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NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

TECHNICAL NOTE 2342

EVALUATION OF PACKED DISTILLATION COLUMNS

I - ATMOSPHERIC PRESSURE

By Thaine W. Reynolds and George H. Sugimura

Lewis Flight Propulsion Laboratory
Cleveland, Ohio

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SUMMARY

The 6- and the 7-foot-packed-height laboratory glass distillation columns and the 30-foot-packed-height stainless-steel distillation columns in use at the NACA Lewis laboratory were evaluated. Four column-packing combinations of the glass columns and four column-packing combinations of the steel columns were investigated at atmospheric pressure using a test mixture of methylcyclohexane and 2,2,4-trimethylpentane. Theoretical-plate values and pressure-drop data were obtained for the column-packing combinations investigated.

It was confirmed that preflooding of a column was necessary to obtain maximum separating efficiency. The glass column with wire-coil packing had theoretical-plate values ranging from about 80 to 190 over its operating range. These values were from 2 to 4 times the theoretical-plate values obtained for the other glass columns of the same length.

The 30-foot-packed-height steel column with a 2-inch diameter and stainless-steel helix packing had theoretical-plate values from $1\frac{1}{3}$ to 2 times greater than those obtained with the 1/4-inch Berl saddles and 2 to 3 times greater than those obtained with the 3/8-inch Raschig rings. The 1-inch-diameter column with helix packing had a maximum theoretical-plate value of over 200 at low reflux rates.

The deviation of the test mixture from ideality is shown to be sufficient to have considerable effect on the calculated theoretical-plate values.

INTRODUCTION

The NACA Lewis laboratory has been engaged in the preparation of pure hydrocarbons for several years in order to obtain correlations of molecular structure with engine performance and with other physical and

chemical properties of the compounds. In the purification process to obtain these pure hydrocarbons, distillation is extremely important. Distillations to obtain maximum purity of a compound often consume the greater part of the total time required for the complete synthesis of the compound. Purification of commercial starting materials also often requires considerable time. Consequently, it is important to know the operating characteristics of the distillation equipment being used in order to obtain the most efficient performance for any particular required separation.

In order to determine efficiency, pressure-drop, and reflux relations of the various types of distillation column and packing in use at the NACA Lewis laboratory, an investigation was conducted under the various operating conditions commonly used. This report describes the 6- and 7-foot-packed-height glass columns and the 30-foot-packed-height stainless-steel columns, which have been in use at this laboratory for over 8 years, and presents the performance data of this equipment at atmospheric pressure.

APPARATUS

Steel column. - A diagram of the typical stainless-steel distillation column used in this investigation is shown in figure 1. All the columns were basically of the same construction but differed in the type of packing used. The height of the packed sections were 30 feet, the over-all heights of the columns were 37 feet, and the column diameters were 1- or 2-inch standard pipe size. Pots of 10- and 20-gallon charge capacity were used.

All of the metal in contact with the distilling mixture was 347 stainless steel with the exception of the flange gaskets, which were of annealed copper. The pot was heated by three resistance elements wound concentrically beneath the pot and connected so as to give continuous control from 0 to 5 kilowatts power input.

The column section was surrounded by a thin sheet-metal jacket, which was wound with three separate lengths of asbestos- and glass-covered resistance heating wire. Each circuit had a capacity of 2 kilowatts and was separately controlled by a variable transformer. The entire column was then lagged with a $1\frac{1}{2}$-inch thickness of magnesia insulation.

The packing was supported at the pot flange by a sieve cone with perforated area equal to the cross-sectional area of the column.

Cooling water was metered to the head condenser through a rotameter and its temperature increase through the condenser was taken as a measure of the rate of reflux in the column. Thermocouples were located in the pot liquid, on the column and the sheet-metal jacket at the center of each heating section, and at the top of the packed section just before the head condenser.

A manometer for measuring pressure drop was connected to the pot and was purged by a very slow stream of nitrogen visually metered through a bubbler system.

Glass column. - The type of glass column used in this investigation is shown in figure 2. The over-all height of all glass columns was from 9 to 10 feet with packed heights of either 6 or 7 feet.

The glass column was surrounded by a length of 2-inch steel tubing around which was wound two separate lengths of resistance heating wire, each of 500-watt capacity and each controlled by a variable transformer. The column was then covered with pipe insulation. A ground-glass joint at the bottom of the column permitted use of various sizes of flasks for the distillations. Pot heat was supplied by a heating mantle controlled by a variable transformer. The condenser unit is described in detail in reference 1.

The columns were set up so that they could be operated at atmospheric or reduced pressures. Pressures below atmospheric were maintained by a commercial metal cartesian-diver-type manostat mounted between a 30-cubic-foot-per-minute vacuum pump and a 4-cubic-foot-capacity surge chamber. In practice, a bank of several columns operating at the same controlled pressure is connected to one surge tank.

Four different sizes of glass columns were tested: (1) 22-millimeter outside diameter, 6 feet long, (2) 22-millimeter outside diameter, 7 feet long, (3) 32-millimeter outside diameter, 6 feet long, and (4) 22-millimeter-inside-diameter, 6 feet long.

The 22-millimeter-inside-diameter column had a silvered vacuum jacket and was not covered with resistance heating wire and pipe insulation as were the other columns.

Packing. - The following packing materials were tested in the steel columns: (1) 1/8-inch single-turn stainless-steel helices, (2) 1/4-inch porcelain Berl saddles, and (3) 3/8-inch porcelain Raschig rings. The packing materials tested in the glass columns were (1) 3/16-inch single-turn glass helices, and (2) a wire-coil packing described in reference 2.

TEST MIXTURE

The mixture used for these experiments consisted of methylcyclohexane and 2,2,4-trimethylpentane, which was chosen because it had a relative volatility of approximately 1.049 and was reported to be ideal (reference 3). In attempting to run check curves on the columns with mixtures covering different composition ranges, however, the mixture deviated considerably from ideality at the low 2,2,4-trimethylpentane end of the concentration range. This deviation is confirmed in figure 3, which is plotted from data of reference 4, and shows this trend of relative volatility with concentration.

The theoretical-plate values reported herein were calculated using the following Fenske equation (reference 5) and a relative volatility of 1.049:

$$N = \frac{1}{\log_e \alpha}\left[\log_e \left(\frac{x_d}{1-x_d}\right)\left(\frac{1-x_r}{x_r}\right)\right]$$

where

N number of theoretical plates including the still

α relative volatility of mixture

x_d mole fraction of more volatile component in distillate

x_r mole fraction of more volatile component in residue

Both components of the test mixture were purified by fractional distillation and the samples used for the column-testing program had purities of 99.5 mole percent as indicated by freezing-point analysis.

The following refractive index - composition relation was established at 20° C using known mixtures by measuring the refractive index on a five-place precision refractometer:

$$n_D^{20} = 1.39147 + 0.02543x + 0.00612x^2$$

where

n_D^{20} refractive index of mixture at 20° C

x mole fraction of 2,2,4-trimethylpentane in mixture

When a constant value of relative volatility is used, a single plot (shown in fig. 4 and suggested in reference 6), can be made by combining the refractive index - composition curve and the theoretical-plate equation. This curve is useful in making rapid checks on the theoretical-plate values for given residue and distillate compositions and shows the limitations of the range of theoretical-plate values that can be measured without large experimental errors. For example, if distillate and residue compositions are approximately equidistant from a mixture of equal parts and the accuracy of reading refractive index is within ±0.0001, theoretical-plate values of 60 would then have an indeterminancy of ±1 plate and at 160 plates the indeterminancy would be about ±7 plates. The indeterminancy rapidly increases above theoretical-plate values of 160.

Actual-plate values will be lower than those calculated from the value of relative volatility of 1.049, especially when concentrations very low in 2,2,4-trimethylpentane are involved.

PROCEDURE

The test mixture was charged to the column and the pot and column heats were applied until the material was refluxing at the top of the column. The column heats were then adjusted until the jacket temperature and the corresponding column temperature were approximately the same. For the glass columns, the inside temperatures were estimated from the vapor temperatures at the top and the bottom of the column. The pot heat was then increased until flooding occurred at the top of the column, after which it was decreased to give a reflux rate just below the flooding point. The column was refluxed under these conditions until equilibrium was attained, which required from 24 to 72 hours. Test samples from the pot and head were periodically taken until no change in composition occurred. Samples for analysis were then taken and the reflux rate was decreased. The procedure was repeated until the reflux rate had been decreased to a minimum value.

After the flooding point had been determined, the runs without preflooding were made using the procedure previously described except that the initial setting of reflux rate was below the flooding point. The data for the unflooded column with saddle packing were taken at increasing rates rather than decreasing reflux rates.

DISCUSSION OF RESULTS

Glass columns. - In comparing the experimental results of the various columns, it should be noted that the absolute theoretical-plate values are somewhat lower than the values reported herein because of the increase in relative volatility of the mixture at low concentration of 2,2,4-trimethylpentane.

The performance of the four glass columns with and without preflooding is shown in figure 5. The advantage in efficiency to be derived from preflooding the column (references 7 and 2) is evident from this figure; increases in number of theoretical plates from 17 to 55 percent were obtained after preflooding.

In figure 6, a comparison of the theoretical-plate values of the four glass columns after preflooding is shown. The wire-coil-packed column, with theoretical-plate values ranging from about 80 to 190, was from 2 to 4 times more efficient in separating the test mixture than any of the other glass columns. The 7-foot glass column with an outside diameter of 22 millimeters showed an increase in plate efficiency that would be expected for a corresponding increase in length over the 6-foot column of the same diameter. The column with a 32-millimeter outside diameter showed a slight increase in efficiency over the column with a 22-millimeter outside diameter of the same packed height. This result is contrary to the usually expected trend of efficiency with diameter and is undoubtedly a result of the relative size of the packing to column diameter. For any nominal-diameter helix packing there is an optimum column diameter for highest efficiency. At ratios of packing diameter to column diameter less than or greater than the optimum, channeling of the liquid and vapor flows occurs and decreased efficiency results. For the 3/16-inch helix packing used, the optimum column diameter is apparently greater than 22 millimeters. The efficiency of all columns increased with decreasing reflux rates.

The pressure drop through the four glass columns is shown in figure 7. The pressure drop through the wire-coil-packed column was 2 to 3 times greater than that through the other columns at the same vapor velocities.

Steel columns. - The theoretical-plate values plotted against reflux rates for the steel columns are shown in figures 8 to 10. The increase in relative volatility of mixtures low in 2,2,4-trimethylpentane concentration, shown in figure 3, is confirmed in figure 8. As the concentration range of the mixture approaches the low 2,2,4-trimethylpentane contents, the theoretical-plate values calculated from the assumed constant value of relative volatility will increase. As shown

in figure 8, when smaller concentrations of 2,2,4-trimethylpentane were used in the charged mixture, the theoretical-plate values were higher for a given reflux rate. In comparing columns, it was therefore necessary to use as nearly the same concentration range of test mixture as was possible for each test in order to make the comparison valid.

The 2-inch-diameter column with no packing had theoretical-plate values of from 6 to 12 over the reflux-rate range, as shown in figure 10. The empty column could not be flooded with the power available. The 2-inch-diameter column packed with 1/4-inch Berl saddles had theoretical-plate values of about 84 to 94 after preflooding over the reflux-rate range shown, with a minimum value at a reflux rate of about 8 liters per hour (fig. 9(a)). The 2-inch-diameter column packed with 3/8-inch Raschig rings (fig. 9(b)) had theoretical-plate values after preflooding of about 58 to 62 over the reflux-rate range shown with a minimum value at a reflux rate of about 8 liters per hour. This minimum in the curves has been previously observed for saddle and ring packings (reference 8). The efficiency of all packings in the steel columns was increased by preflooding.

The high ratio of packing to column diameter in the case of the ring-packed column caused poor efficiency in comparison with the saddle-packed column. The size rings tested was not considered as optimum but merely representative of the packing that had been in use in the column for some time.

The theoretical-plate values after preflooding for all packings in the 2-inch-diameter column and for the empty 2-inch-diameter column are shown for comparison in figure 10. The efficiency of the helix packing rapidly drops off with increasing reflux rates, whereas the saddle and ring packings are nearly independent of reflux rate although they do exhibit a minimum theoretical-plate value at a reflux rate of about 8 liters per hour. The efficiency of the empty column is also essentially independent of reflux rate, except at very low rates.

The helix packing with theoretical-plate values ranging from about 115 to 179 had efficiencies that were about $1\frac{1}{3}$ to 2 times higher than those of the 1/4-inch Berl saddles and about 2 to 3 times higher than those of the 3/8-inch Raschig ring packing.

The 1-inch-diameter column packed with 1/8-inch stainless-steel helices gave separations at total reflux that were too close to 100 percent to obtain accurate theoretical-plate values from the concentrations. Maximum efficiency exceeded at least 200 theoretical plates at low reflux

rates. The possible separation that can be obtained is shown in figure 11 by the variation in distillate and residue compositions of the test mixture throughout a distillation using this column. The reflux ratio varied considerably during this distillation but averaged about 160 to 1.

The pressure drop through the steel columns is shown in figure 12. The pressure drop through the empty column was too low to be measured with any degree of accuracy on the mercury manometer and therefore was not plotted. Pressure drop through the 1-inch-diameter helix-packed column and the 2-inch-diameter saddle-packed column were about the same below a vapor velocity of about 0.8 feet per second. The pressure drop through the 2-inch-diameter helix-packed column was the highest of the packings tested at vapor velocities below the flooding point.

SUMMARY OF RESULTS

An evaluation was made of combinations of four glass and of four steel distillation columns with various types of packing. The following results were obtained at atmospheric pressure using a test mixture of methylcyclohexane and 2,2,4-trimethylpentane:

1. Preflooding increased the theoretical-plate values for all packings tested. The increase over unflooded operation was as high as 55 percent with the glass column with wire-coil packing.

2. The 6-foot-packed-height glass column with wire-coil packing had theoretical-plate values of about 80 to 190, which were 2 to 4 times the theoretical-plate values of columns of the same height packed with 3/16-inch glass helices.

3. The 1-inch-diameter steel column with 1/8-inch stainless-steel helix packing gave separations indicating a maximum theoretical-plate value of over 200 at low reflux rates.

4. The 2-inch-diameter steel column with 1/8-inch stainless-steel helix packing had theoretical-plate values of 115 to 179. These values were $1\frac{1}{3}$ to 2 times higher than those obtained with the 1/4-inch Berl saddle packing and 2 to 3 times higher than those obtained with the 3/8-inch Raschig ring packing in the same diameter columns.

5. The deviation of the test mixture from ideality is sufficient to have considerable effect on the calculated theoretical-plate values.

Lewis Flight Propulsion Laboratory,
National Advisory Committee for Aeronautics,
Cleveland, Ohio, August 31, 1950.

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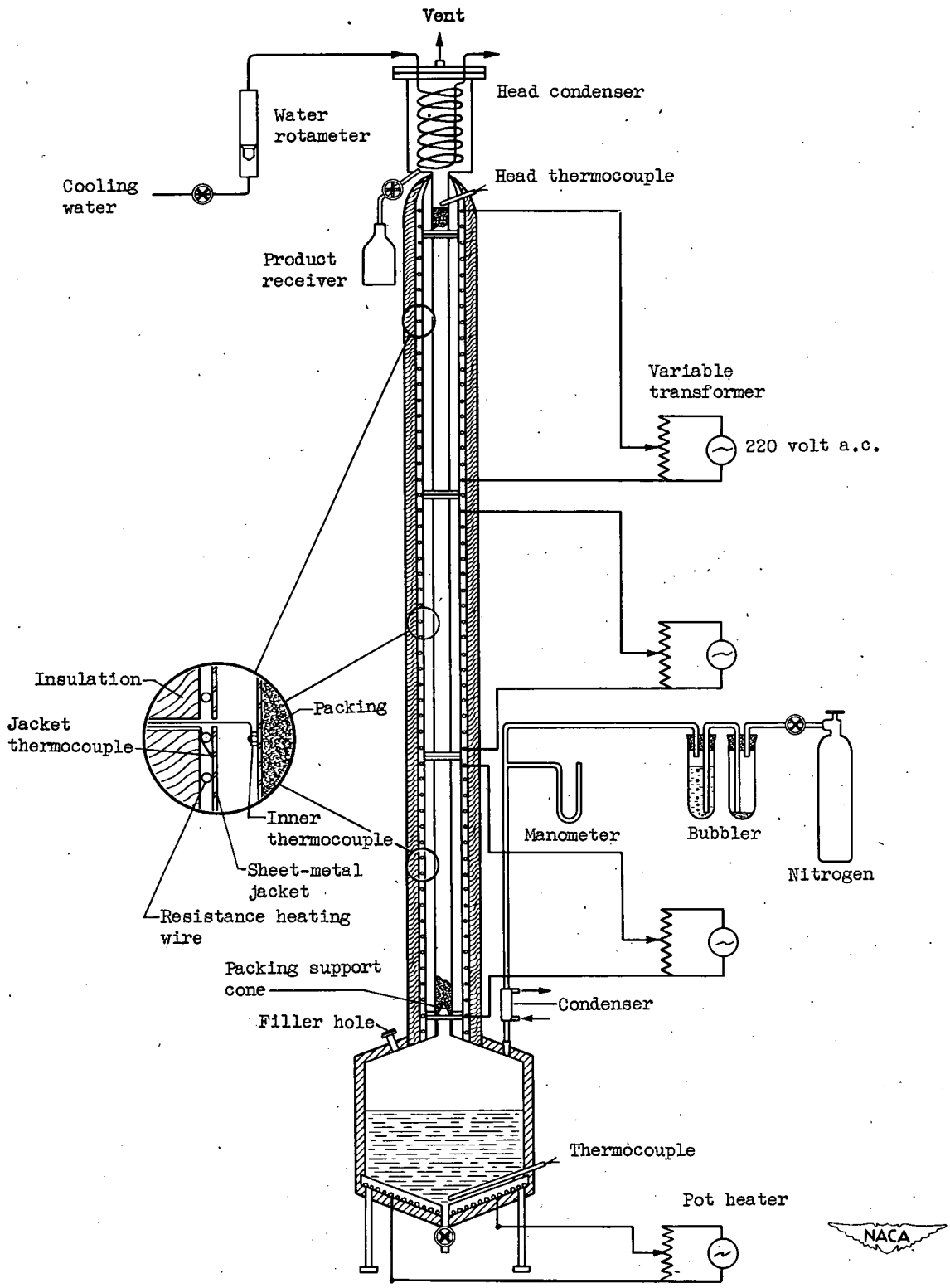


Figure 1. - Stainless-steel distillation column.

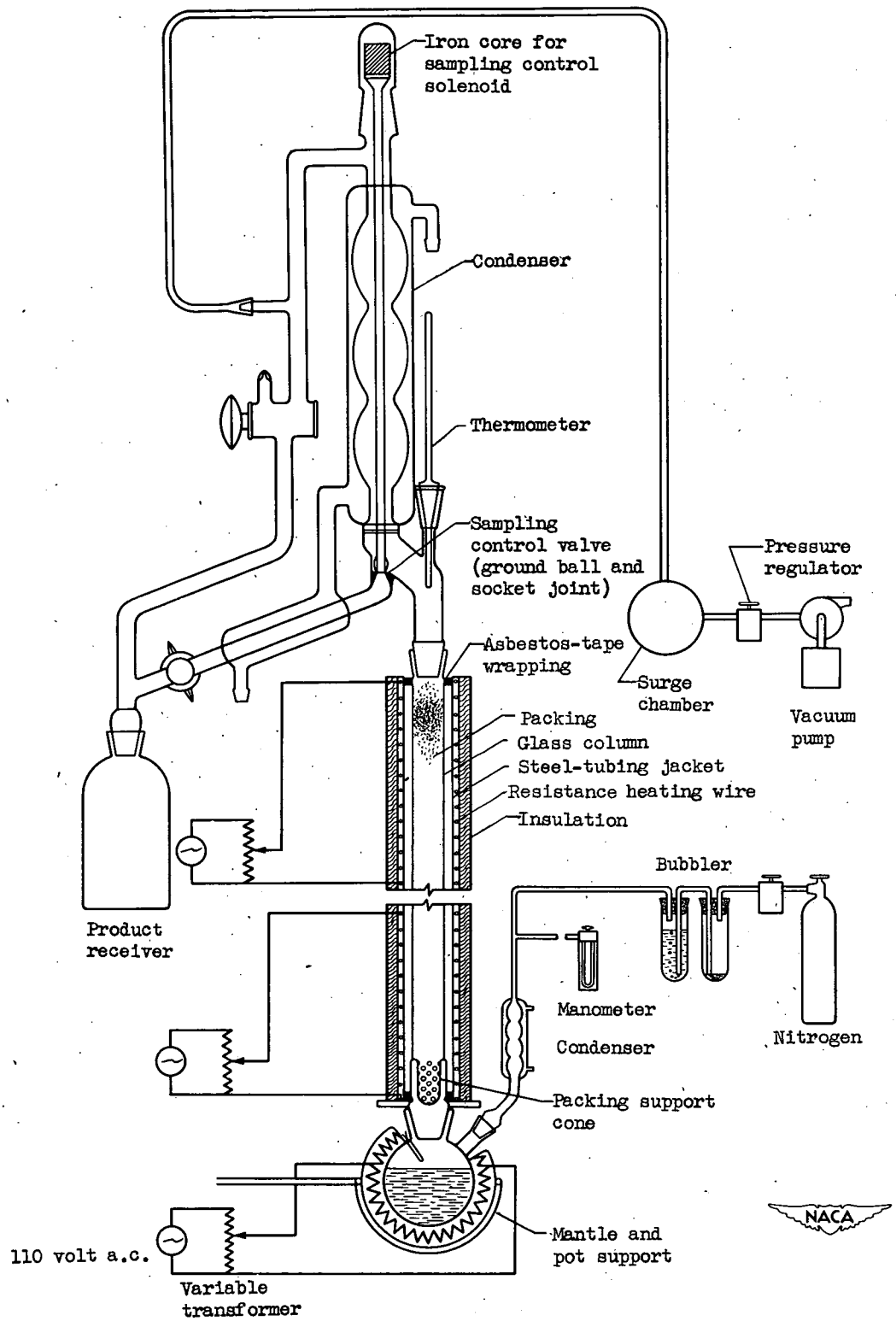


Figure 2. - Glass distillation column.

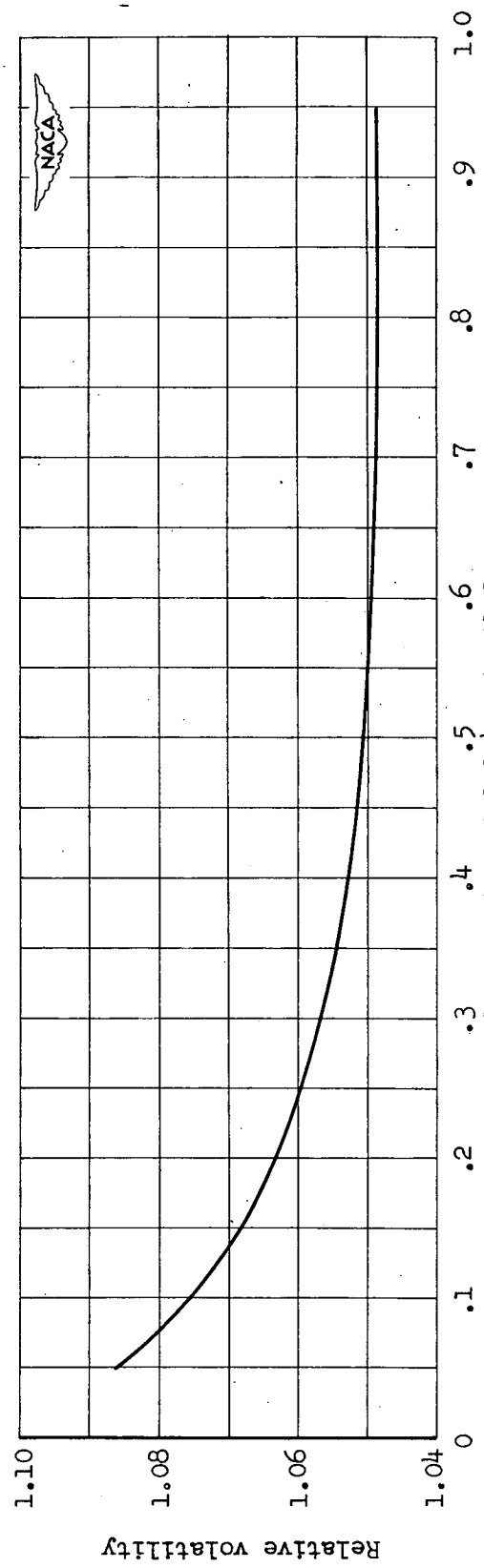


Figure 3. - Relative volatility for mixture of 2,2,4-trimethylpentane and methylcyclohexane at atmospheric pressure. (Data are taken from reference 3.)

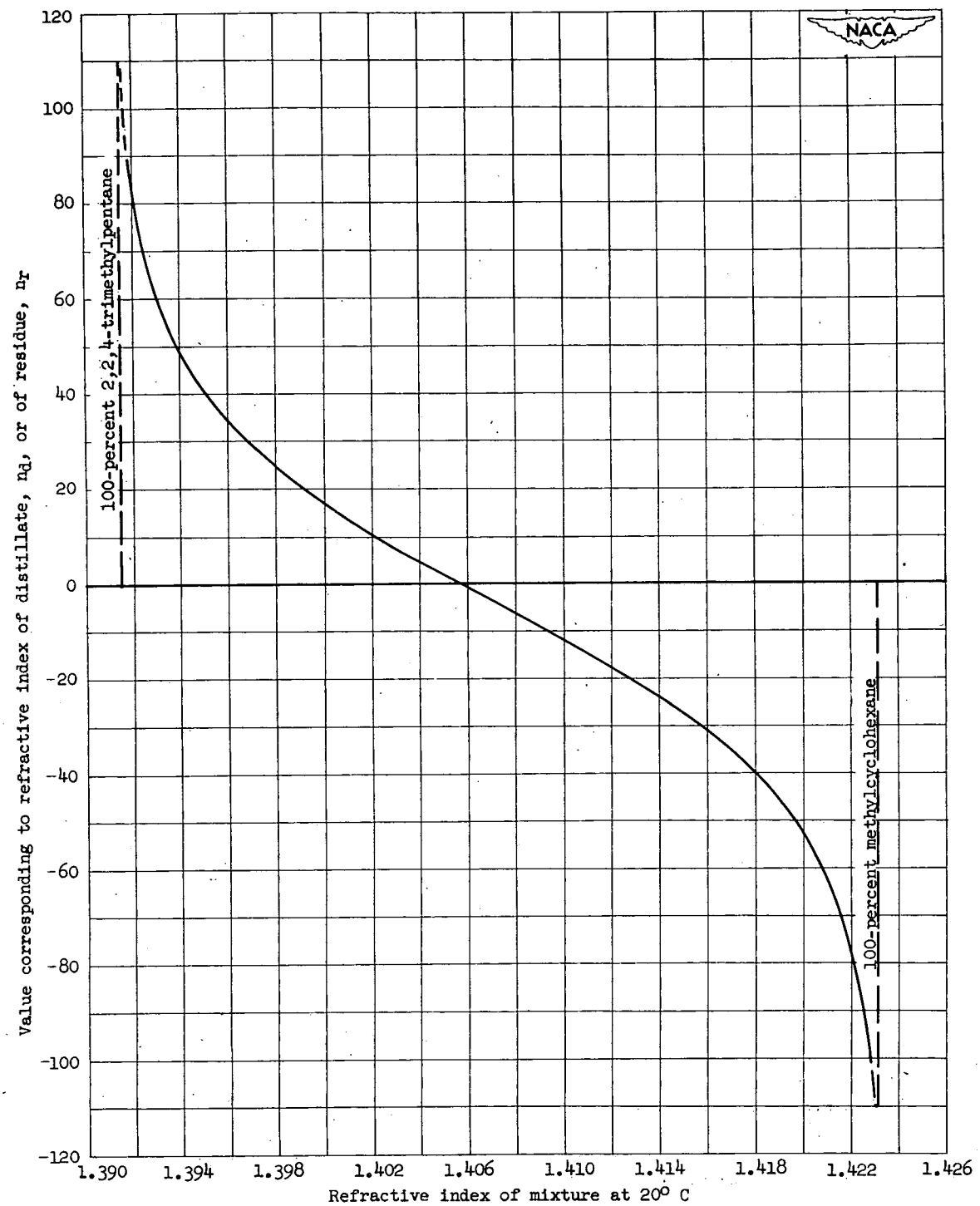
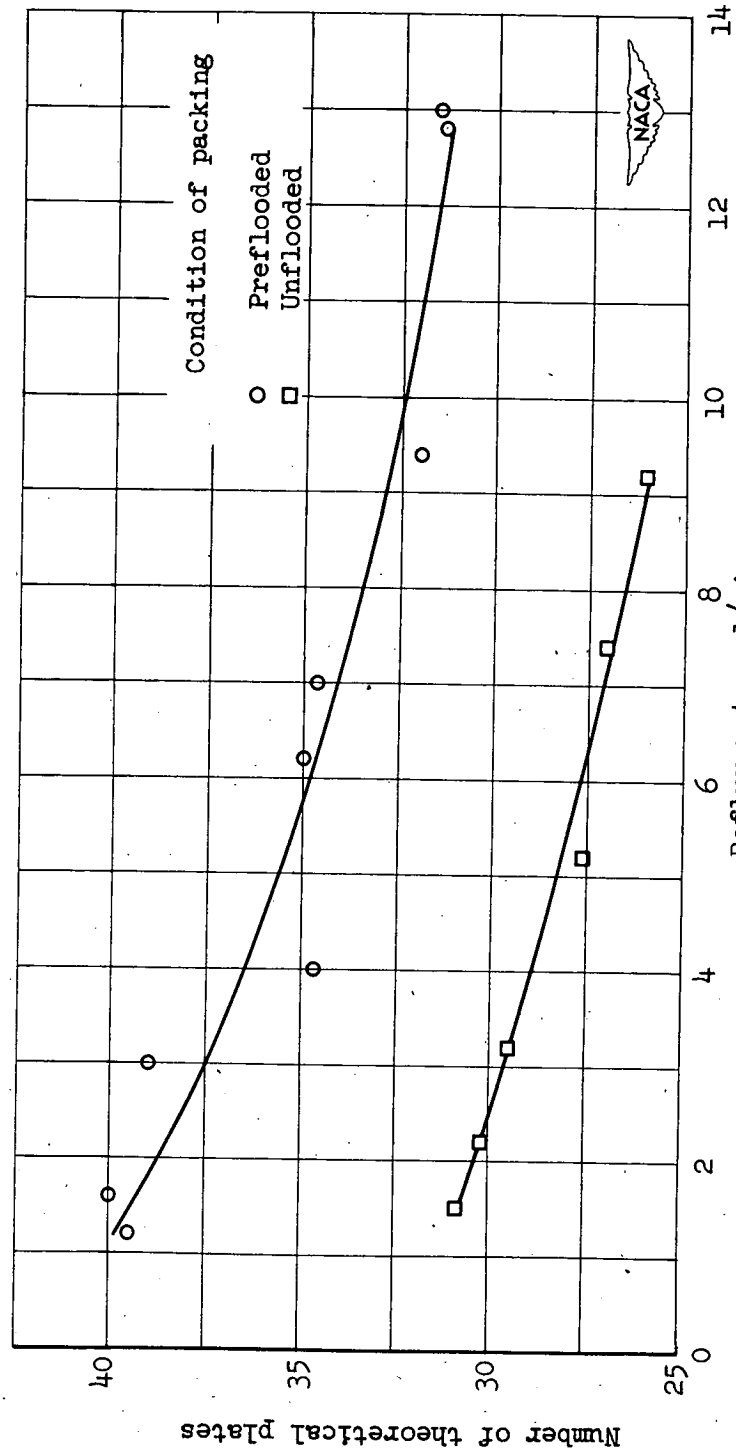
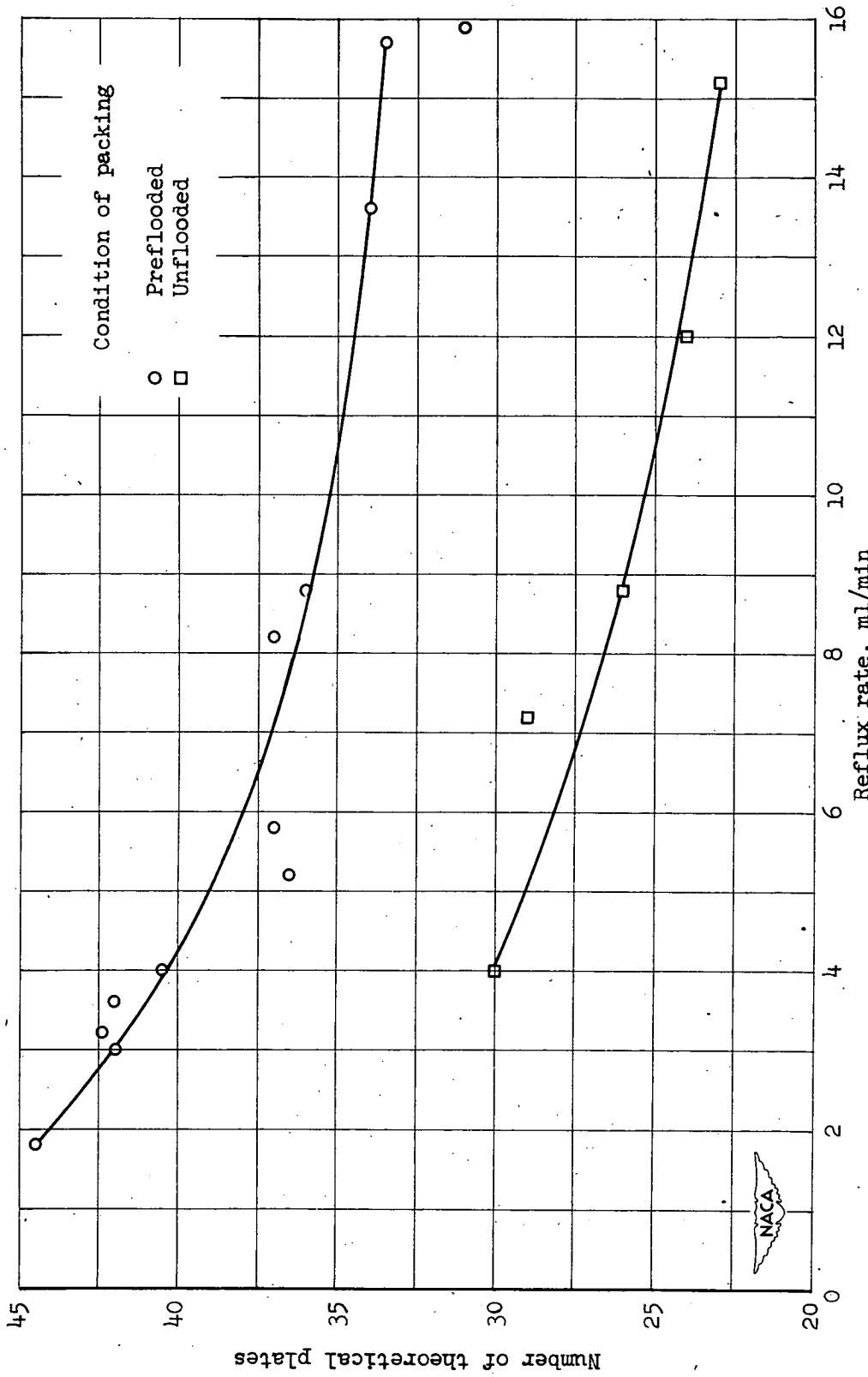


Figure 4. - Curve for determining number of theoretical plates from refractive index of distillate and residue samples for mixture of methylcyclohexane and 2,2,4-trimethylpentane. $N = n_d - n_r$, where N is total number of theoretical plates.



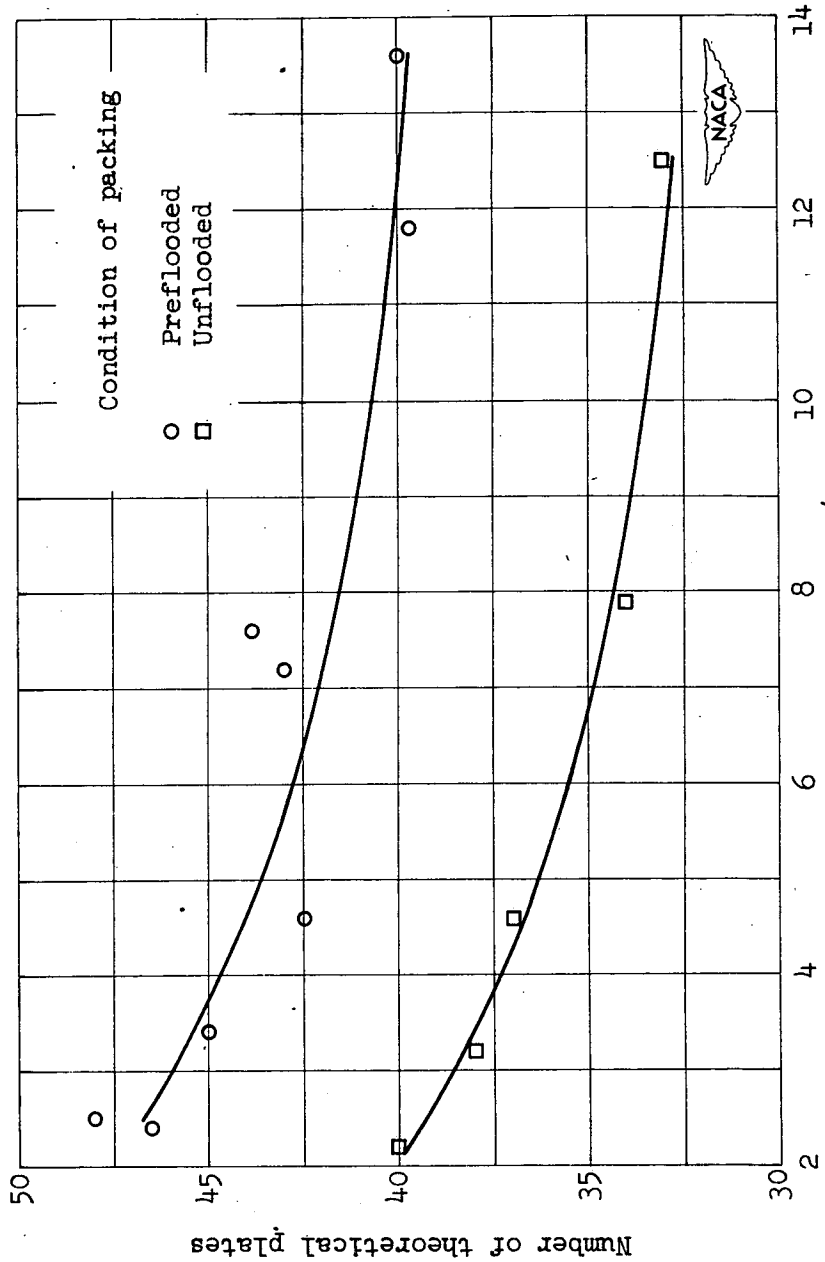
(a) Outside diameter, 22 millimeters; packed height, 6 feet; packing, 3/16-inch glass helices.

Figure 5. - Theoretical-plate values for glass distillation columns.



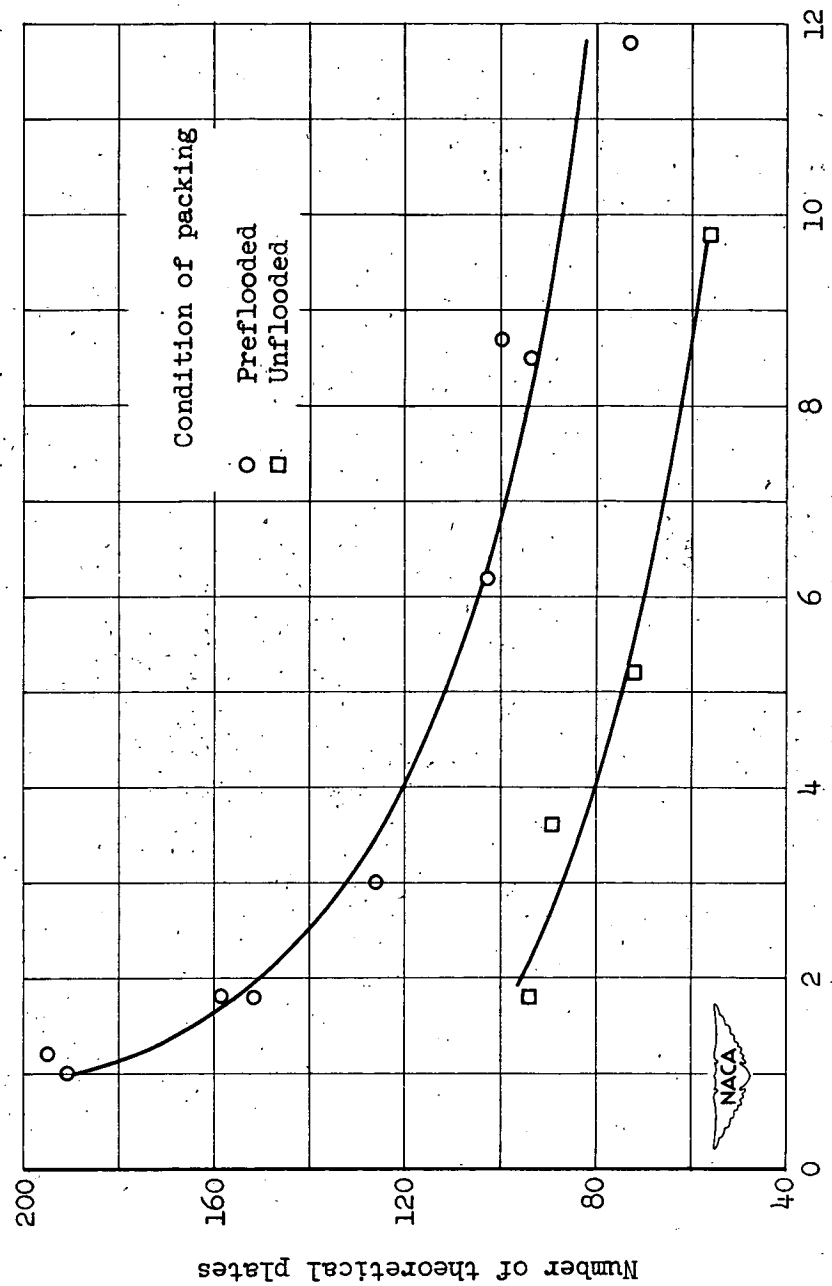
(b) Outside diameter, 32 millimeters; packed height, 6 feet; packing, 3/16-inch glass helices.

Figure 5. - Continued. Theoretical-plate values for glass distillation columns.



(c) Outside diameter, 22 millimeters; packed height, 7 feet; packing, 3/16-inch glass helices.

Figure 5. - Continued. Theoretical-plate values for glass distillation columns.



(d) Inside diameter, 22 millimeters; packed height, 6 feet; packing, wire coil.

Figure 5. - Concluded. Theoretical-plate values for glass distillation columns.

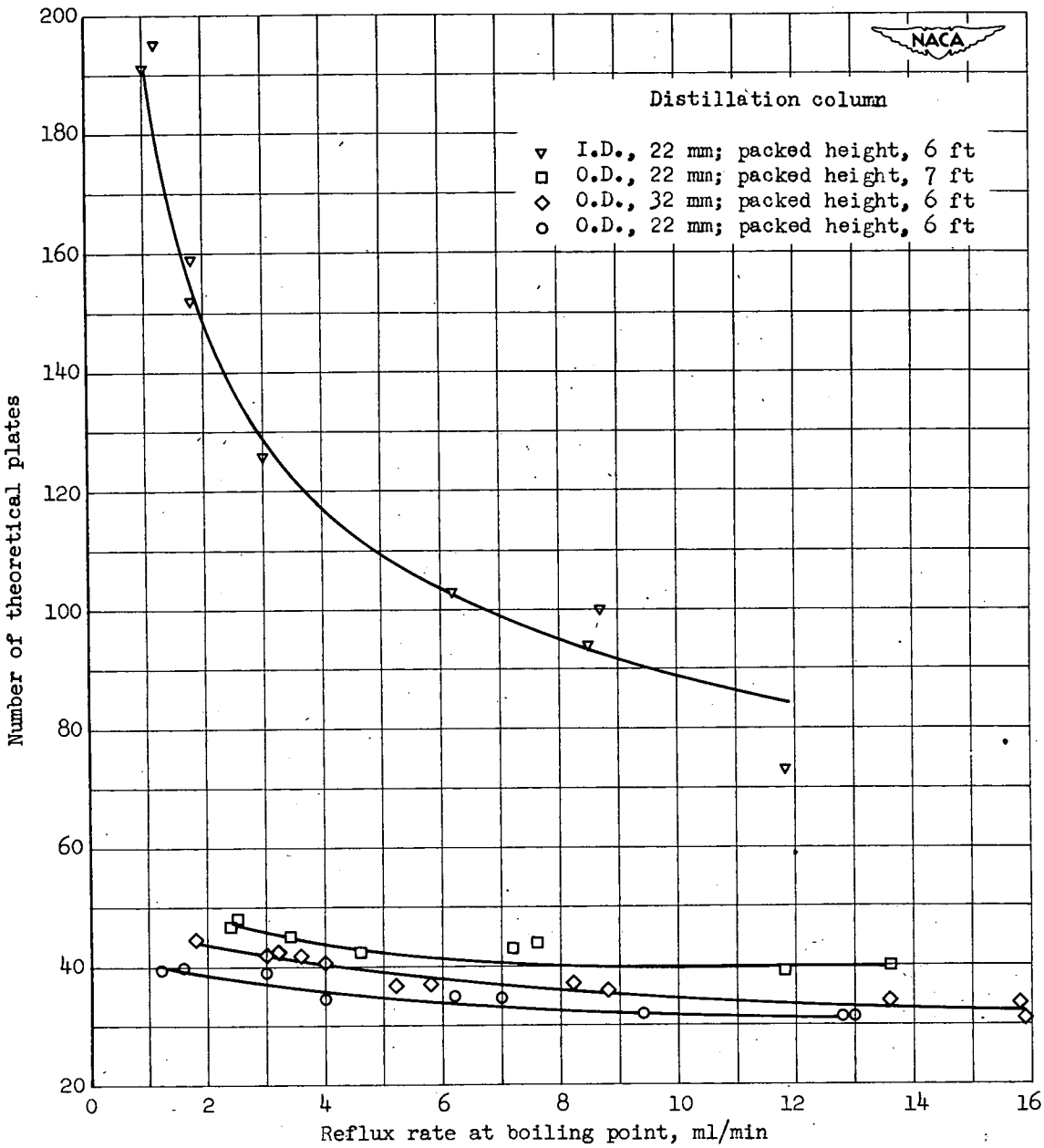


Figure 6. - Comparative theoretical-plate values after preflooding of glass distillation columns.

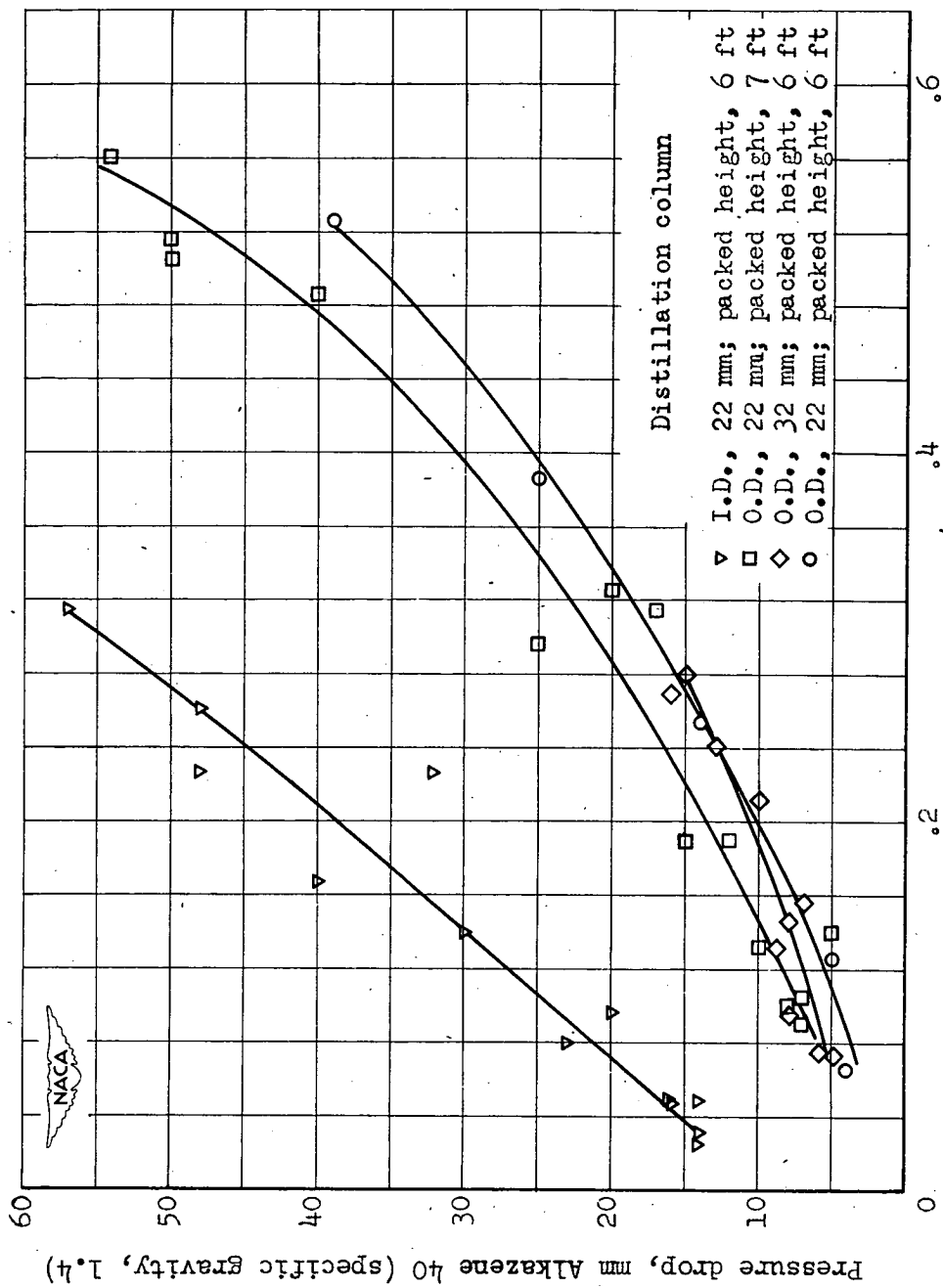


Figure 7. - Pressure drop through glass distillation columns.

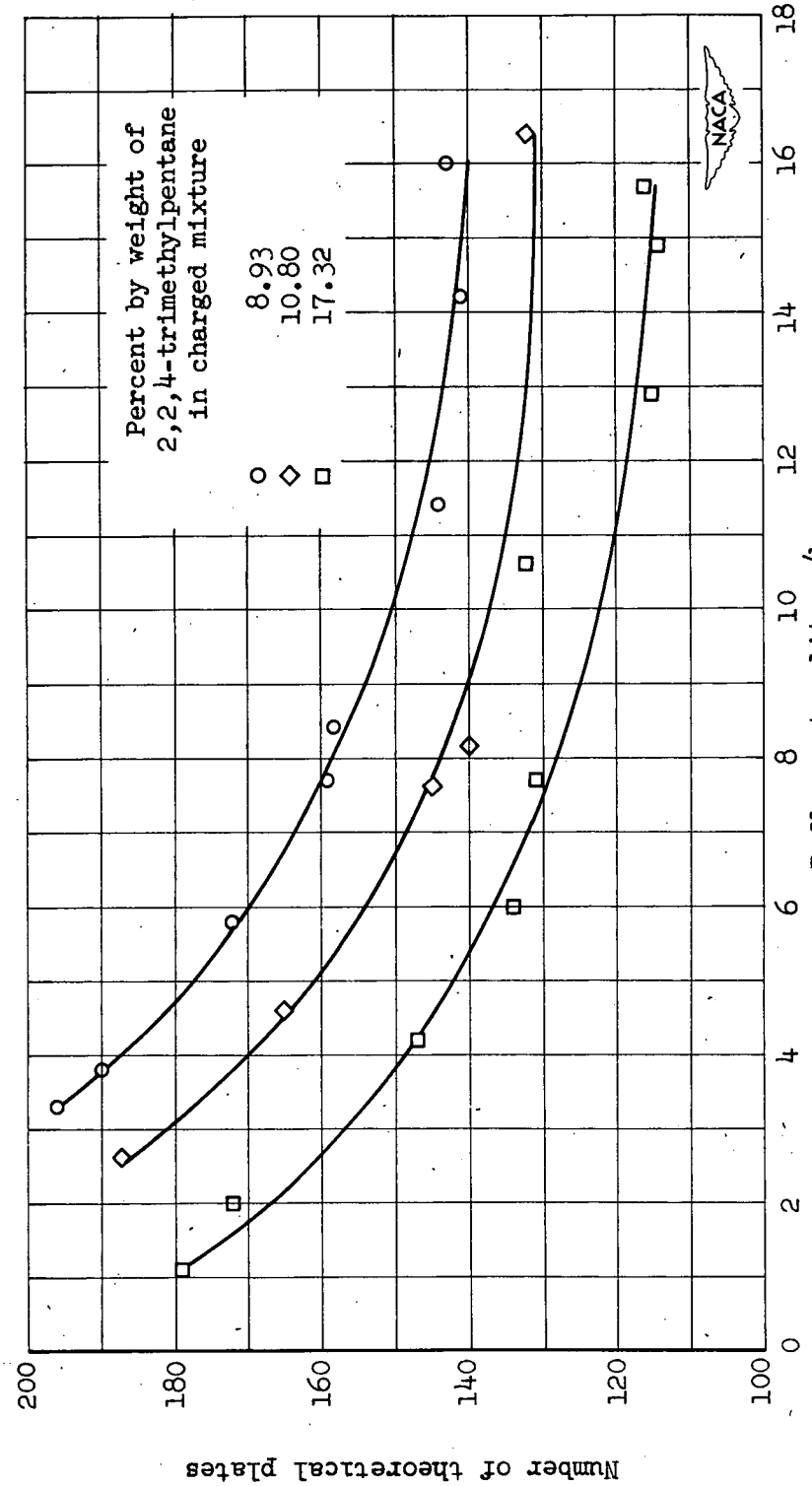


Figure 8. - Effect of mixture composition on theoretical-plate values for stainless-steel distillation column with 2-inch diameter and 1/8-inch stainless-steel helix packing.

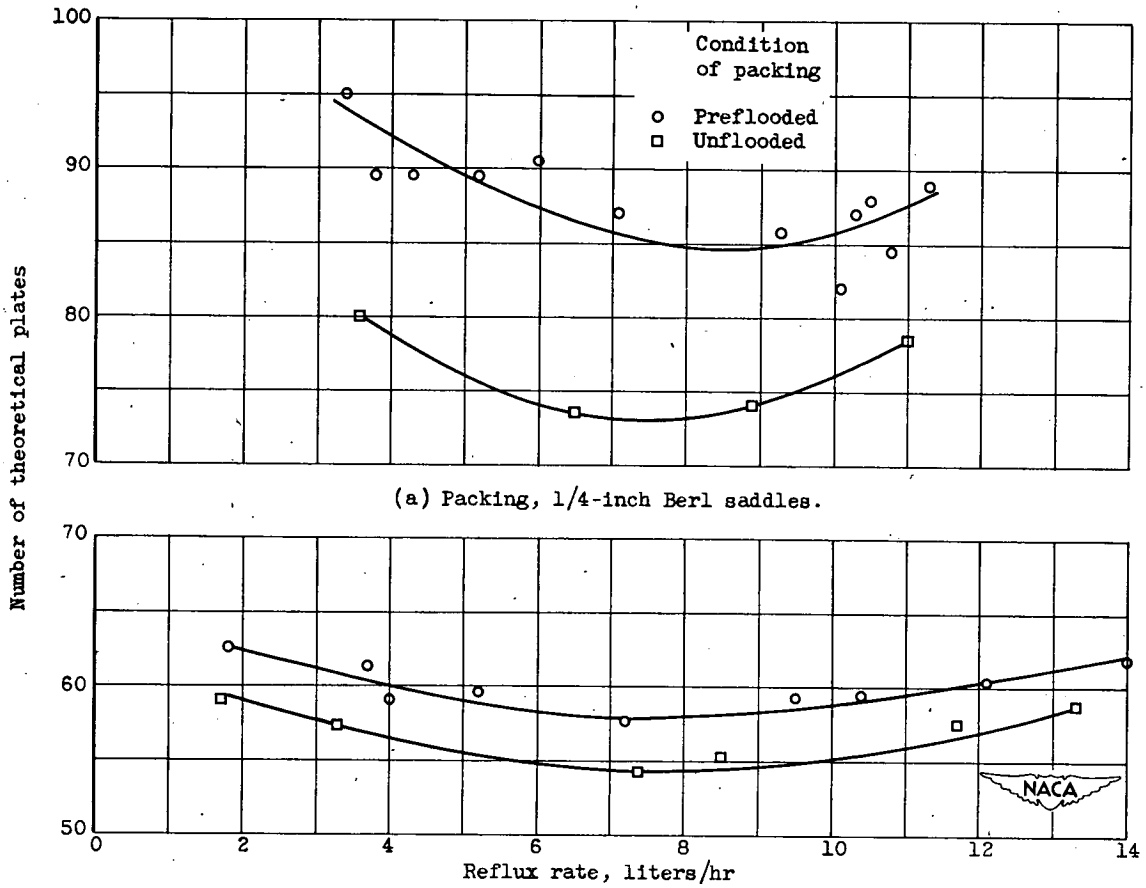


Figure 9. - Theoretical-plate values for stainless-steel distillation columns with 2-inch diameters with two packings.

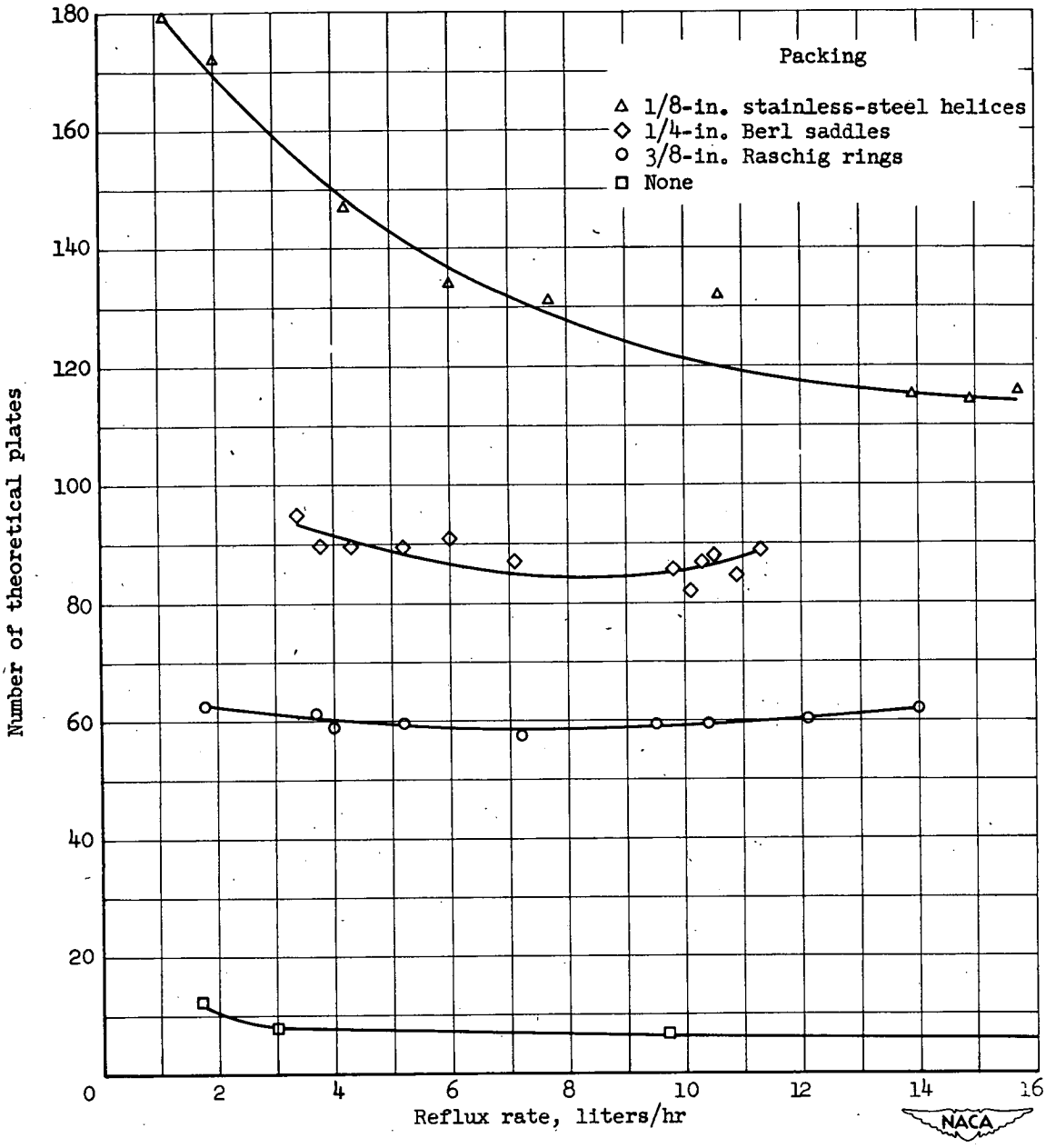


Figure 10. - Comparative performance after preflooding of stainless-steel distillation columns with 2-inch diameters and various packing materials.

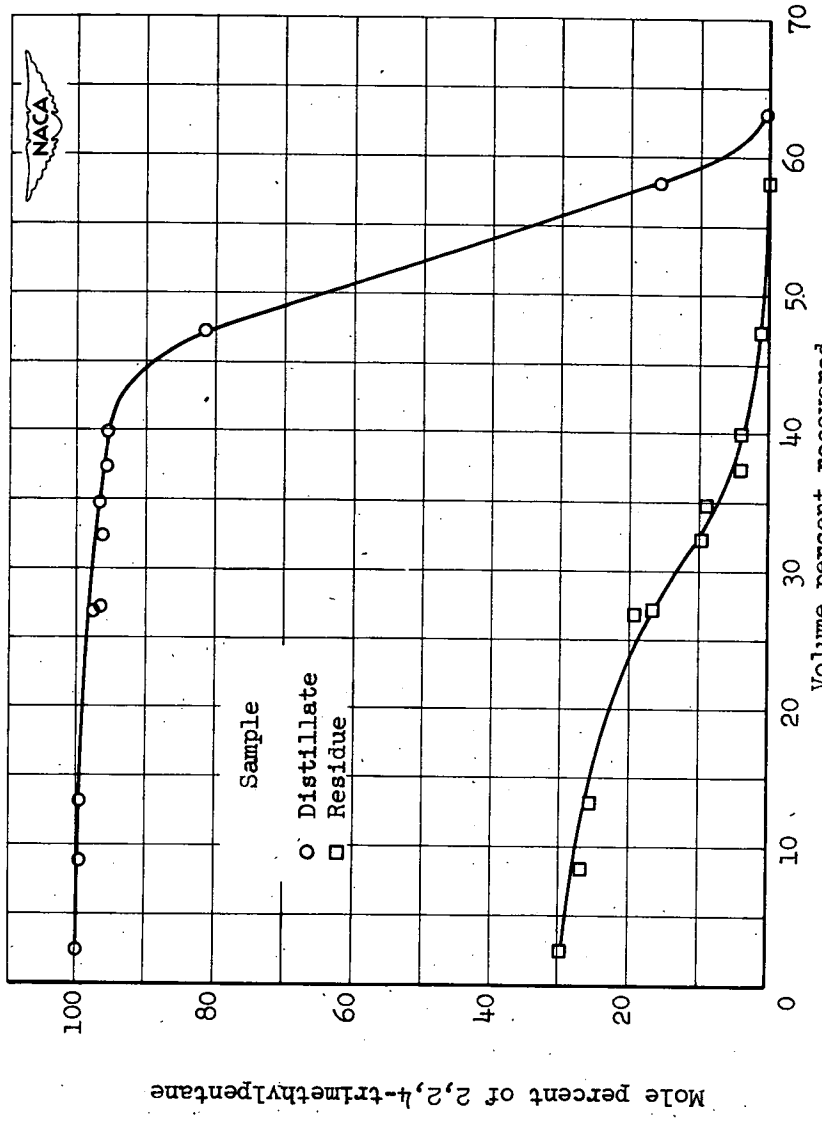


Figure 11. - Variation of residue and distillate composition during distillation of mixture of 2,2,4-trimethylpentane and methylcyclohexane in stainless-steel distillation column. Diameter, 1 inch; packed height, 30 feet; packing, 1/8-inch stainless-steel helices; average reflux ratio, 160 to 1.

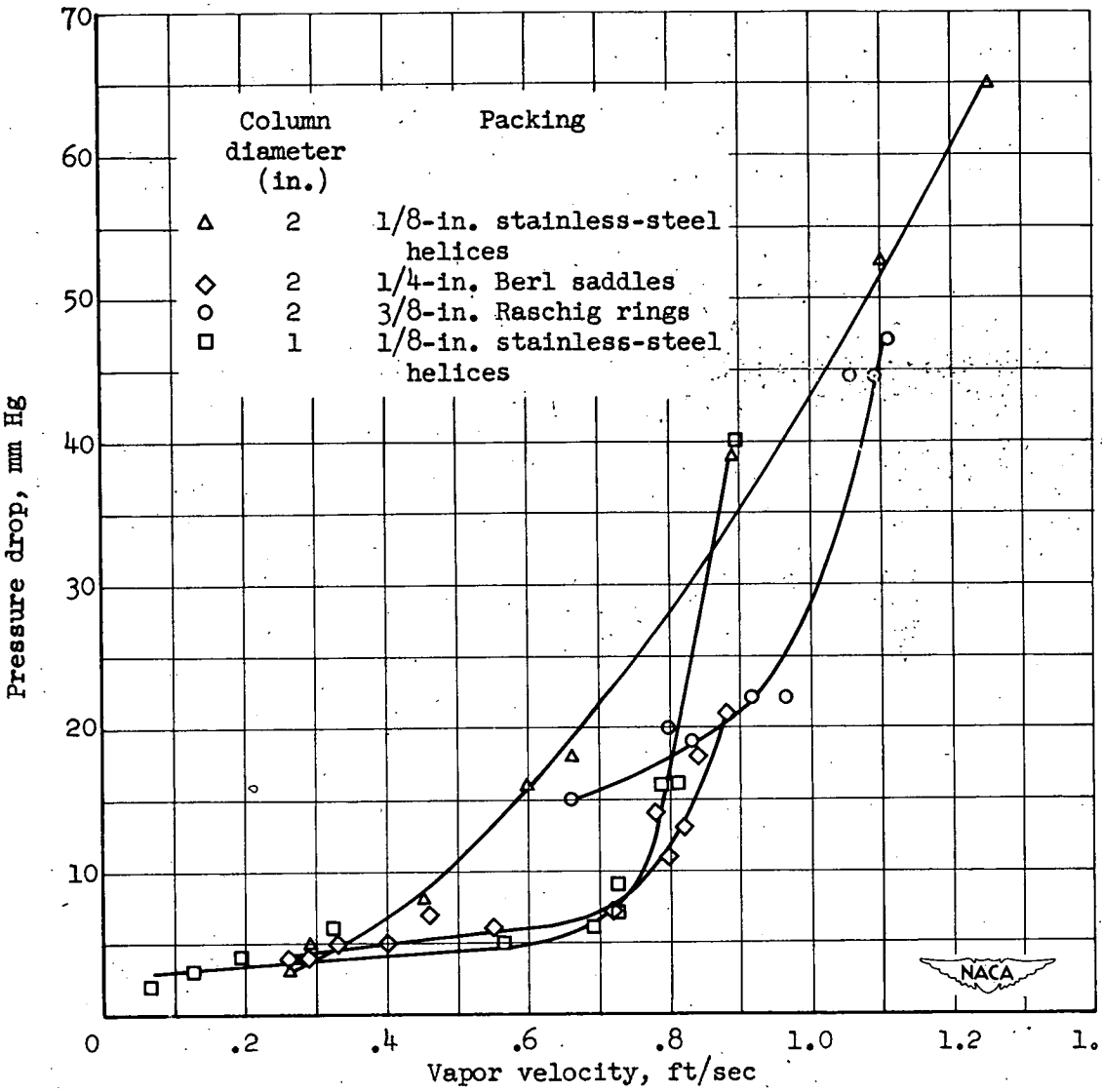


Figure 12. - Pressure drop through various packing materials in 30-foot-packed-height stainless-steel distillation columns.