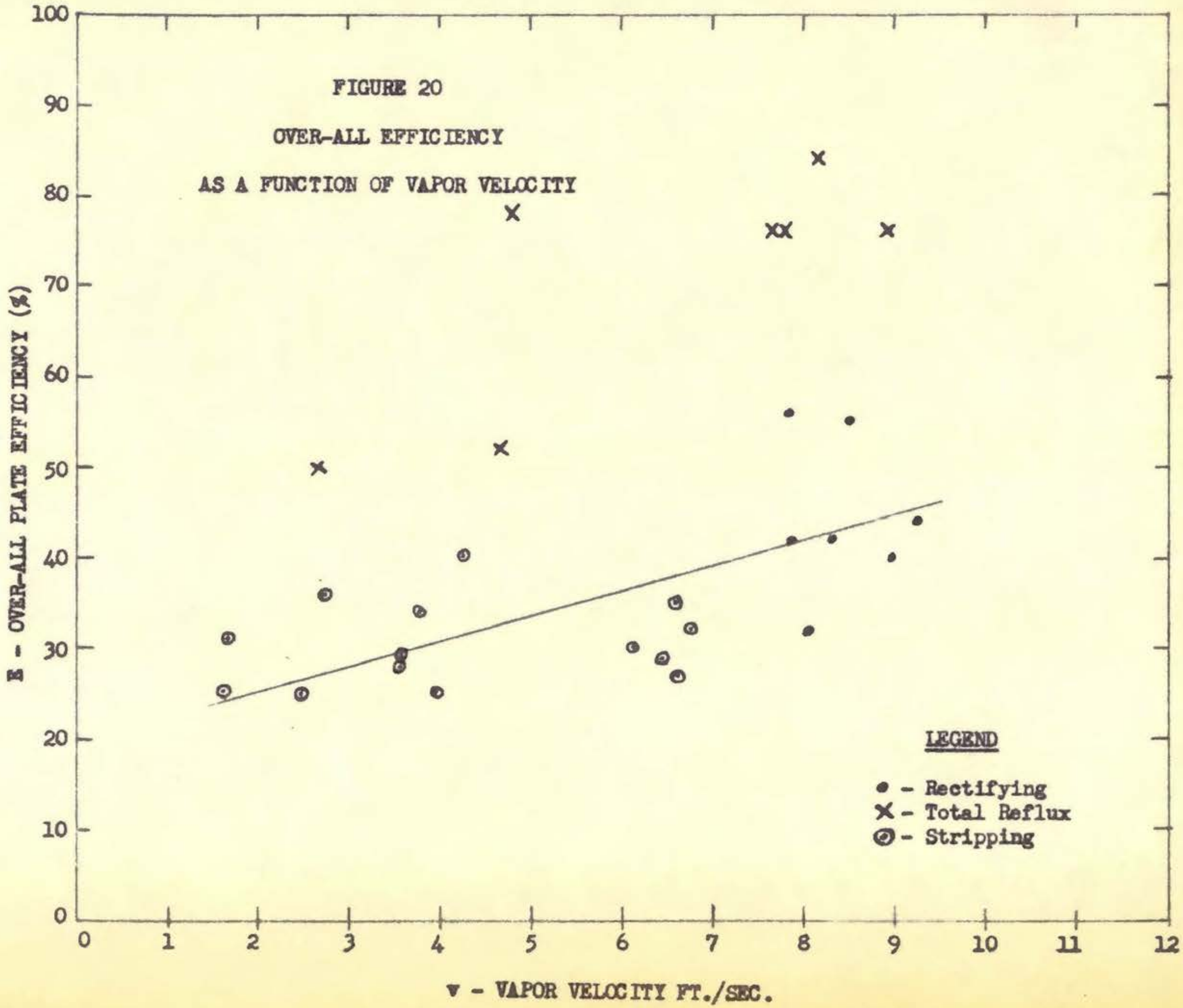


FIGURE 20
OVER-ALL EFFICIENCY
AS A FUNCTION OF VAPOR VELOCITY
E - OVER-ALL PLATE EFFICIENCY (%)
100
90
80
70
60
50
40
30
20
10
0
0
1
2
3
4
5
6
7
8
9
10
11
12
v - VAPOR VELOCITY FT./SEC.
LEGEND
• - Rectifying
X - Total Reflux
⊙ - Stripping

CALCULATIONS

1. Calculation of Liquid Loading L, lb. mol/sec. (Fictitious).

(a) Stripping, rectifying and total reflux runs

$$L = R\left[1 + \frac{c_p M_R (T_2 - T_1)}{\lambda t}\right]$$

where

$R = \frac{R'dm}{60}$ lb. mol/sec. (Fictitious).

R' = Reflux flow, gallons/min.

d = Density of reflux, lb./gal. (Figure 16).

m = Fictitious mols/lb. (Figure 15).

M_R = Average fictitious molecular weight of reflux.

T_2 = Temperature of vapor leaving the column, ^{o}F.

T_1 = Temperature of reflux stream, ^{o}F.

λ_t = Latent heat of vaporization, BTU/lb. mol. (Fictitious).

2. Calculation of Vapor Loading v, lb. mol/sec. (Fictitious).

(a) Stripping and total reflux runs

$$V = \frac{S_n \lambda_s}{M_v \lambda_v}$$

where

S_n = Net steam consumption, lb./sec.

λ_s = Latent heat of vaporization of steam at pressure P_2, BTU/lb.

M_v = Molecular weight of bottoms, lb./lb. mol. (Fictitious).

λ_v = Latent heat of vaporization of the bottoms at pressure P_1, BTU/lb.

(b) Rectifying runs

$$V = \frac{S_n \lambda_s + Lc_{pm}(T_6 - T_1)}{M_v \lambda_v + C_{pm}\,(T_6 - T_1)}$$

where

L from 1.(a)

c_{pm} = Heat capacity of bottoms, BTU/lb. mol ^{o}F.

T_6 = Temperature of tower bottom, ^{o}F.

T_1 = Temperature of reflux stream, ^{o}F.

3. Calculation of Vapor Velocity v, ft./sec.

(a) Stripping, rectifying and total reflux runs

$$v = \frac{10.05VT_{avg.}}{P_{avg.}}$$

where

V from 2.(a) or 2.(b)

$$T_{avg.} = \frac{T_2 + T_6}{2} + 460,\ ^{o}R.$$

$$P_{avg.} = \frac{P_1 + P_5}{2} + 14.4,\ \text{psia.}$$

T_2 = Temperature of tower top, ^{o}F.

T_6 = Temperature of tower bottom, ^{o}F.

P_1 = Pressure in tower bottom, psig.

P_5 = Pressure in tower top, psig.

4. Calculation of Over-All Plate Efficiency

E = (Actual Plates/Theoretical Plates)100, %

5. Calculation of Heat Balance Across the Column

Heat in $= S_n \lambda_n \times 60$, BTU/min.

Heat out $= M_1 (T_5 - T_4)$, BTU/min.

where

M_1 = Cooling water flow, lb./min.

T_4 = Temperature of cooling water leaving the condenser

T_5 = Temperature of cooling water entering the condenser

VITA

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Master of Science

Thesis: PLATE EFFICIENCIES OF KOCH KASKADE FRACTIONATOR USING THE SYSTEM METHANOL-WATER

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Graduate:
The writer entered the graduate school of Oklahoma Agricultural and Mechanical College in June 1950 and completed the requirements for Master of Science in Chemical Engineering in May 1956.

Experience:
Prior to entering the Navy in January 1945, the writer worked in the laboratories of Halliburton Oil Well Cementing Company, Duncan, Oklahoma as a laboratory assistant. After serving 18 months in the Navy he was honorably discharged as Aviation Electronics Technician 3/c. While attending Oklahoma A. and M. he worked part time as an analyst with Agricultural Research Department and during summers worked with Halliburton Oil Well Cementing Company as a Junior Chemist. He went to work for Monsanto Chemical Company, Texas City, Texas in February 1951 and is serving as a Research Engineer.