TITLE: **LIQUID DISTRIBUTION STUDIES IN PACKED BEDS**

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RECENT ADVANCES IN ABSORPTION
AND DISTILLATION

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ABSTRACT

LIQUID DISTRIBUTION STUDIES IN PACKED BEDS

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Recent research has shown that liquid distribution is even more important to the apparent efficiency of packed columns than was previously believed. An "Adjustable Liquid Distributor"* which allows distribution to be changed with the column in operation has been designed and is being used to gain more insight into packed column operation. Preliminary conclusions are that discontinuities and zonal flows have the most severe impact on efficiency.

*Patent Pending

INTRODUCTION

The effect of liquid distribution on packed tower performance has been the subject of both experimental and theoretical work for over 50 years. One of the earliest significant studies, and one which is still being cited today was that of Baker, Chilton, et al(1) in 1935 which established 8 as the ratio of tower diameter to packing size below which significant wall flow developed. A great deal of the work since then such as Billet(2) has concentrated not only on distribution but also on how it affects scaling up laboratory column results to a commercial size. Maldistribution, which becomes more and more of a concern as column diameters become larger, is often cited as the reason for the extensive scatter in published efficiency data(3) and is blamed for the failure of commercial columns to produce the results predicted from laboratory tests(2).

Most authors who have attempted to model the flow in random dumped packed beds start with a fine scale statistical approach. Cihla and Schmidt(4) employed a Gaussian distribution function for flow through a differential element of a packed bed and found that the resulting expression had the form of the equations used to describe molecular diffusional processes. They then obtained series solutions for several particular boundary conditions. Hoek(3) used numerical solutions of their differential equation to model his experimental results in which an imposed "large scale" maldistribution (such as 20 percent non-irrigation at the top of the bed) slowly evolved into "small scale maldistribution" or channeling which he concluded is an inherent and stable property of the packing. Albright(5) used a random number technique to model flow through a packed bed and obtained similar results to Hoek's, except that he chose to utilize the term "natural distribution" to describe the fine scale flow pattern which he also considered to be a property of the packing. He concluded that an initial distribution that is better than natural will quickly degrade to it and one that is worse will achieve it but sometimes at a very slow rate. For example, he computed that initial regions of non-homogeneous flows such as a non level drip-pan could double the distance required to reach "natural flow" compared to homogeneous initial distribution. This could seriously impact the anticipated overall column efficiency. The issue thus facing the distributor designer/column operator is not only how to design a good distributor but, perhaps more importantly, what to set as the fabrication and installation tolerances.

IDEAL DISTRIBUTION

An example of the effect of initial liquid distribution on packed column performance comes from FRI's data bank. Early experimental work(6) employed a notched trough distributor (Figure 1) almost exclusively. At the suggestion of Zuiderweg(7), a Tubed Drip Pan Distributor similar to that described by Billet(8) was fabricated (Figure 2). However, Billet's 600 pour points/m^2 were reduced to approximately 100 corresponding to the inherent channel density or "natural distribution" found by Hoek. The dramatic results are presented in Figure 3. Using this distributor, it is now possible to determine the inherent efficiency of any packing under ideal distribution conditions. However, the Tubed Drip Pan Distributor as designed, fabricated, and assembled for this study is commercially impractical. For example, the problem of warpage during welding was solved by employing 1/2 inch (12.7 mm) thick steel plate for the floor of the pan. The major deviation from a commercial distributor, however, lies in the unique construction of the commercial size experimental column. The entire 4 foot (1.22 m) diameter top head is removable. This means that the distributor does not have to be fabricated in sections which must be passed through a manway for assembly inside the column. The pan was fabricated in one piece for calibration and testing on the ground and then lifted intact and placed in the column where final leveling took place. Therefore, it was decided to initiate a program to quantify the penalty for deviation from ideal distribution and thereby provide guidelines for the design, fabrication and installation of commercial distributors.

THE ADJUSTABLE LIQUID DISTRIBUTOR

From the moment that a controlled maldistribution program was first contemplated, it was recognized that the necessity of shutting down and opening up a four foot diameter column for each distribution change would be prohibitively expensive and inordinately time consuming. Therefore, it would be necessary to change the distribution in a controlled manner while the column was in operation. The design finally chosen consisted of supplying liquid to each pour point by means of a slotted cylinder and piston arrangement operated by a rod extending through the top head of the column. This was made possible by equipping the column with a temporary flat head and sealing each rod by passing it through a "Swagelok" fitting with a teflon ferrule. Operation was restricted to atmospheric pressure. Figure 4 is a drawing of the device and Figures 5 and 6 are photographs. The cylinders were installed with the bottom of each slot 1/2 inch (12.7 mm) above the pan floor to avoid plugging by scale or dirt. The slotted cylinder piston pairs were then adjusted so that the bottom of each slot and the bottom of each piston were the same distance from the floor of the pan. Piston adjustment (slot opening control) was accomplished by means of a wing nut and brace arrangement. The piston was bottomed against an internal stop nut with the wing nut in an elevated position. The wing nut was then screwed down until it met resistance from the brace and the piston was raised by the appropriate number of turns of the wing nut. Since the threading on the rod is 20 revolutions/inch, it is believed that this arrangement allowed certainty of piston elevation to about 0.5 mm (0.02 inches). The device was calibrated with water at grade by measuring the flow through each drip point and making adjustments until satisfactory uniformity of flows was achieved. Figure 7 is a photograph of the calibration and Figure 8 shows the adjusting rods at the top of the column. Figure 9 presents the results of the final calibration prior to installation in the column. The small triangles point in the direction of deviation from the mean and their height is proportional to the magnitude. The standard deviation is 6.4 percent of the mean which was felt to be as low as could be practically achieved. This was tested by replicating the previous Tubed Drip Pan runs at the outset of the experimental program. Figure 10 presents a comparison of the ALD results with the previous results. The excellent agreement provided the necessary confidence to embark on the full experimental program. There were two intermediate shut downs for minor adjustments during the program and the baseline run was repeated after each one. The average of these runs is referred to hereafter as "Base".

EXPERIMENTAL PROGRAM

As mentioned previously, the necessity of sealing all of the adjustment rods mandated running at atmospheric pressure. The cyclohexane/normal heptane system which has been an FRI standard for 30 years was utilized with a 12 foot (3.658 m) bed of one inch (25.4 mm) stainless steel Pall Rings. Liquid samplers (Figure 11) which have been described previously(6) were installed at two foot intervals in the four foot column. Figure 12 is an installation drawing. The experimental program was designed to furnish design basis guidelines and insight into allowable tolerances. In all, a total of 104 runs were made, all at total reflux.

Three rates were used for each pattern. The target values were 30, 60, and 90 percent of the maximum useable capacity. Higher values were avoided because no holddown device was used and lower values were not desired because very small liquid rates magnify small differences in piston settings. Rates are presented as percent of useable capacity following the approach of Strigle(9). Our observations confirm his: modern, high open area packings remain hydraulically operable at rates well past the point where the efficiency has totally deteriorated, thus percent of flood is irrelevant to efficiency comparisons. Useable capacity is the loading at which separating efficiency begins to rapidly deteriorate.

INTERPRETATION OF RESULTS

The results were analyzed in two ways. The apparent overall HETP was determined and individual composition profiles were analyzed. The apparent overall HETP is, of course, the only thing the operator of a commercial column would observe, but as Silvey and Keller(6) pointed out, it is very sensitive to the handling of end effects and in this case, to changing distribution.

Analysis of bed profiles was done as follows: At total reflux, making the usual assumptions, the Fenske equation is:

$$N_{Theo} = \frac{1}{\ln\alpha} \ln\left[\left(\frac{X}{1-X}\right)_T \left(\frac{1-X}{X}\right)_B\right]$$

and

$$N_{Theo} = \frac{\text{Bed depth}}{\text{HETP}}$$

If α and HETP are constant, a plot of ln(X/1-X) vs bed depth should yield a straight line with slope $\ln\alpha/\text{HETP}$.

In a reasonably close boiling, almost ideal system, such as cyclohexane/normal heptane, α may be taken as constant and it is generally accepted that the HETP should be almost constant. Therefore, a curving line implies that the effective L/V is changing. Consequently, if a packed bed achieves natural flow almost immediately, a plot of ln(X/1-X) vs bed depth will be a straight line. If an initial maldistribution is being corrected, the slope of the ln(X/1-X) vs bed depth plot will increase from its initial value at the top of the bed until natural flow is achieved and then it will remain constant (unless wall flow develops which will flatten it out again).

The data were examined in this manner and exhibited the anticipated behavior. Figure 13 shows the results from a baseline run where every effort was made to achieve good initial distribution. As may be seen, the profile is a straight line. (Note reflux and bottoms compositions are indicated for information only but not used in any of the computations).

Figure 14 is a case of maldistribution with recovery. The profile in the lower portion of the bed is a straight line with a slope numerically equal to the good initial distribution case. The profile at the top of the bed shows the effect of moderate maldistribution, in this case, simulation of a uniformly sagging distributor.

Figure 15 presents a case where the maldistribution is so severe that there is no indication of recovery in 12 feet (3.658 m) of packing. This was an extreme case in which liquid flow to one half of the bed was shut off. However, it dramatically illustrates the fact that recovery can be very slow.

Initially, it had been hoped that a simple correlation such as type of maldistribution vs. distance to reach natural flow could be developed. Unfortunately, this is not the case. There is a complex loading effect as may be seen in Figure 16 which presents the results of simulating leakage of a seam (all of the slots at a minimum opening with one row in the center wide open). For this situation there is complete recovery after about six feet (1.8 m) at the high and medium rate and no recovery at all after 12 feet (3.658 m) at the low rate.

Figure 17 illustrates the fact that different types of maldistribution have greater or lesser effects. Results are presented in terms of the ratio of the apparent overall HETP to the base value. As may be seen, an 11 percent chordal blank (an old seal pan left in a column that formerly had trays) has a more harmful effect than blanking off 16 percent of the area in the center (malfunctioning ring distributor). Note also that chordal blanking was the only type of maldistribution in which the apparent overall HETP deteriorated with increasing loading. For all others, it either was essentially constant or improved as the rates were increased.

IMPLICATIONS FOR THE DESIGNER/INSTALLER/OPERATOR

Analysis of the detailed profiles from a fundamental point of view is proceeding. Additional experimental work is planned to extend the work to other packing sizes and to explore certain anomalies in the data. However, review of the apparent overall HETP results leads to some general observations of importance to the designer/installer/operator.

First, comfort may be derived from the fact that there appears to be a reasonable tolerance for a uniform maldistribution pattern. Figure 18 shows that there is virtually no penalty if a distributor uniformly tilts or uniformly sags (center to wall or vice versa) such that the ratio of highest to lowest flow is 25 percent. However, if a discontinuity occurs, the results could be disastrous. A tilting distributor will ultimately begin to lose flow in a manner identical to a chordal blank. Figure 19 compares the 25 percent tilt, which is virtually identical with the base, to an 11 percent chordal blank of an otherwise level distributor.

Another form of discontinuity is the creation of a zonal flow. This could be caused, for example, by an obstruction in a major branch point of a pipe grid type of distributor. Figure 20 compares tilts with a ratio of maximum to minimum flow of 1.25 and 1.5 to a situation where one half of the distributor is passing 25 percent more liquid than the other half. As may be seen, the loss of efficiency for the zonal flow case is twice as great as for a "uniform" maldistribution with twice as much variation from maximum to minimum.

Two runs which should be of particular interest to installers of distributors were made at the suggestion of a member company. They reported that inspection of a large distributor following installation showed that the variations followed a Gaussian pattern. To investigate this situation, a random number generator was used to generate a Gaussian flow distribution (maximum/ mean = 2) which was then randomly assigned. A second case divided the distributor into six zones, three designated high and three low. All randomly generated flows greater than the mean were randomly assigned to a high zone and all less than the mean to a low zone. Figure 21 presents the results which show practically no effect of the purely random variation but the zonal flow had a 20 percent decrease in efficiency.

Runs were also made in which pour point density was decreased. It was found that a significant reduction was possible before the effects could be observed. However, it must be remembered that this was achieved with a distributor that had been adjusted and leveled much more painstakingly than is usually practical or even possible commercially. Future work will test the combination of density reduction and non-ideal distribution.

IN SUMMARY

Studies of controlled maldistribution of reflux to a packed bed being used for distillation have shown that recovery to natural flow and constant HETP is an extremely complex function of many variables. However, a general conclusion which provides guidance to designers/ installers/operators is that a packed bed has a reasonable tolerance for both a uniform or smooth variation in liquid distribution and for one that is totally random. However, the impact of discontinuities or zonal flow is much more severe. Additional work is in progress to obtain a more complete understanding.

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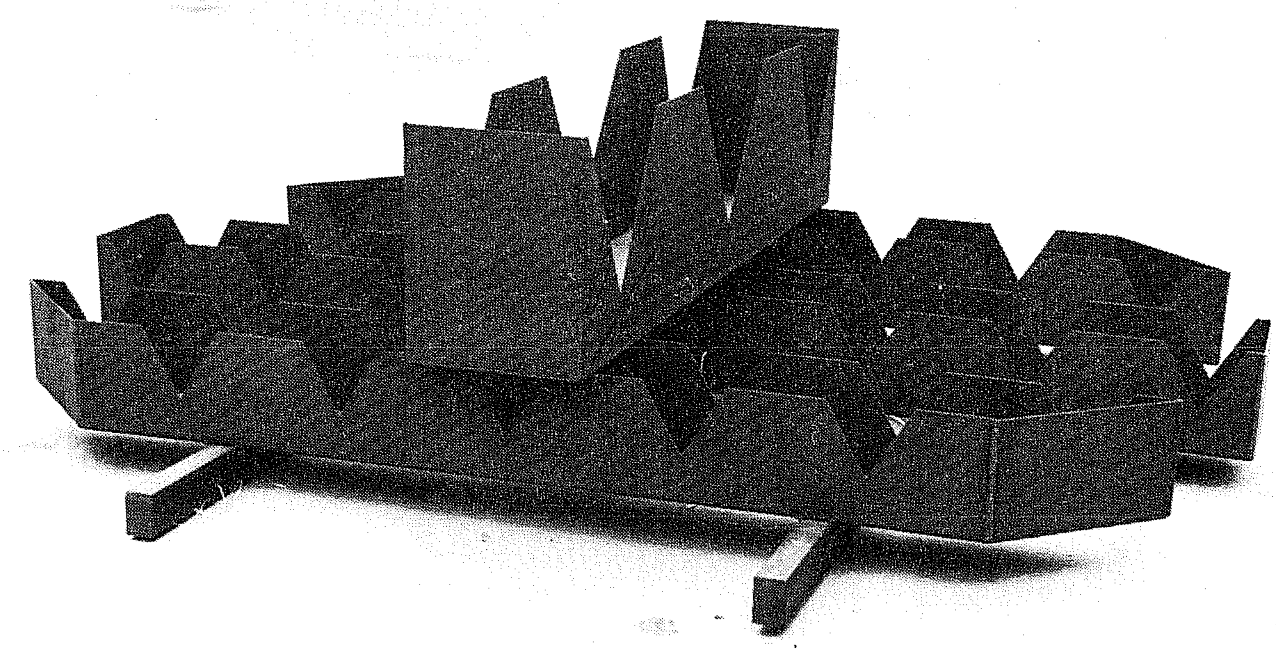


NOTCHED TROUGH DISTRIBUTOR

Figure 1

TUBED DRIP PAN IN SERVICE

Figure 2

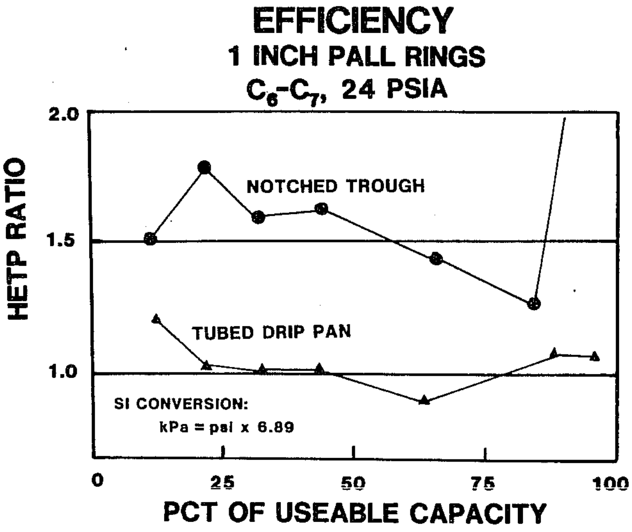
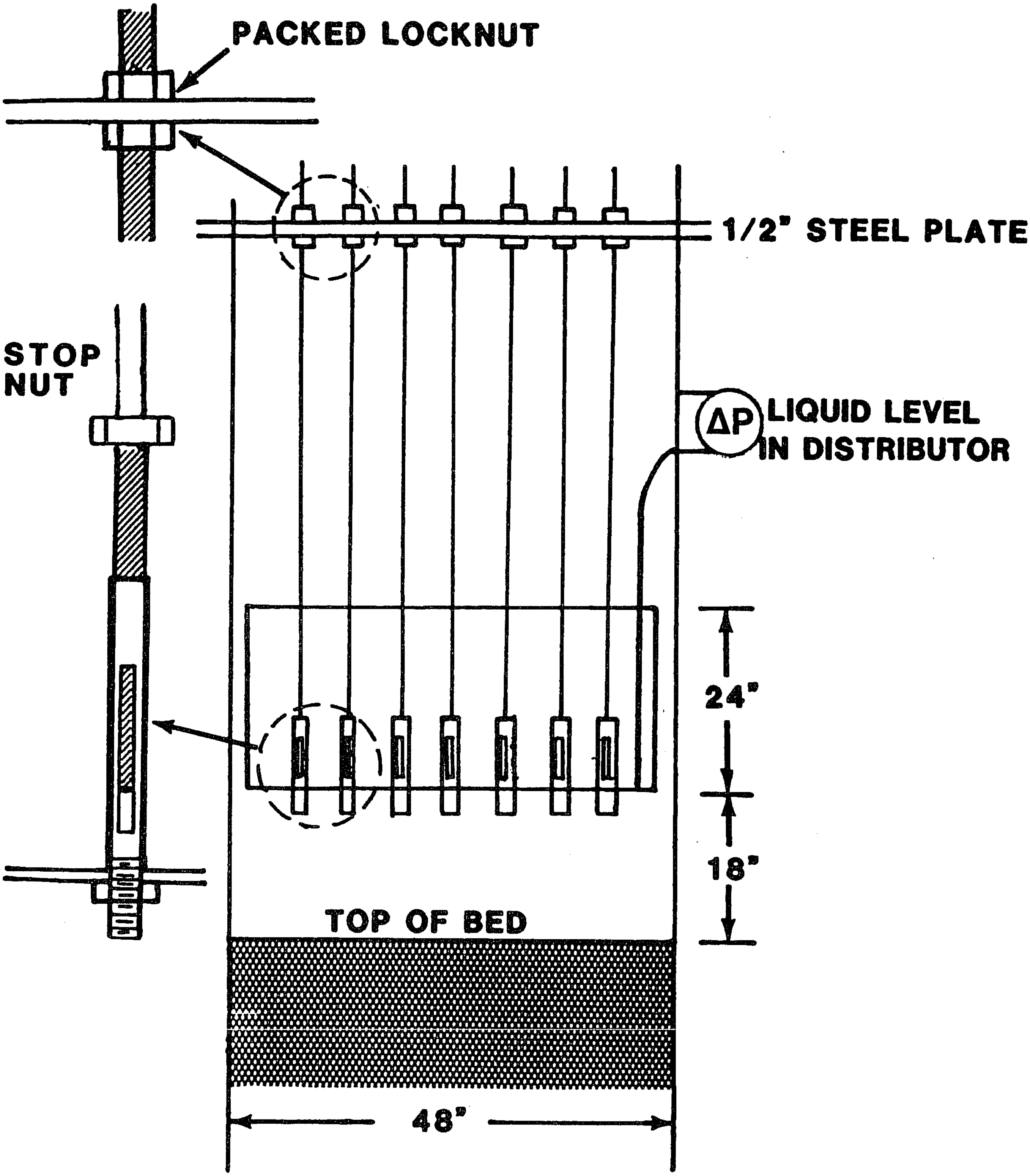


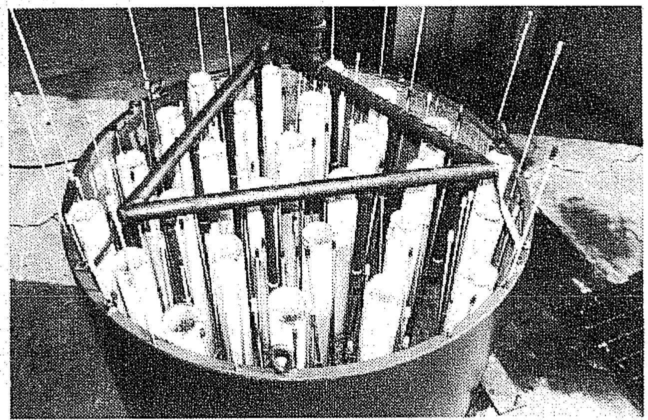
Figure 3

ADJUSTABLE LIQUID DISTRIBUTOR



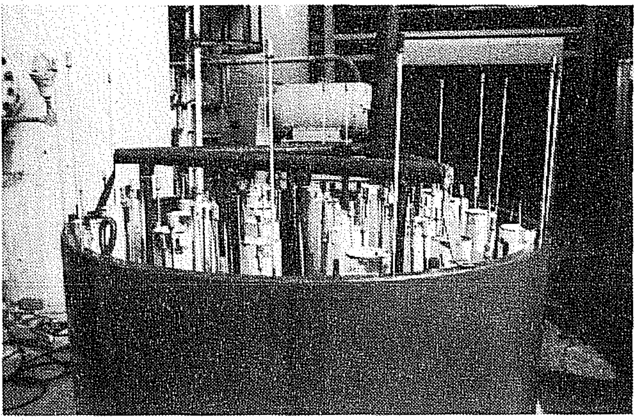
SI CONVERSION: mm = in x 25.4

Figure 4



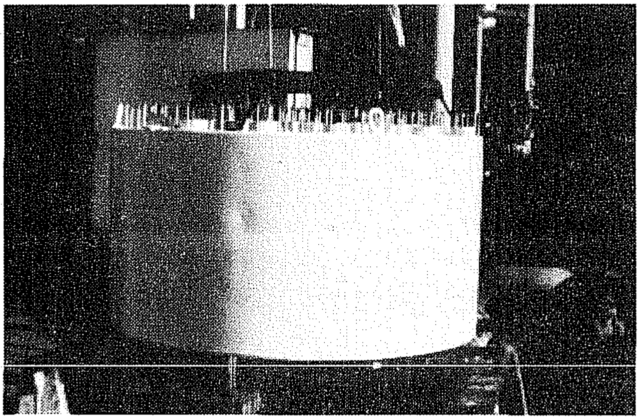
ADJUSTABLE LIQUID DISTRIBUTOR

Figure 5



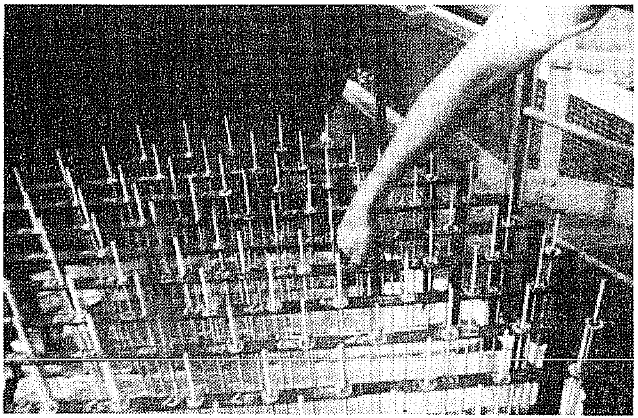
ADJUSTABLE LIQUID DISTRIBUTOR

Figure 6



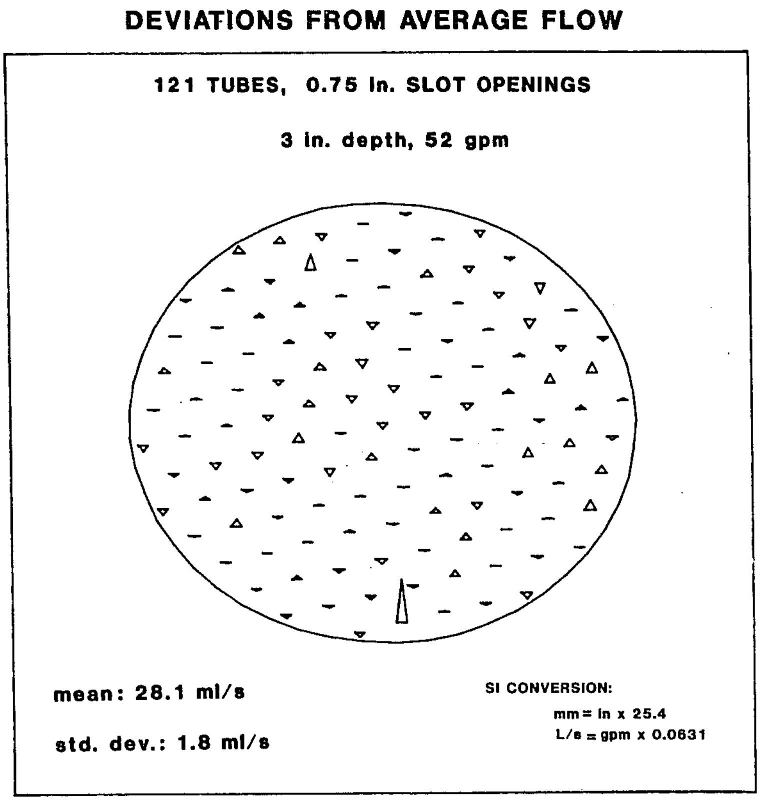
WATER CALIBRATION

Figure 7



SETTING PISTONS AT TOP OF COLUMN DURING OPERATION

Figure 8



CALIBRATION RESULTS

Figure 9

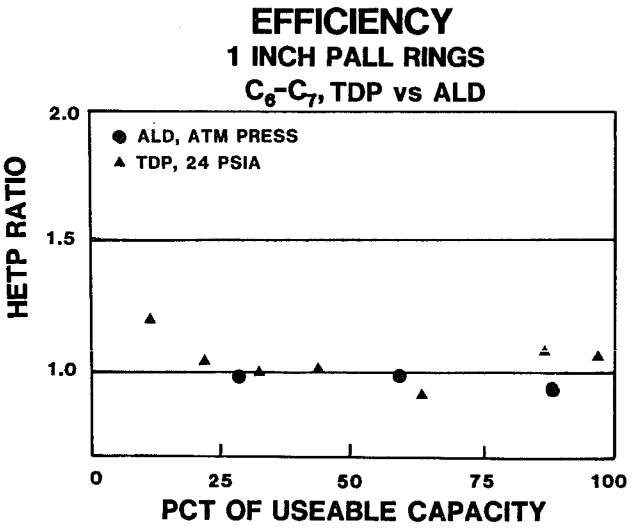
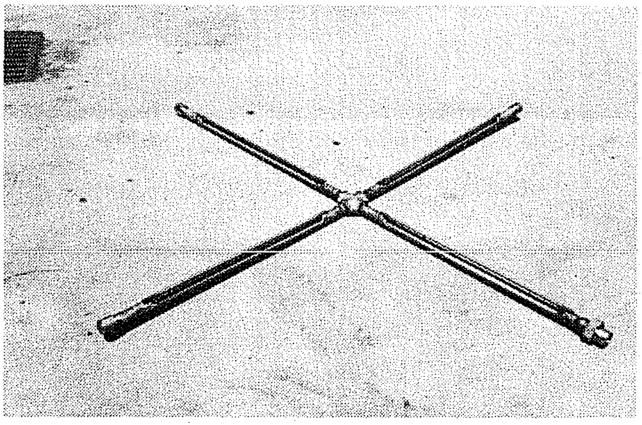


Figure 10



LIQUID SAMPLING DEVICE

Figure 11

INSTALLATION DIAGRAM

ADJUSTABLE LIQUID DISTRIBUTOR

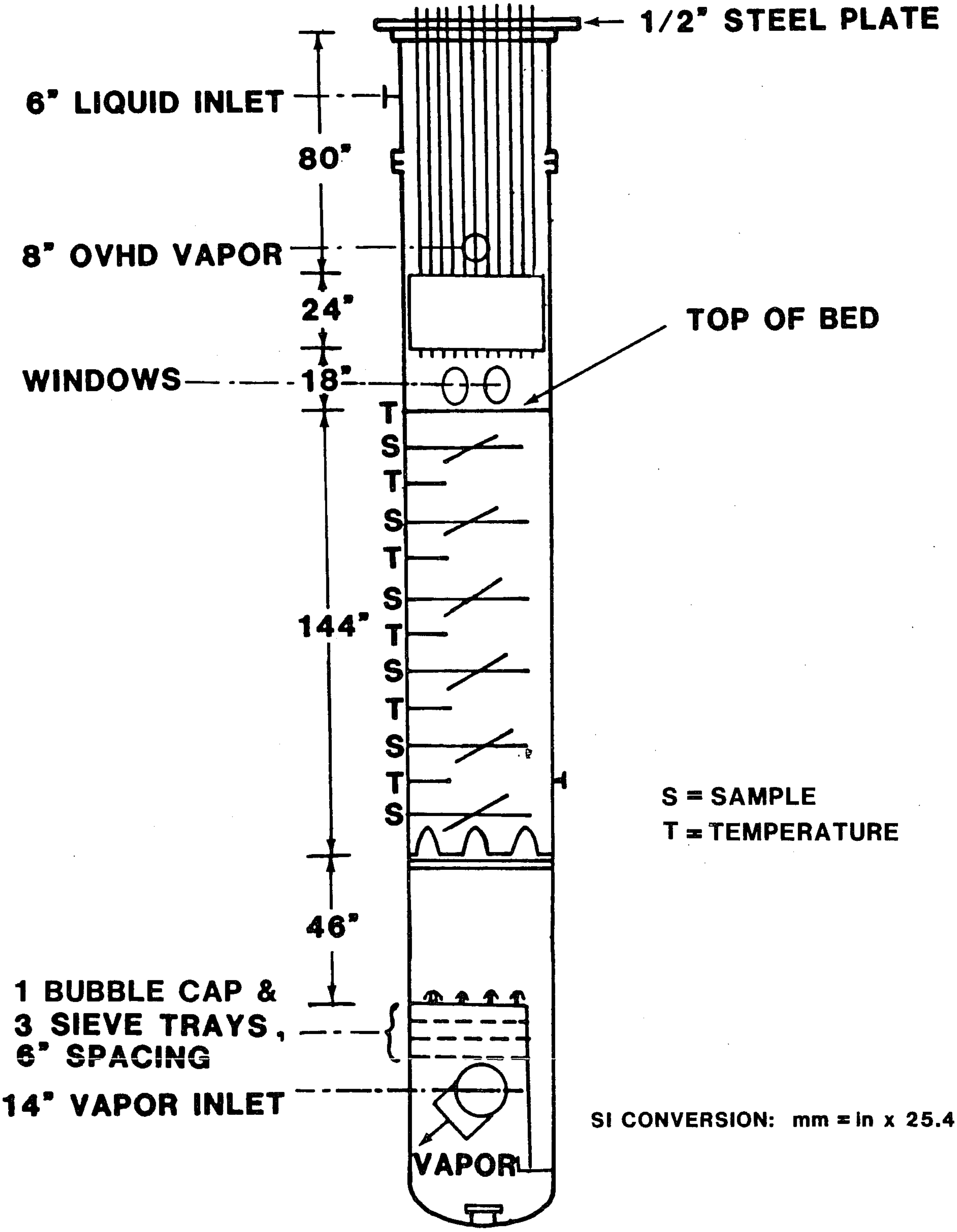


Figure 12

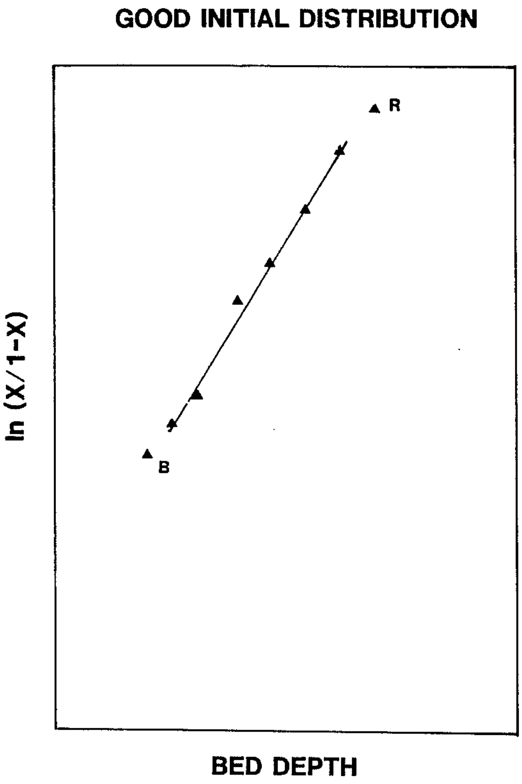

GOOD INITIAL DISTRIBUTION

Figure 13

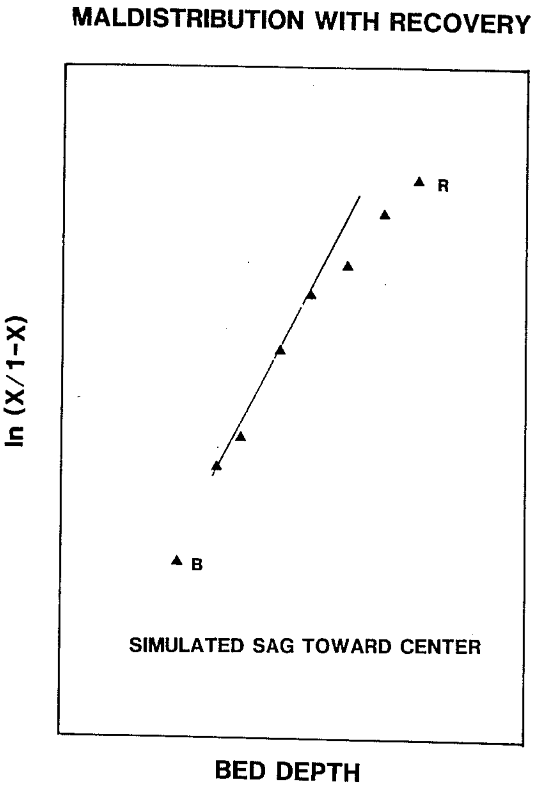

MALDISTRIBUTION WITH RECOVERY

Figure 14

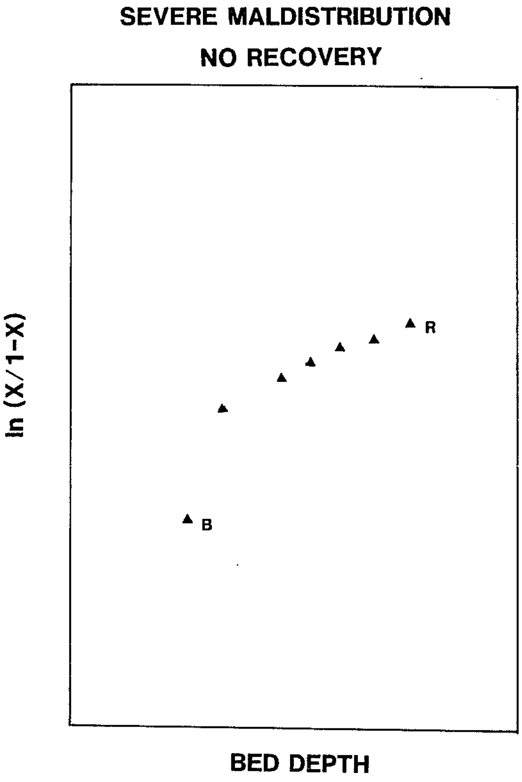

SEVERE MALDISTRIBUTION
NO RECOVERY

Figure 15

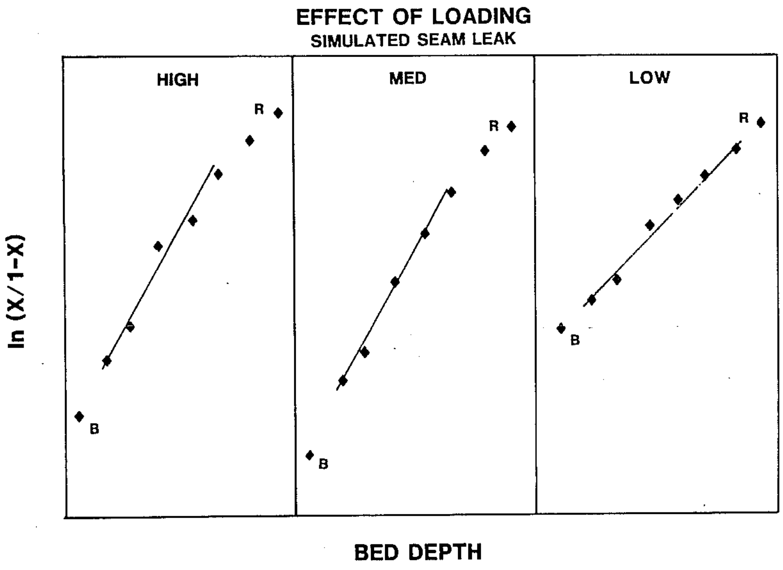


Figure 16

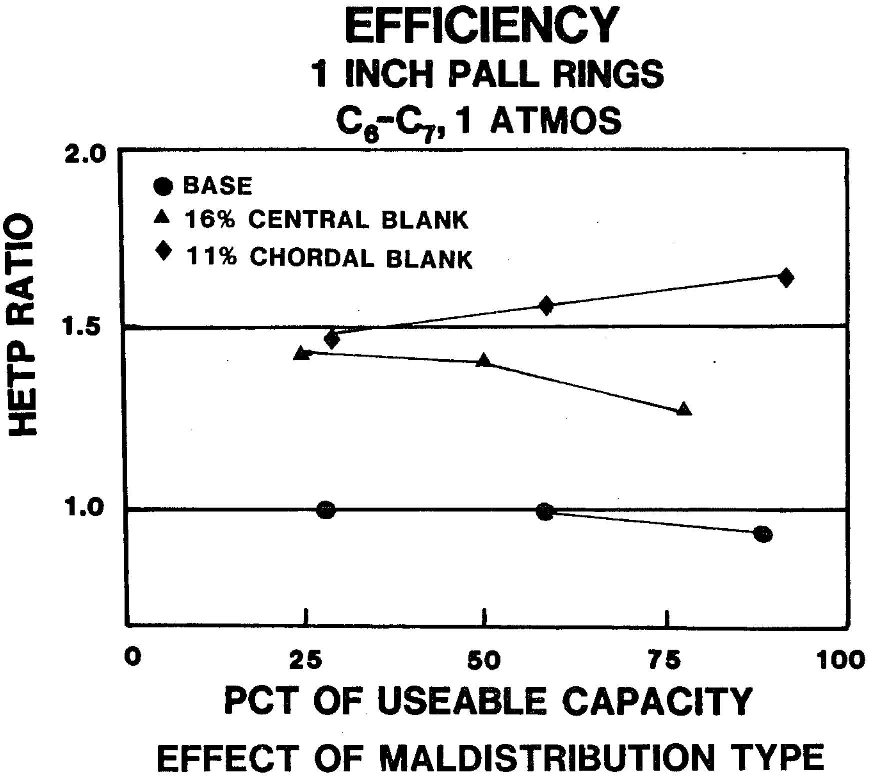


Figure 17

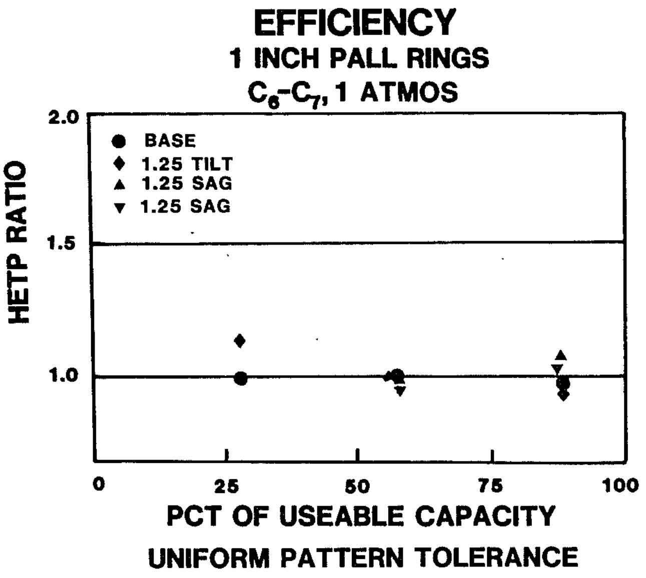


Figure 18

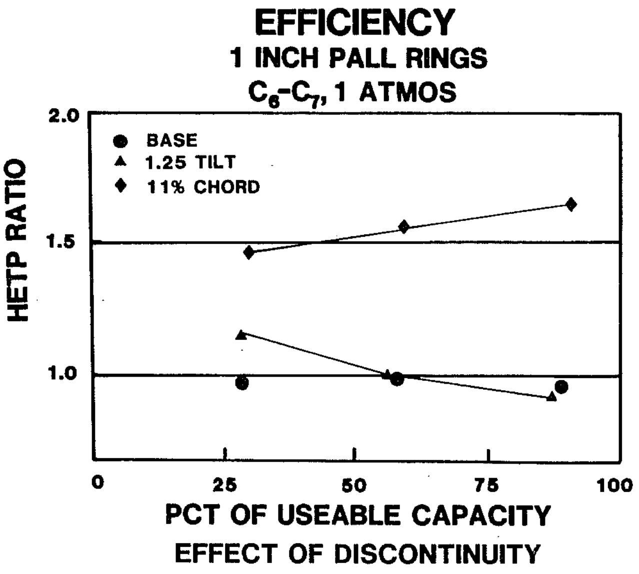


Figure 19

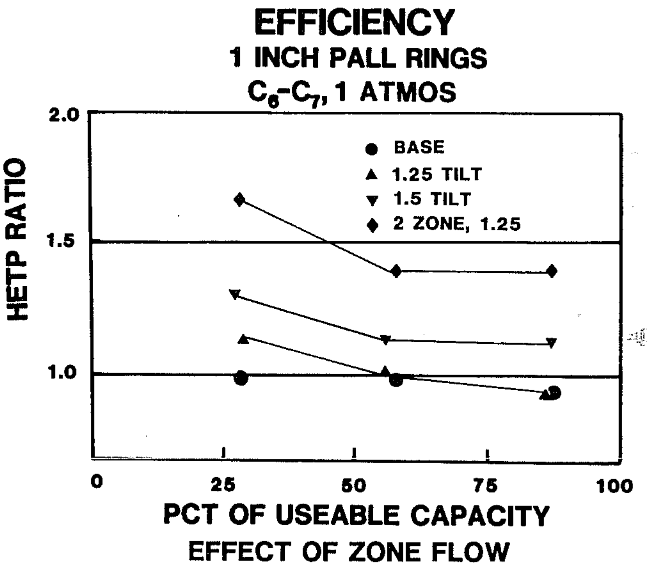


Figure 20

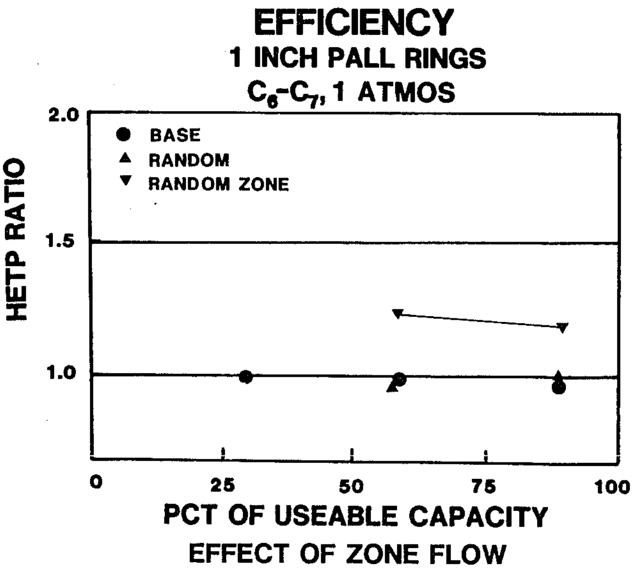


Figure 21