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MASTER

PROGRESS REPORT NO. 16

covering the specific topic of

COMPARATIVE PERFORMANCE OF EXPANDED-METAL
PLATES IN A PULSE COLUMN EXTRACTOR

Contract AT-(40-1)-1320
The Performance of Contactors for
Liquid-Liquid Extraction

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DEPARTMENT OF ENGINEERING RESEARCH
NORTH CAROLINA STATE COLLEGE
RALEIGH, NORTH CAROLINA

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August 3, 1956

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COMPARATIVE PERFORMANCE OF EXPANDED-METAL
PLATES IN A PULSE COLUMN EXTRACTOR

PROJECT SUMMARY

This report covers work on an auxiliary aspect of the pulse column project of Contract AT-(40-1)-1320. It was organized as a thesis program for Mr. J. C. Park, in partial fulfillment of the Master of Science Degree in Chemical Engineering. As a thesis, it represented a cooperative effort between three groups. The Graduate School provided a review and control through a Committee, the Department of Chemical Engineering furnished a research assistantship for Mr. Park, and the research project furnished its resources and personnel under the direction of Dr. F. P. Pike.

The accomplishments of the work are adequately described in the thesis, which is the main body of this report.

From the point of view of the project, two deductions concerning future work are pertinent. The work should be extended to provide a comparison utilizing rate of mass transfer data. The procedures intended to characterize plate resistance offer some promise of providing a relatively simple procedure for testing plate designs outside of a pulse column.

COMPARATIVE PERFORMANCE OF EXPANDED-METAL PLATES IN A
PULSE COLUMN EXTRACTOR

by

Jong Chul Park

A thesis submitted to the Faculty of the North Carolina State College
in partial fulfillment of the requirements for the degree of Master
of Science in the Department of Chemical Engineering.

Raleigh

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I wish to express my sincere appreciation to Dr. E. M. Schoenborn, my thesis advisor, and to Dr. F. P. Pike, Senior Investigator for the A. E. C. project, for their guidance in the experimental work and their help with the thesis preparation.

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4. SUMMARY

A new type of plate construction has been tested, with promising results, for its performance in a pulse column extractor, in comparison with a conventional perforated plate design. The new plates were made of expanded metal, a commercial product which serves particularly to give a side-component to the velocity of the liquid, hence a new flow pattern between plates. In addition, they give more free space along the path of flow, per unit of plane area, greater stiffness, and other physical differences. Refer to Figures 4 and 5, page 18 and 19 for a comparison of the appearance of the expanded-metal and perforated plates employed.

The expanded-metal plates were designated by the symbol EX and constructed of material specified as 1/8" x 22 gage AISI 302 x 5/64". The comparison perforated plates (P-25) were punched with round 1/8" holes in a triangular pattern providing 25 percent free space. Measurements on plates P-25 and EX are presented in Tables 2 and 3. The pulse column consisted of a 1.92" i.d. Pyrex column containing 23 plates spaced 2.00" apart. It was operated at room temperature on carefully purified benzene-water, with the benzene dispersed. Special care was taken to reduce and control the trace impurities to the minimum levels. The reproducibility obtained implied success in these efforts.

For all the runs the flow rates of the benzene and water phases were set equal to each other, a procedure which facilitated the runs and the calculations, and reduced the effort required. It was assumed that the comparative performance of the two plates would be roughly the same at all flow ratios.

The major performance factors measured were C , the flooding capacity (efficiency) and ΔP , the average pressure drop, as functions of π_D , the average pulsed flow past the plates. See the text for the specific definitions of these terms. Additional observations were made on holdup, and on the flow patterns and column appearance. Pulse frequencies were varied from 20 to 80 cycles per minutes, pulse amplitude from 0.10 to 2.0". For expanded-metal plates **EX**, flooding capacity was found to decrease as expected with increasing π_D , yet to exhibit two Regions, I and II, characterized by an abrupt transition. In Region I, where the pulse action was relatively slow, it was found necessary to correct the flooding capacities and the pulsing flow rates for the amplitude equivalent of the plates, about 0.07". The corrected values were denoted as C' and π_D' .

For the flooding studies, it was found to be immaterial whether the plates were arranged in alternate or the same directions, or whether the punched side was up or down. In Region I, some flooding capacities were obtained greater than the ideal value of 100 percent, as a consequence of a burst of flow that often persisted in time past the forward pulse stroke. Plots of the logarithm of the corrected flooding capacity, C' , gave straight lines against the logarithm of π_D' , the pulsed flow rate corrected for plate amplitude equivalent, in both Regions I and II. In Region I, the results varied from $C' = 1.293$ at $\pi_D' = 40.7$ ft/hr to $C' = 0.585$ and 0.596 at $\pi_D' = 292$. The transition was calculated to occur at $C' = 0.572$ for $\pi_D' = 304.3$. In Region II, the results varied from $C' = 0.377$ at $\pi_D' = 389$ to $C' = 0.038$ at $\pi_D' = 1210$ ft/hr. Pulse column operation is often in the region $\pi_D' = 400$ to 600 ft/hr.

For the pressure drop studies, the same as for the flooding studies, it was found to be immaterial whether the plates were arranged in alternate

or the same directions, or whether the punched side was up or down. For the two-phase operation, the average dynamic pressure drop in inches of water, ΔP_{avg} , across the 23 plates both for the upstroke and the downstroke, was found to be uniquely a function of π , ft/hr, according to the equation:

$$\Delta P_{avg} = 6.733 \times 10^{-5} \pi^{1.827}$$

Measurements were made from $\Delta P = 0.67$ to 13.90 inches of water, and π from 209 to 1045 ft/hr. Some of the data could be interpreted as indicating a transition to streamline flow when the flow approached the punched side of the plate at π_D values below about 300. However, the indications were by no means conclusive.

The holdup measurements and the visual and photographic studies showed clearly that the flow patterns between the plates were different between cases where the plates were arranged in alternated or the same directions. The observations were reproducible, but the pattern of behavior was obscure. The results appeared to be of value in implying that differences might exist between the two arrangements, in the case of mass transfer.

The comparison between the performance of the expanded-metal plates and the conventional perforated plates (from previous work) was made on two bases, average pressure drop and an arbitrary power function, the product of the average pulsed flow rate by the average pressure drop. In both cases, the expanded metal plates were superior in permitting higher flooding capacities. This indicates that for a given power input, expanded metal plates would require pulse columns of smaller diameter.

No measurements were made on the rate of mass transfer, the per-

formance factor which controls column height.

A number of attempts were made to characterize the resistance of plates, under unpulsed circumstances, so as to indicate their behavior in a pulse column. It was concluded that single and dual plates, unpulsed, do not act like a bundle of plates when pulsed. Therefore, plates to be assessed must be tested under actual pulse-column conditions.

5. STATEMENT OF PROBLEM

The purpose of the thesis program was to explore the comparative behavior of plates made of expanded metal, in substitution for conventional perforated plates in an existing liquid-liquid pulse-column extractor operating on the benzene-water system.

The performance factors presumed to be of major importance were flooding capacity, pressure drop and rate of mass-transfer. However, rate of mass-transfer data were excluded as being too difficult for this program.

6. BACKGROUND

6.1 General

The increased importance in recent years of liquid-liquid extraction, as a tool for effecting separations, has focused attention on refinements in operating procedures and in equipment details. Extraction equipment of commercial importance for continuous counter-current operation may be classified into two competitive types, the mixer-settler type, and the column type. There are other types, but they are of less industrial importance. The column type, of which there are many variations, may be itself grouped into agitated and non-agitated variants. Agitation is the key to better performance, so where separations are relatively difficult, agitated column units are preferred. The pulse column is a pulse-agitated, perforated-plate, continuous, counter-current extraction column. The basic patent for the pulse column was taken out by Van Dijck (17) in 1935. Incidentally, that patent describes the pulsation of the plates themselves, not the fluids, as is the general practice. The first substantial description (and naming) of the pulse column and its operation was provided by Burns, Groot and Slansky (2) in 1949 in a classified report. In 1954, Sege and Woodfield (13) provided in the open literature a number of details on the operation of a pulse column. By now the pulse column is established as one of the more effective liquid-liquid contacting devices. Yet, improvement in pulse column details are worth seeking.

6.2 Functions of the Plates

Since the work of Sherwood, Evans and Longcor (15) in 1939, it has been known that for drops, the regions of formation and of collapse often

contribute more to the extraction process than does the region of free fall (or rise), particularly for short columns. A number of workers have since confirmed and expanded this find. A natural deduction from this fact is that an effective extraction column could be made by providing for frequent drop-formation and drop-collapse, at the cost of the free-fall contribution. This the pulse column does, and is the reason for its effectiveness.

The idealized behavior of the dispersing action at the plates of a pulse column is presented in Figure 1.

Figure 1

Idealized Picture of Dispersing Action at Plates

In practice the action is rarely as clear-cut and simple as that pictured. The jets start straight but quickly acquire a wavy path several times as wide as the formation sites. As the jets elongate they disintegrate with the formation of a spectrum of sizes of droplets. Some of these droplets are somewhat smaller than the perforations in the plates,

hence stand a chance of passing the next plate without coalescence. Some of the droplets are smaller still, and rise so slowly that they are retained between two plates for several pulse cycles. Some droplets are so tiny that their motion is primarily Brownian, and they are swept back by the continuous phase flow. Consequently, each burst of droplets produces a wide variety of droplet sizes and velocities, some backward.

In regard to coalescence, this takes time, usually more time than allowed for completion. The larger droplets seem to coalesce faster than the smaller ones. Often, the coalescing layer underneath a plate coalesces fairly completely right at the plate in the allotted time, but less so at increasing distance away. The bottom layers of the coalescing droplets are almost invariably emulsions of fine droplets, sometimes containing a large fraction of continuous phase. As a consequence, the pulse stroke generates jets that are in part emulsions to begin with, particularly toward the end of the stroke.

We see that the functions of a plate in a pulse column are essentially two: (1) to form a barrier of sufficient resistance to arrest, collect and at least partially coalesce a good portion of the droplets, and (2) to serve with the pulse action to disperse suitably the collected material into droplets. The geometry of the plate and its openings, plus its wettability, must be such as to facilitate these functions.

If the perforations are too small, the result will be excessive pressure drop, and a dispersion containing a preponderance of droplets too small. In turn, the too-fine dispersion causes both excessive entrainment and too slow a coalescence. In extreme cases, emulsification results. If the perforations are too large, the plates are not a sufficient barrier to lead to the essential coalescence and reformation. In

that case, the rate of mass transfer drops severely.

If the perforations are too far apart, the percent free area is too low and low throughput capacity results. If the perforations are too close, the jets coming from them interfere and merge, reducing the degree of dispersion and the rate of mass transfer.

The controlling spacing between perforations is basically the spacing between an opening and its nearest neighbor. This fact tends to impose somewhat of an equilateral triangular pattern to the openings.

The choices for plate geometry thus represent balances of opposing influences, leading to certain ranges of general suitability. For instance, for plates perforated with round holes in a triangular pattern, the spacings generally provide between 10 and 25 percent free area. The hole diameters in practice range from 1/16 to 1/4 inch, with 1/8 and 3/16 inch quite common. With these ranges of geometry, the pulse frequency is usually within 20 to 80 cycles/minute, and the plate spacings from 0.5 to 3.0 inches.

6.3 Plate Variations

It is instructive to consider the plate variants that have been used so far in pulse or similar columns.

<u>Plate</u>	<u>Reference</u>
Perforated plates*	Many
Countersunk holes	Li (7)
Dished-hole plates	Mayfield and Church (8)
Expanded metal	Thornton (16)
Teflon-stainless perforated plate	Pike (10)
Polyethylene perforated plates	Werning (18)
Fluorothene perforated plates	Sege and Woodfield (13)

*Traditionally, with round holes in a triangular pattern, and of stainless steel.

Some further plate variants that might be used for pulsed liquid-liquid extraction are as follows:

<u>Plate</u>	<u>Reference</u>
Screen	Laird (6)
Slotted (Turbogrid)*	Shell Dev. Co. (14)

At the beginning of this work, we were unaware of the report of Thornton (16) on expanded-metal plates for liquid-liquid work. The use of expanded-metal plates for liquid-liquid contact is a logical extension of its recent usage for vapor-liquid contacting, under the designation "Spraypak", as reported by McWilliams (9). Another tower packing quite similar to Spraypak, and also successful for distillation use, is "Panapak", described by Scofield (12). Panapak does not, however, appear to be adapted to liquid-liquid contacting.

Samples of expanded metal were obtained and inspected in careful detail. By visual observations, it appeared that they gave promise of providing a novel and perhaps superior variation in plate construction.

6.4 Coalescing Aids

As expressed in Section 6.2, a popular concept is that coalescence aids mass transfer, perhaps by mechanically mixing material to achieve the same results as the slower diffusion process. Therefore, consideration is sometimes given to improving coalescence as a means of improving pulse column efficiency. Certain operating techniques are favorable. Mechanical devices and techniques may be employed. Cannon (3) speaks of screens being placed between plates to aid in coalescing. Pike, Withers and

*Not yet reported for liquid-liquid service.

Beatty (11) demonstrated that drops coalesce neatly when impaled on spikes. With glass laboratory columns, internal swirls are often observed. It is reasonable to suppose that different mechanical arrangements will provide different swirls and different coalescence. In fact, for large diameter columns, Woodfield and Sege (20) showed that added "swirl" plates did in fact control internal flow patterns, and overcame channelling. Perhaps expanded-metal plates would provide a novel mechanical action leading to improved coalescence.

6.5 Influence of Impurities

It was found by Pike (10) and by Edwards and Beyer (4) that trace impurities profoundly influence the flooding capacity of a pulse column. Beyer and Edwards ascribed the effect to differences induced in plate wettability. That plate wettability is an important factor has been reported by Sege and Woodfield (13) for pulse columns, and for unpulsed columns by Garner et al (5). However, it could also be that the impurities affect the interfacial tension of the droplets and through the interfacial tension, the coalescence rate and thus the flooding capacity.

The important point is that trace impurities can seriously affect all results; and, hence, should be controlled during the experimentation.

6.6 Performance Factors

The major performance factors of a pulse column are three: flooding capacity, specific rate of mass transfer, and pressure drop. For a given system, these three factors control, respectively, the diameter and height of the column, and (in part) the power requirements for the pulsing action.

Performance factors of less importance are entrainment, backmixing,

holdup, end effects, and changes of behavior with length.

In considering possible testing of expanded-metal plates, it was decided that a reasonable evaluation of the new plates could be made on the basis of flooding and pressure-drop studies. Rate of mass transfer data, while desirable, are attained only with a much greater effort and time consumption than was judged warranted at this time.

6.7 Summary

Pulse columns are such effective extraction units that appreciable effort is warranted in seeking improvements in mechanical and operating details. Plate construction is an important mechanical detail that has been the object of appreciable study already. It was speculated that expanded metal could well serve the functions of the plates in a pulse column, and might even be a superior variant. It was decided to attempt an evaluation on the basis of flooding and pressure-drop studies, giving due attention to the disrupting influence of impurities during experimentation.

7. EQUIPMENT AND MATERIALS

7.1 Main Apparatus

The pulse column apparatus employed was an existing one constructed under an Atomic Energy Commission contract AT-(40-1)-1320. The equipment is shown schematically in Figure 2 and is pictured in Figure 3.

The two feed streams, benzene and water, were stored in 55-gallon stainless steel tanks. They were pumped by steady-flow, variable-speed piston pumps to and through the pulse column, then back to their respective feed or other tanks. The lines were stainless steel tubing fitted together with "Koncentrik" tubing fittings which possess a Teflon ring seat. A few valves and other items were of brass and polyethylene. Except for six synthetic rubber gaskets in the pulse column, all liquid contact was with stainless steel, glass, brass, Teflon and polyethylene.

The two aqueous, and one benzene, pumps were Milton-Roy "Constametric" piston pumps controlled by D-C motors and General Electric "Thymotrol" controls. These pumps possess dual pistons so geared that the resultant flow is supposedly constant, without even a sinusoidal character, independent of the discharge pressure. By means of an auxiliary set of gears, the speed range of these pumps was rated to be 58-fold downward from the maximum. To further lower the minimum capacity of the benzene feed pump (which had larger pistons than the water-phase pumps), it was fitted with a variable resistance, up to 100 ohms, in the field circuit of the D-C drive motor. The packings for these pumps was stated to be "pure" Teflon. They were installed practically without the use of lubricant, and were leached by many months of operation.

The pulsing pump was a Milton-Roy "Simplex" pump equipped with a

FIGURE 2
PULSE COLUMN EQUIPMENT LAYOUT

Figure 3
Pulse Column Unit

203 027

special microadjustment of the stroke length permitting the piston stroke to be changed during operation from 0 to 4 inches, equivalent to a range of amplitudes in the 1.92-inch column of 0 to 2.54 inches. By means of the "Thymotrol" control unit, the frequency could be varied from about 7 to about 148 strokes per minute. This pump was also equipped with "pure" Teflon packing. However, interfering substances continually leached slowly out of the packing. Therefore, an isolation chamber was installed between the pump and the column, and fitted with a Kel-F diaphragm.

The pumps possessed the flow capacities indicated in Table 1.

Table 1
Pumping Capacities of the Pumps

<u>Phase Pumped</u>	<u>Pump Capacity Ranges</u>	
	<u>Gallons/hour</u>	<u>Feet/hour^a</u>
Benzene	0.72 ^b - 57.0 ^d	5 - 394
Water	0.56 ^c - 32.5 ^d	8 - 225

^aIn the 1.917 inch diameter column with 0.377 inch diameter spacers

GPH x 6.938 = FPH,
GPH x 0.1337 = CFH,
FPH x 0.0003211 = CFM,
CFM x 3114 = FPH

^bMinimum without irregular action, with added control in motor field.

^cMinimum without irregular action, as rated.

^dRated maximum of pump.

For flow indication, rotameters were used. For flow measurement, a system of calibrated gage glasses was employed. See Figure 3, which shows

two of the three gage glasses for a tank. Each tank was equipped with 3-inch, 1.5-inch, and 0.75-inch internal diameter gage glasses, each about forty-eight inches long. The connecting tubing included a quick-opening valve, so arranged that the flow could be obtained from either the tank or any one of the gage glasses.

Temperatures were obtained by mercury-in-glass thermometers installed in entrance and exit lines of each stream, near the column. The thermometers were graduated to 0.2°C .

7.2 Pulse Column

The pulse column is shown in Figure 4. The primary element was a precision-bore tube of chemical-grade (No. 7720) Pyrex, 1.917 inches in internal diameter and forty-eight inches long. The ends for about 2.5 inches were larger, about 2.00 inches, by reason of their not being shrunk on the mandrel used for the 1.917-inch section. Measurements made at positions throughout the length, and at various orientations, gave diameters from 1.916 to 1.921, but 1.917 seemed the most appropriate single figure for the precision-bore section. The cross-sectional area for $d = 1.917$ inches is 2.886 square inches. When calculating a flow rate in the column, a subtraction was made for the area of the spacers between the plates. With the spacers of 0.377-inch diameter, the subtraction is 0.112 square inches, and the effective area = 2.774 square inches. There were 23 plates spaced 2.00 inches apart.

7.3 Perforated Plates

The three perforated plates and two expanded-metal plates used in this program are pictured in Figure 5. The perforated plates were designated to indicate the approximate percent free space for each plate.

FIGURE 4
COLUMN AND PLATE DETAILS

PERFORATED PLATES

P-10

P-25

P-40

EXPANDED METAL PLATES

EX

EX-R

FIGURE 5
PLATE VARIATIONS TESTED

Plates P-10, P-25 and P-40 had, respectively, 9.8%, 24.6% and 41.9% free space. All were the same thickness, 0.0317 inches, the same hole size of 1/8 inch, and the same alloy, type 316 stainless steel. They differed only in hole spacing, hence percent free space.

The characteristics of plate P-25 are presented in Table 2.

Table 2

Details of Perforated Plate P-25

Plate Diameter = 1.912 inches	Hole Space = 0.244 ± 0.0789
Plate Thickness = 0.0317 ± 0.00122	Percent Free Space = 24.6
Hole Diameter = 0.127 ± 0.0163	Diameter/spacing Ratio = 7.84
Plate Gage = 21.8 U. S. Std.	

7.4 Expanded Metal Plates

7.41 Background

Expanded metal is a common and widely available industrial product known also as expanded mesh and metal lath, listed in those names in Thomas' Register of American Manufacturers. Most of the expanded metal possesses openings from 1/4 to 2 inches, and is used industrially for reinforcing walls and insulation, for partitions and walls, and for landings, stairs, gratings and the like. Smaller sizes are used for decorative grilles and shapes.

Denholme Incorporated, 5711 Boulevard East, West New York, New Jersey, specializes in the smaller designs, in precision work, and will supply relatively small orders. In particular, Denholme specialized in stainless steel, which is almost always the material of construction for pulse column plates.

7.42 Manufacture

Expanded metal (as supplied by Denholme) is precision cut by a

specially-designed vertical press of English development that cuts and stretches one row of holes at a time. Metal lath is made on a different machine. With stainless steels, 20 gage is about the maximum thickness of metal for meshes under $1/4$ inch.

The final form is primarily controlled by three factors: the die, the die setting, and the machine setting. The dies come in graded sizes, such as $1/16$, $1/8$, $3/16$, $1/4$, etc., inch which designate approximately the maximum expansion (see Figure 4, rotated views) of the opening (not the length of the slit). Stainless steel has some tendency to fracture when using the regular dies, so there is a special series of dies for it which provides a slightly longer slit. Incidentally, the gage of the metal influences this expansion somewhat. The die setting may be changed to give various corrugated or ridged effects, or to omit various openings. The machine setting controls the strand width to, say, $1/32$, $3/64$ or $1/16$ inch, hence the meshes per foot in the expanded direction. Depending upon the setting of the machine, the strand width can be varied from 0.010" up to about 0.25" depending upon the size of the die being used and the open area required.

7.43 Nomenclature

Expanded metal (as supplied by Denholme) carries designations such as

$1/8$ " x 0.032 aluminum x $1/16$ "

$1/8$ " Ridged x $2 1/4$ gage steel 0.056"

$3/16$ " Special x $1/32$ " AISI 302 x $3/32$ "

The first part indicates the die used. When a standard die is used

often only the size is listed, such as $1/16$ ", or the designation "regular" or "standard" is added. Special settings of the dies carry designations such as ridged, texmesh, special, etc. Figure 5 shows Plate EX-R, which is ridged. The second part provides the thickness of the sheet material, and the metal designation. Often the thickness is indicated by the gage number. Unfortunately, the various metals and alloys have different gage scales, so a simple gage designation is often ambiguous. The third part provides the strand width. In summary, besides picking the metal, there are four specifications to be placed on expanded metal, the die, the die setting, the metal thickness and the strand width.

7.44 Construction of Plates

It proved to be impossible to construct plates out of expanded metal by means of a lathe. It was necessary to develop a technique using a grinder, at a labor cost of about \$1.50 per plate. The steps were as follows:

- (a) Shear the expanded-metal sheet into squares about one inch wider than the finished plates. See that no slots are crushed that would cross the circumference of the finished plate.
- (b) Orient all expanded-metal squares the same way. Pick a standardized location approximately where the plate center would lie, then drill a hole ($1/8$ "). With the hole as center, scribe a circle with a radius about $1/32$ " greater than the finished plate.
- (c) Cut out circles on a "Do-All", using a fine-toothed blade installed with the teeth backwards. Otherwise, it jerks and tears the unsupported strands of the plate. Follow the circle described in (b).
- (d) Grind one plate at a time to within 0.001 " in a grinder using a jig with a center pin to fit the $1/8$ " center hole of the plate.
- (e) After grinding, each plate-edge has burrs sufficient to spoil a close fit. These burrs are removed by hand filing.

7.45 Summary of Plate Measurements

The EX plates tested in the pulse column were prepared from expanded metal designated as 1/8" x 22 gage AISI 302 x 5/64". Measurements on the expanded metal are presented in Section 9. A summary of the results is given in Table 3. Some EX-R plates were made from stock designated 1/8" Ridged x 22 gage AISI 302 x 3/64".

Table 3

Summary of Measurements of Expanded-Metal EX

Meshes per foot, expanded direction	=	69
Meshes per foot, unexpanded	=	22.5
Angle of opening	=	32.7 $\pm$ 0.7 degrees
Strand Width	=	0.0700 $\pm$ 0.0003 inches
Metal Thickness	=	0.0359 $\pm$ 0.0002 inches
Metal Thickness	=	20.5 U.S. Std. Gage
Specific Weight	=	0.5845 $\pm$ 0.0052 gm/cm ²
Percent Free Area, Maximum	=	41.9 $\pm$ 2.3 percent
Degree of Expansion	=	1.24
Plate Designation	=	1/8" x 22 Gage AISI 302 x 5/64"

7.5 Chemicals

The water was a good grade of distilled water prepared from a Barnstead still, and transported in stainless-steel drums specially cleaned and used only for that purpose.

The benzene was purchased as thiophene-free, 1° benzene from the Barrett Division of Allied Chemical and Dye Corporation. By the time of this program, it had been used several times in the pulse column. Therefore, it had been washed thoroughly with distilled water, and redistilled several times to remove water and adventitious material.

7.6 Auxiliary Details

7.61 Connecting Parts

As is customary, and to reduce entrainment, the column proper was connected with standard Corning flanges to top and bottom bell-shaped end sections, as shown in Figure 4. In this case, stainless-steel collars (Figure 7) serving as pressure taps were installed between the column proper and the end sections. Above the top end sections were additional sections which by their size insured that the pressure fluctuations above the top tap would be minimized, whether arising from flow resistance or from fluctuations in liquid level due to the pulsating volume (see Figure 4).

Gaskets were necessary at various connections of the column installation. There were six in all, made from 1/8" "Ameripol" rubber, a solvent-resistant synthetic. These gaskets had been exposed for several years to the leaching action of benzene and water. Even so, they were suspect as sources of contamination. However, the area exposed was small, and no leaching from installed gaskets could be detected spectrophotometrically.

The inlet distributors introduced feed on the axis of the column. Since the ends were capped, a distributor blocked the flow impact of the stream and introduced it radially from the distributor end..

7.62 Plate Installation

The 23 plates were strung on a stainless-steel 1/8" (1/4" for perforated plates) center rod of the same length as the column. The spacing between the plates was maintained by the use of 2.00-inch lengths of 0.377" od stainless-steel tubing. The spacers should have fitted the center rod snugly, for otherwise the unevenness of the plate was expressed

by the plates not being level by differing degrees. To counter this tendency, the space between the center rod and spacer was filled with a wrapping of 0.01" thick Teflon strip. By tightening the nuts on the tie-rod ends, the plates and spacers could be readily squeezed enough to cause the plate assembly to bind against the column wall with a holding power sufficient to withstand the pulsing action.

It was noted that the apertures of the expanded-metal plates possessed an angle of inclination from the plate, and that one side of the plate showed punch marks from the dies. Based upon the direction of the plate apertures, the plates were arranged in approximately two different ways, (1) plates in alternate (180°) direction, punched side down, and (2) plates all in the same direction, punched side down. The idealized arrangements are illustrated in Figure 6.

Figure 6

Expanded-Metal Plate Arrangements

In practice, it was impossible to exactly attain the idealized arrangements because the plates had a tendency to turn as they were being inserted into the column.

7.63 Pressure-Measuring Devices

The dynamic pressure drop across the plates of the pulse column was measured by equipment and technique carefully developed previously on the pulse column project. Pressure waves were recorded at locations across the plates, then subtracted to give the dynamic pressure drop. This procedure measures pressure effects due to acceleration and friction between the tap locations.

At each end of the column, as illustrated in Figure 4, were installed stainless-steel collars drilled and tapped to receive a "Pressuregraph" pickup, which is a pressure-detecting element. The collars are illustrated in Figure 7.

The "Pressuregraph" was an Electro-Products Laboratories, Model 3700-C unit, consisting of a cabinet, Model 3709-D pick-ups, standard cables, and a Standard Mounting Base, Model 3704-B, for 115/220 volts, 50/60 cycles. Additional equipment consisted of one Model 30808 Sola Constant Voltage Transformer, 500 VA, one Model 304-A DuMont Cathode Ray Oscillograph, one EXAKTA VX 35 mm Camera, one Model 3710 8-position Pickup switch, supplementary pickups, cables and copper-beryllium diaphragms of 0.008" thickness.

Several important modifications were made in the Pressuregraph circuit. A 50-microfarad condenser was placed across the oscilloscope input, to ground. This change served to filter out nearly all of the extraneous vibrations, or "noise", without altering the pressure signals. A second change was the addition of an external variable condenser, 1 to 30

FIGURE 7
COLLAR WITH PRESSURE TAP

microfarads, across the external terminals of the horizontal sweep adjustment circuit. This served to permit the recording of the traces of complete pressure cycles for the low frequencies encountered, around 0.2-2 cycles per second. A 7500-ohm potentiometer was installed in series with the vernier horizontal sweep control, to permit a finer adjustment. An auxiliary vacuum-tube voltmeter, set for 0.5 volts ranges, served to aid one of the adjustments.

A microswitch was carefully positioned on the stroke arm of the pulse pump in such a manner as to close precisely at the far back position of the pulse piston. This contact switch was wired into the output circuit of the Pressuregraph in such a way as to break the circuit at the back end of the pump stroke. As a result, a "pip" showed on the oscilloscope trace, marking the beginning of each stroke, permitting the pressure-wave image to be timed.

A view of the main Pressuregraph equipment is shown in Figure 8. The pickup assembly is shown in Figure 9.

The Pressuregraph detects a pressure wave through the deflection of the thin (0.008") metallic diaphragm. This deflection varies slightly the thickness of an air gap between the diaphragm and a solid core in the pick-up, changing the inductance and capacitance of the system. These changes reflect instantly in the signal from the pickup, which is visible as a trace of a wave on the oscilloscope screen. The camera records an entire wave from the single trace. The photographic evidence of a pressure wave is calibrated against known pressures placed on the column taps. Unfortunately, frequent calibrations were necessary.

203 041

Figure 8

Main Pressuregraph Equipment

203 042

Figure 9
Pressuregraph Pickup

7.64 Photographic Devices

The camera used in the photographic studies was an EXAKTA VX 35 mm camera with a Zeiss Tessar f:2.8 coated 50 mm lens, and extension tube. A shutter time of 1/250 second was employed at a lens opening of about f:2.8. The camera was placed about six inches from the column wall, which was floodlighted from two sides by G.E. No. 1 photofloods. Eastman Kodak Tri-X film was used.

7.7 Provisions for Safety

Benzene is a toxic chemical for which the recommended maximum, average, daily, 8-hour concentration is 35 parts per million, as reported by the American Industrial Hygiene Association (1). Fortunately, this limit is above the threshold odor limit of about 20 ppm, as determined by Pike (10). Benzene is also an explosive hazard, with an explosive range of about 1.4-7.1 volume percent.

As part of a safety program, the room containing the pulse column unit was ventilated with two large explosion-proof ventilating fans, one exhausting to the outside. The pump motors, electric-lines and switches were explosion-proof. Adequate warning signs and indoctrination were employed. At intervals, blood tests were made on research personnel to provide a means of detecting possible benzene poisoning.

8. TESTS WITH INDIVIDUAL PLATES, UNPULSED

8.1 Purpose

Aside from experience, there is as yet no rational basis for choice of the dimensions of the plate openings. Thus, it was a problem to select the expanded-metal plate most suitable for comparison with a typical perforated plate. This section of work was undertaken to explore the factors involved in plate choice, to search for a simple test for plate suitability, and to select a specific expanded-metal plate for pulse column tests.

8.2 Cleaning Procedure

Assuming that plate wettability was a major factor, a great deal of attention was paid to cleaning procedures. At first the plates and parts were washed in hot Alconox solution, then carefully rinsed in tap and distilled water. Yet the test results were erratic. It was thought that more stringent cleaning procedures were necessary, but this might not have been true.

The final cleaning procedure was as follows: The parts were washed in boiling Alconox solution, then flushed with water and transferred to cleaning solution (H_2SO_4 - $\text{Na}_2\text{Cr}_2\text{O}_7$) for prolonged soaking, usually overnight. After that, the plates were flushed in running tap water for hours, then with distilled water. Often, the plates were stored under benzene, although this factor showed no influence. In the handling of the plates, contact with fingers and other objects was carefully avoided.

8.3 Preliminary Tests

8.31 Basis

It was assumed that the only factor of importance was the resistance caused by interfacial tension. This resistance was to be measured by the hydrostatic head of benzene necessary to overcome it. Dynamic effects were ignored.

8.32 Apparatus

The preliminary testing apparatus is shown in Figure 10. The test column consisted of two five-inch lengths of two-inch internal-diameter Pyrex pipe connected by Corning aluminum flanges. Test plates were installed between the pipe sections, using Ameripol synthetic rubber gaskets cut to the pipe size.

Figure 10

Preliminary Plate Testing Apparatus

The gaskets had previously been extracted with benzene. These gaskets were of the same material as those used in the pulse column, where

experience indicated no complications due to the gasket extractables.

8.33 Procedure and Observations

At first the test column was installed entirely submerged. Benzene was introduced slowly about four inches underneath the test plate. To our surprise, the rising benzene passed through the plates with hardly a hesitation. A number of typical perforated plates, as well as the expanded metal plate, gave the same results. Included was a plate with 1/8 inch holes, 24.6 percent free space (P-25). This plate was known to perform well in a pulse column, in which it stopped the upward flow of benzene quite effectively during pulsing cycles.

When the test column was raised so that its top was three inches above the water surface, the test plates started to function properly. In this case, the liquid in the column was isolated from the main body, except through the column bottom. As benzene was introduced slowly, a little benzene passed the plate, then started to accumulate underneath the plate. A measurable layer could be built up before more benzene passed the plate. The impact of the rising benzene appeared at times to drive some benzene through the plate. However, several different types of nozzles and their arrangement did not noticeably influence the behavior. Later, some brief tests indicated that the depth of immersion of the plate might be a factor. If so, this would appear to involve the inertia of liquid above a plate.

8.34 Deductions

This simple test demonstrated that more was involved than merely the play of interfacial forces. Only when a back hydrostatic

pressure was built up above a plate did it begin to function. Also, it appeared that the kinetic energy of the drops as they rose and struck the plate, and the inertia of the liquid above the plate, were factors.

8.4 Tests on Single Plates

8.41 Apparatus

Based upon the findings of Section 8.3, an apparatus was constructed as shown in Figure 11 and photographed in Figure 12. (However, in Figure 12, the test column is shown with a two-plate installation, instead of a single-plate unit.) The central item was the glass column made of two sections of two-inch i.d., five-inch long Pyrex pipe sections joined together by Corning aluminum flange connections and 1/8" Ameripol rubber gaskets. In contrast to the installation of Section 8.3, this test column was suspended so that the top was above the water level, with the plates about two inches below the surface.

The water level was initially set at the point of incipient overflow through a one-half inch diameter side hole about 13 inches above the bottom. When benzene was fed to the interior of the test column, all of it was trapped therein. But the water level was maintained constant by the overflow feature.

The Ameripol gasket was previously well extracted. The neoprene connections on the glass tubing were made with the barest exposure of rubber. The use of the Teflon stopcock avoided the need for a stopcock lubricant. Great care was taken to avoid dirt, grease and other contamination.

8.42 Procedure

Benzene was fed slowly underneath the plate. In the beginning

FIGURE 11
EQUIPMENT FOR TESTING PLATES , UNPULSED

Figure 12

Plate-Testing Equipment

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benzene passed rather freely through the plate. Soon, however, enough collected underneath the plate to block the perforations. When the bottom layer was continuous across the column, a fairly reproducible behavior began.

As more benzene was added slowly and in increments, a hydrostatic pressure built up, tending to drive benzene through the perforations. Readings were taken of the layer depth after each addition. Finally, an increment of benzene to the underneath layer exceeded the equilibrium amount. Benzene then was driven through the perforations, passing a volume much larger than the last increment. When the burst of flow subsided, the increment passed was measured, then added by calculation to the underneath layer to give the depth of layer there at the point of breakthrough. Similarly the top layer depth was calculated back to the point of breakthrough.

8.43 Mathematical Treatment

At the moment before breakthrough, a pressure equilibrium existed across the plate, with a situation as depicted in Figure 13.

Figure 13

Illustration of Layers, Single Plate

Let X_1 = thickness of upper benzene layer, above water datum at breakthrough, cm.

X_w = thickness of upper benzene layer below water datum, cm.

X_2 = Thickness of lower benzene layer at breakthrough, cm.

ρ_w = density of water, gm/cm³.

ρ_b = density of benzene, gm/cm³.

$\Delta\rho$ = $\rho_w - \rho_b$.

C = resistance of plate, gm/cm².

At pressure equilibrium, in units of gm/cm², $X_1 \rho_b + C = (X_2 + X_w)$

$(\rho_w - \rho_b) = (X_2 + X_w)(\Delta\rho)$. In practice X_w could be positive, negative or zero. It appeared to be close enough to zero to neglect. In that case

$$X_1 \rho_b + C = X_2(\rho_w - \rho_b) ; X_1 \rho_b + C = X_2 (\Delta\rho)$$

Rearranging,

$$X_2 = \frac{\rho_b}{\Delta\rho} X_1 + \left(\frac{C}{\Delta\rho} \right)$$

As illustrated in Figure 14, a plot of equilibrium values of X_2 against X_1 ($X_w = 0$), should allow a determination of the plate resistance, C .

The slope $\left(\frac{\rho_b}{\Delta\rho} \right)$ is fixed by measurable properties.

Figure 14

Mathematical Illustration

8.44 Theoretical Plate Resistance

The theoretical value of C for static conditions may be derived from the equation for the maximum bubble-pressure method for measuring surface tension, assuming that the derivation applies to the liquid-liquid case. The details of this method are explained by W. D. Harkins in the treatise by Weissberger (19).

The equation given is as follows

$$\sigma = \frac{\bar{p} r}{2} \left[1 - \frac{2}{3} \frac{r}{h} - \frac{1}{6} \left(\frac{r}{h} \right)^2 \dots \right]$$

where r/h is small compared to 1.0,

and σ = surface or interfacial tension, dynes/cm

r = radius of opening, cm

$$h = \frac{\bar{p}}{(\Delta\rho)(g)}$$

$\bar{p}$ = maximum pressure difference, dynes/cm².

$\Delta\rho$ = density difference for the two phases,

In our case, $\bar{p} = Cg$, hence

$$h = \frac{\bar{p}}{\Delta\rho g} = \frac{C}{\Delta\rho}$$

Therefore,

$$\sigma = \frac{C g r}{2} \left[1 - \frac{2}{3} \left(\frac{r \Delta\rho}{C} \right) - \frac{1}{6} \left(\frac{r \Delta\rho}{C} \right)^2 \dots \right]$$

For 20°C, for benzene-water and 1/8" holes

$$\sigma = 34.96 \text{ dynes/cm}$$

$$r = 1/16" = 0.1587 \text{ cm}$$

$$\Delta\rho = 0.1181 \text{ gm/cm}^3$$

Substituting the above numerical values, and solving,

$$C = 0.459 \text{ gm/cm}^2.$$

8.45 Results and Observations

In attempts to measure the plate resistance, C , measurements were made on perforated plates P-10, P-25 and P-40, and expanded metal plates EX and EX-R. These data are presented in Figures 15, 16, 17, 18 and 19.

Particularly in the case of the perforated plates, it was observed that the results scattered rather badly, both within runs and from run to run. For a time, this random behavior was attributed to differences in plate wettability reflecting different degrees of cleanliness. Efforts to minimize contamination and to standardize the procedure were doubled and redoubled, to no effect; the onset of breakthrough always showed more or less of a random behavior. Furthermore, breakthrough always occurred at a single location, or perhaps two locations, during a run, and tended strongly to recur at the same locations during that run.

Inspection of Figure 15, for example, shows that the results are quite sensitive to values of X_1 . An error in ± 1 mm in measuring X_1 would account for the spread in results. Referring to Section 8.43 and the equations there, such a variation might have arisen by X_w differing from zero and varying by ± 1 mm during runs. It was difficult to observe X_w with our technique. Also, the depth of immersion of the plate was not controlled. Later information indicated that this may have been an important factor. See Section 8.33.

For each reading of each run on each plate, a value was calculated for the plate resistance, C . Run averages of these individual values are

FIGURE 15
TEST ON SINGLE, UNPULSED PLATE P-10

2.0 34

FIGURE 16
TEST ON SINGLE, UNPULSED PLATE P-25

FIGURE 17
TEST ON SINGLE, UNPULSED PLATE P-40

FIGURE 18
TEST ON SINGLE, UNPULSED PLATE - EX

FIGURE 19
TEST ON SINGLE, UNPULSED PLATE EX-R

given in Table 4, leading to a single average value for C for each plate.

Table 4
Plate Resistances - Single, Unpulsed Plates

<u>P-10</u>	<u>P-25</u>	<u>P-40</u>	<u>EX</u>	<u>EX-R</u>
0.204	0.250	0.086	0.330	0.280
0.138	0.332	0.176	0.359	0.260
0.138	0.295	0.209	0.346	0.298
0.223	0.309	0.175		0.263
0.247	0.313	0.163	Avg = 0.345	
	0.296	0.132	$\sigma = 0.015$	Avg = 0.275
Avg = 0.190	0.336	0.302		$\sigma = 0.018$
$\sigma = 0.050$				
	Avg = 0.304	Avg = 0.178		
	$\sigma = 0.029$	$\sigma = 0.067$		

σ = Standard deviation

The resistances are in units of $C = \text{gm/cm}^2$.

On Figure 20 are plotted the results of Table 4, where C for plate EX is plotted against the percent free space measured in the direction of flow (see Section 9.7). The ranges of values calculated for 95 percent confidence are also shown.

8.46 Deductions

The results exhibited in Figure 20 are difficult to interpret. Perhaps the flaws in technique, or the extreme sensitivity to X_1 values, might have combined to give spurious results. Yet the reproducibility of the data seemed sufficient to demonstrate the broad features of a behavior pattern.

The theoretical derivation of Section 8.44 gave $C = 0.46 \text{ gm/cm}^2$, a value much above the observed results. This is understandable if it is

FIGURE 20
AVERAGE PLATE RESISTANCES
FOR SINGLE PLATE TESTS

assumed that kinetic effects, the impact of the drops, the inertia of the fluid above the plate, are important. Kinetic effects would reduce the observed C values to less than the equilibrium value.

The maximum in the curve can, so far, only be interpreted as the result of pressure wave interactions and resonance.

Definite conclusions were as follows:

1. The curve of Figure 20 probably represents a real behavior.
2. The change in technique consisting of raising the test column until its top was above the water level caused a startling and major change in the performance of the plates.
3. The two expanded metal plates, EX and EX-R, seemed to perform in about the same manner as the perforated plates.
4. The test technique should be extended to include tests of two plates acting together.

8.5 Tests on Two Plates

8.51 Apparatus

In view of the anomalous results of Section 8.4 in measuring the resistance of single plates, it was decided to test for plate interactions by testing the resistance of a two-plate installation. The apparatus of Section 8.4 was modified in the detail of the test column, which became a three-section column as depicted in Figure 21. The photograph of Figure 12 illustrates the two-plate installation.

8.52 Procedure

The procedure was so similar to that for the single-plate installation (Section 8.42) that no detailed description is necessary. As before, benzene increments were added and measured until breakthrough occurred, then the layer depths were calculated to the conditions before

Figure 21

Two-Plate Installation

breakthrough.

8.53 Mathematical Treatment

At the moment before breakthrough, the equilibrium conditions are shown in Figure 22.

Figure 22

Illustration of Layers, Two Plates

Let X_1 = thickness of top benzene layer at breakthrough, cm.

X_2 = thickness of benzene layer under top plate at breakthrough, cm.

X_3 = thickness of benzene layer under bottom plate at breakthrough, cm.

X_w = thickness of a water layer as indicated, cm.

ρ_w = density of water gm/cm³.

ρ_b = density of benzene, gm/cm³.

$\Delta\rho$ = $\rho_w - \rho_b$

C = resistance of one plate, cm/cm².

At pressure equilibrium, in units of gm/cm²,

$$(X_1 + X_2 + X_3)(\rho_b) + X_w \rho_w + 2C = (X_2 + X_3)(\rho_w)$$

$$X_1 \rho_b + X_w \rho_w + 2C = (X_2 + X_3)(\Delta\rho)$$

Rearranging,

$$X_3 = \left(\frac{\rho_b}{\Delta\rho} \right) X_1 + [2C + X_w \rho_w - X_2 \Delta\rho] \left(\frac{1}{\Delta\rho} \right)$$

As illustrated in Figure 23, a plot of equilibrium values of X_3 against X_1 , using a measured $\left(\frac{\rho_b}{\Delta\rho} \right)$ value, should define an intercept value from which the resistance, C , of a single plate could be calculated.

Figure 23

Mathematical Illustration, Two Plates

8.54 Results and Observations

The results quickly brought out a deficiency in the apparatus. The X_3 layer tended to be so deep that only one or two readings could be taken before the holding capacity of the test column was exceeded. Therefore, there were usually not enough points for plots like Figure 23. Moreover, where several points were obtained, the apparent slope was much greater than $(\frac{\rho_b}{\Delta p})$ and the constancy of the C values became questionable.

Individual values of C for each run point were calculated, using the theoretical values of $(\frac{\rho_b}{\Delta p})$. By experimentation it was found that the C values were a function of X_3 , the benzene depth under the bottom plate. Figure 24 shows a plot of all the C values for the five different plates tested. The indication is that for this test procedure quite different plates give the same performance. Apparently the plates interact in some manner. According to Pike (10), plates with 1/4" holes can be used in a pulse column, where a bundle of plates serve to cause coalescence even when a single plate cannot. Thus, pulse column operation confirms some kind of plate interaction.

8.55 Deductions

It was noticed that, on Figure 24, the C values climbed as the depth (X_3) of the benzene layer increased. This is consistent with the idea that kinetic effects are important, and tend in these installations to lower the observed C values. As the X_3 depth increased, it should act as a buffer and damp out some of the kinetic effects, as was observed.

The two plate and the single plate tests did not give the same re-

sults. It appeared that plate EX might perform properly in a pulse column.

8.6 Conclusions

The conclusions from Section 8.0 work are as follows:

1. Various plates exhibited widely different behavior patterns in three different test situations intended to characterize their resistance to unpulsed flow in a two-phase system. Apparently, the action of one plate influenced the action of a second plate. Plates in a bundle may act quite differently from a single plate.
2. Although needing further refinement, test procedures were developed that gave reasonably reproducible results in characterizing the ability of one or two plates to retain a dispersed phase. Possibly, such tests might be developed to an extent sufficient to rate new plate designs for pulse column feasibility.
3. Kinetic effects combine to give single and paired plates less resistance than they would have under equilibrium conditions.
4. Expanded-metal plate EX was judged to be a fair first guess for a suitable new plate to be tested in a pulse column assembly in comparison with regular perforated plates with $1/8$ " holes.
5. At the present, plates being considered for pulse column work should be tested in actual pulse column operation.

9. MEASUREMENTS ON EXPANDED-METAL EX

9.1 General

As a result of the work described in Section 8.0, the expanded metal selected for major study was that designated as 1/8" x 22 gage AISI 302 x 5/64". This specification was denoted EX. In the following sections 9.2 to 9.9, the measurements are described for those factors thought to be pertinent to the use of the material as plates in a pulse column.

9.2 Meshes per Foot

Meshes are the openings in the form. In the unexpanded direction, the meshes per foot are the slits per foot. In the expanded direction, the meshes per foot is generally several times larger, and is influenced by the degree of expansion and, to a small extent, by the thickness of the base metal. See Figures 4 and 5.

By measurements over a length of about six inches, these results were obtained:

Meshes per foot, expanded direction	69
Meshes per foot, unexpanded direction	22.5

9.3 Angle of the Openings

The openings in expanded metal have an axis inclined α degrees from the plane of the plate, as illustrated in Figure 25.

The angle α was measured as follows. A plate was placed in a holder and installed about a foot in front of a cathetometer so that the line of sight coincided with the plane of the plate. While sighting through the instrument the plate was rotated slowly from a position edge-on until the maximum opening was observed. The extent of the rotation was the angle α .

Cross Section Through Line of Maximum Opening in the Direction of Expansion.

Figure 25

Angle of Opening

The walls of the openings are not precisely parallel to the axis of the openings, since the strands are not bent uniformly. Also the punched side of the form differs slightly from the other side. Therefore, the angle α differed slightly as measured from the two sides. The results are presented in Table 5.

Table 5

Measurement on the Angle of the Opening - Plates EX

32.5° 31.5° <u>33.0°</u>	32.6° 33.2° <u>33.4°</u>
32.3° average	33.1° average

Final average = 32.7°

Standard deviation = 0.68°

9.4 Strand Width

The strand width is half of the width of metal between holes, in the expanded direction. An equivalent definition is that, of the two possible interpretations, the strand width is the smaller width.

At the edges of sheets, or cut forms, of expanded metal, there are exposed strips of metal which are the width of the metal between holes, which is twice the strand width. These exposed strips are the only measures of strand width that are accessible to micrometers. To determine strand width, exposed strip widths are measured, then the measurements divided by two. The results of such measurements are given in Table 6, on four samples chosen at random.

Table 6
Measurements on Strand Width - Plates EX

<u>Strip Width (Twice Strand Width)</u>			
<u>Sample 1</u>	<u>Sample 2</u>	<u>Sample 3</u>	<u>Sample 4</u>
0.1409"	0.1385"	0.1415"	0.1387"
0.1410	0.1385	0.1420	0.1380
0.1411	0.1383	0.1420	0.1387
<hr/>	<hr/>	<hr/>	<hr/>
0.1410 avg	0.1384 avg	0.1418 avg	0.1385 avg

Average strip width = 0.1399 inches
 Average strand width = 0.0700 inches = $\frac{4.48^u}{64}$
 Standard deviation, strand width = 0.00026

9.5 Metal Thickness

Metal thickness is the same as the thickness of the original metal sheet, except where distorted by the punching operations. It is best measured by micrometer at exposed strips. The results of such measurements on four samples chosen at random are given in Table 7.

Table 7

Measurements on Metal Thickness - Plates EX

<u>Sample 1</u>	<u>Sample 2</u>	<u>Sample 3</u>	<u>Sample 4</u>
0.0362"	0.0362"	0.0357"	0.0364"
0.0361	0.0359	0.0353	0.0359
0.0362	0.0358	0.0356	0.0358
<u>0.0362 avg</u>	<u>0.0360 avg</u>	<u>0.0355 avg</u>	<u>0.0360 avg</u>

Average metal thickness = 0.0359" = 20.5 U.S. Standard Gage
 Standard deviation = 0.00022"

9.6 Weight per Unit Area

The weights were measured for ten expanded-metal plates selected at random. The diameters = 1.912" $\pm$ 0.001, and the hole for the center rod had the diameter 0.125" $\pm$ 0.002. The results are given in Table 8, where the area is that of the plate minus that of the center hole, both calculated as circles.

Table 8

Measurements on Specific Weight - Plates EX

Area = 2.859 $\pm$ 0.003 in.² = 18.44 $\pm$ 0.02 cm²

<u>Plate</u>	<u>Grams</u>	<u>Plate</u>	<u>Grams</u>
1	10.780	6	10.759
2	10.794	7	10.665
3	10.793	8	10.603
4	10.794	9	10.891
5	10.819	10	10.695

Average weight = 10.778 grams = 0.3802 ounces
 Standard deviation 0.085 grams = 0.0030 ounces
 Grams/cm² = 0.5845 $\pm$ 0.0052
 Ounces/in.² = 0.1330 $\pm$ 0.0025

9.7 Percent Free Area

Free area can be interpreted either as the open area in the plane of the plate, or that perpendicular to the axis of the openings. The latter seems to be the value pertinent to the flow problems.

Free area was measured graphically by integrating the free area seen on photographs, using a polar planimeter. For the photographs of the area in the direction of flow, the plates were mounted and turned until, by eye, the photograph would show the maximum free area. The measurements are shown in Table 9. The maximum percent free area = $41.9\% \pm 2.3$.

Table 9
Measurements on Percent Free Area - Plates EX

In Plane of Plate		Perpendicular to Axis of Flow		
Pictures	Percent	Pictures	Percent Free Area	
	Free Area		Punched Side	Other Side
1	21.8	3 and 4	38.6	39.4
2	19.6	5 and 6	40.4	46.9
	<u>20.7 avg.</u>	7 and 8	40.0	43.2
		9 and 10	41.2	43.2
			<u>40.5</u>	<u>43.2</u>
Measurements made by different people.			41.9 avg.	
			Standard deviation = 2.33	

9.8 Shape and Size of Openings

In Figure 26 are shown openings and measurements as viewed on photographs such as in Figure 4. In Figure 4 the punched side is shown in the "top view."

Dimensions are Approximate

Shape and Size of Openings

9.9 Degree of Expansion

The degree of expansion can be determined by two methods, as follows:

Method A - Strand Count

By measurement on a plate, or a photograph of a plate, the spread of 20 strands (beginning to beginning, or end to end) in the direction of expansion was 1.75 inches. By calculation, since the strand width is 0.0700 inches, 20 strand widths before expansion = $20 \times 0.070 = 1.400$ inches. Hence,

$$\text{degree of expansion} = \frac{1.75}{1.40} = 1.25$$

Method B - Specific Weight of Plate

The area of a plate was found in Section 9.6 to be 2.859 square inches, excluding the center hole. The thickness of a strand was found in section 9.5 to be 0.0359 inches. Therefore the volume is $(2.859)(0.0359) = 0.1026$ cubic inches. The specific gravity of Type 302 stainless steel is 7.93 (Metal Handbook, page 312). If it were a solid

plate of the metal thickness, the weight would be $(7.93)(.1026)(16.387) = 13.333$ grams. However, the measured weight (section 9.6) = 10.778 grams.

Hence,

$$\text{degree of expansion} = \frac{13.333}{10.778} = 1.237$$

Assume that the best value for degree of expansion = 1.24.

10. PULSE COLUMN TESTS, EXPANDED-METAL PLATES

10.1 Background

10.11 Choice of Expanded Metal

While the results of Section 8.0 were inadequate in permitting the selection of the most appropriate expanded metal for comparison, it did appear that the EX plate, 1/8" x 22 gage AISI 302 x 5/64", was a reasonable choice.

10.12 Impurity Problem

As discussed in Section 6.5, the flooding capacity of a pulse column is greatly influenced by trace impurities. Because of this, rather extreme care had been taken in the construction of the pulse-column installation to eliminate or reduce all manner of contamination. The construction was entirely stainless steel, Teflon, polyethylene tubing, and glass, with a little brass and six Ameripol synthetic rubber gaskets. The benzene and water feeds were brought to an extreme degree of purity, for extraction work of this magnitude.

Experience indicated that while the Ameripol rubber could contribute some material by leaching, its contribution was so small as to be of no consequence. The negligible physical exposure, the prior leaching of years, and the reproducibility attainable in previous flooding studies ($\pm 5\%$) combined to rule out the gaskets as a factor in the impurity problem. Some coalescence tests in 250 ml stoppered graduates indicated that polyethylene contributed no significant contamination.

The one weakness lay in the pump packings, which were supposedly pure Teflon and which were installed with virtually no lubricating grease

some years previously. However, it had been discovered that foreign substances could be extracted from the "pure" Teflon packing that could not be extracted from sheet or bar Teflon. Apparently, some binder had been employed in its fabrication. These extractables were found by experience to influence somewhat the flooding behavior. The pulse pump in particular contributed trace impurities, being larger and operating on a volume of fluid not changed often. Therefore, the pulse pump was isolated from the pulse column by the chamber shown in Figure 4. The feed pumps, with smaller packing and in-and-out flow were found to give little trouble (that is, they permitted reproducible data), particularly if the benzene and water feeds were used only once, not recycled.

In spite of the concern with impurities, it should be emphasized that the level of impurity was probably an order of magnitude or two less than with any other pulse column unit. When all the precautions were observed, flooding data could be reproduced from one week to the next within 3 percent. According to Pike (10), this is much better than the usual case, where ± 20 percent is considered good, and ± 50 percent is often observed. Another evidence of cleanliness was the small amount of interfacial precipitate (crud) that would build up in a day. Ordinary pulse column installations will develop far greater quantities of precipitate in that time. Interfacial crud is accepted as one of the facts of life in extraction work, and provision usually has to be made for its periodic removal.

10.13 Provisions for Cleanliness

For insurance in cleanliness, the following steps were taken:

1. In the beginning the apparatus was taken apart and cleaned.

The water tank was wiped clean of a slight precipitate, and the water flow lines flushed with water. The pump heads were opened and cleaned, particularly the check valves. The benzene tank and flow lines were flushed with clean benzene. The glass column and plates were washed with hot Alconox, then H_2SO_4 - $\text{Na}_2\text{Cr}_2\text{O}_7$ cleaning solution, then with tap and distilled water. Upon reassembly, the entire system (except for benzene lines) was flushed with tap water, then distilled water.

2. After cleaning, and when assembling, contact was avoided between bare hands and fingers with parts such as the plates and column insides.

3. Most of the batches of benzene were checked for impurity, by spectrophotometer.

10.14 Checks on Behavior

As a check for accidental contamination, the following steps were taken:

1. Samples of both exit streams were analyzed spectrophotometrically at least every two days for contamination, some (at least) of which showed a 270 mμ absorption peak.

2. The quantity of interfacial crud was observed carefully. It should be barely perceptible within several days.

3. With new batches of feed materials, the flooding point was checked with previous behavior.

4. Failure to reproduce previous results led to the discard of feed material.

10.2 Flooding Capacity Measurements

10.21 Technique

The column was operated with the interface at the top, and the benzene phase dispersed at the bottom. However, the column was started up with no benzene in it. The desired frequency and amplitude were carefully set at the beginning of a run. Next, the inlet V_D and outlet V_C , both at the bottom of the column, were set at selected test values. For this thesis program, the flow ratio was fixed at $V_D = V_C$ for lack of

time to cover other ratios. Then the inlet V_C flow rate was set closely equal to the outlet V_C rate. The interface was held in about the same position during a run by adjusting the inlet V_C .

With the settings made this way, the flows into and out of the bottom end section were set precisely at known values successively increased by small increments. If the column was operating below flooding, the column served to provide flows matching the end-section flows without visible disturbance within the column. However, if conditions were beyond flooding, the column could not pass the proper quantities of V_D and V_C . In that case, the relative inventory of material within the column, and consequently its appearance, started changing. These changes generally also changed the interface level, but not fast enough for useful measurement there. The flooding point was determined by visual observations of the column action.

10.22 Criterion for Flooding

The criterion for flooding was visual, and depended somewhat upon experience. When conditions were definitely below flooding, the column appearance stabilized quickly, say within five minutes. When conditions were definitely above flooding, there were two classes of behavior. When $\overline{\pi}_D^*$ was low, a benzene layer continued to increase between the plates, leading to a double dispersion and finally pushing out the bottom of the plate section, as shown in Figure 41. When $\overline{\pi}_D$ was high, the emulsion never cleared but instead, became thicker and grew until it also pushed out of the plate section at the bottom. See Figure 34. When near flooding, it was said that flooding did not exist if the flooding signs did not show within 30 minutes.

*See Section 10.23 for definition of $\overline{\pi}$.

10.23 Mathematical Treatment, Perforated Plates

Some terms of general utility are defined below, in consistent units. In previous pulse column studies, it had been found that the flooding capacity of perforated plates is well correlated by the relationship

$$C = f[\bar{\pi}_D, V_D/V_C].$$

V_D = flow rate of the discontinuous phase, ft/hr.

V_C = flow rate of the continuous phase, ft/hr.

F = pulse frequency, cycles per minute

A = pulse amplitude, ft., in the column measured between extreme stroke positions. The column area is calculated assuming plate spacers but no plates.

q_D = total volume of flow that passes a plate in the direction of V_D during one pulse stroke.

θ_D = time in minutes during which flow is in the direction of V_D . For a symmetric or ideal stroke, $\theta_D = 1/2F$ (see Fig. 27A)

$\bar{\pi}_D$ = flow rate in the column in the direction of V_D , averaged for the time that the net flow is in that direction. Hence
 $\bar{\pi}_D = q_D/\theta_D$. For the symmetric stroke and $V_D = V_C$,
 $\bar{\pi}_D = 2FA$.

θ_t = total cycle time, minutes. $\theta_t = 1/F$, independent of symmetry and V_D/V_C . For the symmetric case, and $V_D = V_C$, $\theta_t = 2\theta_D$ (see Figure 27A)

Δ_D = flow rate in the column in the direction of V_D , averaged over the total cycle time. Hence, $\Delta_D = q_D/\theta_t$. When $V_D = V_C$,
 $\Delta_D = FA$.

$C = V_D/\Delta_D$, the flooding capacity, also the flooding efficiency

q_C , θ_C , $\bar{\pi}_C$, and Δ_C are comparable terms for the continuous phase.

Originally, it was assumed that a pulse stroke created a perfect, sinusoidal velocity wave through a plate, as shown in Figure 27A. (Only the case where $V_D = V_C$ is shown there). In the ideal case,

U = instantaneous velocity past plate, ft/hr.

$$U = \pi FA \sin(2\pi F\theta) + (V_D - V_C). \quad (\pi = 3.1416).$$

when $V_D = V_C$, and $\sin(2 \pi F \theta) = 1$,

$$U = \pi FA = \text{maximum velocity, ft/hr. } (\pi = 3.1416)$$

In that case,

$$\pi_D = 2 FA, \text{ in consistent units}$$

and $\Delta_D = FA$, in consistent units.

In practice, it was observed that the actual velocity curve differed from the ideal curve to an appreciable degree. The observed curve, on Figure 27A, is irregular and crosses the axis at 172° , not 180° . The actual curve defines, for $V_D = V_C$

$$\pi_D = 2.09 FA = \text{ft/hr, when } FA = \text{ft/hr.}$$

and $\pi_C = 1.91 FA = \text{ft/hr, when } FA = \text{ft/hr.}$

In the actual case, regardless of the V_D/V_C ratio,

$$\Delta_D = \Delta_C = FA$$

10.24 Mathematical Treatment, Expanded-Metal Plates

The expanded-metal plates possess a holding capacity for liquid that is sufficient to influence its behavior, and to require modification of some of the equations of Section 10.23, which were developed for perforated plates with negligible holdup. As shown in Section 10.25 the void space of plate EX is equivalent to an amplitude of 0.07 inches. When a pulse flow of coalesced discontinuous phase starts through the plate, it does not emerge on the other side until after a flow equivalent to 0.07 inches. Thus, this depth of amplitude does not contribute to the plate actions of jet formation and dispersion, nor of flow capacity. However, when there is hardly a coalesced layer, the plate equivalent amplitude would not have the same influence.

Consider the example of Figure 27-B. If the pulse stroke were set

FLUID VELOCITY PAST PLATE

UPFLOW

0

DOWNFLOW

ANGULAR POSITION OF DRIVE CRANK, DEGREES

for an amplitude of 0.20" in the column, 0.07" of the amplitude would be absorbed to displace the amount of liquid filling the void space of the plate. The remaining 0.13" would be the effective amplitude.

Coalesced Phase Assumed

Figure 27-B

Action of Plate Amplitude Equivalent.

Taking the phenomenon of plate holdup into consideration, the mathematical quantities of Section 10.23 were expressed as shown in Table 10. For expanded plates in general, substitute the amplitude equivalent of the plate for the specific value of 0.07.

Table 10
Expressions Pertinent to Flooding

<u>Quantity</u>	<u>Perforated Plates</u>	<u>Expanded-Metal Plates EX</u>
Amplitude	A	$A' = A - 0.07$
Average flow of D phase	2.09 FA	$2.09 F(A - 0.07)$
Average flow of C phase	1.91 FA	$1.91 F(A - 0.07)$
Total net flow, D direction	FA	$F(A - 0.07)$
Total net flow, C direction	FA	$F(A - 0.07)$
Flooding Capacity	$C = \frac{V_D}{\Delta_D}$	$C' = \frac{V_D}{\Delta'_D}$

10.25 Amplitude Equivalent of Plate EX

Two methods were used to measure the amplitude plate equivalent as follows:

Method A - From Plate Dimensions

By sandwiching an EX plate between two planes, the apparent plate thickness was found to be 0.096 inches. Using the data of Sections 9.6 and 9.9, the total weight of a solid plate was calculated to be $(2.859)(0.096)(2.54)^3(7.93) = 35.67$ grams. The actual plate weight was 10.778 grams. Therefore, the void volume = $\left(\frac{35.67 - 10.78}{35.67} \right) \times 100 = 69.8 = 70$ percent. To fill this void volume, an amplitude of $0.096'' \times 0.70 = 0.067 \approx 0.07$ inches is required.

Method B - From Flooding Data

Unfortunately, the only data for this analysis was under suspicion of contamination, and was less reproducible than the other sets. Refer to Runs 9, 7 and 8 of Table 11. Note that these runs were all

made at the same $\overline{\pi}_D$ value, but different amplitudes down to the very low value of $A = 0.10$ inches. According to Pike (10), previous pulse-column flooding data using perforated plates had clearly indicated that flooding was controlled uniquely by $\overline{\pi}_D$, at a given V_D/V_C ratio. Here was a major discrepancy, presumably greater than experimental error, when amplitude, A , affected flooding in addition to $\overline{\pi}_D$. In Figures 28 and 30, the use of an amplitude corrected for plate voids (0.07 inch) apparently gave the desired unique relationship of $C = f[\overline{\pi}_D]$, at a given V_D/V_C ratio. It appeared that a straight line is a fair approximation, using the logarithmic scales. Hence, we might assume

$$\left(\frac{V_D}{A - X} \right) = a[\overline{\pi}_D]^{-b} = a[10.45 F(A - X)]^{-b}$$

where A = amplitude in inches

X = correction for plate equivalent amplitude

F = cycles per minute

From the data of Figure 30, $b = -0.398$ about. Using this exponent and run data for

Runs 8 and 7, $X = 0.075$ inches

Runs 8 and 9, $X = 0.077$ inches

Considering both Methods A and B, it was judged that the proper amplitude equivalent of Plate EX was 0.07 inches, and that the value is most appropriately obtained from the dimensions of the plate.

10.26 Flooding Data

The flooding data using the expanded metal plates are presented in Tables 11, 12, 13 and 14.

Table 11

Flooding Capacities in a Contaminated System

Plates **EX** in Alternated Direction, Punched Side Down
Benzene-Water System, Benzene Dispersed

Run No.	Amplitude <u>A = Inches</u>	Frequency <u>F = cpm</u>	$V_D = V_C$ <u>ft/hr</u>	$\overline{V}_D$ <u>ft/hr</u>	C <u>Ratio</u>	$\overline{V}_D'$ <u>ft/hr</u>	C' <u>Ratio</u>
1	1.00	60	70.1	627.	0.234	584	0.251
2	0.63	40	90.6	264.	0.719	234	0.809
3	2.00	50	31.1	1045.	0.062	1008	0.065
4	1.00	40	79.1	418.	0.396	389	0.425
5	1.00	20	84.4	209.	0.844	194	0.907
6	0.65	20	58.2	136.	0.896	131	1.004
7	0.20	30	23.4	62.7	0.779	40.8	1.198
8	0.10	60	13.5	62.7	0.449	18.8	1.497
9	0.30	20	27.2	62.7	0.906	48.1	1.182
10	0.25	80	62.3	209.	0.623	151	0.865
11	0.50	80	83.5	418.	0.417	360	0.485

Table 12

Flooding Capacities

Plates EX in Alternated Direction, Punched Side Down
Benzene-Water System, Benzene Dispersed

Run No.	Amplitude A = Inches	Frequency F = cpm	$V_D = V_C$ ft/hr	π_D ft/hr	C Ratio	π'_D ft/hr	C' Ratio
12	1.00	80	35.7	836	0.089	778	0.096
13	2.00	60	22.4	1254	0.037	1210	0.038
14	2.00	40	34.6	836	0.086	806	0.090
15	2.50	40	29.0	1045	0.058	1008	0.060
16	1.00	72	36.8	753	0.102	700	0.110
17	1.00	30	81.6	314	0.544	292	0.585
18	1.00	60	46.1	627	0.154	583	0.165
19	1.00	40	70.1	418	0.350	389	0.377
20	1.00	30	83.1	314	0.554	292	0.596
21	1.00	80	35.8	836	0.090	777	0.096
22	2.00	50	28.0	1045	0.056	1009	0.058
23	2.00	25	58.2	523	0.233	504	0.241
24	0.20	30	25.2	62.7	0.841	40.7	1.293
25	0.30	20	27.7	62.7	0.924	48.0	1.205
26	0.50	20	38.9	104.5	0.779	90.0	0.905
27	0.50	40	59.2	209	0.592	180	0.688

Table 13

Flooding Capacities

Plates EX in Alternated Direction, Punched Side Up
Benzene-Water System, Benzene Dispersed

<u>Run No.</u>	<u>Amplitude A = Inches</u>	<u>Frequency F = cpm</u>	<u>$V_D = V_C$ ft/hr</u>	<u>$\overline{TT}_D$ ft/hr</u>	<u>C Ratio</u>	<u>$\overline{TT}'_D$ ft/hr</u>	<u>C' Ratio</u>
28	2.0	50	28.0	1045.	0.056	1009	0.058
29	1.0	40	70.1	418.	0.350	389	0.377
30	0.5	20	38.2	104.5	0.760	90	0.887

Table 14

Flooding Capacities

Plates EX in Same Direction, Punched Side Down
Benzene-Water System, Benzene Dispersed

<u>Run No.</u>	<u>Amplitude A = Inches</u>	<u>Frequency F = cpm</u>	<u>$V_D = V_C$ ft/hr</u>	<u>$\overline{TT}_D$ ft/hr</u>	<u>C Ratio</u>	<u>$\overline{TT}'_D$ ft/hr</u>	<u>C' Ratio</u>
31	2.0	50	27.7	1045.	0.055	1009	0.057
32	1.0	40	70.7	418	0.351	389	0.380
33	0.5	20	38.0	104.5	0.760	90	0.883

The flooding runs were made at room temperature, about $24 \pm 3^\circ\text{C}$, with recycle of both phases. In all cases $V_D = V_C$, and the benzene was dispersed into the water. The effort was made to cover a wide range of behavior, from a flooding efficiency (ϵ) of about 100 percent down to a few percent. Tests were made with plates installed in various ways, to see if that arrangement was a factor.

After an extensive cleaning of the apparatus, it was charged with fresh benzene and fresh water, and Runs 1 through 11, Table 11, carried out. These runs appeared at first to satisfactorily define the flooding, but when fresh benzene and water were used, different flooding capacity data were obtained. All subsequent flooding data, covering two more batches each of benzene and water, were self-consistent and were more reproducible. It was concluded that Runs 1 through 11 were made with some unknown trace impurity, perhaps a result of the excessive handling at the start.

10.27 Discussion

The distinction between the first and the subsequent sets of data is illustrated on Figures 28 and 30. Figure 30 shows the internal consistency of all the data, except for the first set (Table 11). Figure 28 provides a comparison between the first set, judged to be slightly contaminated, and a later portion for exactly the same geometric condition. With trace contamination, flooding capacities up to 50 percent higher were obtained. According to Pike (10), this finding is in agreement with previous experience on the benzene-water system, using perforated plates, where also it took most strenuous efforts to insure cleanliness sufficient for reproducibility, and where also trace impurity led

to higher flooding capacities.

The desirability of correcting the C and π_D terms for the equivalent plate amplitude is argued as follows. On Figure 28 are shown results for five runs made at two constant π_D values, but different amplitudes down to the very low value of 0.10 inch. Points are plotted on both the corrected and uncorrected bases, with connecting lines to show the shift involved. While these data were obtained on the system judged slightly contaminated, the corrections are much larger than the unreliability of the data. When corrected, the results indicate that a single curve, independent of amplitude, suffices to correlate the data.

Visual observation of the column operating at very low amplitudes further substantiated the need for such a correction. Under these conditions, the layer of benzene below a plate could be seen pulsating in the free space in the plate. Sometimes a few drops would be released thru a few of the plate openings with the pulse, but more frequently the benzene would be forced thru the plate on every second or third pulse only. This applied to a pulse amplitude of 0.1 inch, and it is easy to visualize that, if the amplitude were reduced to that equivalent to the plate holdup, about 0.07 inch, no benzene would pass thru.

Figure 29 shows the results of Table 12 plotted uncorrected. Figure 30 shows the same set, plus other data, plotted on the corrected basis. These data are considered to be as satisfactory as can be obtained. Only in Region I were the amplitudes run at values low enough (0.20 inches) for the 0.07-inch correction to be important. Within Region I, the degree of fit of the results to a straight line is some measure of the comparative worth of the corrected and uncorrected values of C and π_D . Accordingly, lines were fitted to Regions I and II by the

method of least squares, and the degree of fit examined statistically.

Table 15 contains the results, assuming that the relationships are as follows:

$$C = a(\pi_D)^b \quad \text{and} \quad C' = a(\pi_D')^b$$

Table 15

Constants for Flooding Behavior Equations, $V_d = V_c$

Variables	Region	Constant a		
		Value	95% Confidence Limits	95% Limits on log a
C, π_D	I	3.101	2.263 to 4.249	± 0.1368
C, π_D	II	38,050	24,230 to 59,770	± 0.1961
C', π_D'	I	5.572	4.808 to 6.459	± 0.0641
C', π_D'	II	32,360	20,250 to 51,700	± 0.2035

Variables	Region	Exponent b	
		Value	95% Confidence Limits
C, π_D	I	-0.3029	± 0.0629
C, π_D	II	-1.9302	± 0.0692
C', π_D'	I	-0.3980	± 0.0305
C', π_D'	II	-1.9137	± 0.0725

The degrees of fit for the best-fitting lines were such as to give standard deviations as follows in Table 16:

Table 16
Standard Deviations for Equations

<u>Variables</u>	<u>Region</u>	<u>Standard Deviation</u>
c, π_D	I	0.0179
c', π'_D	I	0.0101
c, π_D	II	0.0211
c', π'_D	II	0.0226

While visual inspection of the plotted data and comparison of the standard deviations indicate a slight improvement thru the use of the correction, the small number of points involved prevents this conclusion from being supported statistically.

On Figure 30 are plotted all the data presumed to be without trace influence. The runs for the plates alternated, in the same direction,

and reversed, all fall on the same lines. Therefore, plate arrangements do not influence flooding capacity. However, as shown in Sections 10.3 and 10.4, the appearance of the column was appreciably affected by plate arrangements, while pressure drop was not.

Figures 28, 29 and 30 show clearly two distinct regions of behavior. The break occurs sharply at $\pi_D' = 304$ ft/hr. Region II seemed to be characterized by flooding because of excessive emulsification. Region I showed much better coalescence, but tended toward so much buildup of dispersed phase that double dispersion would occur. The abrupt transition was not observed with perforated plates, but perhaps because of insufficient information.

In the region of low π_D' , capacities higher than the theoretical value of 1.00 were obtained. An interpretation of this behavior is that, at these slow pulsing rates, once the plate resistance is overcome by the pump action, the subsequent resistance is much lower and the hydrostatic head suffices to drive some benzene through the holes. This type of action was observed in the Section 8.0, during tests of unpulsed plates where, once the plate resistance was overcome, a burst of flow followed.

Figure 31 shows how the V_D value increases as π_D' increases, at $V_D = V_C$. The theoretical relationship for 100 percent flooding efficiency is (see Table 10)

$$V_D = \left(\frac{1}{2.09}\right) \pi_D' = 0.479 \pi_D'$$

Figure 32 shows a manner of plotting which is well adapted to locating the transition point, and in predicting the settings for pulse column runs.

10.28 Deductions

Expanded-metal plates showed a flooding behavior characterized by an abrupt transition between flooding due to excessive emulsification (Region II) and that due to inadequate pulsing (Region I).

In Region I flooding, it was necessary to correct the actual amplitude by the amplitude equivalent of the expanded-metal plate. In Region II, the results did not show a need for such a correction, but there its use is generally insignificant.

In regard to flooding capacity, it made no difference how the plates were directed, or whether the punched side was up or down.

10.3 Visual and Photographic Studies

10.31 Flow Patterns at Plate

The orientation of one plate in regard to its neighbors made a big difference in the appearance of the column during normal operation (although neither flooding capacity nor pressure drop were so affected).

Figure 33 shows schematically the differences observed for the two plate arrangements employed, as viewed perpendicular to the direction of plate expansion. When the plates were placed in the same direction, the upstroke and downstroke impulses reinforced each other and tended to give a continuous, regular, circulation pattern. When π_D' was low, say < 100 , the circulation pattern was scarcely evident. But when in Region II, for instance, distinct circulation patterns were evident. Circulation appeared to give a higher average residence time per drop, between plates, than for perforated plates, since many drops completed more than one circulation before passing upward. Also, during portions of the cycle the continuous and discontinuous phases segregated somewhat in opposite sides

Figure 33

Circulation Patterns Between Plates

of the column.

When plate directions were alternated, the upstroke and downstroke impulses bucked each other, reversing the pattern of circulation each cycle. This eliminated the segregation observed during part of the cycle, when plates were in the same direction, and gave a more uniform appearance at all times. It appeared that the average residence time between plates was less than with the equi-directional plates, but that agitation was more vigorous.

Figure 46 shows that plate direction also influences holdup profoundly.

10.32 Appearance Near Flooding

During the flooding runs, some photographs were taken when at or near flooding. When this close to flooding, the appearance was independent of the nearness to flooding. Mostly the photographs show a particular plate location, but a few show the flooded condition in the bottom end section. There were 23 plates, two inches apart, numbered from the top.

The plates were installed with alternated directions. The timing of the picture in regard to the pulse cycle was rough and done by eye. In regard to interpretation, several aspects are worth noting. Quite frequently, relatively large stationary drops adhered to the inside glass walls. In the dynamic view, these wall-drops were easily ignored and the true dispersion seen. In the pictures, the distinction is often difficult to make. Also the glass walls curved, only the center part being in good focus, and the larger drop images were distorted.

A summary of the pictures is as follows in Table 17. The sequence of results is best appreciated when reference is made to Figure 30, and

the relative position of a run on that plot.

Table 17
Summary of Pictures of Flooding Appearance

Figure Number	Run No.	π_D' ft/hr	F cpm	A inches	Flooding Capacity $C' = V_D/\Delta_D'$
34	13	1210	60	2.00	0.038
35	16	700	72	1.00	0.110
36	19	389	40	1.00	0.377
37	11	360	80	0.50	0.485
38 and 39	17	292*	30	1.00	0.585
40 and 41	26	90	20	0.50	0.905

*Practically at the transition point of $\pi_D' = 304$. See Figure 30.

The pictures are arranged in sequence, starting with a high π_D' , low C' value. It is seen that in Region II, Figure 30, the emulsion becomes finer and finer as π_D' becomes greater. It was observed that the degree of dispersion also became finer from bottom to top. Figure 35 illustrates the appearance at two locations in the column, one higher than the other. Also, often a difference could be seen around the column, at one location, due to the directing influence of the plate.

Figure 38 shows that at $\pi_D' = 292$, in Region I close to the transition point, a definitely coalesced layer existed for the bottom few plates, but not above. Also, there was often a difference radially and circumferentially at a given location, due to the directing influence of the plates (note Figure 38-C). Figure 38-c was taken at a direc-

tion which reveals a localization of coalesced material at a wall, and a gradation of coalescence across the plate.

10.33 Deductions

The visual and photographic studies revealed clear differences between operation with plates arranged in the same direction, and in alternated directions. When in the same direction, regular circulation swirls resulted. In the other case, swirl reversal occurred each cycle. Presumably, these differences ought to be reflected in a difference in rate of mass transfer as well as holdup (Figure 46).

With the directing influence of expanded-metal plates, they should be of advantage in fairly large columns where adequate mixing is hard to obtain, providing π_D' is large enough.

10.4 Pressure-Drop Measurements

10.41 Background

The measurements were planned with the prior knowledge that for perforated plates

$$(1) \Delta P_{\text{avg}} = K \pi_D^n = K \pi_C^n$$

$$\text{where } n = 1.827 \pm 0.060$$

$$\text{and } \Delta P_{\text{avg}} = \frac{\int \Delta P d\theta}{\Delta \theta}, \text{ for the flow time.}$$

(2) the upflow and downflow pressure-drop relationships become the same when due consideration is given to the irregularities of the pulse wave. See Section 10.23.

(3) the average pressure drop under two-phase flow conditions are satisfactorily expressed in units of the continuous phase, except when quite near the flooding point.

FIGURE 34
FLOODING APPEARANCE AT $\pi_b' = 1210$
CAPACITY AT FLOODING = 0.038

PLATE NO. 12

NOTE THE FINER
DISPERSION

35 - A

APPEARANCE DOES NOT
CHANGE THROUGHOUT
PULSE CYCLE

PLATE 12 IS 8"
HIGHER THAN PLATE 16

PLATE NO. 16

35 - B

FIGURE 35
FLOODING APPEARANCE AT $\pi_b' = 700$
FLOODING CAPACITY = 0.110

PLATE NO. 14

DROPS ATTACHED
TO WALL

PLATE NO. 15

36 - A

DOWN STROKE

PLATE NO. 15

36 - B

UP STROKE

FIGURE 36

FLOODING APPEARANCE AT $\pi'_0 = 389$

CAPACITY AT FLOODING = 0.377

PLATE NO. 16

NOTE WATER
BEING DISPERSED

DOWN STROKE

37 - A

PLATE NO. 20

NOTE WATER
BEING DISPERSED

DOWN STROKE

37 - B

FIGURE 37

FLOODING APPEARANCE AT $\Pi'_d = 360$

FLOODING CAPACITY = 0.485

PLATE NO. 13

38 - A
UP STROKE 38 - B
DOWN STROKE

PLATE NO. 21

NOTE SLANT
OF LAYER ↗ 38 - C
UP STROKE 38 - D
DOWN STROKECOALESCED LAYER
EVIDENT ONLY AT
BOTTOM FEW PLATES

FIGURE 38
FLOODING APPEARANCE AT $\pi_0' = 292$
FLOODING CAPACITY = 0.585

BOTTOM END SECTION AT FLOODING

FIGURE 39

FLOODING APPEARANCE AT $\pi_b' = 292$

FLOODING CAPACITY = 0.585

PLATE NO. 14

WATER PHASE
WETS AND FLOWS
DOWN WALL

40-A

DOWN STROKE

PLATE NO. 14

NOTE COALESCED
LAYER

40-B

UP STROKE

FIGURE 40
FLOODING APPEARANCE AT $\pi_p' = 90$
FLOODING CAPACITY ≈ 0.905

APPEARANCE OF BOTTOM END SECTION
AT FLOODING

FIGURE 41
FLOODING APPEARANCE AT $\pi_d' = 90$
FLOODING CAPACITY = 0.905

10.42 Technique

The measurements of the dynamic pressure-drop wave, across the bundle of 23 plates, were carried out by an involved technique using the equipment described in Section 7.63. Reference is made to the manufacturer's directions. A sample calculation in Section 15 provides some details on the procedure.

The pressure-drop wave was measured as the difference between the pressure waves at two locations, using at the two locations equipment items (mainly the pickups) that were prone to individual differences in their calibration changes. Therefore, it was necessary to shut the equipment down and recalibrate each pickup assembly shortly after each pressure determination.

The calibration was accomplished under static conditions, while the measurements were under dynamic conditions. However, the equipment was designed to give no lag in response for frequencies much more rapid than those employed here. Presumably, then, the static calibration was applicable.

10.43 Pressure-drop Data

The conditions for the pressure-drop measurements are presented in Table 18. The conditions were chosen so that, when plotted on Figure 42 the loci formed lines in Regions I and II intended to represent about 75 percent of flooding. Sets A and B were planned, but the first attempts under those conditions seemed to indicate too small a pressure drop for accurate measurements.

10.44 Discussion

The data are plotted on Figure 43. Omitting the two obvious

Table 18

Conditions for Pressure Drop Measurements

23 ~~EX~~ Plates, Punched Side DownBenzene-Water System, Benzene Dispersed, $V_D = V_C$

Set No.	A Inches	F Cpm	$V_D = V_C$ ft/hr	$\frac{\Delta P}{\rho V^2}$ ft/hr	$\frac{V_D}{\Delta' D}$		Percent Flooding
					Ratio	Flooding*	
A [‡]	0.30	20	22.8	48.1	0.991	1.190	83
B [‡]	0.50	20	31.8	89.9	0.739	0.930	79
1	0.50	40	48.3	180	0.561	0.705	80
2	1.00	35	53.9	340	0.331	0.466	71
3	1.00	60	30.2	583	0.108	0.166	65
4	2.00	50	19.0	1008	0.039	0.0584	67

*Values calculated from equations fitted to flooding data.
See Section 10.27.

[‡]Pressure drop too small for accurate measurements.

Table 19

Pressure Drop Measurements - 23 EX Plates

Plates in Alternated Direction, Punched Side Down

Set No.	Run No.	$V_D = V_C$ ft/hr	Upstroke			Downstroke		
			$\overline{\Pi_D}$ ft/hr	Avg. ΔP "H ₂ O	Mean "H ₂ O	$\overline{\Pi_C}$ ft/hr	Avg. ΔP "H ₂ O	Mean "H ₂ O
1-A	7	48.3	209	1.60	1.53	191	0.53	0.68
	//			1.47			0.83	
2-A	6	53.9	366	2.31	2.22	334	1.57	1.53
	10			2.13			1.48	
3-A	5	30.2	627	5.22	5.24	573	4.12	4.20
	9			5.26			4.29	
4-A	4	19.0	1045	12.96	12.96	955	12.63	11.36
	8			12.96			10.09	

Plates in Same Direction, Punched Side Down

1-S	2	48.3	209	1.53	1.43	191	0.59	0.67
	3			1.33			0.75	
2-S	4	53.9	366	2.50	2.37	334	1.71	1.75
	5			2.24			1.78	
3-S	6	30.2	627	6.84	6.15	573	5.94	5.63
	7			5.45			5.32	
4-S	8	19.0	1045	13.90	13.90	955	10.34	10.34

points, the best-fitting equation for these data is:

$$\Delta P_{\text{avg}} = 6.733 \times 10^{-5} \pi^{1.7544}$$

where ΔP_{avg} = inches of water

and π = either π_D or π_C ft/hr

The 95 percent confidence limits applied to the slope give the range 1.7544 ± 0.0978 , which includes the value 1.827 derived for perforated plates. Using the slope 1.827, and omitting the obvious two points, the best-fitting equation is

$$\Delta P_{\text{avg}} = 4.287 \times 10^{-5} \pi^{1.827}$$

This latter equation was judged to be the most appropriate one, since the slope value of 1.827 had been derived from a much larger body of data, and its use gave a satisfactory visual fit to the expanded-metal data.

Omitting the two wild points, the data for plates with alternated directions led to the same pressure drop as plates arranged in the same direction.

A peculiarity was in question in regard to the upstroke and downstroke measurements. The relationships should have been exactly the same for these two cases, if judged by the perforated-plate results. For these plates, the punched side was down in each case, and the upstroke flow approached the punched side, but the downstroke flow approached the other side. In appearance, the punched side differed a bit from the other side. It was hard to express this difference tangibly, but it appeared that the punched side offered a slightly less abrupt flow transition. This appearance may well have been an illusion.

Examination of the results, excepting the two wild points, gave no indication of a difference in pressure drop due to the direction of flow approach.

The two wild points are hard to explain, if they need explanation. Since each plotted point is an average of two runs, the two wild points actually represented four runs which reproduced themselves well. They all applied to the case of flow approach to the punched side, and could be interpreted as a transition to streamline flow. More data are needed to settle the point. This ought to be possible, since during the measurements with perforated plates useful, though rather inaccurate, measurements were made down as far as 0.2 inches of water.

Values of Reynolds' number for flow through the perforations may be approximated, assuming that the fluid flowing is benzene.

The previous work on perforated plates (not shown) showed erratic evidence of a possible transition at about $Re = 200$. The two wild points on Figure 43 might be taken to imply a transition, for flow toward the punched side, at about $Re = 300$. The evidence is quite inconclusive. But certainly above $Re = 300$, there is no transition.

10.45 Deductions

As a result of the pressure-drop measurements, the following deductions were made.

(1) As for perforated plates, the average pressure drop across expanded-metal plates is a unique function of $\overline{w}_D$, the average flow rate, for two-phase operation.

(2) The numerical relationship is this:

$$\Delta P_{\text{avg}} = 4.287 \times 10^{-5} \bar{V}_D^{1.827}$$

where ΔP_{avg} = time average ΔP , inches of water
 $\bar{V}_D$ = ft/hr for benzene flow through column

(3) In regard to pressure drop, it makes no difference whether the plates are alternated or placed all in the same direction. Presumably, no other arrangement would make any difference.

(4) In regard to pressure drop, it makes no difference which side of the plate is approached by the flow, except that there exists a weak possibility that a transition between turbulent and streamline flow might occur sooner when flow approaches the punched side.

(5) With the materials and equipment employed, practically all flow conditions through expanded-metal plates are turbulent. The transition between streamline and turbulent flow certainly does not occur above $Re = 200$ to 300 . The calculation of Reynolds' criterion for flow through the openings is as follows:

Perforated Plates

Let d = hole diameter

V = average velocity through individual holes

ρ = density of benzene phase

μ = viscosity of benzene phase

f = percent free space

$\bar{V}_D$ = average velocity through column

An average Reynolds' number thus becomes

$$(Re)_{\text{avg}} = \left(\frac{d V \rho}{\mu} \right) = \left(\frac{d \bar{V}_D \rho}{f \mu} \right)$$

For the perforated plates, using benzene-water,

$$d = 1/8" = 0.01041 \text{ ft}$$

$$\rho/\mu = 37.4 \text{ hr/ft}^2, \text{ approx., for benzene}$$

$$f = 0.246$$

$$(\text{Re})_{\text{avg}} = \frac{(0.01041)(37.4)(\pi_D)}{0.246} = 1.58 \pi_D \text{ approx.}$$

Expanded-Metal Plates

Here $F = 41.9$ percent in the direction of flow. Assuming the validity of its use,

$$(\text{Re})_{\text{avg}} = \frac{(0.246)}{(0.419)} (1.58) (\pi_D) = 0.93 \pi_D, \text{ approx.}$$

Using a similar Reynolds' number, but ρ/μ averaged for the two intermingling phases, and using a measure of the peak pressure at the column bottom, Li (7) obtained a transition from streamline to turbulent flow at about 1200 for the average Re . Li's plates differed from ordinary pulsed-column perforated plates in possessing a plate thickness $(5/32")$ about five times normal, a percent free area (8.5 percent) about $1/3$ normal, and $1/16"$ diameter holes countersunk $1/16"$ on each face. It is to be expected that his plates would indicate a transition from streamline to turbulent flow at a higher Re than more customary plates.

10.5 Holdup Measurements

10.51 Background

Holdup is defined as the relative amount of discontinuous phase in the column, at steady state. It may be expressed as a ratio to the

continuous phase, as a fraction of the total column volume, or as quantity per plate or column.

Holdup data are not of primary interest here, because holdup is not often a factor influencing the choice of an extractor. Holdup becomes important only in exceptional cases, such as where radiation damage to the solutions is involved, or where practical factors of cost or time restrict the quantities or processing time available.

Nonetheless, holdup information is of general interest and is usually obtained rather simply. Holdup measurements have often been shown to be closely related to flooding capacity and to rate of mass transfer. Therefore, holdup data were obtained when the opportunity arose.

10.52 Technique

When steady state had been reached, a switch was thrown to cut off all power to the pulse column, stopping each pump at the same time. The emulsion between the plates separated more or less rapidly, building up a clear layer of discontinuous phase underneath each plate. Except for a few drops just after the power was shut off, none of the discontinuous phase ever leaked through a plate. Measurements on the depth of the clear phase beneath plates gave the holdup for each plate.

The holdup for the entire column was obtained by a displacement method. In practice, this was done by quickly and carefully marking the benzene-water interface position and the benzene level in the overflow chamber at shut down, closing off the benzene overflow line and pulsing the column to clear it of benzene. Water was then added to the column (necessarily equal to the benzene removed) to raise the interface to its original position. The benzene displaced was measured directly as that amount that had

to be drained from the overflow chamber to lower the benzene level to its shutdown position.

10.53 Holdup Measurements, Near Flooding

After the flooding capacities in Section 10.2 had been determined, the flow rates, V_D and V_C , were each reduced to about 97 percent of the flooding value. Column operation was continued for 10 to 15 minutes until the appearance of steady-state operation. Then the operation was stopped and holdup measured for each plate, and for all plates together.

On Figures 44, 45, and 46 are shown the individual holdup for each plate for runs with several plate arrangements.

In Tables 20, 21, and 22 are presented the total holdup data for three plate arrangements. These results are plotted on Figure 47.

10.54 Holdup Measurements, Below Flooding

During the runs for measurement of pressure drop, the holdup results were also measured. The run conditions were such that operation was from 65 to 80 percent of flooding, approximately. See Table 18. The results are placed in Table 23.

10.55 Discussion

Inspection of Figures 44, 45 and 46 show generally a trend of increasing plate holdup near flooding, from top to bottom. Possibly part of this effect was due to insufficient time being allowed to reach steady state. However, the effect was probably at least partly real, since the column appearance always changed along the column, being more finely dispersed toward the top. To settle the point, it would be necessary to make runs demonstrating that steady state had been reached.

Table 20

Holdup Measurement Near Flooding

23 EX Plates in Alternated Direction, Punched Side Down

Run No.	A Inches	F cpm	$\frac{V}{D}$ ft/hr	Total Holdup	
				ml	percent*
1	0.2	30	40.7	1400	0.63
2	0.3	20	48.0	1490	0.67
3	0.5	20	90.0	1620	0.73
4	0.5	40	180.0	1080	0.49
5	1.0	40	389.	815	0.37
6	1.0	60	583.	755	0.34
7	1.0	72	700.	630	0.28
8	1.0	80	777.	600	0.27
9	1.0	80	777.	590	0.27
10	2.0	40	806.	580	0.26
11	2.5	40	1009.	700	0.32
12	2.0	50	1009.	930	0.42
13	2.0	60	1210	690	0.31

*Assuming the total volume involved is that between pressure taps, 2220 ml. The distance was 50 1/8 inches.

Table 21

Holdup Measurements Near Flooding

23 EX Plates in Alternated Direction, Punched Side Up

<u>Run No.</u>	<u>A Inches</u>	<u>F cpm</u>	<u>ft/hr</u>	<u>Total Holdup</u>	
				<u>ml</u>	<u>percent*</u>
14	2.0	50	1009	765	34.5
15	1.0	40	389	760	34.2
16	0.5	20	90	1560	70.2

*Assuming the total volume involved is that between pressure taps, 2220 ml. The distance was 50 1/8 inches.

Table 22

Holdup Measurements Near Flooding

23 EX Plates in Same Direction, Punched Side Down

<u>Run No.</u>	<u>A Inches</u>	<u>F cpm</u>	<u>ft/hr</u>	<u>Total Holdup</u>	
				<u>ml</u>	<u>percent*</u>
17	2.0	50	1009	845	38.0
18	1.0	40	389	1030	46.5
19	0.5	20	90	1670	75.2

*Assuming the total volume involved is that between pressure taps, 2220 ml. The distance was 50 1/8 inches.

Table 23
Holdup Measurements Below Flooding
23 EX Plates, Punched Side Down

Set* No.	$V_D = V_C$ ft/hr	π'_o ft/hr	V_D/Δ'_D		Alternate Direction Total Holdup		Same Direction Total Holdup	
			Ratio	% Flooding	ml	percent**	ml	percent**
1 A & S	48.3	180	0.561	80	312	14.1	180	8.1
					290	13.1	130	5.9
2 A & S	53.9	340	0.331	71	324	14.6	320	14.4
					320	14.4	330	14.9
3 A & S	30.2	583	0.108	65	240	10.8	400	18.0
					245	11.0	520	23.4
4 A & S	19.0	1008	0.039	67	525	23.6	395	17.8
					517	23.3		

*See Tables 18 and 19. Here A = alternated, S = same directions.

**Calculated as percent of total volume of 2220 ml.

Nonetheless, the holdup data near flooding were obtained under substantially the same conditions, hence they have comparative value. Figure 47 shows the total holdup near flooding. Clearly, when the plates are in the same direction the holdup is substantially increased, compared to the alternated plates. This finding is consistent with the visual studies in indicating a difference between the two arrangements.

The holdup measurements taken below flooding, Table 23, disclose no simple pattern. Yet the results are reproducible, hence, real. They show differences between plates alternated and plates in the same direction, although sometimes one, sometimes the other provide the greater holdup.

10.56 Deductions

Holdup measurements indicate that the conditions between plates differ depending on whether plates are alternated or in the same direction, agreeing with the visual observations. The pattern of difference for ordinary operation could not be derived from the limited data obtained.

The holdup differences imply that rate of mass transfer differences would exist for the two plate arrangements.

11. PULSE COLUMN TESTS, PERFORATED PLATES P-25

11.1 Flooding Capacity Measurements

Data were obtained from Pike (10) for the flooding behavior on the same system by the same procedures, in the same equipment, except for the fact that the 23 plates were the P-25 plates. The data, at $V_D = V_C$, are presented in Figure 48, with lines for comparison giving the EX plate performance, plotted on a corrected basis, as explained in Section 10.24. The correction is negligible under ordinary operation.

11.2 Pressure-Drop Measurements

As in Section 11.1, data were obtained from Pike (10) for pressure drop across 23 plates P-25. A single equation holding equally for single-phase and two-phase operation is as follows:

$$\Delta P_{\text{avg}} = 3.313 \times 10^{-5} \pi^{1.827} \text{ inches of water}$$

$$\Delta P_{\text{avg}} = \frac{\int \Delta P d\theta}{\Delta \theta} \text{ for the flow period}$$

Figure 49 shows a plot of the perforated plates data and equation, plus a line for the final equation expressing the expanded-metal data.

12. DISCUSSION

12.1 Problem of Plate Comparison

Both the expanded-metal plates and the perforated plates may be considered to be characterized by two major factors, the percent free space and some measure of the size of the opening, such as hole diameter. The multiple choices compel any comparison between the two types of plates to be an arbitrary comparison. Assume, for instance, specific expanded-metal plate and any performance characteristic such as flooding capacity. Within limits, equivalent flooding performance can be obtained for perforated plates either by changing the hole diameter, keeping the free space constant, or changing the free space at constant hole size. Also, both can be changed according to some relationship, such as maintaining a given hole size to hole spacing ratio. Therefore, it is an arbitrary choice as to which perforated plate design to use as a comparison.

Considering the above complications, it was thought best to compare the two types of plates on the basis of the quality of the major performance obtained per unit of effort to attain that performance. The major performance for the present consideration is flooding capacity, which serves to control column diameter. (The rate of mass transfer is equally important, but was not measured.) The effort to attain desired performance is properly measured by the power input required, although sometimes the pressure drop is used (assuming fixed equipment features).

The power input required by the plate resistances themselves, as opposed to that required in auxiliary lines, is given as follows:

$$HP = \frac{1}{\theta_t} \int_0^{\theta_t} S \rho U \Delta P d \theta_t = \text{horsepower}$$

where the energy requirement is positive for both up and down strokes,

HP = horsepower requirement

S = column cross-section

ρ = average density of the material pumped

U = instantaneous flow rate past the plates

A check on the units is as follows:

$$HP = \left(\frac{1}{hr}\right)(ft)^2\left(\frac{lbs}{ft^3}\right)\left(\frac{ft}{hr}\right)(ft)(hr) = \frac{ft-lbs}{hr}$$

Since S and ρ are constant for a given situation

$$HP = \frac{C}{\Theta_t} \int U \Delta P d \Theta_t$$

For a symmetric pulse stroke,

$$HP = \frac{C}{\Theta_D} \int U \Delta P d \Theta_D$$

For our purposes, we assume that the average value of U can be employed as follows:

$$HP \cong k U_{avg} \frac{\int \Delta P d \Theta_D}{\Theta_D} = k U_{avg} \Delta P_{avg}$$

Since U_{avg} is symbolized by π_D ,

$$HP \cong k \pi_D \Delta P_{avg} = k[ft^2/hr]$$

The $[\pi_D \cdot \Delta P]$ product thus defined is termed for the power factor. While it is not correct to say that horsepower is strictly proportional to power factor, there is considerable symmetry in the U vs Θ and ΔP vs Θ curves, and it appears reasonable to say that as the power factor increases, so does horsepower. Therefore, a comparison at constant power factor is

reasonably a comparison at constant horsepower, or power input to the pulse column.

12.2 Plate Comparisons

The comparative results for the two types of plates are compiled into Table 24, showing at constant π_D or π_D' the values of C or C' , ΔP_{avg} , and the power factor (ΔP) (π_D or π_D') in units of ft^2/hr . It is of little importance here that π_D , C and π_D' , C' , are on slightly different bases, differing by the small correction for the 0.07" plate amplitude equivalent, for in practical cases amplitudes around 1 - 2.5 inches are employed.

Figure 50 shows flooding capacity plotted against average pressure drop across the 23 plates. At $\Delta P = 5.0$ inches of water, a typical operating condition, the EX plates provide a flooding capacity over 40% greater than does the P-25 plates.

Figure 51 shows flooding capacity plotted against the arbitrary power function, in ft^2/hr . At $\pi_D = 500$, a typical operating condition, the EX plates provide a flooding capacity about 40% greater than do the P-25 plates.

On either basis employed, and as well on the basis of comparison of capacity at constant π_D according to Figure 48, the expanded metal plates give substantially better performance than perforated plates.

12.3 Flooding and Pressure Drop Behavior

Both flooding capacity and pressure drop were independent of the manner of plate arrangement, yet the holdup and the appearance between plates differed appreciably. This could only be so if both flooding and pressure drop are controlled by the actions at the plate, rather

Table 24
Comparative Performance of Plates EX and P-25

π_D, π_D' ft/hr	ΔP_{avg} Inches H ₂ O	$\pi_D \times \Delta P_{avg}$ ft ² /hr	C ratio	ΔP_{avg} Inches H ₂ O	$\pi_D' \times \Delta P_{avg}$ ft ² /hr	C ratio
100	0.149	1.24	0.72	0.193	1.61	0.892
200	0.534	8.90	0.562	0.691	11.51	0.675
300	1.11	27.7	0.414	1.44	36.0	0.575
400	1.88	62.7	0.293	2.43	81.0	0.339
600	3.95	197.5	0.151	5.11	255.3	0.156
800	6.68	445.	0.077	8.64	576.	0.0900
1000	10.03	836.	0.041	12.98	1082.	0.0587
1200	14.00	1400.	0.0225	18.10	1810.	0.0414
1500	21.05	2630.	0.010	27.22	3402.	0.0270

than by behavior between the plates.

The sharp break that appears in the flooding behavior at $\pi_D' = 304$, as shown on Figure 30, has not been reported previously. It may be peculiar to expanded-metal plates. Inspection of previous data on perforated plates revealed that no run conditions for them were such as to clearly prove that perforated plates do or do not exhibit a sharp break.

12.4 Inferences concerning Mass Transfer

Both the holdup results and the visual evidence clearly indicated that conditions between plates were markedly dependent upon whether the plates were arranged alternately or in the same direction. The circulation patterns reproduced on Figure 33 illustrated one of the differences. Figure 47 showed how holdup patterns could vary. Such differences would logically lead to differences in rate of mass transfer, in spite of the fact that neither flooding capacity nor pressure drop were affected. Since rate of mass transfer is a major performance factor, expanded-metal plates cannot be assumed to be properly evaluated until the two plate arrangements are tested for differences in rate of mass transfer.

12.5 Impurity Problem

The experiences of this study confirm previous findings that trace impurities exert a relatively large effect on flooding capacity, and presumably also on rate of mass transfer. The effort necessary to control the level of impurities makes this point a major one in pulse column work.

12.6 Plate Characterization

The results of Section 8.0, wherein the attempt was made to charac-

terize plates by their resistance to unpulsed flow, showed that this was a difficult task but still one that might be developed sufficiently to give standardized results. However, kinetic effects of drop impact and fluid inertia were shown to be of such magnitude that probably the only way to evaluate an unknown plate is to test it in a pulse column.

13. NOMENCLATURE

Abbreviations

FPH	=	ft. per hour, based upon column minus spacer cross section.
CFM	=	cubic feet per minute.
GPH	=	gallons per hour.
HP	=	horsepower.
i.d.	=	internal diameter.
P-10	=	perforated plate, 1/8" holes, approximately 10% free space.
P-25	=	perforated plate, 1/8" holes, approximately 25% free space.
P-40	=	perforated plate, 1/8" holes, approximately 40% free space.
EX	=	expanded metal plate, 1/8" x 22 gage AISI 302 x 5/64".
EX-R	=	ridged expanded metal plate, 1/8" ridged x 22 gage AISI 302 x 3/64".

Superscripts

'	denotes basis employing correction for plate amplitude equivalent
"	denotes inches
n	denotes exponent

Subscripts

D	refers to discontinuous phase
C	refers to continuous phase
t	refers to total cycle, as illustrated in Figure 27

Symbols

A, A'	=	pulse amplitude, inches (or feet), in column with spacers and no plates.
C, C'	=	flooding capacity, flooding efficiency, $C = \frac{V_D}{V_C}$, $C' = \frac{V_D}{\Delta' D}$
d	=	diameter

Symbols (Cont.)

- FA = frequency-amplitude product, ft/hr.
 F = pulse frequency, cycles per minute.
 f = percent free space for plates.
 S = column cross-section, total minus spacer, ft² or inch².
 U = instantaneous velocity through column, ft/hr.
 V = average velocity through plate perforations.
 V_D, V_C = clear phase flow, ft/hr as for A.
 q_D, q_C = clear phase flow past plate during a pulse stroke.
 $\overline{\pi}, \pi'$ = π_D or π_C , π'_D or π'_C .
 π_D, π_C = q_D/θ_D , q_C/θ_C , average flow rates past plate, ft/hr.
 Δ_D, Δ'_D = q_D/θ_t , average flow rate over the total cycle in the direction of V_D .
 θ_D, θ_C = actual time of flow past plates, during a pulse stroke in the direction of the clear phase flow, minutes.
 θ_t = total cycle time, minutes.
 σ = interfacial tension, dynes/cm.
 ρ = density, gm/cm³.
 μ = viscosity, consistent units.
 ΔP = pressure drop in inches of continuous phase.
 ΔP_{avg} = $\frac{\int \Delta p d\theta}{\Delta \theta}$, where $\Delta \theta$ = time involved.

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15. APPENDIX

15.1 Pressure Drop Calculations

As an example of the details of proceeding from the observed data to the final pressure drop information, the calculations for Set No. 4, Run No. 4, of the pressure drop series are presented here. The operation was the conventional two-phase pulsing operation.

Run Conditions

$$V_D = V_C = 0.00610 \text{ ft}^3/\text{min.} \frac{(0.00620)(60)(114)}{(2.774)} = 19.0 \text{ ft/hr.}$$

Frequency = 50 cycles per minute.

Amplitude = 2.00 inches.

Temperature = 23.8°C.

$\bar{V}_D = 1045 \text{ ft/hr.}$

Pressure Wave Records

The record of the pressure wave at the bottom tap is shown in Figure 52; that for the top tap, in Figure 53. Note the pip indicating the far back position of the pulse piston.

Total Benzene Holdup

The benzene holdup, measured by the technique described in Section 10.52, was 32.0 cubic inches. The cross sectional area being 2.774 square inches (Section 7.2), the corresponding height in the column was 11.5", equivalent to 10.1" of water at the fluid temperature of 23.8°C.

Figure 52

Observed Pressure Wave - Bottom Tap

Figure 53

Observed Pressure Wave - Top Tap

Calibration Results

The calibration was carried out by imposing increments of pressure on the column, after shutdown, and taking the static reading on the pressuregraph scale. In order to be able to subtract the pressure waves directly, they were first put on the same basis. The pressure units chosen were inches of water measured above the top tap, for the operating condition involving benzene holdup between taps. The operating condition thus differed from the static, calibrating condition, wherein the fluid between the taps was entirely water.

The correction to the bottom, static reading to convert it to the bottom, operating condition was 11.5-10.1, or + 1.4" H₂O. Since the distance between taps = 50.1", the bottom, static pressures were corrected by subtracting 50.1 - 1.4 = 48.7" H₂O.

The calibration results for the two locations are presented in Figure 54, in units of inches of water above the top tap, during operation.

Pressure Wave Measurements

The pressure waves at the two locations are plotted on Figure 55, in the common units described. The pressure wave at the top was small, reflecting as it did only the inertia and friction of the liquid flowing in a low-resistance channel of relatively large cross section.

The subtraction of the two waves on Figure 55 gave the single pressure drop wave shown on Figure 56.

Average Pressure Drop

The average pressure drop was obtained by graphical integration of the areas involved. The average upflow pressure drop was the sum of the two areas above the zero axis, divided by the time that the pressure drop curve was above this axis. The average downflow pressure drop was the area below the zero axis divided by the corresponding time.

From Figure 56, the values were found to be:

$$\text{Avg. } \Delta P, \text{ upflow} = 12.92'' \text{ H}_2\text{O}$$

$$\text{Avg. } \Delta P, \text{ downflow} = 12.63'' \text{ H}_2\text{O}$$