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Packing Materials for Fractionating Columns

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The many variables in distillation and in the determination of H. E. T. P. render the drawing of definite conclusions hazardous unless a large number of experiments have been made. Based only on the results presented here, the following conclusions have been drawn: (1) New type packing materials give double or triple the efficiency of former packings. (2) The best packings are one-turn and two-turn wire helices, one-turn and two-turn glass helices, carding teeth, and No. 19 jack chain. (3) An increase in height reduces the efficiency of a packing, the effect being much more

pronounced with $\frac{3}{4}$ -inch (small) diameter columns than with 2-inch (large) diameter columns. (4) An increase in the diameter of a column reduces the efficiency of a packing. (5) Different hydrocarbon mixtures give approximately the same H. E. T. P. value. (6) The effect of the rate of distillation varies with different packings and with the diameter of the column. (7) Laboratory columns made and operated by different persons have given similar results, showing that efficient ones can be made easily and so that they give reproducible results.

IN DESIGNING columns for the fractionation of Pennsylvania gasoline it was found necessary to develop a new type of packing material of a much higher efficiency than any previously used in either laboratory or commercial work. A large number of packing materials have been in use or have been tried (3, 5, 8, 11, 14, 15, 21). Data on the efficiency of the packings are incomplete, are not always comparable, and fail to take into consideration the effect of the height and diameter of the column in which the test was made. Accordingly, a study was made of various types of packing material.

The purpose of the packing in a fractionating column is to bring about as intimate contact as possible between the ascending vapor and the descending liquid without too great a reduction in the throughput or capacity of the column. The vapor-liquid contact or the scrubbing efficiency may be expressed in terms of H. E. T. P. (height equivalent to a theoretical plate) (10) and is the most important feature of a packing. It has been repeatedly shown (1-5, 17, 18) that to separate close-boiling substances a large number of theoretical plates is necessary. Regardless of any other advantages a packing may have, unless it has a low H. E. T. P. it will not make a good separation. Two other important points are throughput and holdup. A packing, unless it will allow a superficial vapor velocity under operating conditions of at least 0.6 foot per second, is of little practical use except in special cases where the time factor is unimportant. The question of holdup has been very well discussed by Podbielniak (11). For a sharp separation between two substances the operating holdup should be low. Packed columns occupy an intermediate position between the low holdup of indented and spiral packed columns and the high holdup of bubble cap columns. The operating holdup of various packings is not greatly different. Since very efficient packings have been developed having a large number of plates in a given height, it is possible in making a given separation to reduce considerably the height of packed section necessary and therefore

the holdup. If, in addition, proper consideration is given to the volume of the charge in relation to the size of the column, the problem of holdup will have been overcome.

The study of packing materials was based, therefore, mainly on their H. E. T. P. value under total reflux and their throughput. It is realized that the H. E. T. P. under operating conditions may be different from that under total reflux. However H. E. T. P. values under total reflux can be obtained easily, and, although they do not show the complete picture of fractionation, they are an important guide in studying and designing fractionating columns.

It is appreciated that in the final analysis the test of a fractionating column is the actual separation it will make in a reasonable amount of time. The packings found in this work to have low H. E. T. P. values have been in considerable actual use in the chemical laboratories of the Pennsylvania State College and have given sharp as well as complete separation. Figure 1 is a photograph of the control room for fractionating equipment charging 40 gallons and packed with jack chain.

COMMERCIAL APPLICATIONS

The packings studied are of commercial as well as laboratory importance. Several of the packings are already in industrial use. In addition to its plant use, jack chain is used in many semiworks installations. The results obtained here should furnish useful information to the operators of plant and semiworks packed columns and should emphasize the importance of periodic tests of the efficiency of the columns by means of two liquids whose vapor-liquid equilibria are known.

Many of the packings used in this work were relatively inexpensive. The iron carding teeth cost approximately \$40 per cubic foot. The cost of the nickel wire necessary to make up one cubic foot of this type packing is about \$80. The metal helices were readily made by machine in this laboratory.

DEFINITION OF TERMS

The terms used are defined below in order to avoid repetition in the text.

VELOCITY is expressed as superficial linear vapor velocity in feet per second at the top of the column and as cubic inches per hour of liquid condensed at the top of the column, under operating conditions. Attention is called to the fact that with different liquids, while the linear velocity in feet per second may be the same, the cubic inches per hour of condensate may vary considerably. In each instance it is indicated in the tables whether the original reflux was obtained as a volume of liquid at its boiling point (sharp-edged orifice measurements) or determined from the amount of heat picked up in the condenser water. In the former case the letter *o* follows the heading "vapor velocity," in the latter, *h*. In either case, to change from one to the other it is necessary to know the densities of the boiling liquids. These densities have been calculated from data and equations given in the International Critical Tables (7) since it was necessary in several cases to use extrapolated values. The density of methylcyclohexane at its boiling point was obtained by interpolation between values obtained using equations given in the International Critical Tables for thermal expansion of petroleum products (7) and the expansion data given in the Tag Manual (for oils) (16). The approximate densities thus obtained for the liquids at their normal boiling point are as follows:

<i>n</i> -Heptane	0.614	Carbon tetrachloride	1.489
Methylcyclohexane	0.704	Acetone	0.749
Benzene	0.814	Methanol	0.749
Toluene	0.777		

TOTAL REFLUX. This indicates all condensate returned to the column as reflux; i. e., no product is withdrawn.

REFLUX RATIO. This is the ratio of volume of condensate returned to the column to that withdrawn as product.

FREE SPACE. The volume per cent of packed section not occupied by packing is the free space.

HOLDUP. The operating holdup is the amount of liquid and vapor in the column while the column is operating. The static holdup is the amount of liquid required to wet the walls and packing of a column.

H. E. T. P. This stands for height equivalent to a theoretical plate; it was determined under total reflux.

DIAMETER OF COLUMNS. Unless otherwise stated, the diameter given is always the inside diameter of the column.

HEIGHT OF COLUMNS. Unless otherwise stated, the height given is the height of packed section.

REFRACTIVE INDEX. This property is measured by an Abbe refractometer maintained at 68° F. $\pm 0.2^\circ$ (20° C.).

BOILING POINT. Boiling points were obtained by a modified Cottrell boiling point apparatus which could be maintained at any desired pressure. The temperature was read by a Bureau of Standards calibrated copper-copel thermocouple and is accurate to within $\pm 0.2^\circ$ F.

***x-y* DIAGRAM.** This is the experimentally determined diagram expressing the vapor-liquid equilibria of the liquids being examined.

TEST LIQUIDS

Several sets of liquids were used since H. E. T. P. is known to vary with different liquids. These are given below.

CARBON TETRACHLORIDE AND BENZENE. The *x-y* diagram was constructed by Varteressian (19) from the data of Rosanoff and Easley (13). This mixture is cheap and readily obtained in the pure state, and analysis is easy and accurate (5). Both liquids were Baker's analyzed and were used without further purification since the results thus obtained and results using redistilled materials were identical. Analysis was by refractive index. The properties of the two substances are as follows:

	CARBON TETRACHLORIDE	BENZENE
Boiling point at 1 atm., ° F. (° C.)	168.8 (76.0)	176.2 (80.1)
Refractive index, n_D^{25}	1.4595-1.4600	1.4995-1.4998

BENZENE AND TOLUENE. The *x-y* diagram was calculated from the data of Rosanoff, Bacon, and Schulze (12) using 230.7° F. (110.4° C.) as the normal boiling point of toluene at which temperature the vapor pressure of benzene is 2.323 atmospheres, and 176.5° F. (80.3° C.) as the normal boiling point of benzene at which temperature the vapor pressure of toluene is 0.387 atmosphere. This mixture follows Raoult's law; on this basis, boiling point-composition curves were calculated for pressures near 1 atmosphere. Analysis of this mixture was by measurement of boiling point in a modified Cottrell apparatus, followed by reference to these curves. The benzene was the same as that used above. The toluene was either Baker's analyzed or some fractionated in a 27-foot packed column which had the equivalent of at least thirty theoretical plates when tested in the usual way with a mixture of *n*-heptane and toluene. The fractionated toluene had the following properties:

Boiling point at 1 atm., ° F. (° C.)	231.1 (110.6)
Freezing point, ° F. (° C.)	-139.0 (-95.0)
Density, d_{4}^{25}	0.8657
Refractive index, n_D^{25}	1.4964

***n*-HEPTANE AND TOLUENE.** The *x-y* diagram was determined by Bromiley and Quiggle (2). The toluene was the same as that used above. The *n*-heptane was that used for knock rating purposes. It was obtained from the California Chemical Company and had the following properties:

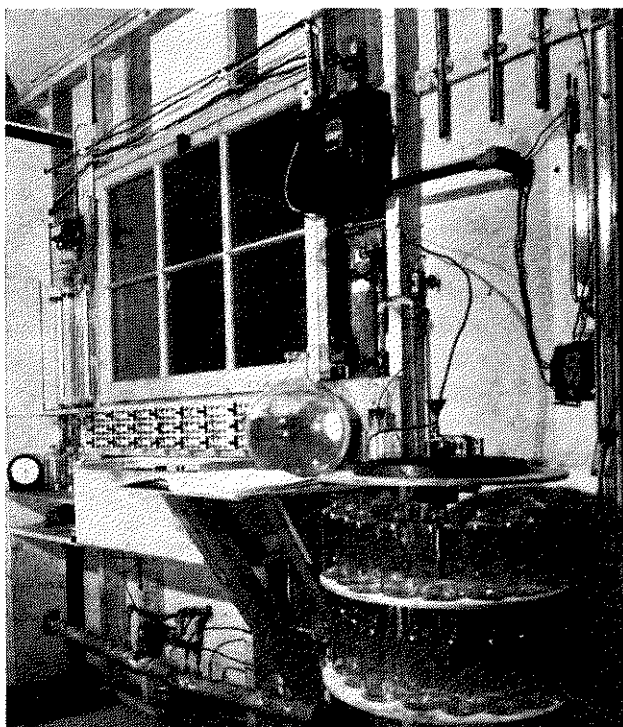


FIGURE 1. CONTROL ROOM FOR FRACTIONATING EQUIPMENT CHARGING 40 GALLONS

Boiling point at 1 atm., ° F. (° C.)	209.1 (98.4)
Freezing point, ° F. (° C.)	-131.4 (-90.8)
Density, d_{4}^{20}	0.6839
Refractive index, n_D^{20}	1.3878

n-HEPTANE AND METHYLCYCLOHEXANE. The equation,

$$\frac{X_{nA}}{X_{nB}} = (\alpha^{n-1}) \frac{X_{1A}}{X_{1B}}$$

where $\frac{X_{1A}}{X_{1B}}$ = molal ratio of A to B on any plate

$\frac{X_{nA}}{X_{nB}}$ = molal ratio of A to B on any plate *n* plate removed from the first

n = number of perfect plates required for separation

α = relative volatility (4)

1.07 = the value for α given by Beatty and Calingaert (1)

was used for the determination of the number of perfect plates required for the observed separation. Analysis was by means of refractive index using the data of Bromiley and Quiggle (2). The *n*-heptane was the same as that used above. The methylcyclohexane was Eastman's technical grade purified by stirring with concentrated sulfuric acid, by washing with water and sodium carbonate solution, and by drying over calcium chloride and fractionating. The fractionation was carried out in a column having the equivalent of ten theoretical plates when tested in the usual way with a mixture of carbon tetrachloride and benzene. The purified methylcyclohexane had the following properties:

Boiling point at 1 atm., ° F. (° C.)	213.4 (100.8)
Refractive index, n_D^{20}	1.4231-1.4232

ACETONE AND METHANOL. The *x-y* diagram was constructed from equilibrium data given by Hausbrand (6). Analysis was by refractive index. The acetone and methanol were redistilled and had the following properties:

	ACETONE	METHANOL
Boiling point at 1 atm., ° F. (° C.)	133.7 (56.5)	150.8 (66.0)
Refractive index, n_D^{20}	1.3594	1.3289

COLUMNS

The influence of the height and the diameter of a column on the efficiency of a packing is an important consideration in designing a column. Accordingly, the packings were tested in columns of varying height and diameter, and are described below.

COLUMN 1. This column consisted of a glass tube 0.76 inch in diameter and 48 inches tall. The height of packed section was approximately 27 inches, the packing being held in place by a roll of monel screen. A straight copper condenser was used. On the lower end of the condenser a 0.12-cubic-inch (2-cc.) glass cup was attached; during the test the condensate ran into the cup and overflowed onto the packing so that when equilibrium had been reached a sample of the distillate was present in the cup. In some cases a reflux gage was attached to the condenser and then the 0.12-cubic-inch cup was attached to the gage. The reflux gage consisted of a glass tube about 8 inches tall and 0.16 inch in diameter with a platinum sharp-edged orifice sealed in one end. The height of liquid in the tube was a measure of the rate of distillation, the gage having been previously calibrated. The column was jacketed with a glass tube but was not wound with resistance wire. A 61-cubic-inch (1-liter) short-neck flask was used as the still, and was heated by a Bunsen burner.

COLUMN 2. This was similar to column 1 except that the diameter was 0.70 inch.

COLUMN 3. This was similar to column 1 except that the diameter was 0.66 inch.

COLUMN 4. This column consisted of Shelby steel tubing 0.67 inch in diameter and 32 inches tall. It was connected to a glass head by a litharge and glycerol joint. In all other respects it was similar to column 1.

COLUMN 5. This column was made of glass and was 0.80 inch in diameter. The height of packed section was 55 inches. The packing was held in place by indentations in the wall of the tube. Product could be withdrawn by means of a side arm which extended into the column and received the condensate as it ran off the condenser. The amount of condensate withdrawn as distillate was regulated by a stop-cock. The column was jacketed with a glass tube wound with chromel resistance wire, which in turn was jacketed with a plain glass tube. A 61-cubic-inch (1-liter) flask was used as the still and was heated by a Bunsen burner.

COLUMN 6. This was similar in all respects to column 5 except that the height of packed section was 68 inches. The still was heated by an electric heater.

COLUMN 7. This was a regular fractionating column used in the organic research laboratory. A similar type column has been described by Whitmore and Lux (20). This column was 0.55 inch in diameter and had a packed section of 17.7 inches.

COLUMN 8. This column was made of Shelby steel tubing 0.67 inch in diameter and had a packed section of approximately 113 inches. The packing was held in place by a roll of monel screen. The head of the column was a glass tube 2 1/8 inches in diameter and 24 inches tall and was connected to the Shelby tube by an inverted stopper and litharge and glycerol joint. The condenser was a copper spiral, a sample of the distillate being obtained as above by means of the 0.12-cubic-inch (2-cc.) cup. The rate of distillation was measured by the reflux gage. The column was jacketed with 1-inch pipe which was covered with asbestos paper, wound with chromel resistance wire, and lagged with 85 per cent magnesia pipe covering. The still was a 122-cubic-inch (2-liter) short-neck flask and was heated by a Bunsen burner. A mercury U-tube was connected to the top of the flask so as to measure the pressure drop in the column.

COLUMN 9. This column consisted of a glass tube 2.12 inches in diameter and 48 inches tall. The packing was held in place by indentations in the wall of the column on which a piece of monel screen rested. The height of packed section was 26 inches. A sample of the distillate was obtained by a 0.30-cubic-inch (5-cc.) cup as previously described. The column was jacketed with a glass tube wound with chromel resistance wire. A 183-cubic-inch (3-liter) flask was used as the still and heated by a Bunsen burner.

COLUMN 10. This column was made of standard 2-inch iron pipe, and had a diameter of 2.07 and a height of 102 inches. Product could be withdrawn from different sections of the column by side arms placed 12, 42, and 72 inches from the bottom of the column. These side arms were attached to small copper cups which extended into the packing and caught some of the descending liquid. By means of valves the rate of take-off could be regulated. This method of take-off has the disadvantage that the cups may not catch a representative sample of the liquid in that particular cross section of the column, but only one small stream which may have a different composition.

The reflux composition was a coil of 1/4-inch copper tubing. By measuring the rate of water through the condenser and the temperature of the inlet and outlet water, the B. t. u. to the condenser, and therefore the rate of distillation, could be determined. When the height of packed section was only 30 to 42 inches, the condenser was lowered down into the column until it was just above the packing. The column was wound with chromel resistance wire and lagged. The

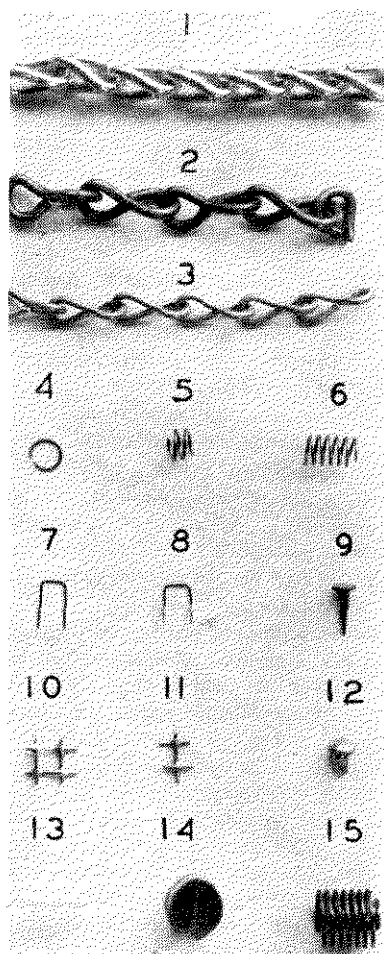


FIGURE 2. PACKING MATERIALS (ACTUAL SIZE)

1. No. 16 double-link brass jack chain
2. No. 16 single-link iron jack chain
3. No. 19 single-link aluminum jack chain
4. One-turn metal helix
5. Two-turn metal helix
6. Six-turn metal helix
7. Straight carding tooth
8. Bent carding tooth
9. No. 2 cut tack
10. Hollow-square wire form
11. Double cross wire form
12. Bifurcated rivet
13. Glass tube
14. Lessing ring
15. No. 1 Cholet-Vanderhoef ring

pipe coupling with $\frac{1}{4}$ -inch boiler plate welded on to form the top and bottom and had a capacity of 427 cubic inches (7 liters). The still was welded to the column proper and was heated electrically by strip heaters in series with chromel resistance wire. In all other respects the column was similar to column 10.

MISCELLANEOUS COLUMNS. Various columns in the organic research laboratory were also tested. These columns were similar to column 7 and differed mainly in height and diameter.

PACKING MATERIAL

Many different kinds of packing material have been tried and they are described below. Average dimensions are given. A photograph of some of the packings is given in Figure 2.

ONE-, TWO-, AND SIX-TURN HELICES. No. 24 B & S Lucero wire was wound on a $\frac{1}{8}$ -inch rod and cut into the desired helix.

GLASS HELICES. These were similar to the metal helices. They were made by winding molten glass on a $\frac{1}{8}$ -inch rod as described by Wilson, Parker, and Laughlin (27).

still consisted of a 12-inch piece of standard 6-inch pipe with $\frac{1}{4}$ -inch boiler plate welded on to form the top and bottom. A $\frac{1}{4}$ -inch pipe in the bottom of the still allowed for drainage and the withdrawal of samples. The top of the still was connected to a mercury U-tube so as to measure the pressure drop. The still was connected to the column proper by a 2-inch flange, the packing being held up by a piece of monel screen inserted between the flanges. The still was heated electrically by a winding consisting of 80 feet of No. 16 asbestos-covered monel wire.

COLUMN 11. This column was made of standard 2-inch pipe and had a packed section of 156 inches. Product could be withdrawn only at the top of the column; it was withdrawn as vapor and condensed in a water-jacketed product line. The still consisted of a standard 10-inch

CRIMPED WIRE. No. 24 B & S gage copper wire was fashioned to give ten crimps per inch, each $\frac{1}{16}$ inch high. The crimped wire was then cut into pieces about $\frac{1}{4}$ inch long.

STRAIGHT CARDING TEETH. A carding tooth is essentially a staple with a square top. Those used here were $\frac{1}{8}$ inch wide and had either a $\frac{5}{32}$ or a $\frac{7}{32}$ -inch leg. The size of the wire was No. 28 B & S gage.

BENT CARDING TEETH. These were the same as the straight carding teeth except that the legs were $\frac{1}{4}$ inch long and bent $\frac{1}{8}$ inch in from the open end.

MISCELLANEOUS BENT CARDING TEETH. These were a mixture of various sizes of carding teeth, of approximately the dimensions indicated for the straight and bent teeth.

DOUBLE-CROSS WIRE FORMS. These were made by cutting $\frac{1}{8}$ -inch galvanized screen in the form of double crosses. Hollow square wire forms were $\frac{1}{8}$ -inch galvanized screen cut into single squares.

No. 1 CHOLET-VANDERHOEF RINGS. These rings were double coils of No. 20 B & S gage wire. The inner ring was 0.19 inch wide and 0.46 inch long, and the outer ring 0.40 inch wide and 0.46 inch long.

LESSING RINGS. These rings were of brass, 0.25 inch wide and 0.28 inch long.

GLASS TUBES. These tubes were 0.27 inch long and had an outside diameter of 0.23 and an inside diameter of 0.17 inch.

No. 2 CUT TACKS. These were ordinary cut tacks having a total length of 0.261 inch. The diameter of the head was 0.139 inch.

No. 3 CUT TACKS. These were ordinary cut tacks having a total length of 0.380 inch. The head diameter was 0.164 inch.

B. B. SHOT. These were air rifle shot No. 5 having an average diameter of 0.176 inch.

BIRD SHOT. These were No. 7 shot obtained from Winchester Leader 12-gage loaded shells and had an average diameter of 0.098 inch.

BIFURCATED RIVETS. These rivets had a total length of 0.183 and a prong length of 0.125 inch. The diameter of the head was 0.154 inch.

TUBULAR RIVETS. These rivets had a total length of 0.172 and a prong length of 0.141 inch. The diameter of the head was 0.222 inch.

OUTSIDE PRONG RIVETS. These rivets had a total length of 0.431 and a prong length of 0.130 inch. The diameter of the head was 0.171 inch.

JACK CHAIN. Various sizes and kinds of jack chain were used. No. 16 single-link iron jack chain was made of No. 15 B & S gage wire and had links 0.51 inch long. No. 18 single-link iron jack chain was made of No. 17 B & S gage wire and had links 0.43 inch long. No. 19 single-link aluminum jack chain was made of No. 17 B & S gage wire and had links 0.40 inch long. No. 20 single-link iron jack chain was made of No. 19 B & S gage wire and had links 0.33 inch long.

TEST PROCEDURE

There are many sources of error and variables in the determination of H. E. T. P. values. Some of these are as follows: analysis of samples, purity of test liquids, method of obtaining samples, jacket temperature, measurement of rate of distillation, uniformity of rate of distillation, uniformity of packing columns, possible corrosion of the packing, distribution of liquid from the condenser or overflow cup, distribution of descending liquid in the column, accuracy of x - y diagram, effect, if any, of concentration on H. E. T. P., and establishment of equilibrium conditions in the columns.

It was possible, by following a definite procedure, to obtain values for H. E. T. P. which could be checked consist-

TABLE I. FREE SPACE AND HOLDUP

PACKING	FREE SPACE %	HOLDUP ^a	
		Cu. in. Cu. in.	
Miscellaneous carding teeth	92.1	0.023	
6-turn No. 24 Lucero wire helix	91.2	0.054	
No. 1 Cholet-Vanderhoef rings	90.0	0.014	
2- and 3-turn No. 24 Lucero wire helices	86.5	0.046	
Straight 1/32-in. carding teeth	84.9	...	
Lessing rings	84.7	0.044	
Bent 1/4-in. carding teeth	84.0	0.047	
1-turn No. 24 Lucero wire helix	82.0	0.046	
Straight 7/32-in. carding teeth	81.6	0.042	
Hollow square wire form	79.6	0.051	
No. 16 single-link iron jack chain	72.3	0.033	
Double-cross wire form	71.9	0.056	
Glass tubes	69.8	0.037	
No. 20 single-link iron jack chain	69.5	0.044	
No. 18 single-link iron jack chain	69.4	0.035	
No. 3 cut tacks	68.0	0.047	
Outside prong rivets	66.2	0.054	
No. 2 cut tacks	64.5	0.058	
Crimped wire	64.0	...	
Tubular rivets	60.0	0.053	
Bifurcated rivets	54.5	0.046	
B. B. shot	42.0	0.041	
Bird shot	39.4	0.036	

^a Ratio of volume of liquid to volume of packing.

ently, although occasionally a value was obtained which for no apparent reason deviated considerably from the usual value. In the following tables every run represents a determination made after ample time was allowed for equilibrium to be established. The various runs represent either considerable time intervals between runs, or else the starting of the experiment anew. This was found necessary to insure that the data obtained were correct, that the results were reproducible, and that the experimental conditions indicated were the principal conditions leading to the results obtained.

The columns were carefully cleaned and, if wound with resistance wire, were heated approximately to the boiling point of the test liquids. Tests on some of the short 27-inch columns with and without external jacket heat gave the same H. E. T. P. value. The still was then charged with 12 to 24 cubic inches (200 to 400 cc.) of sample for the small columns, 30 to 50 cubic inches (500 to 800 cc.) of sample for the medium size columns, and 120 to 180 cubic inches (2000 to 3000 cc.) of sample for the large ones. The concentrations of the two liquids were chosen so as to operate in the middle portion of the *x-y* diagram. The material was allowed to reflux for 15 minutes and then 0.6 to 0.9 cubic inch (10 to 15 cc.) of distillate was withdrawn to remove any trace of moisture or low-boiling material. This was possible only when the column was equipped with a side arm. In the other columns the glass cup was emptied and then replaced. Refluxing was then continued for 1 to 1.5 hours or in the case of those taller than 60 inches for 1.5 to 2.5 hours to establish equilibrium. Dur-

TABLE II. AREA

PACKING	AREA	
	Sq. in./cu. in.	(Sq. cm./cc.)
Crimped wire	69.5	(27.4)
Straight 1/32-in. carding teeth	56.5	(22.3)
Straight 7/32-in. carding teeth	47.3-60.0	(18.6-23.6)
Bent 1/4-in. carding teeth	43.2-53.3	(17.0-21.0)
Double-cross wire form	47.3	(18.6)
Outside prong rivets	47.3	(18.6)
1-turn No. 24 Lucero wire helix	46.6	(18.4)
Miscellaneous bent carding teeth	38.1	(15.4)
No. 2 cut tacks	36.1	(14.2)
Bird shot	35.9	(14.1)
No. 20 single-link iron jack chain	35.9	(14.1)
2- and 3-turn No. 24 Lucero wire helix	35.0	(13.8)
Hollow square wire form	34.3	(13.6)
Bifurcated rivets	32.5	(12.8)
Tubular rivets	31.7	(12.5)
No. 18 single-link iron jack chain	28.7	(11.3)
No. 3 cut tacks	25.2	(9.9)
6-turn No. 24 Lucero wire helix	23.1	(9.1)
Lessing rings	22.4	(8.8)
Glass tubes	21.9	(8.6)
No. 16 single-link iron jack chain	20.8	(8.2)
B. B. shot	20.3	(8.0)
No. 1 Cholet-Vanderhoef rings	14.0	(5.6)

ing this time the rate of distillation was kept as constant as possible.

When equilibrium had been established and with the column operating, samples of the still material and distillate were taken. These were withdrawn simultaneously or within as short an interval of time as possible. The still samples were obtained

TABLE III. EFFICIENCY TESTS USING BENZENE AND TOLUENE

PACKING	VELOCITY AT TOP, <i>h</i>		PRESSURE DROP <i>In. Hg</i>	BENZENE		TOTAL THEORETICAL PLATES	H. E. T. P. <i>Inches</i>
	Vapor	Liquid at B. P.		Distillate	Still		
	<i>Ft./sec.</i>	<i>Cu. in./hr. (l./hr.)</i>		<i>Mole %</i>	<i>Mole %</i>		
IN COLUMN 10, DIAMETER 2.07 INCHES, HEIGHT OF PACKED SECTION 42 INCHES							
No. 16 single-link iron jack chain	0.2	88 (1.4)	0.02	99.6	17.1	7.0	7.0
	0.7	333 (5.5)	0.08	100.0	10.5	9.0	5.2
	1.1	525 (8.6)	0.16	100.0	8.2	9.0	5.2
	1.2	573 (9.4)	0.20	98.0	6.0	6.5	7.6
	1.5	723 (11.9)	0.35	99.4	10.1	8.0	6.0
IN COLUMN 2, DIAMETER 2.12 INCHES, HEIGHT OF PACKED SECTION 26 INCHES							
No. 16 single-link iron jack chain	Fast	94.0	22.0	4.5	7.4

TABLE IV. EFFICIENCY TESTS USING CARBON TETRACHLORIDE AND BENZENE

PACKING	—VELOCITY AT TOP—		CARBON TETRACHLORIDE		TOTAL THEO- RETICAL PLATES	H. E. T. P. Inches
	Vapor	Liquid at B. P.	Distillate	Still		
	Ft./sec.	Cu. in./hr. (l./hr.)	Mole %	Mole %		
A. IN COLUMN 1, DIAMETER 0.76 INCH, HEIGHT OF PACKED SECTION 27 INCHES						
No packing	Fast ^a	..	24.5	17.8	2.0	37.0 ^b
No. 16 single-link iron jack chain	Fast	..	42.0	20.0	7.5	4.2
Double-cross wire form	Fast	..	56.5	17.5	14.0	2.1
Miscellaneous carding teeth	Fast	..	55.0	17.0	13.5	2.2
Bent 1/4-in. carding teeth	Slow	..	53.8	15.2	14.0	2.1
	Fast	..	57.0	15.2	16.5	1.7
B. SAME AS A, HEIGHT OF PACKED SECTION 28.5 INCHES						
Bent 1/4-in. carding teeth	Fast	..	65.0	14.0	20.0	1.5
	Slow	..	54.0	14.0	15.0	2.0
Straight 7/32-in. carding teeth	Fast	..	67.0	14.0	21.0	1.4
Straight 5/32-in. carding teeth	Slow	..	59.0	15.0	17.0	1.8
	Fast	..	60.5	14.0	18.0	1.7
C. IN COLUMN 2, DIAMETER 0.70 INCH, HEIGHT OF PACKED SECTION 28 INCHES						
Bent 1/4-in. carding teeth	Fast	..	50.0	16.5	12.0	2.5
	Fast	..	55.5	15.0	15.0	2.0
D. IN COLUMN 3, DIAMETER 0.66 INCH, HEIGHT OF PACKED SECTION 27 INCHES						
Straight 5/32-in. carding teeth	Fast	..	51.5	14.5	13.0	2.2
E. SAME AS D, HEIGHT OF PACKED SECTION 28 INCHES						
Bent 1/4-in. carding teeth	Fast	..	55.3	14.5	15.0	2.0
	Fast	..	48.0	13.0	12.5	2.4
F. SAME AS D, HEIGHT OF PACKED SECTION 29 INCHES						
Double-cross wire form	Fast	..	61.5	13.0	18.0	1.7
	Fast	..	55.0	12.0	16.0	1.9
G. IN COLUMN 4, DIAMETER 0.67 INCH, HEIGHT OF PACKED SECTION 29 INCHES, VAPOR VELOCITY 0						
Straight 7/32-in. carding teeth	1.4	76 (1.3)	47.2	12.0	13.0	2.4
	1.7	92 (1.5)	49.0	12.0	13.5	2.3
	1.6	87 (1.4)	50.0	11.0	14.5	2.1
	1.5	79 (1.3)	40.0	8.5	12.0	2.6
Bent 1/4-in. carding teeth	Fast	..	51.5	15.0	13.0	2.4
Double-cross wire form	Fast	..	46.5	9.5	13.5	2.3
Straight 5/32-in. carding teeth	Fast	..	55.0	14.5	14.5	2.1
	Slow	..	49.0	15.0	12.0	2.6
	Fast	..	46.5	15.0	11.0	2.9
H. SAME AS G, HEIGHT OF PACKED SECTION 28 INCHES						
Bent 1/4-in. carding teeth	Fast	..	46.5	14.0	11.5	2.7
Double-cross wire form	Fast	..	49.0	16.5	11.5	2.7
	Fast	..	46.5	9.5	13.5	2.2

^a Same rate as when packed.^b Calculated on basis of 37 inches of column height.

TABLE V. SUMMARY OF CARBON TETRACHLORIDE-BENZENE RESULTS ON COLUMNS 1 TO 4^a

PACKING	H. E. T. P. IN INCHES			
	Column 1 (0.76 in. diam.)	Column 2 (0.70 in. diam.)	Column 3 (0.66 in. diam.)	Column 4 (0.67 in. diam.)
Double-cross wire form	1.9-2.2	1.7-2.0	2.3
Bent 1/4-in. carding teeth	1.5-2.0	2.0-2.1	2.0-2.4	2.4-2.7
Straight 7/32-in. carding teeth	1.4-1.6	2.0-2.3
Straight 1/2-in. carding teeth	1.7-1.8	2.2-2.3	2.2-2.6

^a Columns 1 to 3 are glass, column 4 is metal.

TABLE VI. EFFICIENCY TESTS USING CARBON TETRACHLORIDE AND BENZENE

PACKING	—VELOCITY AT TOP—		PRES- SURE DROP	CARBON TETRACHLORIDE Distillate	TOTAL THEORETICAL PLATES	H. E. T. P.	
	Vapor Ft./sec.	Liquid at B. P. Cu. in./hr. (l./hr.)					In. Hg
A. IN COLUMN 5, DIAMETER 0.80 INCH, HEIGHT OF PACKED SECTION 55 INCHES, VAPOR VELOCITY 0							
Double-cross wire form	1.1	85 (1.4)	..	66.0	14.4	20.5	2.8
Hollow-square wire form	43.6	12.5	11.5	5.2
No. 2 cut tacks	0.7	52 (0.9)	..	72.5	15.0	25.0	2.3
2-turn and 3-turn No. 24 wire helices	1.0	79 (1.3)	..	60.0	14.0	17.0	3.4
B. IN COLUMN 6, DIAMETER 0.80 INCH, HEIGHT OF PACKED SECTION 56 INCHES, VAPOR VELOCITY 0							
Straight 7/32-in. carding teeth	1.2	92 (1.5)	..	67.0	12.0	23.0	3.0
	1.2	92 (1.5)	..	69.0	8.5	26.5	2.6
	1.5	116 (1.9)	..	65.5	6.0	25.5	2.7
C. SAME AS B, HEIGHT OF PACKED SECTION 58 INCHES							
No. 16 single-link iron jack chain	36.0	6.0	12.0	6.2
	47.0	15.0	11.0	6.8
D. IN COLUMN 8, DIAMETER 0.67 INCH, HEIGHT OF PACKED SECTION 115 INCHES, VAPOR VELOCITY 0							
No packing	1.8	92 (1.5)	0.04	23.0	9.5	6.0	23.0 ^a
	1.8	92 (1.5)	0.04	20.0	8.5	5.5	26.0 ^a
Bent 1/4-in. carding teeth	1.8	98 (1.6)	..	64.0	5.5	25.0	4.8
	1.6	79 (1.3)	..	67.0	5.5	27.0	4.5
E. SAME AS D, HEIGHT OF PACKED SECTION 113 INCHES							
Straight 7/32-in. carding teeth	1.6	84 (1.4)	1.77	73.5	6.0	30.5	3.8
	2.01 ^b	72.0	3.5	31.5	3.7
	1.4	76 (1.3)	1.18	67.0	6.0	26.5	4.4
No. 16 double brass jack chain	1.2	64 (1.1)	0.55	66.0	12.0	21.0	5.6
No. 18 double brass jack chain	1.5	79 (1.3)	0.51	68.0	10.5	23.0	5.1
F. SAME AS D, HEIGHT OF PACKED SECTION 111 INCHES							
No. 18 single-link brass jack chain	1.6	85 (1.4)	0.47	73.0	17.5	24.5	4.7
	1.5	79 (1.3)	0.39	70.5	17.4	22.5	5.2
No. 18 single-link iron jack chain	1.1	61 (1.0)	0.55	72.0	15.2	25.0	4.6
	1.4	76 (1.3)	1.05	72.5	12.5	26.5	4.4
No. 19 single-link iron jack chain	1.2	65 (1.1)	0.67	78.5	23.3	27.0	4.3
	1.1	61 (1.0)	..	80.0	22.0	29.5	3.9
Glass tubes	0.9	49 (0.8)	0.43	64.0	11.0	21.0	5.6
	1.3	72 (1.2)	0.75	63.0	9.0	21.5	5.4
G. IN COLUMN 9, DIAMETER 2.12 INCHES, HEIGHT OF PACKED SECTION 26 INCHES							
No. 16 single-link iron jack chain	Fast	28.0	15.5	4.5	7.4
	Fast	30.0	18.5	4.0	8.7
H. SAME AS G, HEIGHT OF PACKED SECTION 27 INCHES							
Double-cross wire forms	Fast	22.0	7.2	7.0	4.5
I. SAME AS G, HEIGHT OF PACKED SECTION 29 INCHES							
Straight 7/32-in. and bent 1/4-in. carding teeth	Fast	34.0	14.0	7.5	4.5
	Slow	31.5	14.0	6.5	5.3
J. IN COLUMN 10, DIAMETER 2.07 INCHES, HEIGHT OF PACKED SECTION 30 INCHES, VAPOR VELOCITY 1							
Double-cross wire form	0.3	170 (2.8)	0.08	48.6	19.8	10.0	3.3
	0.6	272 (4.5)	0.20	18.5	5.0	7.0	5.0
	0.7	340 (5.6)	0.39	44.0	20.8	8.5	4.0
	0.8	384 (6.3)	0.59	16.8	5.0	6.5	5.5
	0.8	388 (6.4)	0.63	36.5	16.2	7.5	4.6
K. SAME AS J, HEIGHT OF PACKED SECTION 56 INCHES							
Double-cross wire form	0.2	123 (2.0)	0.32	60.5	12.8	18.5	3.2
	0.4	183 (3.0)	0.47	51.0	8.8	16.0	3.7
	0.6	295 (4.8)	0.79	19.8	1.0	14.5	4.1
	0.6	298 (4.9)	0.95	55.0	15.6	15.0	4.0
L. SAME AS J, HEIGHT OF PACKED SECTION 40 INCHES							
No. 16 single-link iron jack chain	0.4	178 (2.9)	0.12	38.4	17.1	7.5	6.2
	0.8	383 (6.3)	0.24	35.9	15.2	7.5	6.2
	1.1	527 (8.7)	0.31	32.1	13.9	7.0	6.7
	1.3	655 (10.7)	0.47	24.8	11.7	5.5	8.9
M. SAME AS J, HEIGHT OF PACKED SECTION 72 INCHES							
No. 16 single-link iron jack chain	0.2	120 (2.0)	0.12	49.0	15.3	12.5	6.3
	0.7	350 (5.7)	0.20	51.2	13.7	13.5	5.8
	1.0	492 (8.1)	0.31	46.2	11.8	14.0	5.5
	1.0	515 (8.4)	0.35	33.8	10.6	10.5	7.6
	1.2	601 (9.9)	0.55	48.2	13.0	13.5	5.8
	1.4	698 (11.5)	0.55	29.4	9.7	10.0	8.0

^a Same rate as when packed.^b Column probably flooded.

by a tube extending into the boiling liquid in the flask; the portion of the tube outside the flask was bent down so that a siphon could be started either by the pressure drop in the column or by a slight suction. The line was closed by a stopcock or pinch clamp when not in use. The first sample obtained was discarded since it served to clean out the line; two samples, each of 0.12 cubic inch (2 to 3 cc.), were then taken. The sample of distillate, if the cup was used, was obtained by simply raising the condenser

and removing the cup. If the column had a side arm, which was as short as possible, two or three 0.06-cubic-inch (1-cc.) samples were withdrawn rapidly, a previous sample having cleaned out the product line. In some cases the distillate was obtained by withdrawing product very slowly so that essentially total reflux was maintained. These results agreed with those obtained when product was withdrawn rapidly.

The samples were analyzed by refractive index and occasionally checked by density. In the case of benzene and toluene, analysis was by boiling point, making it necessary to obtain larger samples.

This did not introduce a serious error since this mixture was used only in the column 2 inches in diameter. Knowing the composition of the still and distillate, the total number of theoretical plates was obtained from the *x-y* diagram using the graphical stepwise method of McCabe and Thiele (9). The number of inches of packed section divided by the total number of plates minus one gave the H. E. T. P. One plate was subtracted because of the enrichment in going from the still to the column. The enrichment in the unpacked sections of the column was neglected since these sections were small and since, as will be shown later, the enrichment itself was negligible.

The tests were made at different rates of distillation. In cases where the rate was not measured, the column was operated first as close to flooding as possible, and second at 50 to 70 per cent of the first rate. These will be designated as fast and slow, respectively. Check runs were made in all cases. In some cases, when testing any one packing, determinations were made on different days, the test solution was changed, and the columns were repacked with new packing. Every effort was made to insure the reliability of the results.

Unfortunately the results are not complete, and correlation between the different columns is frequently lacking. This is because the paper represents the data obtained in the course of various distillation projects in this laboratory rather than one particular study.

PHYSICAL MEASUREMENTS OF PACKINGS

It would be expected that a packing with a large area would be more efficient than one with a small area and that a packing with considerable free space would allow a high throughput in a column. Accordingly, measurements of area, free space, and holdup were made on a number of packings. The free space and holdup were measured by filling a 6.1-cubic-inch (100-cc.) graduate with

the packing and noting the volume of kerosene at ordinary temperatures required to fill the graduate to the 6.1-cubic-inch mark. From the volume the percentage free space was obtained. The kerosene was then poured off the packing which was allowed to drain for 15 minutes. The volume of kerosene added minus that recovered was equal to the holdup of the packing. Since packing materials settle differently in columns of various diameters, the values for holdup and free space thus obtained are only relative.

The area of 6.1 cubic inches of the packing was determined by measuring many individual pieces by means of a micrometer to obtain the average area of one piece and then ascertaining the number of pieces in this volume either by counting or by weighing. It should be remembered that the area thus measured may not be the effective area of the packing when it is in use in a column, since there is a certain unknown common area of contact between adjacent pieces of packing. The results obtained are given in Tables I and II.

H. E. T. P. TESTS USING BENZENE AND TOLUENE

Benzene and toluene can be practically completely separated in columns having the equivalent of twelve theoretical plates when tested with a mixture of benzene and toluene. This mixture could only be used, therefore, in short or inefficient columns. In addition, the analysis by boiling point required samples considerably larger than 0.06 to 0.12 cubic inch (1 to 2 cc.). The mixture was used only in the short 2-inch diameter columns whose capacity is seven times that of the $\frac{3}{4}$ -inch diameter columns. The results are given in Table III.

EFFICIENCY TESTS USING CARBON TETRACHLORIDE AND BENZENE

This mixture could be used in a large variety of columns; it could be analyzed easily and was, therefore, the one most frequently used. It was not employed with the aluminum jack chain because of the reaction of aluminum with carbon tetrachloride. If it is used for any length of time in metal columns, there may be some corrosion. The results are given in Tables IV to VI.

EFFICIENCY TESTS USING *n*-HEPTANE AND TOLUENE

The use of *n*-heptane and toluene, since they are both hydrocarbons, is well adapted to columns employed in fractionating gasoline. This mixture offers no corrosion difficulties, can be analyzed accurately by refractive index, and except for the high cost of the *n*-heptane is in all respects one of the best possible mixtures. The results obtained are given in Table VII.

EFFICIENCY TESTS USING *n*-HEPTANE AND METHYLCYCLOHEXANE

Mixtures of *n*-heptane and methylcyclohexane are very suitable for testing fractionating columns, particularly if the columns are to be used in petroleum work. Since these two substances boil only 4.3° F. (2.4° C.) apart, they are particularly suited to the testing of very efficient fractionating columns. Analysis by density or refractive index measurements is easy and accurate. The only serious objection is

TABLE VII. EFFICIENCY TESTS USING *n*-HEPTANE AND TOLUENE

PACKING	—VELOCITY AT TOP—		PRESSURE DROP In. Hg	<i>n</i> -HEPTANE		TOTAL THEORETICAL PLATES	H. E. T. P. Inches
	Vapor	Liquid at B. P.		Distillate	Still		
	<i>Ft./sec.</i>	<i>Cu. in./hr. (l./hr.)</i>		<i>Mole %</i>	<i>Mole %</i>		
A. IN COLUMN 7, DIAMETER 0.55 INCH, HEIGHT OF PACKED SECTION 17.7 INCHES							
1-turn glass helix	Fast	84.5	25.0	10.5	1.9
B. IN COLUMN 10, DIAMETER 2.07 INCHES, HEIGHT OF PACKED SECTION 40 INCHES, VAPOR VELOCITY <i>h</i>							
No. 16 single-link iron jack chain	0.3	183 (3.0)	0.08	65.6	2.7	8.5	5.3
	0.6	405 (6.6)	0.12	68.8	2.0	8.0	5.7
	1.0	680 (11.2)	0.20	55.1	2.6	6.5	7.3
	1.3	868 (14.2)	0.39	38.3	2.0	5.5	8.9
C. SAME AS B, HEIGHT OF PACKED SECTION 42 INCHES							
No. 19 single-link aluminum jack chain	0.6	452 (7.4)	0.20	73.3	2.3	9.5	4.9
	0.8	567 (9.3)	0.24	73.0	2.1	9.5	4.9
	1.1	732 (12.0)	0.31	70.3	2.8	9.0	5.2
	1.3	876 (14.4)	0.51	64.9	2.5	18.0	6.0
Straight 7/32-in. carding teeth	0.3	233 (3.8)	0.16	83.5	3.8	12.5	3.7
	0.8	587 (9.6)	0.47	82.1	1.8	3.0	3.5
	0.9	537 (8.8)	1.18	41.4	1.7	6.5	7.6
	0.9	553 (9.1)	1.18	33.4	1.3	5.0	10.5
D. SAME AS B, HEIGHT OF PACKED SECTION 70 INCHES							
No. 16 single-link iron jack chain	0.4	268 (4.4)	0.08	77.5	2.5	12.0	6.4
	0.9	620 (10.2)	0.31	72.5	0.9	12.0	6.4
	1.2	813 (13.3)	0.47	69.0	0.9	10.5	7.4
E. SAME AS B, HEIGHT OF PACKED SECTION 72 INCHES							
No. 19 single-link aluminum jack chain	0.5	372 (6.1)	0.24	83.2	1.8	14.5	5.3
	0.7	477 (7.8)	0.31	86.7	1.5	15.5	5.0
	1.0	716 (11.7)	0.51	88.3	2.4	16.0	4.8
	1.1	767 (12.6)	0.98	77.0	1.5	10.0	8.0
Straight 7/32-in. carding teeth	0.4	287 (4.7)	0.31	94.5	6.0	20.0	3.8
	0.6	471 (7.7)	0.59	93.8	2.3	20.0	3.8
	0.7	551 (9.0)	1.34	93.0	1.2	20.5	3.7
F. IN COLUMN 11, DIAMETER 2.07 INCHES, HEIGHT OF PACKED SECTION 156 INCHES, VAPOR VELOCITY <i>h</i>							
6-turn No. 24 Lucero wire helix ^a	0.7	506 (8.3)	0.12	84.2	24.3	10.5	16.4
	1.0	705 (11.6)	0.12	83.2	27.8	9.5	18.3
	1.3	949 (15.6)	0.20	83.1	26.2	10.0	17.3
	1.7	1240 (20.3)	0.28	93.6	23.5	10.0	17.3

^a Lucero wire is a nickel-copper alloy made by Driver-Harris Company, Harrison, N. J.

TABLE VIII. EFFICIENCY TESTS USING *n*-HEPTANE AND METHYLCYCLOHEXANE

PACKING	—VELOCITY AT TOP, <i>h</i> —		PRESSURE DROP In. Hg	<i>n</i> -HEPTANE		TOTAL THEORETICAL PLATES	H. E. T. P Inches
	Vapor	Liquid at B. P.		Distillate	Still		
	<i>Ft./sec.</i>	<i>Cu. in./hr. (l./hr.)</i>		<i>Mole %</i>	<i>Mole %</i>		
A. IN COLUMN 11, DIAMETER 2.07 INCHES, HEIGHT OF PACKED SECTION 156 INCHES							
6-turn No. 24 Lucero wire helix	0.7	478 (7.8)	0.10	41.8	28.8	9.5	18.4
	1.4	956 (15.7)	0.20	41.7	28.6	9.5	18.4
B. IN COLUMN 7, DIAMETER 0.55 INCH, HEIGHT OF PACKED SECTION 17.7 INCHES							
1-turn glass helix	42.6	31.5	8.0	2.5

the rather high price of *n*-heptane. The results of using this mixture are given in Table VIII.

EFFICIENCY TESTS USING ACETONE AND METHANOL

This mixture was used in only one case where no packing was employed. The results are given in Table IX.

TABLE IX. EFFICIENCY TESTS USING ACETONE AND METHANOL
(In column 8, diameter 0.67 inch, height 118 inches, no packing)

VELOCITY AT TOP, <i>o</i> Vapor Ft./sec.	LIQUID AT B. P. Cu. in./hr. (l./hr.)	ACETONE		TOTAL THEORETICAL PLATES	H. E. T. P. Inches
		Distillate Mole %	Still Mole %		
1.9	55 (0.9)	26.5	6.5	2.5	79.0
2.5	70 (1.1)	21.0	2.4	3.0	59.0
2.6	82 (1.3)	41.0	7.9	3.0	59.0
2.9	89 (1.5)	36.5	3.2	4.5	34.0

EFFECT OF HEIGHT ON EFFICIENCY OF PACKINGS

As would be expected, it has been found that an increase in the height of packed section lowers the efficiency of a column—i. e., increases the H. E. T. P. The effect is very pronounced in the columns 0.75 inch in diameter while in the columns 2 inches in diameter the evidence is inconclusive. This may be due to the fact that in short small-diameter columns there is but little channeling, whereas in short large-diameter columns there is channeling. Hence an increase in height in the small-diameter columns causes channeling and increases the H. E. T. P. value, whereas in the large-diameter column, since channeling is already present, the effect is not as great.

The decrease in efficiency with added height is apparently due only in part to the effect of channeling. In tests using the same concentration of carbon tetrachloride-benzene on columns with and without distributors, H. E. T. P. values were obtained which, while lower for the column with distributors than for the column without distributors, were higher than those obtained in short columns. It is possible that some fundamental relationship in the vapor-liquid equilibria may be responsible. The results are given in Tables X and XI.

TABLE X. EFFECT OF HEIGHT ON EFFICIENCY OF PACKINGS
(In columns 0.66 to 0.80 inch in diameter; test liquids, carbon tetrachloride and benzene)

PACKING	H. E. T. P. AT COLUMN HEIGHT OF:			
	27-29 in.	55 in.	66-68 in.	111-116 in.
	Inches	Inches	Inches	Inches
No. 16 single-link iron jack chain	4.2	...	6.5	...
Double-cross wire form	2.1	2.8
Bent 1/4-in. carding teeth	1.5-2.5	4.6
Straight 7/32-in. carding teeth	1.5-2.1	...	2.8	3.8
No packing	33.7	26.0

TABLE XI. EFFECT OF DISTRIBUTORS ON EFFICIENCY OF PACKINGS

	CARBON TETRACHLORIDE		TOTAL THEORETICAL PLATES	H. E. T. P
	Distillate	Still		
	Mole %	Mole %		Inches
Column 6, diam. 0.80 in., packed for 67 in. with double-cross wire form	65.0	8.5	23	3.0
Column 6, same height of packing, 1 distributor in middle of column	73.0	8.5	28	2.5
Column 1, diam. 0.76 in., packed for 27 in. with double-cross wire form	56.5	17.5	14	2.1
Column 8, diam. 0.67 in., packed for 113 in. with straight 7/32-in. carding teeth	72.0	7.2	29	4.0
Column 8, diam. 0.67 in., with same packing for 103 in., 4 equally spaced distributors	73.5	5.0	31.5	3.4
Column 4, diam. 0.67 in., with same packing for 27 in.	50.0	11.0	14.5	2.1

EFFECT OF DIAMETER OF COLUMN ON EFFICIENCY OF PACKINGS

An increase in the diameter of a column decreases the efficiency of the packing considerably. This is the main reason why packed columns have not found a more extensive use commercially. The results obtained are given in Table XII.

TABLE XII. EFFECT OF DIAMETER OF COLUMN ON EFFICIENCY OF PACKINGS

PACKING	(Test liquids, carbon tetrachloride and benzene)				
	HEIGHT PACKED SECTION	H. E. T. P. AT COLUMN DIAM. OF:			
	Inches	0.67 in.	0.76 in.	2.07 in.	2.12 in.
Double-cross wire form	27-30	2.3	2.1	4.2	4.3
	55-56	...	2.8	3.7	...
No. 16 single-link iron jack chain	68-72	...	6.5	5.9	...
	27-40	...	4.1	6.4	7.9
Straight carding teeth	27-29	...	1.5	...	5.0

EFFECT OF DIFFERENT MIXTURES ON EFFICIENCY OF PACKINGS

Using column 7 (diameter 0.55 inch, height of packed section 17.7 inches), efficiency tests were made with three different mixtures. The columns were all operated at maximum throughput—i. e., just below flooding. It is interesting to point out that under these conditions superficial linear velocity, in feet per second, was high for the water-acetic acid mixture and low for the *n*-heptane-toluene mixture. The results are given in Table XIII.

Tests on column 10 packed with No. 16 single-link iron jack chain, using mixtures of benzene and toluene, carbon tetrachloride and benzene, and *n*-heptane and toluene, gave practically the same H. E. T. P. Using *n*-heptane and toluene in column 11 which was packed with six-turn wire helices, an H. E. T. P. of 17 inches was obtained whereas *n*-heptane and

methylcyclohexane gave a value of 18 inches. In testing an open tube 116 inches long, an H. E. T. P. of 26 inches was obtained with carbon tetrachloride and benzene and 58 inches with acetone and methanol.

TABLE XIII. EFFECT OF DIFFERENT MIXTURES ON EFFICIENCY OF PACKING

(Using column 7 packed with one-turn glass helices)

MIXTURE	MORE VOLATILE COMPONENT		TOTAL THEORETICAL PLATES	H. E. T. P.
	Distillate	Still		
	Mole %	Mole %		Inches
<i>n</i> -Heptane-toluene	84.5	25.0	10.5	1.9
Water-acetic acid	93.8	26.0	8.0	2.5
<i>n</i> -Heptane-methylcyclohexane	42.6	31.5	8.0	2.5

CORRELATION OF PACKING MEASUREMENTS AND H. E. T. P.

As stated above, packings should have a large surface area and a high percentage of free space. It has been found that the product of the number of square inches of surface area per cubic inch of packing and the percentage of free space, divided by 100, gives a numerical value which parallels the efficiency and over-all utility of the packings. Table XIV compares this numerical value with the observed H. E. T. P. and the flooding velocity. The H. E. T. P. was determined in the usual way with a mixture of carbon tetrachloride and benzene.

CORROSION

The problem of corrosion in packed columns is serious. A packing which has corroded even to a small extent behaves quite differently from the original packing. Usually the H. E. T. P. is greater and the throughput is less. Some of the packings used were made of alloys so that no difficulty was experienced with atmospheric corrosion or with the liquids used. For general use in laboratory columns the one-turn glass helices are best in regard to corrosion, although in most cases an alloy wire would be satisfactory. The carding teeth used in the above tests were made of steel wire and because of their large surface area corroded readily when exposed to the atmosphere. An attempt is being made to have them formed from an alloy wire.

LABORATORY COLUMNS

Packed columns are used extensively as laboratory columns. In most cases the packing has been very inefficient, and therefore the number of theoretical plates small. The use of some of the packings described above, instead of the older types of packings, would double or triple the number of theoretical plates in the column. The same would be true of some of the open-type columns which do not use a packing. Efficient, well-designed, packed laboratory columns are used in the organic research laboratory of the Pennsylvania State College. The results obtained on a number of these columns are given in Table XV. The columns were all made and tested by different persons. The tests were all conducted at maximum operating vapor velocity, and the test mixture was carbon tetrachloride and benzene. The columns were packed with three-fourths-turn to two-turn glass helices, and the differences in H. E. T. P. are partly due

TABLE XIV. CORRELATION OF PACKING MEASUREMENTS AND H. E. T. P.

PACKING	AREA \times % FREE SPACE	FREE SPACE	H. E. T. P.	CONDENSATE AT FLOODING	DIMENSIONS OF TEST COLUMN
	100	%		VELOCITY	
			Inches	Cu. in./hr (l./hr.)	Inches
Straight $7/32$ -in. carding teeth	47.0	84.9	1.5	92 (1.5)	0.76×27
Straight $5/32$ -in. carding teeth	46.2	81.6	1.7	...	0.76×27
Crimped wire	44.5	64.0
Bent $1/4$ -in. carding teeth	40.4	84.0	1.7	122 (2.0)	0.76×27
1-turn No. 24 Lucero wire helix	38.2	82.0
Miscellaneous carding teeth	35.1	92.1	2.2	...	0.76×27
Double-cross wire form	34.0	71.9	2.1	110 (1.8)	0.76×27
Outside prong rivets	31.3	66.2
2-turn and 3-turn No. 24 Lucero wire helix	30.3	86.5
Hollow-square wire form	27.3	79.6	5.4	...	0.80×55
No. 20 single-link iron jack chain	25.0	69.5	5.2	...	2.0×53
No. 2 cut tacks	23.3	64.5	2.4	53 (0.9)	0.80×55
6-turn No. 24 Lucero wire helix	21.1	91.2	8.0	...	0.80×66
No. 18 single-link iron jack chain	19.9	69.4	6.5	...	2.0×53
Lessing rings	19.0	84.7
Bifurcated rivets	17.7	54.5
No. 3 cut tacks	17.1	68.0
Glass tubes	15.3	69.7	5.5	73 (1.2)	0.78×27
No. 16 single-link iron jack chain	15.0	72.3	4.2	...	0.73×27
Bird shot	14.2	39.4
Cholet-Vanderhoef rings	12.6	90.0
B. B. shot	8.4	41.5

of this type cannot be increased without a considerable loss in efficiency. When tested with a mixture of carbon tetrachloride and benzene in the usual way, it had the equivalent of six theoretical plates corresponding to an H. E. T. P. of 5.4 inches.

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TABLE XV. EFFICIENCY TESTS OF LABORATORY COLUMNS USING CARBON TETRACHLORIDE AND BENZENE

DIAM.	HEIGHT	TOTAL	H. E. T. P.
Inches	PACKED SECTION Inches	THEORETICAL PLATES	
0.43	10.3	10.0	1.1
0.51	15.0	6.0	3.0
0.32	15.8	7.7	2.4
0.55	17.3	9.6	2.0
0.51	17.3	12.5	1.5
0.55	17.7	12.0	1.6
0.36	20.9	15.0	1.5
0.63	23.6	10.7	2.4
0.51	24.8	10.0	2.7
0.67	32.2	13.5	2.6
0.59	34.7	19.5	1.9
0.55	53.1	22.3	2.5

to this and partly due to the different heights of packed section.

For comparison, data on the following columns are as follows:

COLUMN A. A glass column 0.78 inch in diameter and having a packed section of 27 inches was packed with $1/4 \times 1/4$ inch glass tubes. When tested with a mixture of carbon tetrachloride and benzene in the usual way, it had the equivalent of five and a half theoretical plates, corresponding to an H. E. T. P. of 6.0 inches.

COLUMN B. This was an indented glass column 0.43 inch in diameter and 27 inches tall. The indentations were not made at random but were carefully spaced and shaped so as to give maximum vapor-liquid contact. The diameter of a column

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