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THE EFFECT OF REDUCED PRESSURE ON  
VARIABLES IN DISTILLATION

BY

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### SUMMARY

To determine the effects of reduced pressure on pressure drop and holdup in distillation, seven different liquids were distilled at varying rates at atmospheric pressure and under vacuum at pressures down to 50 mm Hg, using protruded stainless steel packing. Pressure drop and holdup were found to vary exponentially with mass rate at each pressure. At constant mass rate pressure drop was higher for decreased head pressure. Use of material of higher molecular weight led to lower pressure drop at same rate and pressure. Greatest effect of pressure on pressure drop was observed below 300 mm Hg. Liquid holdup was not affected greatly by pressure or molecular weight with the packing used.

Data for the seven liquids at all pressures tested, correlated by a plot of  $\log \Delta p$  vs.  $\log G/\phi$ , defined a straight line within reasonable limits. This line, however, had a slope too steep to be used for design work with such accuracy. Correlation of the data by a plot of  $\log \rho \Delta p$  vs.  $\log G$  also gave a straight line within about the same limits. This has a slope approximately the same as the  $G/\phi$  curve and is not recommended for design work.

Modification of the Reed-Fenske equation to use liquid instead of vapor viscosities appeared to yield a better correlation of data for the individual liquids and gave closer agreement among all the liquids. The curve, defined by  $\rho \Delta p / \mu_1^2 = 0.0001 (G/\mu_1)^{2.4}$ , should be useful for approximating expected pressure drop for any liquid at any rate for the size and type packing used.

Evidence points to diffusion concepts as a basis for correlation

of distillation data. A new relationship is presented to predict the change in pressure drop at constant rate produced by a material of different molecular volume. The data is shown in the form:

$$(\log \Delta P) G/\phi = a - \frac{3}{4} V_M$$

Use of the curves obtained permitted estimation of pressure drop of a liquid with molecular volume greater than twice those used here to within thirty percent over a tenfold change in pressure drop. The curves should apply at any pressure since  $G/\phi$  includes the effect of pressure on vapor density.

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## INTRODUCTION

Distillation, the separation of constituents of a liquid mixture by vaporization of the mixture and separate recovery of vapor and residue, has been used for many centuries. Fractionation, distillation carried out in such a way that successive products are further distilled in the same operation to effect greater purity, is a much more recent innovation. Vacuum distillation, however, can be called a modern operation, since its first industrial usage started in the late nineteenth century. Actually, use of vacuum distillation or fractionation was not widespread until fairly recently. Most of the recorded work on the effects of reduced pressure on distillation has appeared in the past ten or fifteen years.

Vacuum distillation here refers to all distillations performed under pressures less than atmospheric and in equipment not specifically designed for molecular distillation. The principal advantage over atmospheric distillation is the lowering of the boiling point. This permits distillation of substances subject to chemical alteration at the normal boiling point. It is also advantageous for heat stable compounds having low vapor pressures which would boil at inconveniently high (and uneconomical) temperatures at atmospheric pressure. Formation of undesirable azeotropes and polymers may also be avoided by use of low pressure distillation.

A large percentage of batch vacuum distillation is carried out in packed columns due to the lower pressure drop and holdup than in equally efficient bubble-cap or other type columns. In many cases

packed towers are cheaper. Packing also lessens the possibility of the tower collapsing under vacuum. Therefore, packed columns are considered in most of the studies on distillation under reduced pressure.

A moderate amount of work has been reported in recent years on vacuum distillation, but there is still much to be done in order to provide sufficient information for proper equipment design. Some of the more important variables to be considered in batch distillation have been listed<sup>21</sup> as: reflux ratio; number of theoretical plates or separating efficiency; ratio of holdup to charge; vapor velocity or throughput; relative volatility; initial composition of mixture.

The two latter items relate directly to the system being distilled. However, the effects of pressure on physical properties other than relative volatility should be considered. Some physical properties are affected little by pressure changes, but a great deal by variations in temperature. In these cases, it is the reduction of boiling temperature due to the lower pressure which causes differences in properties.

Pressure drop and flooding velocity should be added to the first four variables mentioned when dealing with vacuum work. Of all these variables, a relationship among throughput, holdup, pressure drop and efficiency would be desirable.

Various investigators have considered the effects of diminished pressure on the efficiency of packed columns. Results obtained, in general, show that direction of efficiency variation with pressure and

throughput rate is evidently a function of the individual packing and the liquid test mixture.

The pressure drop becomes quite important in vacuum distillation due to its effects on vapor density. A decrease in pressure causes a decrease in vapor density. This, of course, leads to increased volume flow of vapor with higher vapor velocities. Larger friction losses should result in higher pressure drops. This is extremely important at quite high vacuum since a relatively large pressure drop compared to the desired operating pressure may cause sufficient still pressure to prevent operation under the maximum allowable boiling temperature.

Holdup has a dual effect in batch distillation since it limits the proportion of charge that can be distilled and also affects the sharpness of separation of any two components.

#### Literature Survey

The scope of the present work is an attempt to correlate the effects of reduced pressure in distillation on pressure drop and holdup. Several proposals have been made in the literature. Some of these are based on experiments in gas absorption which can be considered similar in many respects to distillation. Sherwood<sup>15</sup> proposes that since no thoroughly satisfactory method has as yet been developed for correlating pressure drop for hollow commercial packing with countercurrent flow rates of gas and liquid, the simplest and most accurate procedure for equipment design is to refer to plots of flow rate vs. pressure drop for the specific packing. Advanced as the most reliable data are those of Tillson who has made extensive pressure drop measurements in a

20 inch diameter column which is large enough to minimize wall effects. Tillson has presented his data plotted as  $\log \Delta p$  vs.  $\log G/\phi$ , where  $G$  is the mass flow rate of the gas and  $\phi = \sqrt{\rho/0.075}$  is a factor to correct for variation in gas density.

Struck<sup>17</sup> tried to correlate his data on vacuum fractionation of n-decane and trans-decalin by the same type of plot. Using the density of n-decane at 100 mm Hg instead of the density of air (0.075 lbs./cu.ft.) at standard conditions, his correlation was fairly good, particularly at high pressure drops. However, deviation from the correct value increased as pressure deviated from the 100 mm Hg reference. This makes the idea of a reference point selection somewhat questionable.

Hand, Whitt and Gregory<sup>6</sup> have applied the same method to distillation and obtained data at lower liquid rates and higher vapor rates in order to include the ranges generally used in distillation work. These rates are different from those in absorption where control of individual gas and liquid rates is possible. These authors also determined data for phosphorus oxychloride in distillation at atmospheric and reduced pressures in order to compare with the conventional water-air absorption data. The plots of  $\Delta p$  vs.  $G/\phi$  for the two systems cannot be correlated as such since somewhat different slopes are obtained for these two curves. However, the authors claim that the approximately eight percent error can be disregarded since as much variation is obtained in the same equipment with different dumpings of the packing.

For design work, they suggest use of the Fanning-type equation proposed by Chilton and Colburn for flow in packed beds:  $\Delta p = 2f\rho A_p A_1 U^2/g_c d_p$ .

The friction factor is to be obtained from the commonly used friction factor vs. modified Reynolds number chart. This equation involves use of the correction factors  $A_p$  for hollow packing effect and  $A_1$  for flowing liquid effect. Both of these factors vary with flow rate as well as packing diameter. General use would necessitate charts for variation of the factors for all packings considered at various rates. The factors presented are those for water alone. The effect of physical properties of other materials is not definite. No work has evidently been done on the effects of pressure on these variables.

Leva<sup>8,9</sup> has developed an analysis of fluid flow through packed beds and recommends the approach of Brownell and Katz<sup>2</sup> for calculating pressure drop through packed liquid-gas systems. In their method allowance is made for the effect of liquid rate upon effective shape factor of the packing, voidage of the bed and surface roughness of the packing. These effects are included in their expression for pressure drop which is also based on a Fanning-type equation and the friction factor-modified Reynolds number chart. Their procedure is extremely involved and was developed on data for some of the more common types of packing used in absorption towers as well as on sand beds. The newer, high efficiency packings designed for use in packed distillation towers, such as that used in the following experimental work, have much less holdup and pressure drop and have high voids content and surface area. The sphericity and porosity factors as developed by Brownell and Katz do not cover sufficient range to be used for most of these packings. Values of the porosity exponents for the protruded packing used would fall in the range from 50 to

10,000 and their use would be extremely questionable.

Reed and Fenske<sup>11</sup> have presented a correlation based on a dimensional analysis of the factors involved in the hydrodynamics of liquid vapor flow in distillation. This is expressed as an equation involving two dimensionless groups:

$$\frac{P_a M \Delta P (f_0 - H)^3}{R T_a h \mu_v^2 S^3} = k \left( \frac{G}{\mu_v S} \right)^n$$

Data collected over a number of years for wire helix packing was applied and showed quite good agreement for the two liquids involved, benzene and n-heptane.

Data of Struck<sup>17</sup> on n-decane was applied to the correlation and yielded fairly good results. However, Struck tested several different packings and each one of them considered alone gave rise to a separate line of different slope from Reed and Fenske's line indicating there is still some packing characteristic not accounted for. Effect of pressure seemed to be well accounted for since Struck's data included work at atmospheric pressure as well as several reduced pressures. But Reed and Fenske point out that a curve for data on tetraisobutane lies above the benzene curve showing absence of some physical property since the large difference in molecular weight is taken into account.

Struck and Kinney<sup>18</sup> present data on holdup which indicates that holdup varies linearly with distillation rate except at very low rates and increases with decreasing head pressure. This agrees with work by Collins and Lantz<sup>3</sup> and Elgin and Weiss<sup>4</sup>. However, Jesser and Elgin<sup>7</sup> present data which shows exponential variation of holdup with

flow rate. Most investigators in absorption work have found that below the loading point, gas rate had no effect on holdup. As a result most work on holdup in absorption has been carried out with no gas flow. In distillation, vapor rate and liquid rate are related. Theoretically they are equal, if assumption of no heat losses is made. Thus an increased boilup rate raises the reflux rate and holdup should be expressed as function of the vapor mass rate since vapor rate changes would control.

Struck also was not able to correlate holdup at one pressure with that at another by use of Jesser and Elgin's proposed relationship with density, viscosity and surface tension.

From the above review of past attempts at correlation of flow data, it can be seen that more work is needed to obtain sufficient information on design variables. The following objectives are set for this work:

1. Application of previous methods to experimental data on various liquids.
2. Comparison of results of different correlations and selection of preferred method, if any.
3. Investigation of possible new or revised correlations to determine whether such are possible with present data.

## MATERIALS AND EQUIPMENT

### Materials

The following liquids were used in this investigation:

Methyl Alcohol  
Ethyl Alcohol  
Isopropyl Alcohol  
Normal Butyl Alcohol  
Isoamyl Alcohol  
Toluene  
Xylene

All of the above were of reagent quality and physical properties agreed well with accepted values.

These liquids were used for several reasons. Most of the published data includes use of hydrocarbons. Use of other liquids will provide information as to variations due to the influence of physical properties. Investigation of the family of alcohols should provide data on compounds similar in nature chemically, but with increasing differences in physical properties. Toluene and Xylene were included to tie in if possible with some published data.

All liquids were investigated individually, that is, no fractionation or tests on efficiency were attempted. This avoided complicating variables such as heats of solution, variation of still composition and thus of boiling point during a run, and inaccuracies in calculations of densities, viscosities, heats of vaporization of mixtures and so forth.



TABLE 1

PHYSICAL PROPERTIES OF LIQUIDS

	Methyl Alcohol	Ethyl Alcohol	Iso- propyl Alcohol	n- butyl Alcohol	Isoamyl Alcohol	Toluene	Xylene
Molecular Weight	32.0	46.1	60.1	74.1	88.2	92.1	106.2
Specific Gravity 20°/4°C.	0.792	0.789	0.789	0.810	0.813	0.866	0.863
Liquid Viscosity @ 20°C., cp.	0.60	1.20	2.25	2.90	4.40	0.63	0.66
Surface Tension @ 20°C., dynes/cm.	22.6	22.8	21.7	21.6	-	28.5	28.5
Normal Boiling Point, °C.	64.7	78.4	82.5	117.0	132.0	110.8	139.0
Latent Heat of Vaporization, cal/gm.	263	204	172	141	120	87	82
Vapor Density at n B.P., lbs./cu.ft.	0.071	0.099	0.127	0.144	0.162	0.181	0.196

The physical properties of these liquids are shown in Table 1. Variation of properties with temperature and pressure is shown as follows:

Figure 1. Pressure vs. Boiling Temperature  
(Vapor Pressure Curves)

Figure 2. Pressure vs. Vapor Density at Boiling Point

Figure 3. Viscosity vs. Temperature for Liquids

Figure 4. Viscosity vs. Temperature for Vapors

Figure 5. Latent Heat of Vaporization vs. Temperature

### Equipment

A schematic diagram of equipment used is shown in Figure 6.

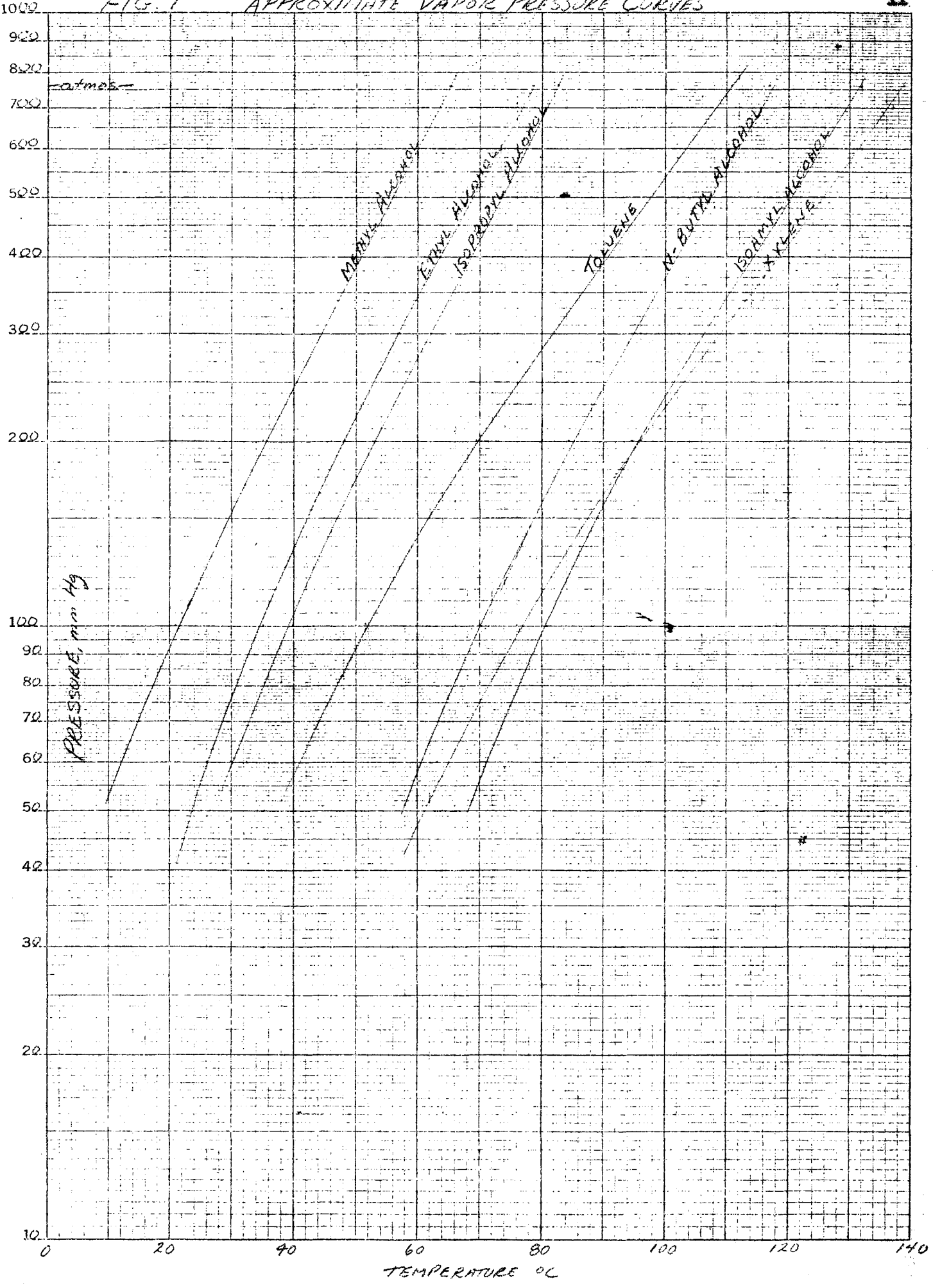
The column consisted of a one inch diameter Pyrex glass tube with a 35 inch packed section, topped by a vacuum-jacketed stillhead with solenoid operated liquid reflux divider. Reflux ratio was controlled by a Flexopulse timer. A 12 inch reflux condenser was found sufficient to condense all the vapors at the boilup rates used. The still was a one liter, three necked Pyrex flask, heated by a Glas-Col mantle. Voltage input to the still heater was controlled by a Variac transformer.

Product was collected in a graduated cylindrical receiver. The reflux trap consisted of a graduated cylinder with stopcock for drainage and a side arm which served as the vapor line to the column.

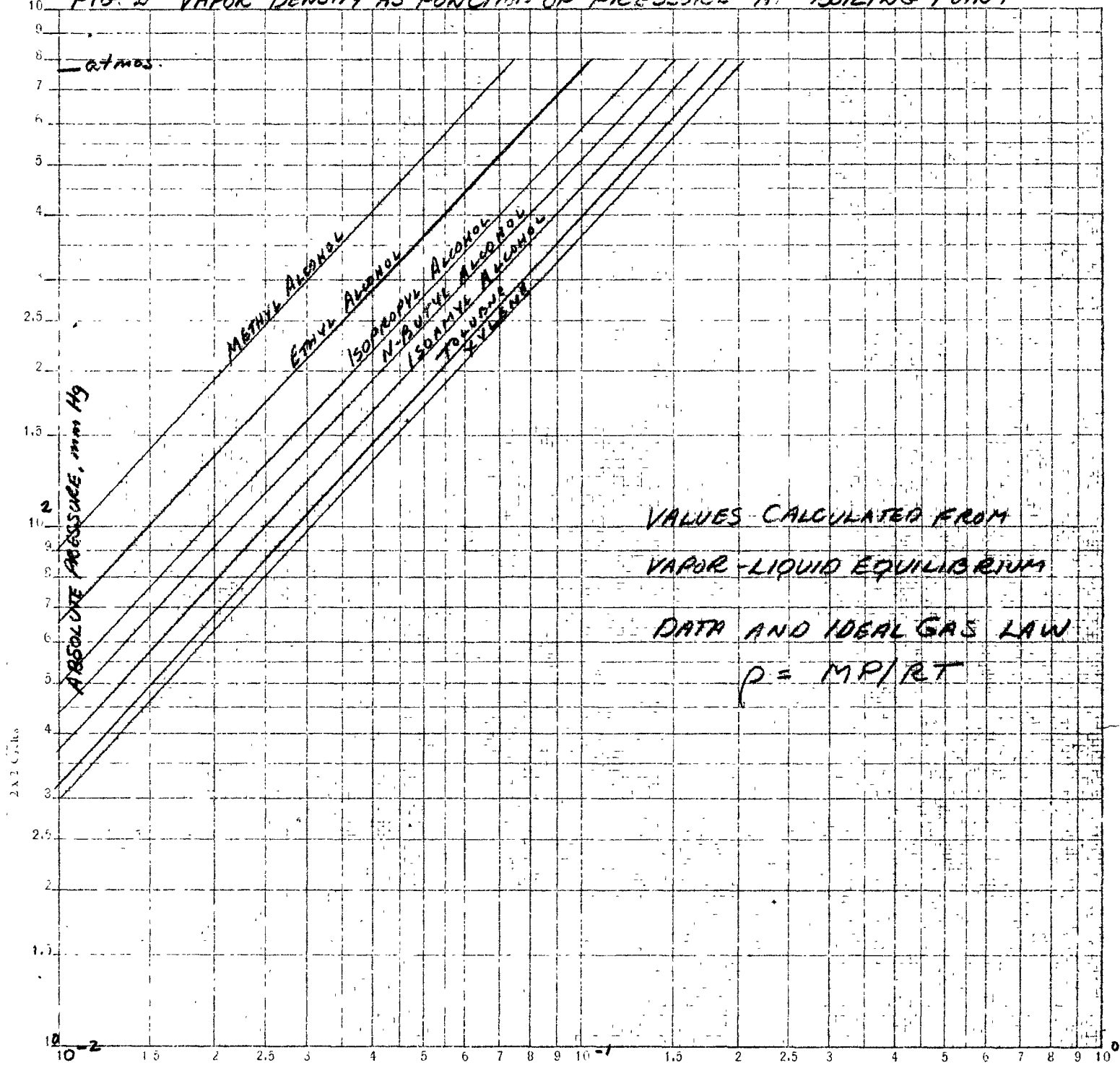
The column was lagged with one inch magnesia insulation. Exposed glass between the still and stillhead was lagged with asbestos rope.

Pressure taps were provided by adapters above and below the

FIG. 1 APPROXIMATE VAPOR PRESSURE CURVES



3 FIG. 2 VAPOR DENSITY AS FUNCTION OF PRESSURE AT BOILING POINT



$\rho$ , VAPOR DENSITY, lbs/ft<sup>3</sup>

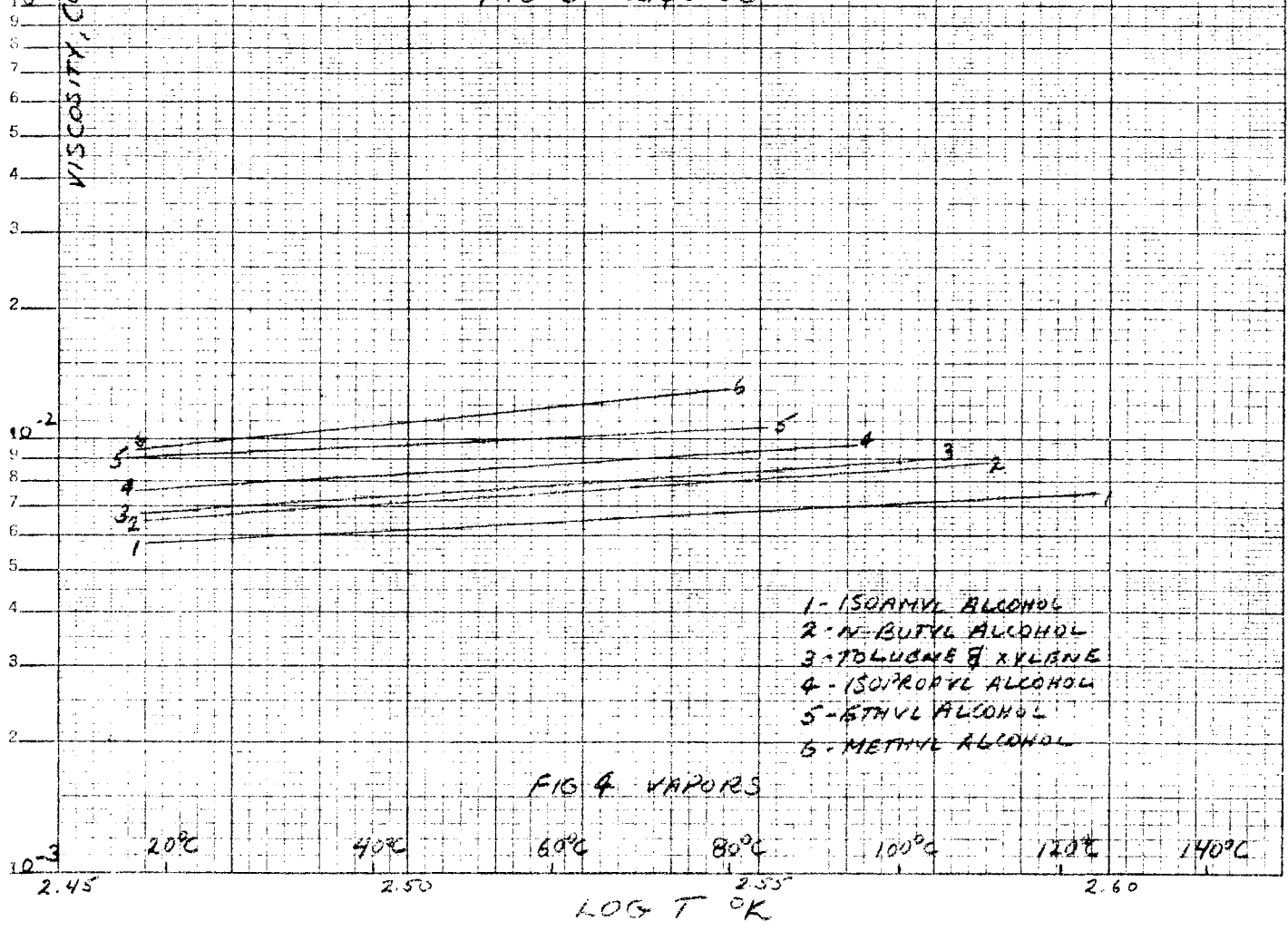
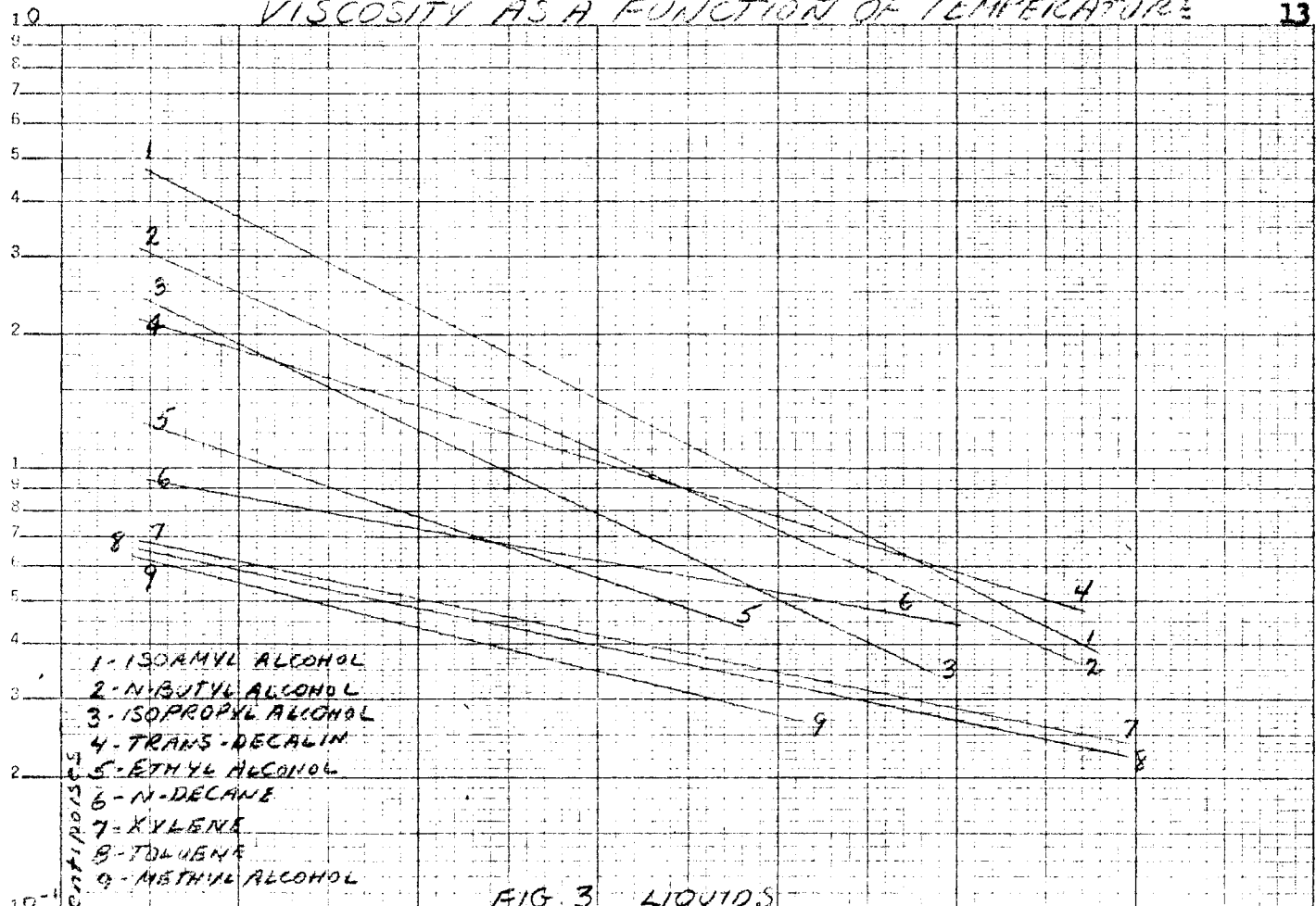


FIG. 5

VARIATION OF LATENT HEAT OF VAPORIZATION  
WITH TEMPERATURE  
Calculated by Method of Watson

LATENT HEAT OF VAPORIZATION, cal/gx.

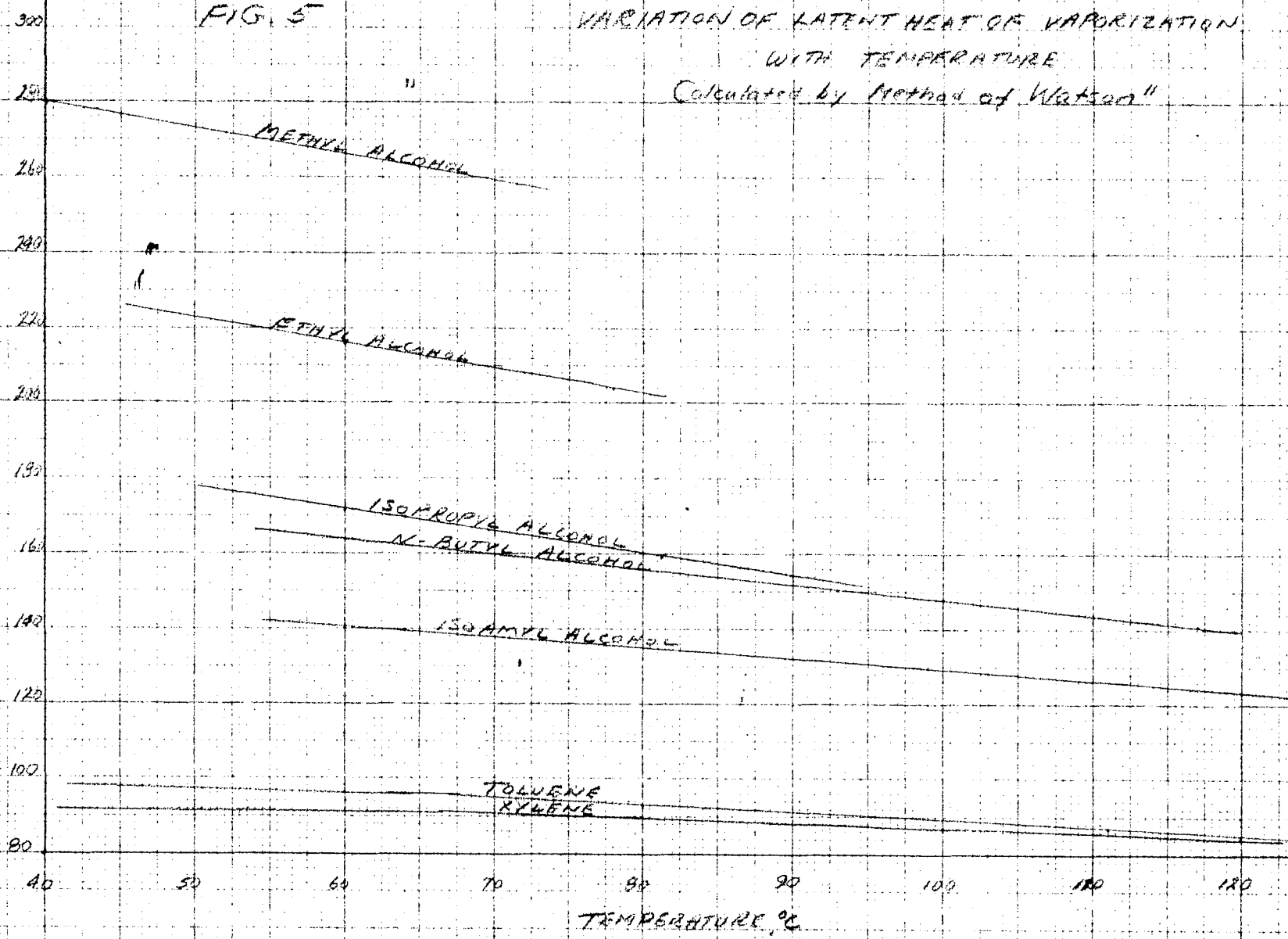
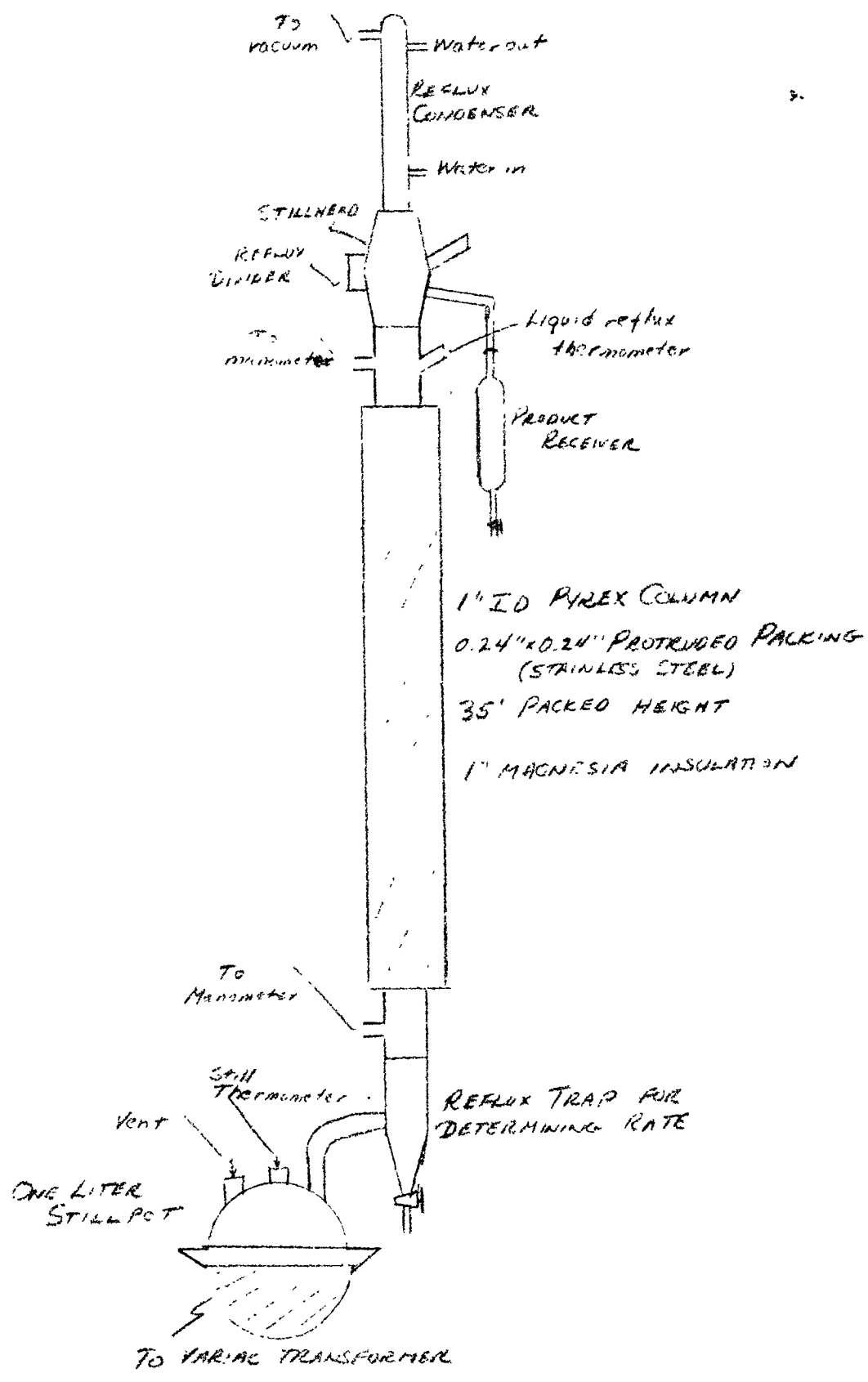


FIG. 6 APPARATUS FOR DETERMINATION OF PRESSURE DROP & HOLDUP IN VACUUM DISTILLATION <sup>15</sup>



column to give the actual column pressure drop. Lines from these taps were connected through vapor traps to a water filled U tube. Operating pressure was determined by a mercury manometer in the vacuum line. Temperatures were read from thermometers in the still, in the vapor line (stillhead) and in the reflux return to the column under the stillhead.

The system was evacuated by a Cenco "Megavac" vacuum pump connected through a dry ice trap and surge tank to the system. Pressure was controlled during vacuum runs by a mercury actuated manostat connected to the vacuum pump and system through a Skinner electric valve.

The column was packed by slowly pouring in 0.24" x 0.24" protruded stainless steel packing, gently tapping the column while filling to a height of 35 inches. A total of 124 grams of packing was used, or approximately 3710 pieces at 0.0335 grams each. The volume of the empty column was 400 cc water at 10°C., while the packed column held 360 cc. The void space of the packing as used was therefore, 90% as compared to the manufacturer's value of 96%<sup>13</sup>. Packed bulk density of the packing was 19.5 lbs./cu.ft. compared to 22.2, and surface/volume ratio was 396 sq.ft./cu.ft. packed volume compared to 372. Static holdup was 25 cc water determined by filling the column with water and allowing to drain for 15 minutes.



### PROCEDURE

The still was charged with approximately 500 cc of the material to be tested. The system was evacuated to the desired operating pressure and the still heated. Material was allowed to reflux at a given rate until the pressure drop was constant. Pressure drop and still, liquid reflux and vapor temperature readings were taken. Bottom rate was obtained by draining the trap and determining the time required to fill trap to marked volume (in duplicate). Top rate was determined by taking off product over a period of 10 to 15 minutes at a reflux ratio of 10:1 (accurately timed). The average boilup rate was determined as the arithmetic average of top and bottom rates to minimize the effects of heat loss through lagging since there was no way to heat the column. The heat loss was considerable with the higher boiling point liquids. Holdup was obtained as volume of drainage from column (less trap holdup) for 15 minutes after heat was removed and the system vented from product receiver. The column was drained and dried under vacuum for 24 hours between runs with different liquids.

During repeat runs at any one pressure, the exact pressure could not always be duplicated. Effects of this small variation are corrected for in the calculations. For example, where density is involved, the density is calculated based on the exact pressure determined from the temperature and vapor pressure charts.

## RESULTS

The results of the individual runs are shown in Table 2 and Figures 9 to 19. For each material, pressure drop and holdup are shown as functions of mass velocity. The mass velocity of liquid and vapor are assumed to be equal.

Change of pressure drop with mass rate for each liquid is shown in Figures 7 to 13. It can be seen that for each liquid at any one pressure, pressure drop increases exponentially with increase of mass rate. At constant rate, pressure drop increased as head pressure was reduced. This effect of pressure was more noticeable below 300 mm Hg. In fact above this pressure, the data are quite close to those at atmospheric pressure.

In general, at any one head pressure, the mass rate required to produce a given pressure drop was higher for a material of higher molecular weight than the one previously tested. This was not strictly true at all pressure drops since the slopes of the individual curves varied at different head pressures and among different materials. Scattering of data did not permit exact determination of slope but the trend indicated decreased slopes at lower head pressures.

Change of liquid holdup with mass rate for each liquid is shown in Figures 14 to 19. These curves also show a definite exponential relationship of holdup to rate in agreement with Jesser and Elgin<sup>7</sup> and Streib<sup>16</sup>. While insufficient data are available to determine the characteristics of each curve at each pressure, again there is a difference in trend of slope at various pressures indicating some effect

of pressure on one of the physical properties affecting holdup. While holdup data were not obtained for all liquids at all pressures, the results show less change of holdup with variation in rate with increase of molecular weight at any one pressure. In absence of a large amount of accurate data, no quantitative conclusions can be drawn, since inaccuracies of the method used for the holdup determination are large. With a small column, a small amount of holdup in parts of the unit outside the packing can lead to a large percentage error in the volume of drainage.

However with this particular packing, of which one of the features is low holdup, there is little effect of pressure on total volume of operating holdup. The holdup observed was of the order of five to twelve percent.

TABLE 2  
EXPERIMENTAL AND CALCULATED RESULTS

Pressure		G	G/ $\phi$	$\Delta P$ $\times 10^3$	$\rho \Delta P$ $\times 10^4$	H $\times 10^3$
<b>METHYL ALCOHOL</b>						
1	atmos.	191	193	150	110	66
2		203	206	110	75	-
3		229	234	210	150	71
4		242	247	130	92	114
5		326	334	300	220	76
6		338	340	370	290	76
7		368	377	300	210	121
8	400mm Hg	179	241	150	62	49
9		200	269	210	88	-
10		214	282	170	74	66
11		262	344	260	110	124
12		262	344	330	140	115
13		271	357	260	110	84
14	300mm Hg	179	306	150	38	61
15		212	366	210	55	89
<b>ETHYL ALCOHOL</b>						
1	atmos.	244	210	110	110	49
2		247	225	110	96	-
3		270	233	130	130	69
4		368	315	370	370	85
5		406	350	300	300	68
6		445	392	430	420	175
7	400mm Hg	100	207	130	66	-
8		161	188	65	37	59
9		161	194	65	28	-
10		223	268	150	76	65
11		226	273	170	88	-
12		288	328	190	110	79
13	300mm Hg	161	207	86	39	59
14		197	252	110	50	72
15		235	313	240	100	-
16		260	324	210	100	71
17		280	378	370	150	-
<b>ISOPROPYL ALCOHOL</b>						
1	atmos.	139	107	32	41	-
2		174	134	43	55	49
3		224	174	110	130	-
4		247	190	150	190	62
5		256	198	86	110	86
6		286	220	150	190	-

(Table 2 cont'd)

		G	G/ $\phi$	$\Delta P$ $\times 10^3$	$P\Delta P$ $\times 10^4$	H $\times 10^3$
7		346	267	170	220	79
8		417	324	210	270	111
9		450	350	240	300	-
10	400mm Hg	179	194	65	42	69
11		183	200	86	54	-
12		248	262	86	54	75
13		249	272	150	95	-
14		346	372	150	97	81
15	300mm Hg	185	244	110	47	63
16		269	350	170	76	72
17		293	378	150	68	-
18		372	455	280	140	76

## n-BUTYL ALCOHOL

1	atmos.	179	130	75	110	63
2		230	175	110	140	75
3		240	196	86	100	-
4		413	324	170	210	81
5	300mm Hg	143	161	32	19	60
6		210	250	53	28	44
7		222	251	43	25	25
8	100mm Hg	93	193	32	5.6	-
9		128	246	53	11	-
10		146	254	53	13	-
11		148	254	65	17	-
12		188	322	110	27	78
13		189	324	130	33	58
14		194	337	86	21	60
15	50mm Hg	103	306	190	16	48
16		126	352	320	31	55
17		150	418	390	38	65
18		187	524	490	47	58

## ISOAMYL ALCOHOL

1	150mm Hg	156	229	54	18	46
2		220	314	86	31	69
3		229	338	110	37	64
4		332	415	190	92	100
5	90mm Hg	94	217	54	8	41
6		150	282	130	28	54
7		159	302	130	27	-
8		252	415	210	59	73
9	40mm Hg	77	298	210	11	44
10		122	325	320	35	-
11		141	378	470	51	54
12		203	553	665	68	-
13		207	495	600	76	64

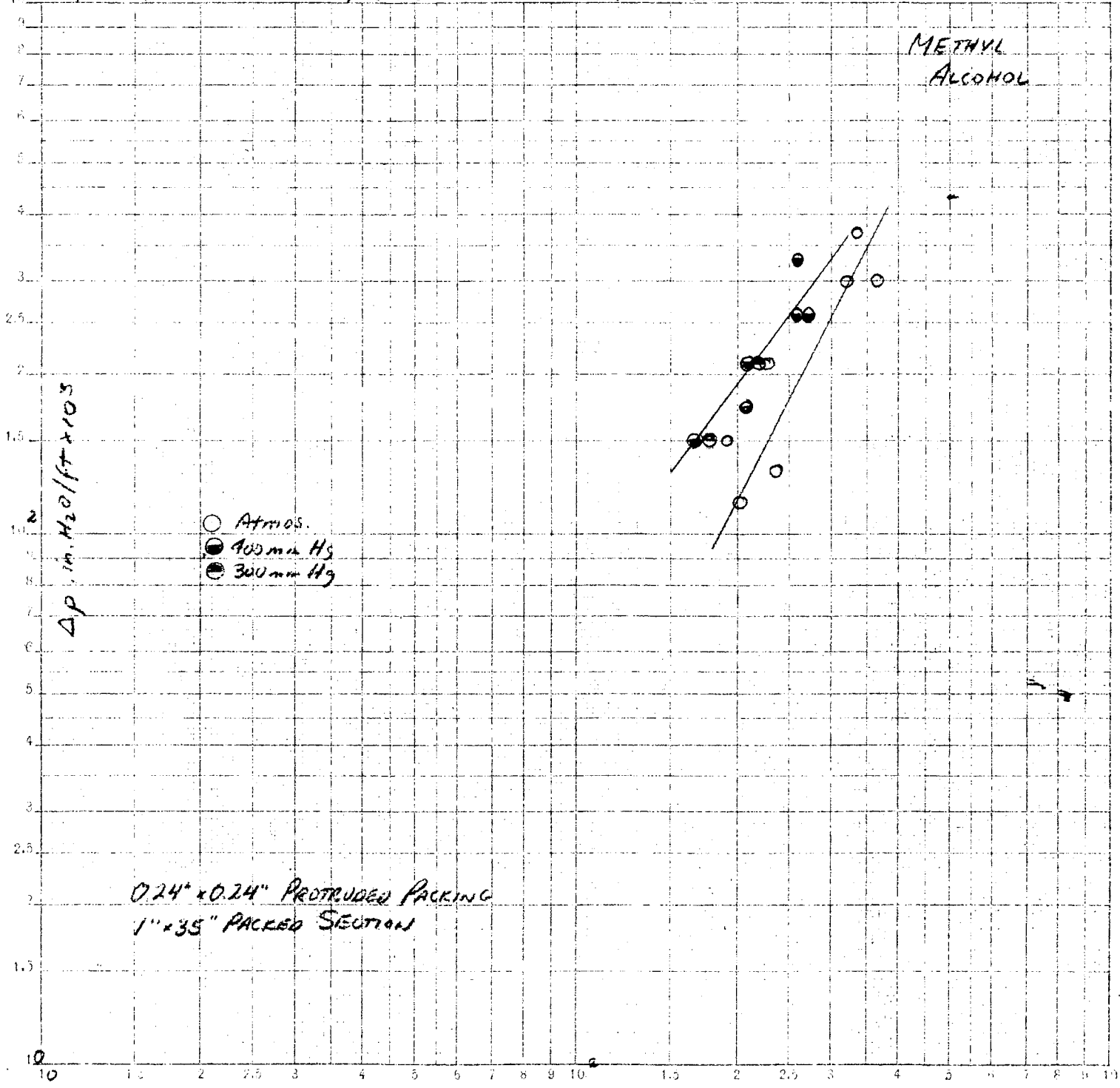
(Table 2 cont'd)

TOLUENE		G	G/φ	$\Delta p$ $\times 10^3$	$\rho \Delta p$ $\times 10^4$	H $\times 10^3$
1	atmos.	225	147	43	75	52
2		274	177	86	160	-
3		292	190	65	110	62
4		338	221	210	380	100
5		376	245	210	380	68
6	300mm Hg	150	162	21	14	40
7		231	232	64	48	90
8		249	265	43	28	-
9		315	344	86	54	56
10		348	370	86	54	30
11	150mm Hg	247	350	110	40	46
12		310	438	170	64	81
13		366	518	510	200	111
14	50mm Hg	143	322	210	52	54
15		161	450	470	60	-
16		202	457	650	60	-
17		203	540	1030	150	-
18		209	498	430	57	61
XYLENE						
1	300mm Hg	148	190	32	15	-
2		274	266	43	43	-
3		374	380	110	110	-
4	100mm Hg	108	196	43	10	-
5		174	266	110	34	-
6		286	426	170	62	-
7	40mm Hg	62	205	110	7	-
8		108	298	170	17	-
9		121	342	240	22	-
10		192	446	340	48	-

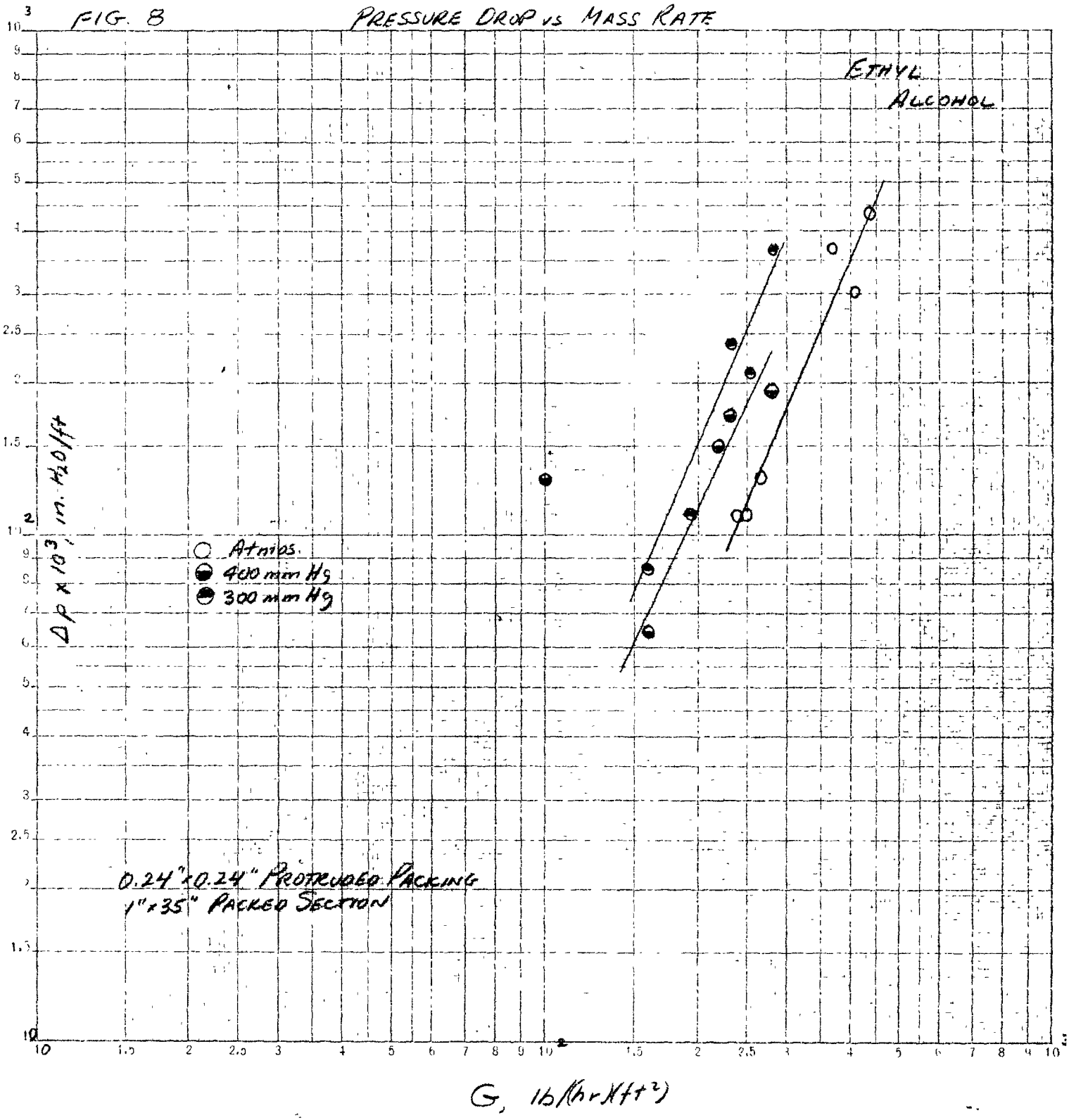
FIG. 7

PRESSURE DROP vs. MASS RATE

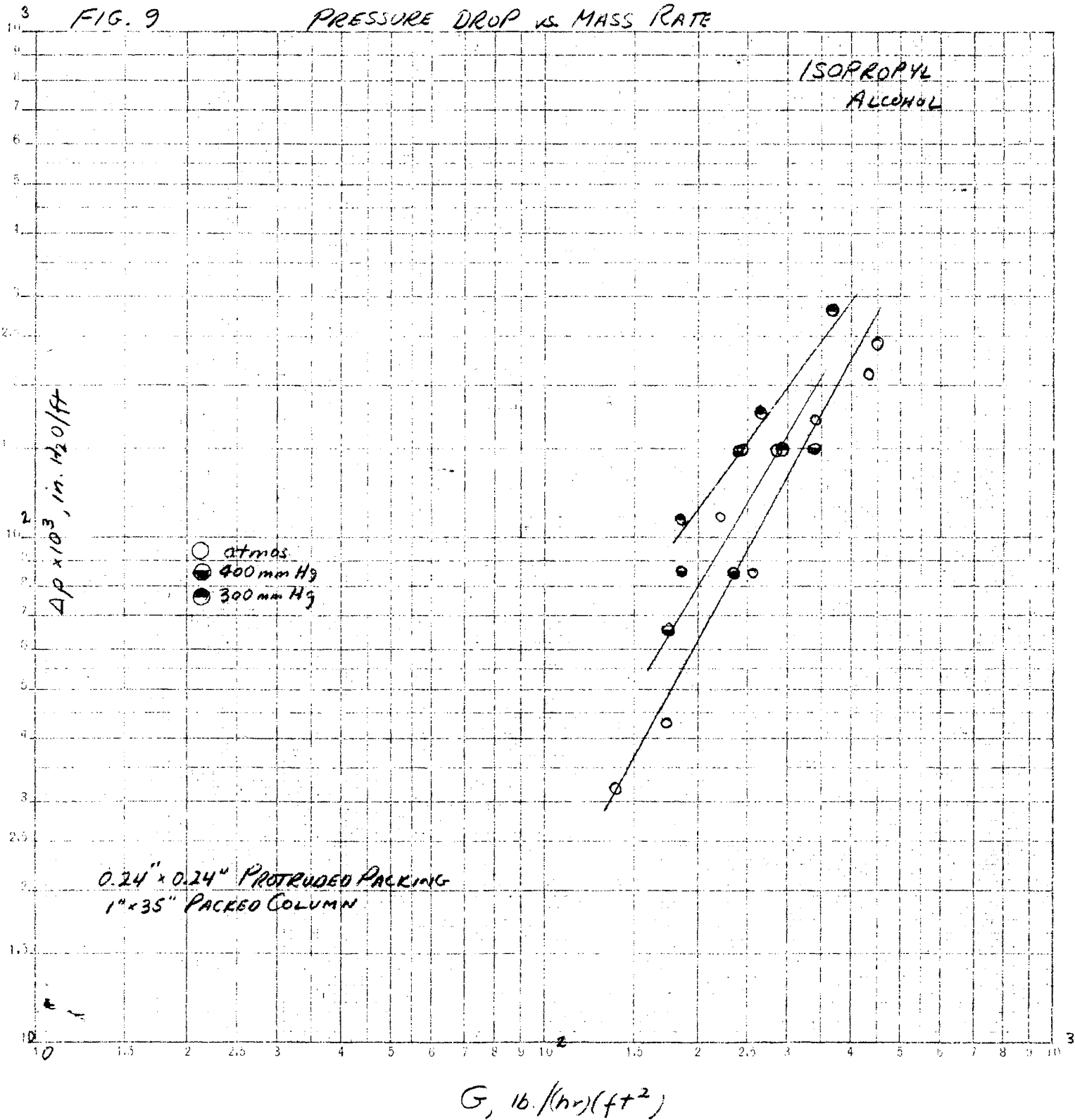
METHYL ALCOHOL



$G, \text{ lb/(hr)(ft}^2\text{)}$







3 FIG. 10

## PRESSURE DROP VS. MASS RATE

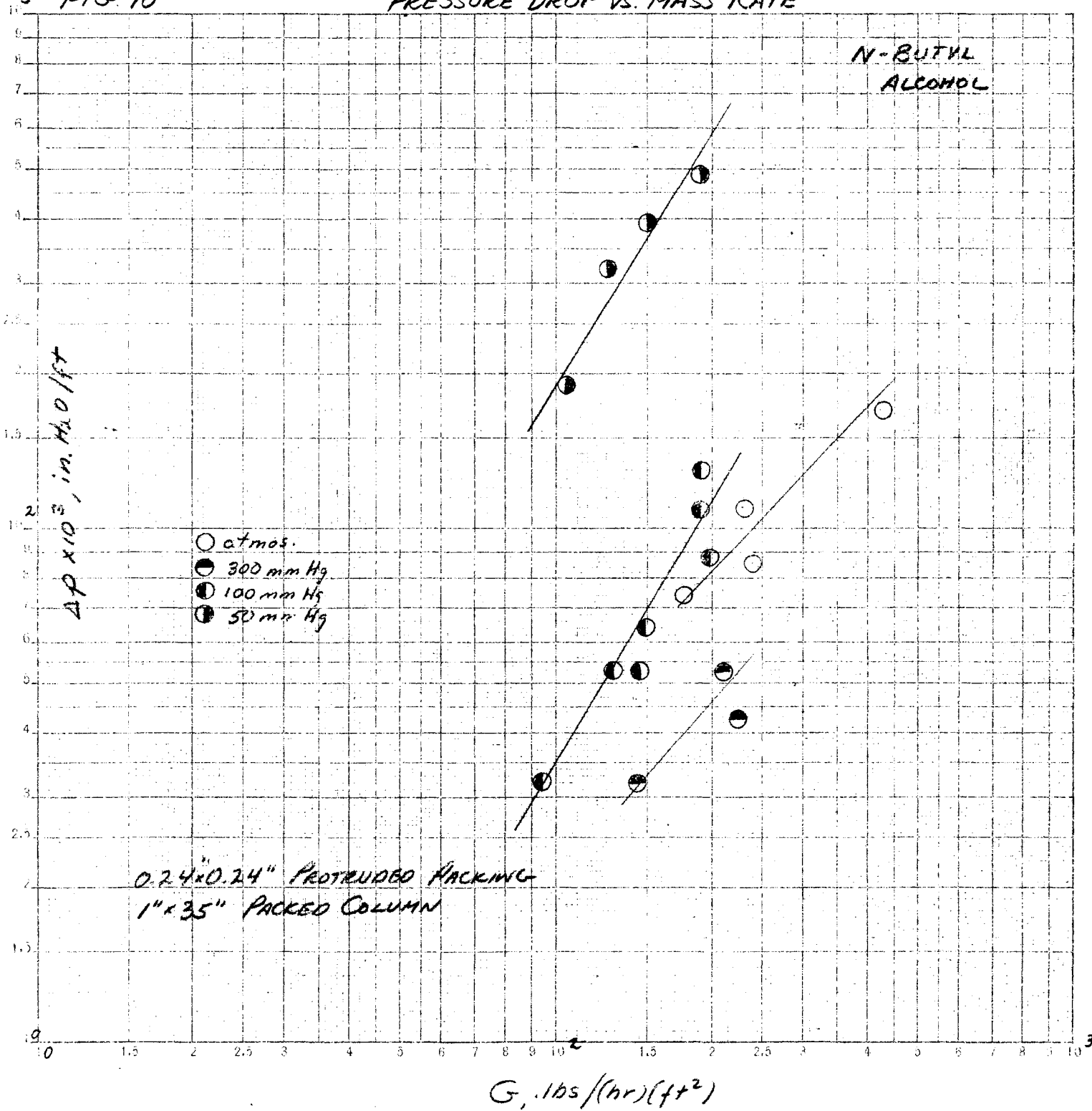
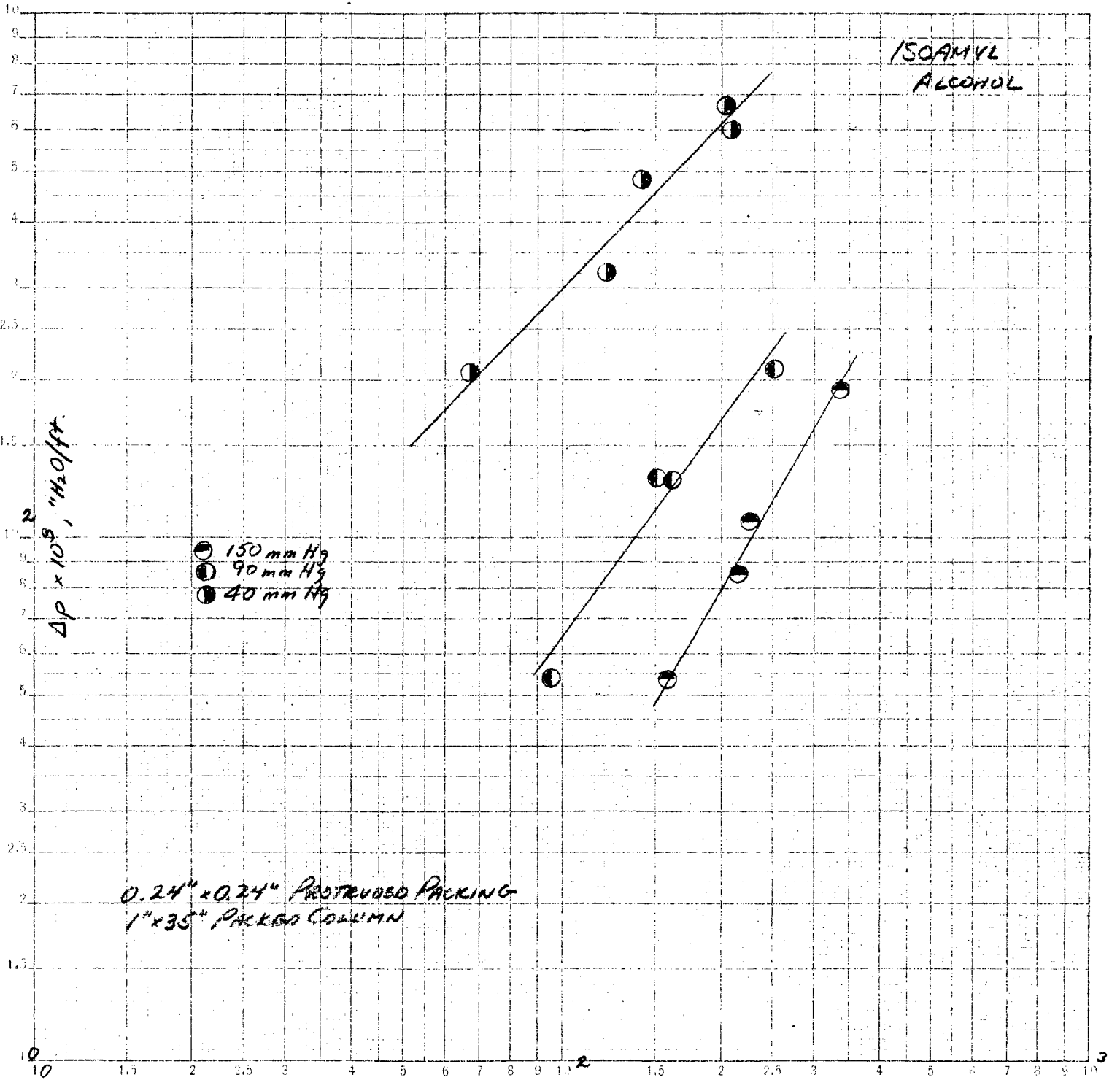


FIG. 11

PRESSURE DROP VS MASS RATE



ISOBUTYL  
ALCOHOL

- 150 mm Hg
- 90 mm Hg
- 40 mm Hg

0.24" x 0.24" PROTRUSO PACKING  
1" x 35" PACKED COLUMN

$G, \text{ lbs}/(\text{hr})(\text{ft}^2)$

$\Delta p \times 10^5, 1/40 \text{ ft.}$

FIG. 12

PRESSURE DROP VS. MASS RATE

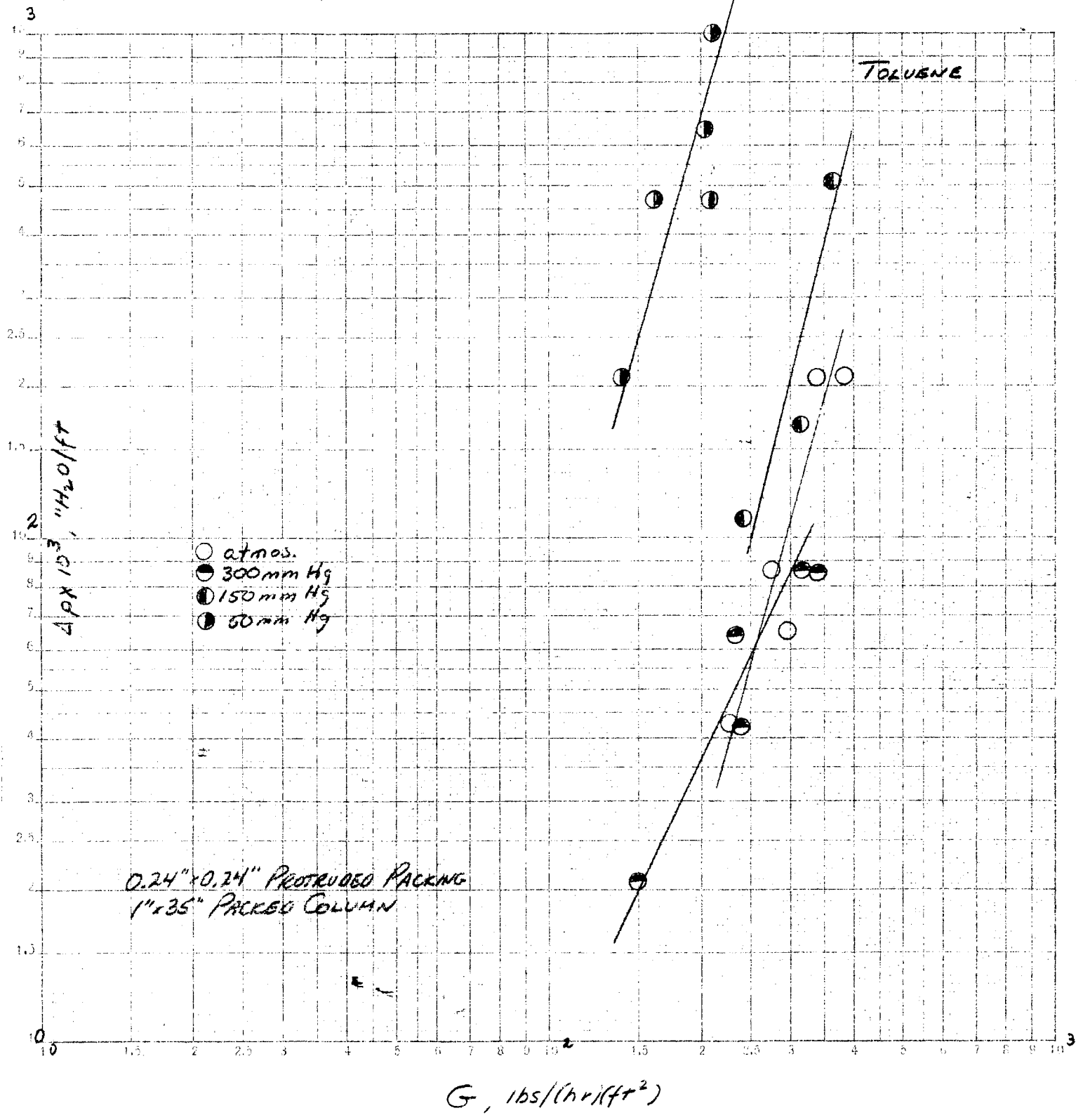
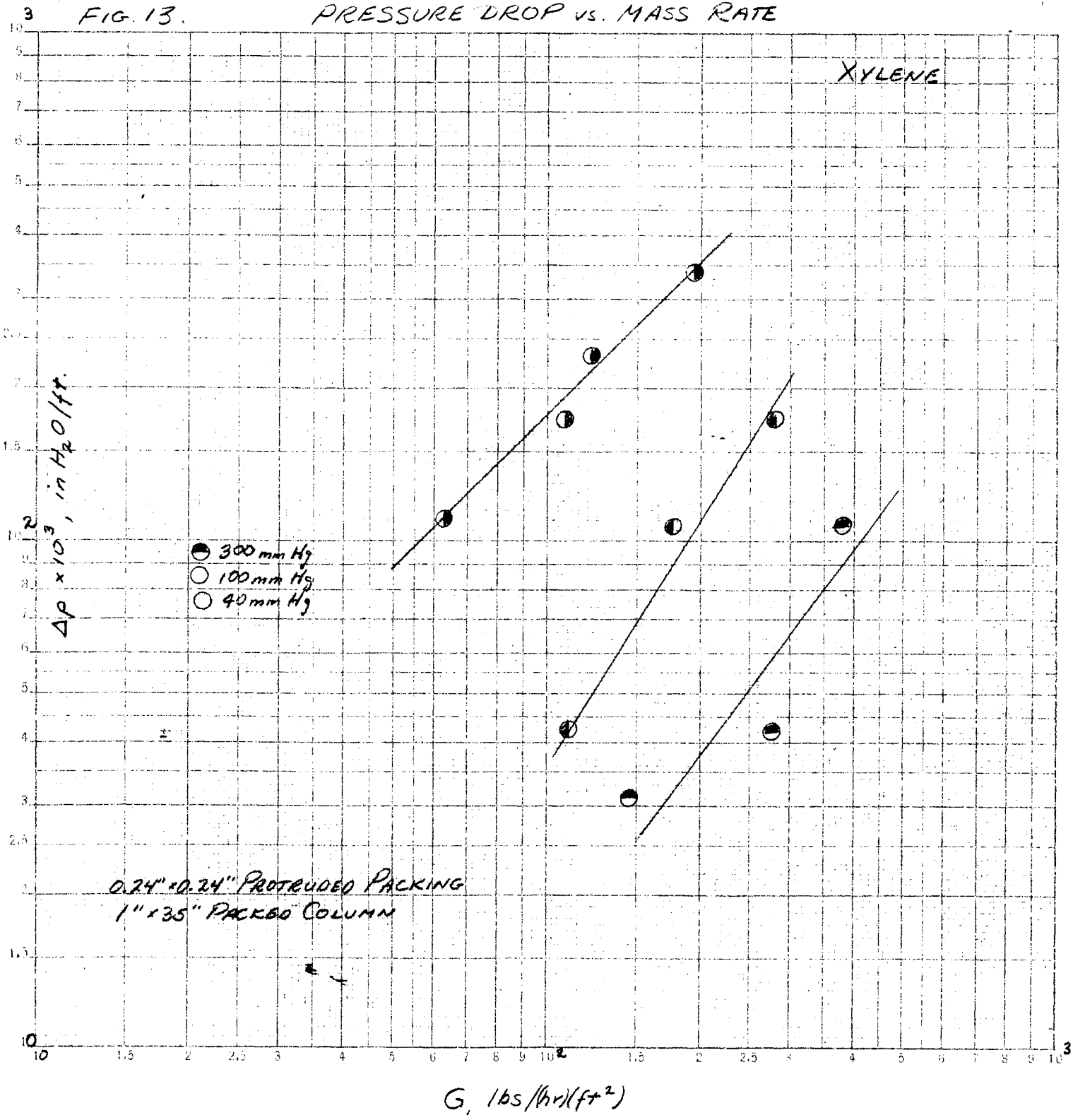


FIG. 13. PRESSURE DROP vs. MASS RATE



HOLDUP VS. MASS RATE

FIG 14 METHYL ALCOHOL

FIG 15 ETHYL ALCOHOL

- atmos
- 400 mm Hg
- 300 mm Hg

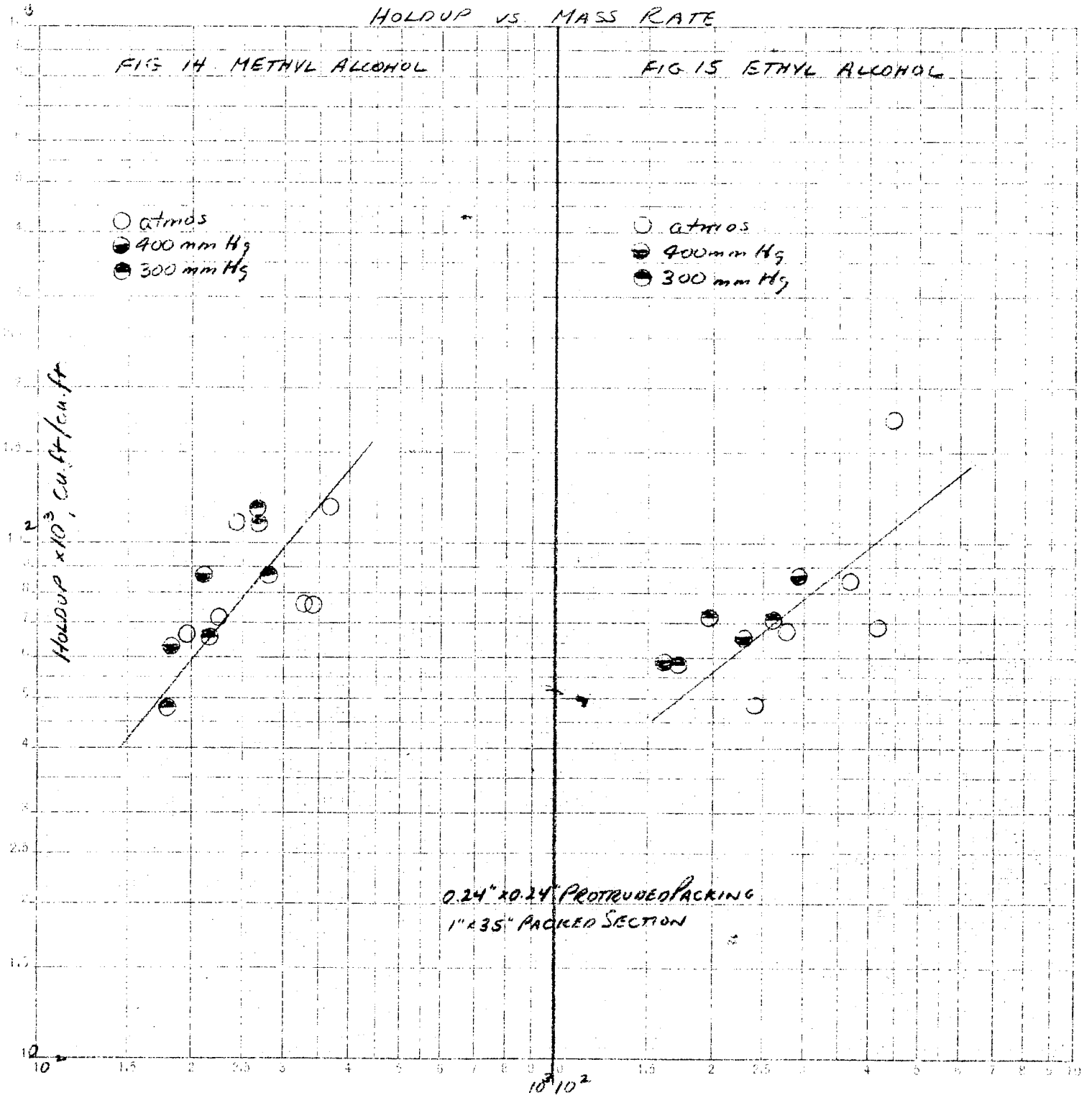
- atmos
- 400 mm Hg
- 300 mm Hg

Holdup  $\times 10^3$ , cu.ft./cu.ft.

0.24" x 0.24" PROTRUDED PACKING  
1" x 35" PACKED SECTION

$G$ , lb/(hr)(ft<sup>2</sup>)

$G$ , lb/(hr)(ft<sup>2</sup>)



HOLDUP VS. MASS RATE

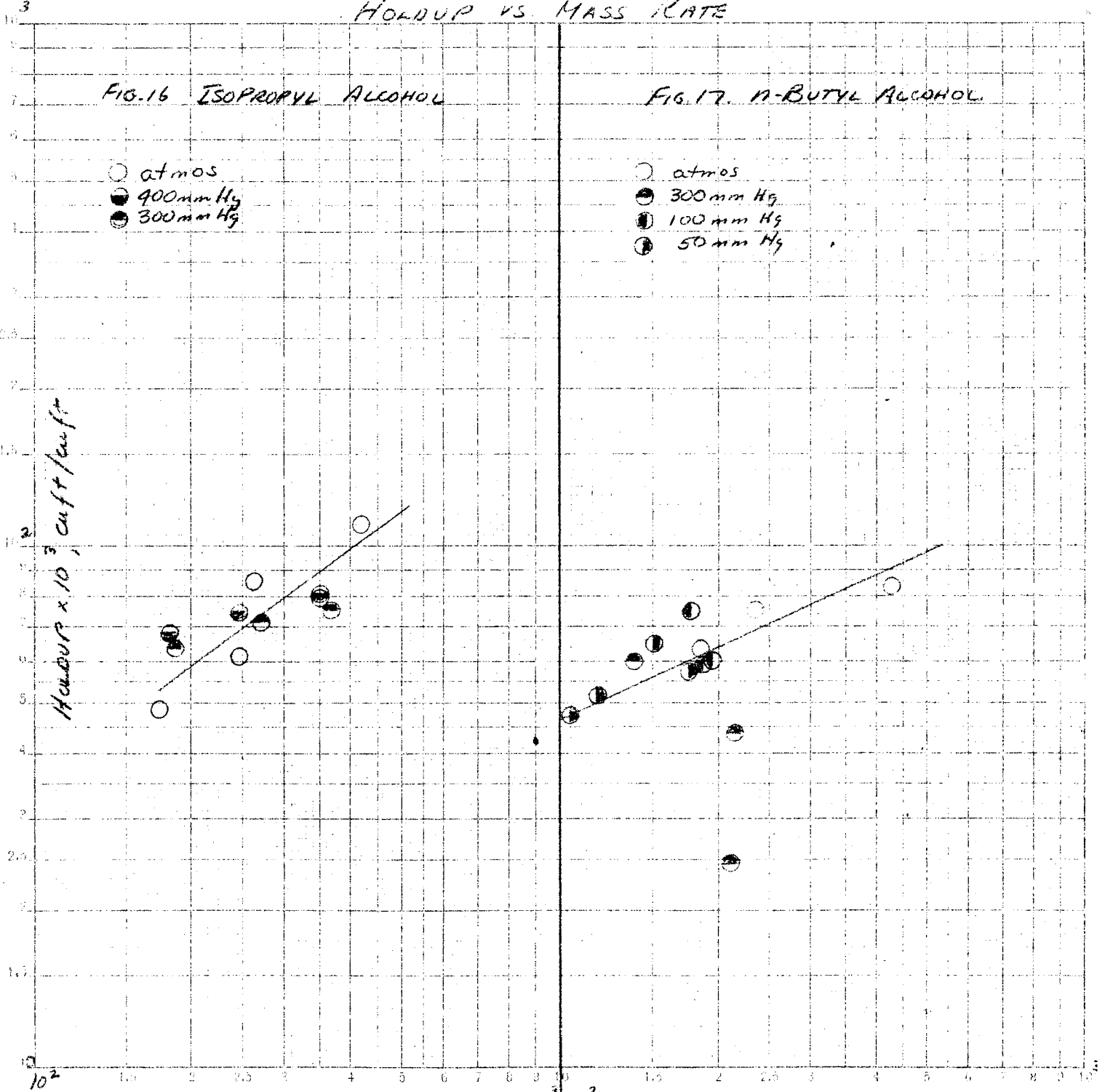
FIG. 16 ISOPROPYL ALCOHOL

- atmos
- 400 mm Hg
- 300 mm Hg

Holdup  $\times 10^3$ , cu ft / cu ft

FIG. 17 n-BUTYL ALCOHOL

- atmos
- 300 mm Hg
- 100 mm Hg
- 50 mm Hg



$G$ , lb/hr/ft<sup>2</sup>

$G$ , lb/hr/ft<sup>2</sup>

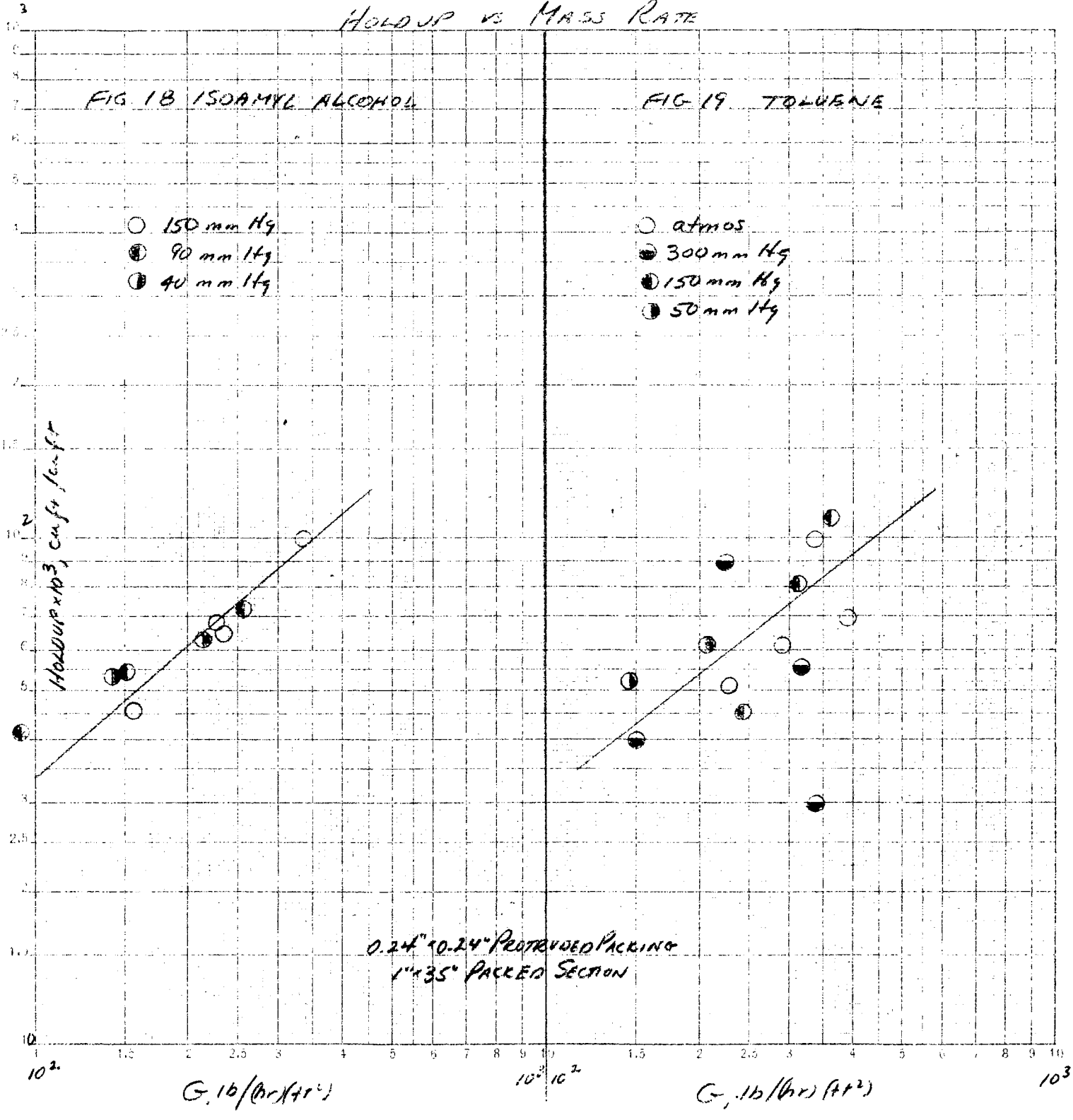
HOLDUP VS MASS RATE

FIG 18 ISOAMYL ALCOHOL

FIG 19 TOLUENE

- 150 mm Hg
- 90 mm Hg
- 40 mm Hg

- atmos
- 300 mm Hg
- 150 mm Hg
- 50 mm Hg





## DISCUSSION OF RESULTS

The effects of reduced pressure on pressure drop as shown above were as expected. At any mass rate, higher vapor velocity due to reduction in pressure should lead to greater friction losses and higher pressure drops. However, the differences between the various materials at different pressures lead to some questions. Among these are reasons for differences in slope of the pressure drop vs. mass rate curves at different pressures and among the different materials. It is assumed that these differences are due to different variations of physical properties with pressure and/or boiling temperature. These differences are applied in the development following.

Comparison of the data at various pressures on a common basis is the first step in a desirable correlation. Vapor density is the property most severely affected by pressure and in addition includes the effect of molecular weight. It would seem that use of vapor velocity (mass rate divided by density) would be valuable. However, a plot of pressure drop against vapor velocity spreads the data in some cases even more, so that the curves at different pressures are more distinct than in the pressure drop vs. mass rate plots. This is due to the large differences in density even at relatively close pressures. For example, with methyl alcohol, data at 300 and 400 mm Hg are quite close together when plotted against mass rate, but are well spread out plotted against vapor velocity. This latter representation accentuates the differences between pressures rather correlating the data.

The use of  $G/\sqrt{p}$  suggested by Forsythe, et al.<sup>5</sup> to correlate data

on pressure drop for various systems also spreads the data to some extent. The similar absorption correlation, using  $G/\phi$  with  $\phi$  based on the density of air at standard conditions yields better results. It would seem that both of these methods would be similar, but the present data when plotted as  $G/\phi$  vs.  $\Delta p$  appear in more of a continuous line over the pressures tested rather than as lines at each pressure as results from use of  $\Delta p$  vs.  $G/\sqrt{\rho}$ . Evidently the factor  $\sqrt{\rho/0.075}$  shows better the effect of density in the relationship between drop and mass rate than does  $\sqrt{\rho}$ .

Attempts to modify  $\phi$  by use of any other factor than 0.5 in the ratio  $(\rho/\rho_{air})^{0.5}$  also spreads the data into groups. Use of the density of air at the conditions of the run in question again has the same effect. These results point up those of Struck mentioned earlier. Struck used the density of n-decane at a specific pressure instead of the density of air and got diverging rather than coinciding curves. Again it would seem that  $\sqrt{\rho}$  should give satisfactory results, especially since the density of air has no connection with the problem. But the results obtained do not agree.

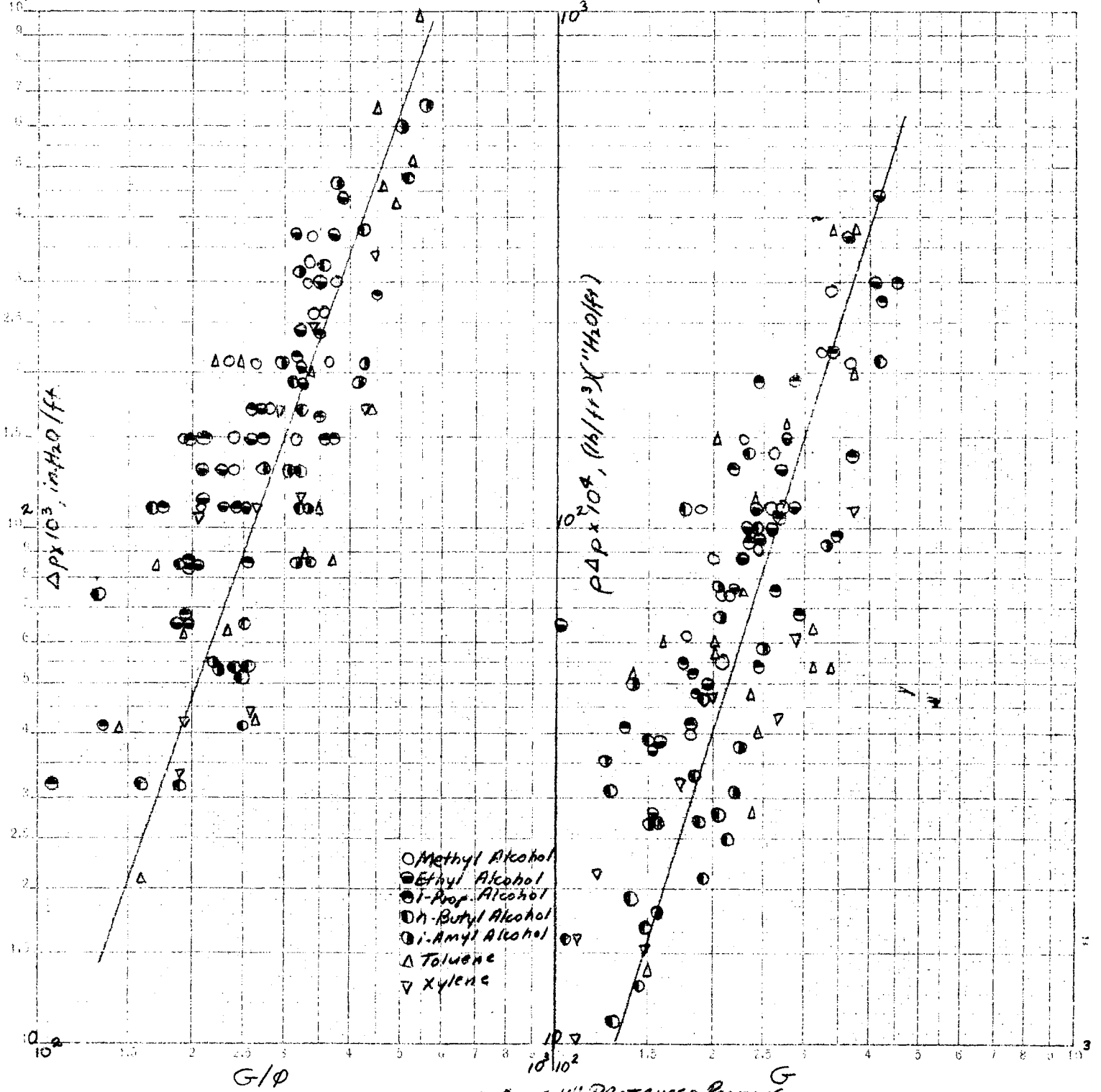
With the  $G/\phi$  vs.  $\Delta p$  curves plotted for each individual material the difference in slope of each curve can still be seen. This is the difference noted by Hand<sup>6</sup> and as shown below no simple correction factor can be applied. This difference can also be seen in the data of Peters<sup>12</sup> for n-decane and trans-decalin and ethylbenzene and chlorobenzene at several different pressures.

The combined plot of  $G/\phi$  vs.  $\Delta p$  for all the materials considered is shown in Figure 20. The data are presented in Table 3.

FIG. 20

$G/\phi$  vs.  $\Delta p$

$G$  vs.  $\rho \Delta p$



0.24" x 0.24" PROTRUDED PACKING  
1" x 35" PACKED COLUMN

TABLE 3  
CORRELATIONS

Pressure		$G/\mu_v$	$\frac{\rho \Delta p}{\mu_v^2} \times 10^6$	$G/\mu_l$	$\frac{\rho \Delta p}{\mu_l^2} \times 10^3$	$G/\mu_v S$	$\frac{\rho \Delta p (f_0 - H)^3}{\mu_v^2 S^3}$
<b>METHYL ALCOHOL</b>							
1	atmos.	5620	49	224	78	18	291
2		6000	37	240	59	-	-
3		6760	68	270	108	22	405
4		7150	41	285	66	23	236
5		9650	100	384	160	31	720
6		10000	130	400	208	32	672
7		10800	94	432	151	35	483
8		5600	31	182	33	18	157
9		6260	46	204	48	-	-
10		6700	38	218	40	21	182
11		8220	58	267	61	26	242
12		8220	72	267	76	26	300
13		8500	59	276	62	27	298
14	300mm Hg	6180	25	165	18	19	87
15		7300	32	195	23	22	115
<b>ETHYL ALCOHOL</b>							
1	atmos.	8220	200	202	120	24	273
2		8320	184	204	110	-	-
3		9100	234	223	140	27	313
4		12400	652	304	390	37	796
5		13700	535	336	320	39	630
6		15000	740	368	440	44	805
7	400mm Hg	3850	150	69	48	-	-
8		6250	75	112	24	17	79
9		6250	78	112	25	-	-
10		8720	178	156	57	23	171
11		8820	204	158	65	-	-
12		11000	253	200	81	30	235
13		5500	72	92	20	17	84
14		6700	93	112	26	21	106
15		8100	173	135	48	-	-
16		9000	186	150	52	28	212
17		9600	266	160	74	-	-
<b>ISOPROPYL ALCOHOL</b>							
1	atmos.	6160	43	115	15	-	-
2		7720	55	144	19	20	107
3		15300	136	285	47	-	-
4		11000	193	204	67	28	355
5		11300	102	210	39	29	198
6		12700	193	236	67	-	-

(Table 3 cont'd)

		$G/\mu_v$	$\frac{\rho \Delta P}{\mu_v^2} \times 10^6$	$G/\mu_l$	$\frac{\rho \Delta P}{\mu_l^2} \times 10^3$	$G/\mu_v S$	$\frac{\rho \Delta P (f_0 - H)^3}{\mu_v^2 S^3}$
7		15400	237	286	82	39	378
8		18500	278	314	96	47	427
9		20000	304	372	105	-	-
10		8220	46	106	7.6	22	79
11		8300	59	107	9.8	-	-
12		11300	57	146	9.4	29	101
13		11400	103	147	17	-	-
14		15800	59	204	9.7	42	177
15	300mm Hg	7700	43	85	5.2	23	90
16		10300	70	124	8.5	33	145
17		12300	63	135	7.6	-	-
18		15500	132	171	16	46	252

## n-BUTYL ALCOHOL

1	atmos.	7150	79	188	55	21	476
2		9100	117	240	81	27	597
3		9500	82	250	57	-	-
4		16000	173	430	120	49	886
5	300mm Hg	6750	9	99	1.9	18	100
6		9900	13	145	2.7	27	144
7		10400	12	153	2.5	28	129
8	100mm Hg	4360	7	40	0.55	-	-
9		6100	13	56	1.1	-	-
10		6900	15	63	1.3	-	-
11		7100	20	65	1.7	-	-
12		9000	31	82	2.6	25	153
13		9050	38	83	3.2	26	212
14		9150	25	84	2.1	26	118
15	50mm Hg	5260	22	30	.72	15	109
16		6500	43	37	1.4	18	203
17		7700	52	44	1.7	21	238
18		9650	65	55	2.1	26	300

## ISOAMYL ALCOHOL

1	150mm Hg	9800	39	93	3.5	24	127
2		13800	63	131	5.7	33	32
3		14300	75	136	6.8	35	94
4		20700	180	196	17	50	186
5	90mm Hg	6200	18	43	0.9	15	86
6		10000	64	69	3.1	24	199
7		10500	60	73	2.9	-	-
8		16700	135	116	6.5	39	438
9	40mm Hg	4500	19	23	0.5	13	110
10		7100	62	36	1.6	-	-
11		8300	88	42	2.3	23	600
12		11800	120	60	3.1	-	-
13		11800	136	60	3.5	34	294

(Table 3 cont'd)

		$G/\mu v$	$\frac{\rho \Delta p}{\mu v^2}$ $\times 10^6$	$G/\mu_1$	$\frac{\rho \Delta p}{\mu_1^2}$ $\times 10^3$	$G/\mu v S$	$\frac{\rho \Delta p (f_0 - H)^3}{\mu v^2 S^3}$
<b>TOLUENE</b>							
1	atmos.	11200	99	372	110	27	135
2		13600	198	452	220	-	-
3		14600	144	485	160	35	192
4		16800	470	560	520	41	615
5		18600	477	622	530	45	597
6	300mm Hg	7400	17	183	11	19	23
7		11300	60	282	37	29	75
8		12200	35	304	22	-	-
9		15400	69	384	43	41	80
10		17100	68	425	42	43	79
11	150mm Hg	14600	72	255	22	34	57
12		18300	115	320	35	42	81
13		21600	346	378	106	50	218
14	50mm Hg	8500	104	118	20	20	66
15		9600	109	133	21	-	-
16		12000	109	167	21	-	-
17		12000	274	167	53	-	-
18		12400	109	172	21	-	-
<b>XYLENE</b>							
1	300mm Hg	6150	14	203	16	-	-
2		13100	49	430	53	-	-
3		18000	134	590	144	-	-
4	100mm Hg	5650	4	127	7.1	-	-
5		9100	50	204	25	-	-
6		15000	87	336	44	-	-
7		2870	8	49	2.4	-	-
8		5050	18	86	5.5	-	-
9		5740	24	98	7.2	-	-
10		8870	54	152	16	-	-

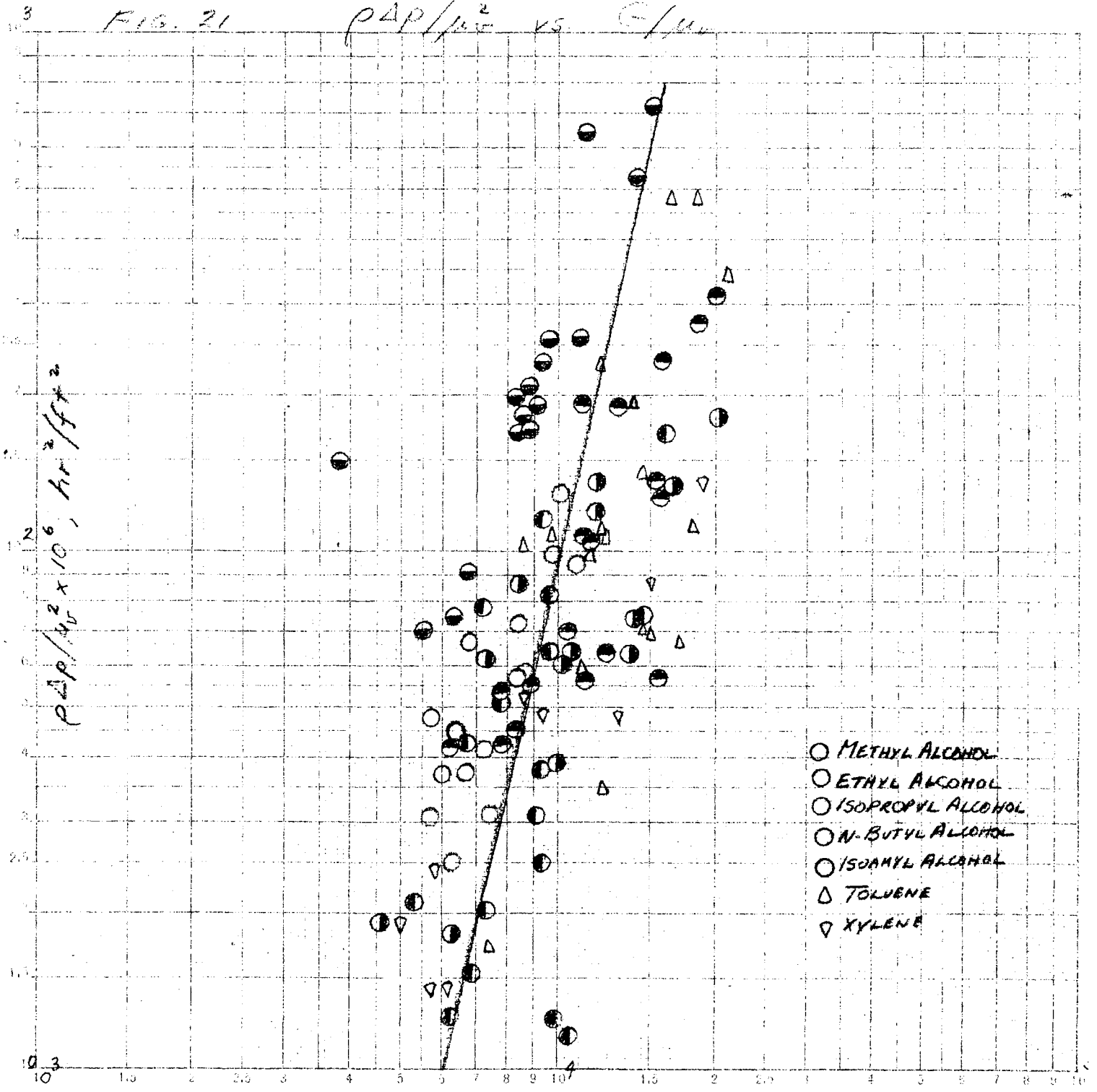
Also shown is a plot of a suggested correlation  $\rho \Delta p = k'' G^n$  based on the Reed-Fenske equation with void space, packing area, holdup and vapor viscosity neglected. The first two items are functions of the packing alone and the third primarily concerns the packing. The correlation obtained here for the seven liquids seems to be as accurate a representation of the data as that presented by Reed and Fenske for benzene and the n-heptane-methylcyclohexane system.<sup>11</sup> Actually the treatment seems to be equally as good as the  $G/\phi$  treatment if either can be considered good. Both curves have a slope of about +3 which is rather high for a curve of this type to be very accurate.

The vapor viscosity correction in the Reed-Fenske equation can be applied in a plot of  $\rho \Delta p / \mu_v^2$  vs.  $G / \mu_v$ . This is shown in Figure 21 and Table 3. The curve shown has a slope of about +5 (exponent n).

It would seem that the liquid viscosity rather than the vapor viscosity would play a large part in affecting holdup. It has been shown by various investigators that holdup is a function of liquid rate alone, below the loading point in absorption. In distillation the vapor rate is rarely above this point, and vapor viscosity should have little effect on holdup or pressure drop. Liquid viscosities vary considerably in temperature and differ in magnitude by as much as 300% among the various liquids investigated, while vapor viscosities change little. With this in mind, the proper values of liquid viscosity were applied to the data by means of the Reed-Fenske correlation in place of the vapor viscosity. The results were plotted as  $\log G / \mu_l$  against  $\log \rho \Delta p / \mu_l^2$  and are shown in Table 3 and Figure 22. Also included are data from Peters<sup>12</sup> on the n-decane and trans-decalin system

FIG. 21

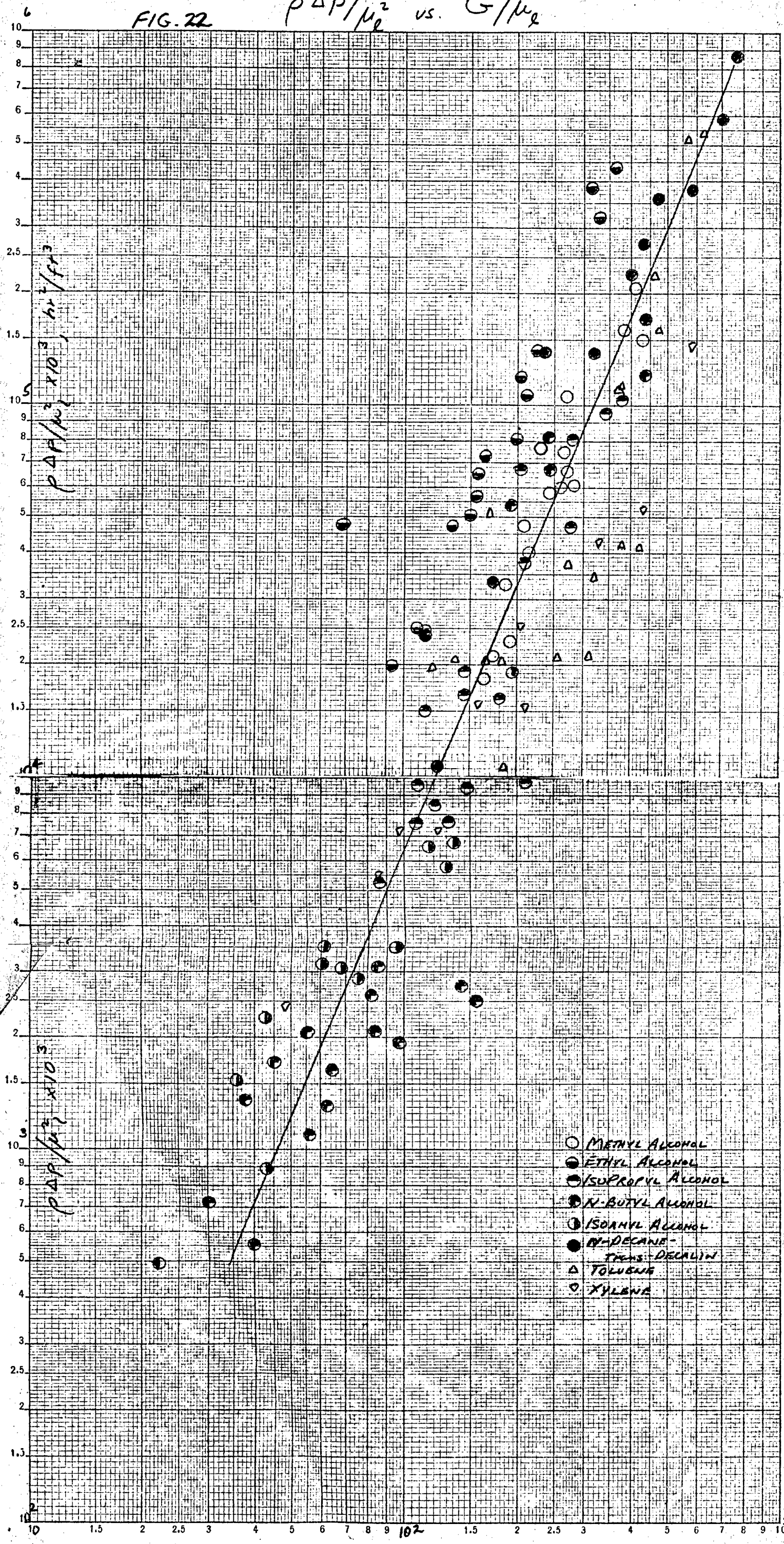
$\rho \Delta P / \mu v^2$  vs  $G / \mu v$



$G / \mu v, [(\text{lb.} / \text{hr. ft}^2)(\text{ft. hr.} / \text{lb.})]$



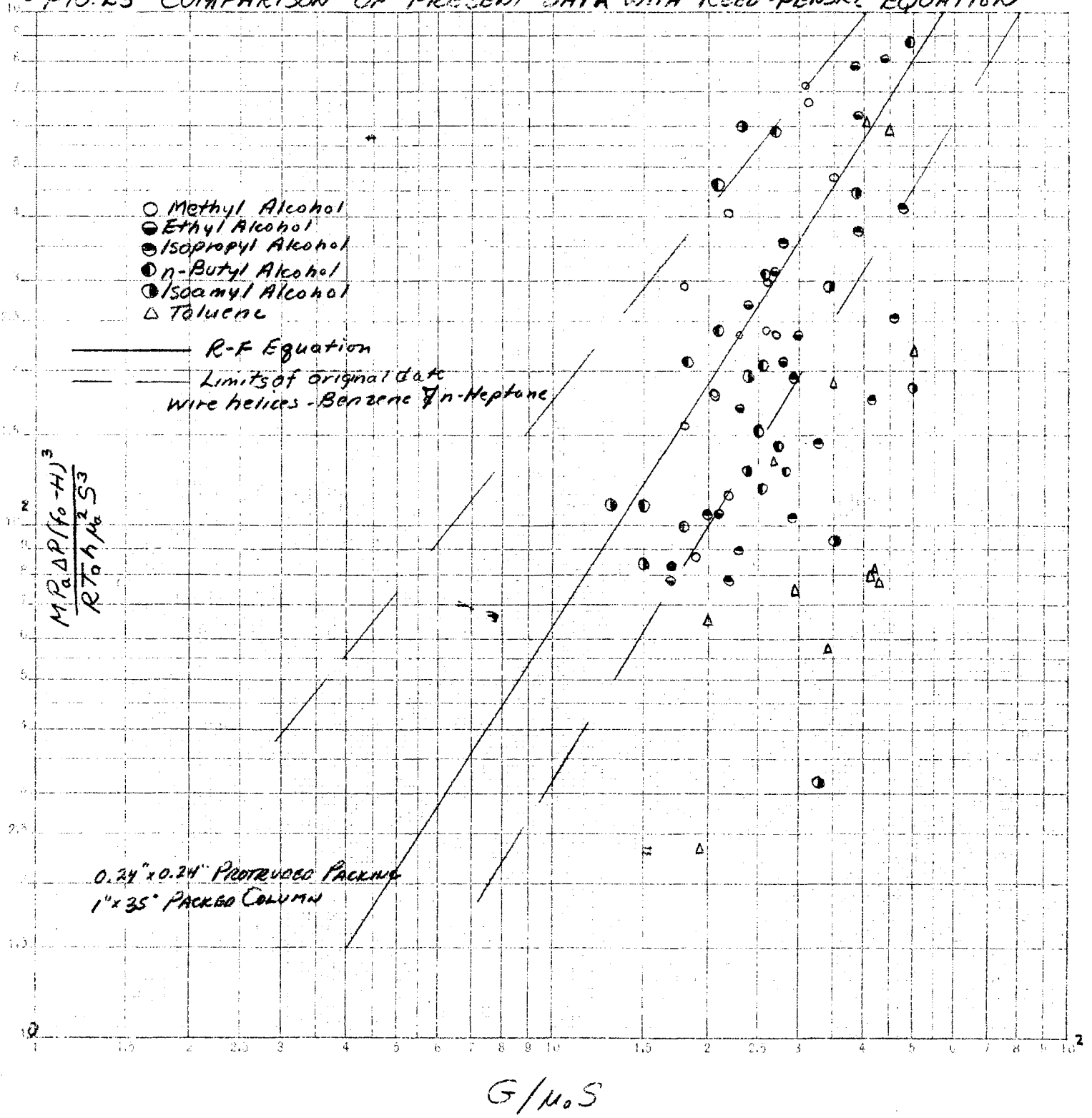
FIG. 22  $\rho \Delta P / \mu^2$  vs.  $G / \mu$



at 50 and 350 mm Hg in a two inch diameter by 24 inch high column with the same packing. This shows a much better correlation than does the use of vapor viscosity and it is felt that this is an improvement over the original proposal by Reed and Fenske. The greatest improvement is the correlation of the data for each individual liquid. This is especially noticeable with methyl alcohol, which points are considerably scattered with any other method of plotting. There is still systematic deviation among the various materials but the individual curves seem to have almost similar slopes. The overall curve has a slope of about +2.5 compared to +.5 with use of vapor viscosity. This is more of the order of that predicted by Reed and Fenske (1.7-1.8). It is felt that the most desirable slope for a curve of this type is about +1 for greatest accuracy. The lowered slope with liquid viscosity is a definite advantage over that for vapor viscosity.

Because of the results found in the  $\rho\Delta\rho$  and  $\mu$  correlations, it is desirable to apply the complete Reed-Fenske equation to the data where holdup information is available. The results are shown in Figure 23 and Table 3. While the majority of the data falls within the limits of the original data, many points for isopropanol, n-butanol and toluene fall well below the line. No holdup data for xylene was obtained and no comparison is possible here. However, the points plotted individually for each material show a definite slope for each material. Close inspection of the original curve<sup>14</sup> shows this possibility between benzene and n-heptane. Data for isoamyl and n-butyl alcohols still show the marked separation of points for runs at various pressures. This may be due to the use of vapor viscosity rather than liquid viscosity as

3 FIG. 23 COMPARISON OF PRESENT DATA WITH REED-FENSKE EQUATION



shown above. The greatest source of error in the overall correlation is probably in the inaccuracies of the holdup data in the small column used. The fact that others have found that each packing gives a different curve indicates that this correlation should be used with caution in applying data from one packing for design work using another packing.

Values of holdup calculated from values of the Reed-Fenske ordinate at  $G/\mu_v S$  for the experimental runs do not show much agreement with the observed values. These values do in general lie within a reasonable range of the true figures however, and the small holdup and overall inexactness of the complete correlation would account for the deviation.

Use of the Chilton-Colburn method as proposed by Hand<sup>6</sup> is not feasible here due to the absence of  $A_p$  factors for the packing used and  $A_1$  factors for the various liquids. Preliminary calculations show that these factors definitely vary with mass rate and  $A_p$  for the protruded packing used should be much lower than those for Raschig rings and Berl saddles. This is expected since protruded packing has been shown to cause a much lower pressure drop than the other two types.

Examination of other physical properties and their variation with temperature leads to the assumption that latent heat of vaporization may be connected with the difference of slopes among the various compounds. This is possible since heat of vaporization certainly plays a part in any distillation. With the materials tested, the latent heat decreased with temperature, and the change in latent heat with temperature decreased as the molecular weight increased. The use of this property

in connection with the  $\Delta p$  vs.  $G/\rho$  plot does not affect the slopes of the various individual curves. Whether considered in mass or molal units, the curves still exhibit the same general differences. It can be reasoned that this also would not affect the relationship of the various points in the  $\rho \Delta p / \mu_1^2$  vs.  $G/\mu_1$  plot.

That there is no great change in the various curves with use of a property differing so widely in magnitude and variation with temperature among the various compounds indicates that it does play a part which is reflected in the throughput rate.

Diffusion concepts may have an answer to the problem. It is known<sup>21</sup> that an increase in the diffusivity in the vapor phase has a favorable effect on enrichment in distillation at lowered pressures. This increase in vapor diffusivity is approximately inversely proportional to the pressure. Liquid diffusivity is essentially unaltered by pressure changes. Docksey and May<sup>21</sup> have assumed that interchange of material between liquid and vapor depends on rate of diffusion through the layer of vapor in which flow is streamline. Their experiments in this field supported this theory and their conclusions were summarized in the relation

$$Z \propto U r^2 / D_v$$

where  $Z$  is the column height for a given separation (an empty tube column was used where height would be the only measure of efficiency of separation),  $U$  is the vapor velocity,  $r$  the column radius and  $D_v$  the diffusivity.

Diffusivity of a binary vapor system has been shown to vary with temperature, pressure, molecular weight and molecular volume<sup>11</sup>. Liquid diffusion is less understood mathematically, but is known to be influenced

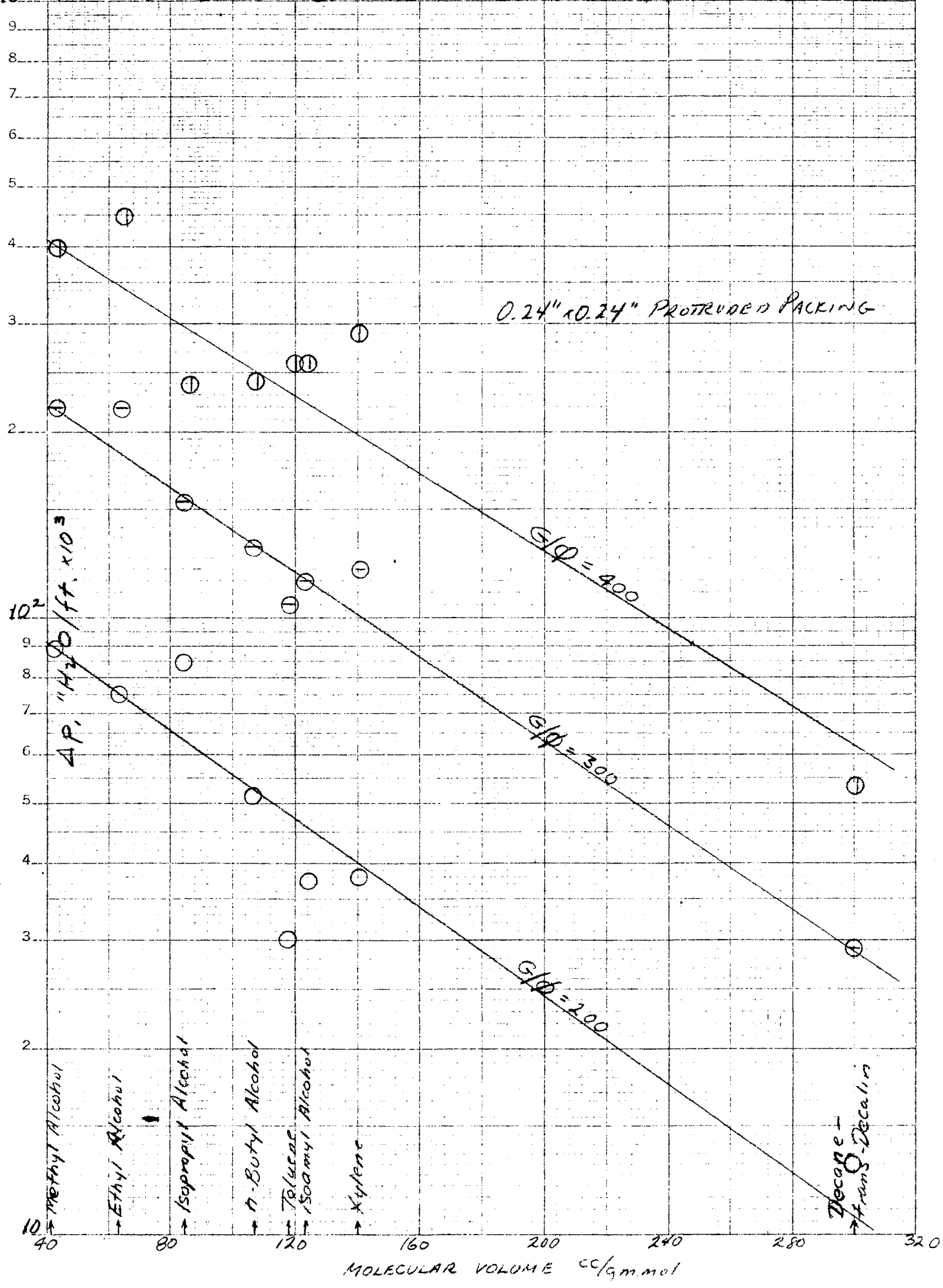
by molecular weight, molecular diameter, solvent viscosity and area of contact.<sup>1</sup> Diffusion of vapors in liquids is also relatively little understood, but it is assumed the factors concerned in both types are combined here.

In an attempt to determine a relationship of pressure drop to diffusivity, it was found that a plot of  $\log \Delta p$  at constant  $G/\phi$  against molecular volume gives approximately parallel lines of negative slope as shown in Figure 24. The deviation of the data from a straight line is much less than in any correlation attempted thus far. These points were taken from plots of  $\Delta p$  vs.  $G/\phi$  for each material at the specified value of  $G/\phi$ . Some of the deviation of the points is due to the fact that, as noted before, the experimental  $G/\phi$  curves are not parallel.

Extrapolation of the  $\log \Delta p$  vs.  $V_M$  curves to the molecular volume of n-decane and trans-decalin (averaged) yields a pressure drop value quite close to that obtained by plotting  $\Delta p$  vs.  $G/\phi$  for the data of Peters at 50 and 350 mm Hg. These data<sup>12</sup> were obtained on the same size and type packing as used in the present experiments. By coincidence, however, the data of Forsythe for benzene-ethylene dichloride in a Stedman-type column fall quite close (not shown) to the lines presented in Figure 24 at the three values of  $G/\phi$  chosen. This indicates that the spacing of the curves is of approximately the right order.

The relationship indicated in Figure 24 for the packing tested leads to an equation of the type:  $(\log \Delta p)_{G/\phi} = a - \frac{3}{4} V_M$ , where the constant  $a$  varies with the desired value of  $G/\phi$ .

10<sup>3</sup> FIG 24 PRESSURE DROP AS FUNCTION OF MOLECULAR VOLUME 47



An equation of this type should be useful in rough approximations of pressure drop for various liquid systems to be run at a known rate through a column with this packing. Modification of the equation to adjust for a different size of the same packing should involve only the constant  $a$ . Use of a different packing would probably change the slope (-0.75) of the curves.

The proposed curves permit estimation of pressure drop to within about thirty percent over a tenfold change in pressure drop. Introduction of the effects of other physical properties may refine the equation even more, since molecular volume and vapor density are the only physical properties presently involved.

The exact physical significance of the influence of molecular volume on the pressure drop is not understood. But diffusivity varies inversely with molecular volume. It seems possible that at a constant rate, the lower diffusivity of a compound would be an indication of less tendency for diffusion of vapor through liquid, less frictional resistance to flow and less pressure drop.



### CONCLUSIONS

Both pressure drop and holdup are exponential functions of mass rate at any pressure. At constant rate, decreased operating pressure leads to increased pressure drop. Higher molecular weight material will cause lower pressure drop at any rate and pressure. Holdup with protruded stainless steel packing is not greatly affected by change in pressure or material being distilled.

Several methods have been presented in the literature to predict pressure drop through a packed column during distillation. Most of these are specific for a given packing or liquid system. From the work presented here, it appears that no single correction factor can be applied to make these correlations general.

The absorption correlation  $\log \Delta P$  vs.  $\log G/\phi$  gives reasonably adequate results for relating data at different pressures. For design work, however, the slope of the curve obtained is rather steep leading to fairly large errors in estimates. The same is true of use of  $\rho \Delta P$  vs.  $G$ , a simplification of the Reed-Fenske equation.

A modification of the latter to include liquid viscosity, presented as  $\rho \Delta P / \mu_1^2 = k' (G/\mu_1)^n$ , yields a curve of slope  $n=2.4$  for the seven liquids tested. The value of  $k'$  for the individual materials varies slightly but a value of  $1 \times 10^{-4}$  would be reasonably accurate. This correlation should give fairly accurate results with any one size and type of packing.

The complete Reed-Fenske equation includes factors for differences in packing systems but uses vapor viscosities. It was developed from

wire helix data but can be used for order-of-magnitude estimates for different packings if accurate holdup data are available. For the packing used the modification presented above using liquid viscosities seems to be preferable.

A relationship between molecular volume and pressure drop at constant density-corrected rate has been presented. This relationship permits estimation of pressure drop at a known rate for liquids with molecular volumes between 40 and 300 cu.cm./gm.mol.

## NOMENCLATURE

$A_1$	factor to correct for effect of liquid on packing, $\Delta p$ wet / $\Delta p$ dry, dimensionless.
$A_p$	factor to correct for effect of hollow packing, $\Delta p$ hollow / $\Delta p$ solid, dimensionless.
$d_p$	individual packing particle, diameter, feet.
$f$	friction factor, dimensionless.
$f_o$	fractional void space of dry packing.
$g_c$	std. acceleration of gravity, lb.(f)ft./lb.(m)sec. <sup>2</sup>
$G$	mass flow rate, lbs./hr.sq.ft.
$h$	height of packed section, ft.
$H$	holdup, cu.ft./cu.ft. of packing.
$k$	constant in Reed-Fenske equation, dependent on packing, dimensionless.
$M$	molecular weight.
$n$	exponent in Reed-Fenske equation.
$P$	absolute pressure, lbs./sq.ft. In Reed and Fenske equation must be expressed as $Pg_c$ , lb.(m)/ft.sec. <sup>2</sup>
$\Delta P$	overall pressure drop, lbs./sq.ft.
$\Delta p$	pressure drop per foot of packed height, "H <sub>2</sub> O/ft., except in $\rho \Delta p / \mu^2$ , where $\Delta p$ is lbs./sq.ft.
$R$	gas law constant, consistent units.
$S$	packing surface area, sq.ft./cu.ft.
$T$	absolute temperature.
$U$	vapor velocity, ft./sec.
$V_M$	molecular volume, cu.cm./gm.mol.
$\rho$	vapor density, lbs./cu.ft.

$\mu$  viscosity, lbs./ft.hr.  
 $\phi$   $\sqrt{\rho / 0.075}$ , where 0.075 is density of air at 70°C and 1 atmosphere.

#### Subscripts

a at arithmetic average of end conditions of column.  
g, v gas or vapor.  
l liquid.

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