LIQUID EXTRACTION IN A PACKED PULSE COLUMN

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LIQUID EXTRACTION IN A PACKED PULSE COLUMN

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Dominik Martin Schuler

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APPRECIATION

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I. INTRODUCTION

Continuous liquid-liquid extraction is used industrially to a great extent where separation or purification by distillation or adsorption is impractical. Extraction is often performed in vertical towers, containing trays, sieve plates, or baffles, or they may contain a packing such as Raschig rings. In any case, one of the main problems is to obtain adequate mass transfer in a reasonable height of tower. To do this it is desirable to have among other things a high interfacial area between the two liquid streams.

Baffle-type columns may be considered as vertical mixer-settlers in which the contact of the two liquid streams is stage-wise. Different types of baffles are in use, but the efficiency, i.e. the number of the theoretical stages, is usually far less than a column of the same height and design would give in distillation. In extraction the effectiveness of an actual stage is usually a small fraction of one theoretical plate.

In spray columns one of the two liquids is dispersed in fine droplets in the second to obtain a high interfacial area. The height required for one theoretical stage in these towers also is usually high. A packed column generally gives a better efficiency than one without packing, and this may be due to an increase in turbulence and interfacial area. However, even in these columns the efficiency is usually poor because the only force available for mixing of the streams is the difference in density of the two liquids.

Mechanical contactors other than towers have also been used to some extent. Extraction in a set of mixers and settlers is common practice. Another device extensively used in some industries is the Podbielniak, Inc., centrifugal contactor, which uses a high speed rotor with a spiral ribbon for contacting the two liquid streams. Bartels and Kleinman (1) report that one of these units was equivalent to about four theoretical stages. The company claims that some machines contain as many as 15 theoretical stages.

The mechanical difficulties involved in devices of this sort and the generally poor performance of extraction columns which are not agitated has stimulated efforts to design vertical extraction columns in which additional mechanical energy is applied to the system. Three ways of doing this have been investigated:

a) Alternate mixing and settling zones, with mechanically operated stirrers (usually with a rotating shaft to operate the stirrers);

b) Vibration of the whole baffle assembly in the tower;

c) Pulsation of the liquid phase while plates or packing are in a fixed position.

A vertical tower with a motor-driven, rotating shaft in the center, on which a number of stirrers have been mounted, was designed and thoroughly investigated by Scheibel (2, 3). The original column contained wire mesh packed calming zones and mixing zones energized by stirrers attached to a shaft reaching from the top of the column to the bottom, with a bearing in the liquid at the bottom.

In this column, one stage, consisting of a mixing and a settling zone, can have an efficiency which exceeds the effect of one theoretical stage because of the additional contacting of the two liquid streams in the settling zone. One actual stage is usually nearly equivalent to one theoretical stage.

In a one-inch column, heights equivalent to a theoretical stage of 2.2 in. have been obtained by Scheibel. In a semi-commercial multistage extraction column, 12 in. in diameter, designed by Scheibel, the heights equivalent to a theoretical stage varied from 9 to 16 inches (4).

For a design of an extraction tower with a vertically vibrated plate assembly, Van Dijck (5) was granted a patent in 1935. In that patent he describes also a system with the plates in fixed position, but with a pulsed liquid phase.

For the system with a vibrated plate assembly no further information was found in the literature. But the other modification with fixed plates and pulsed liquid phase has been studied during recent years. Several atomic energy installations of this kind have been built and operated, but few details have been published (6).

Recently Cohen and Beyer (7) have designed a small pulse extraction column with perforated plates, and investigated its performance. To apply a vibrating motion to the liquid phase the investigators used a cam-driven bellows pulsator. Values of H.E.T.S. as low as 10 in. have been obtained with the system water-isoamyl alcohol. The diameter of the column was 1 in.

A packed extraction column with pulsed liquid phase was investigated by Feick and Anderson (8). They extracted benzoic acid and acetic acid from toluene with water, and operated a column with a diameter of 1 7/16 in., filled with 3/8 in. Raschig rings or ½ in. McMahon saddles. For benzoic acid the reported heights of a transfer unit vary from 13.5 to 3.07 feet for the unpulsed column, and for the pulsed column from 0.837 to 0.558 ft. In the case of acetic acid the height of a transfer unit was 1.1 and 0.4 ft. for the unpulsed and the pulsed column respectively. It is interesting to note that the greatest increase in performance due to pulsation occurred in the case of the benzoic acid where the efficiency without pulsation was very poor.

A similar investigation was published by Von Berg and Wiegandt (9). They give the information that in a packed column of 1¼ in. diameter, filled with ¼ in. Raschig rings, the height of a theoretical stage was 7.8 in. without pulsation and 1.6 in. with pulsation.

In both of these references only a few important details are given about the extractor performance.

The present investigation was undertaken as the first step in a thorough study of the performance of pulsed columns for liquid-liquid extraction.

II. THEORETICAL PART

Calculation of the Efficiency of the Transfer.

The data for the performance of a column may be expressed in H.E.T.S. (height equivalent to a theoretical stage), or as H.T.U. (height of a transfer unit). While H.T.U. possesses some theoretical advantages over H.E.T.S., its correct use requires knowledge of the fluid film resistances. H.E.T.S. has been used here since it may be computed from the compositions of the streams and from equilibrium data. In case the equilibrium line and the operating line have the same slope, the numerical values of H.E.T.S. and H.T.U. are identical. To minimize the uncertainty of data Scheibel (15) proposed to make the operating line nearly parallel to the equilibrium line. Where the slopes of the two lines differ greatly the number of transfer units would differ greatly, depending upon which of the films has been assumed to be controlling.

In all the runs made in this investigation, the extractant (water) had a zero concentration of the solute (acetone) at the start. Furthermore, the distribution coefficient was relatively constant.

In such cases the number of theoretical stages may be calculated according to the formula:

$$E^{(N + 1)} = 1 + (E - 1)\frac{x_o}{x_N}$$

which is developed, for instance, in Perry's Handbook (16). In this formula N represents the number of ideal stages; and x_o and x_N are the concentrations of the solute in the solvent (carbon tetrachloride) for the incoming and leaving streams respectively. E may be called "the extraction factor". It is the ratio of the slope of the equilibrium curve to the slope of the operating line.

It is therefore also given by the ratio $\frac{S\,D}{W}$, where D is the distribution coefficient of the solute (acetone); S is the flow of the solvent (carbon tetrachloride), and W is the flow of the extractant (water). The units used in this report are always gram mols for acetone and liters per hour for solvent and extractant.

III. EXPERIMENTAL PART

1. Description of the Apparatus.

The apparatus designed to get some information about the performance of a packed pulse column is reproduced in detail in Figure 1. It consisted mainly of a column of pyrex glass with a diameter of 5.2 cm and a length of 120 cm. There was only one kind of packing in use, which consisted of Raschig rings made of pyrex glass with inside and outside diameters of 0.55 and 0.78 cm respectively and a length of 0.78 cm. The packing had a height of 81 cm. It was supported by a flat spiral made of monel metal. On this spiral were placed two layers of Raschig rings with a diameter of 1.15 cm, in order to allow a wider spacing in the winding of the spiral. The spiral itself was supported from the bottom of the column by means of two supporting strips made of monel.

Either end of the column was closed with a rubber stopper through which the glass tubing for the different liquid streams extended. The stopper at the lower end of the column was covered with a layer of mercury, about 0.7 cm in height, to keep it protected from the influence of the solvent. The upper stopper was in direct contact with the air space above the liquid.

The pulsator consisted of a brass bellows with an inner diameter of 5.5 cm and about 10 corrugations. The pulsative motion was produced by an adjustable motor-driven eccentric. The bellows was compressed by a spring and drawn back by the pressure of the liquid when the spring

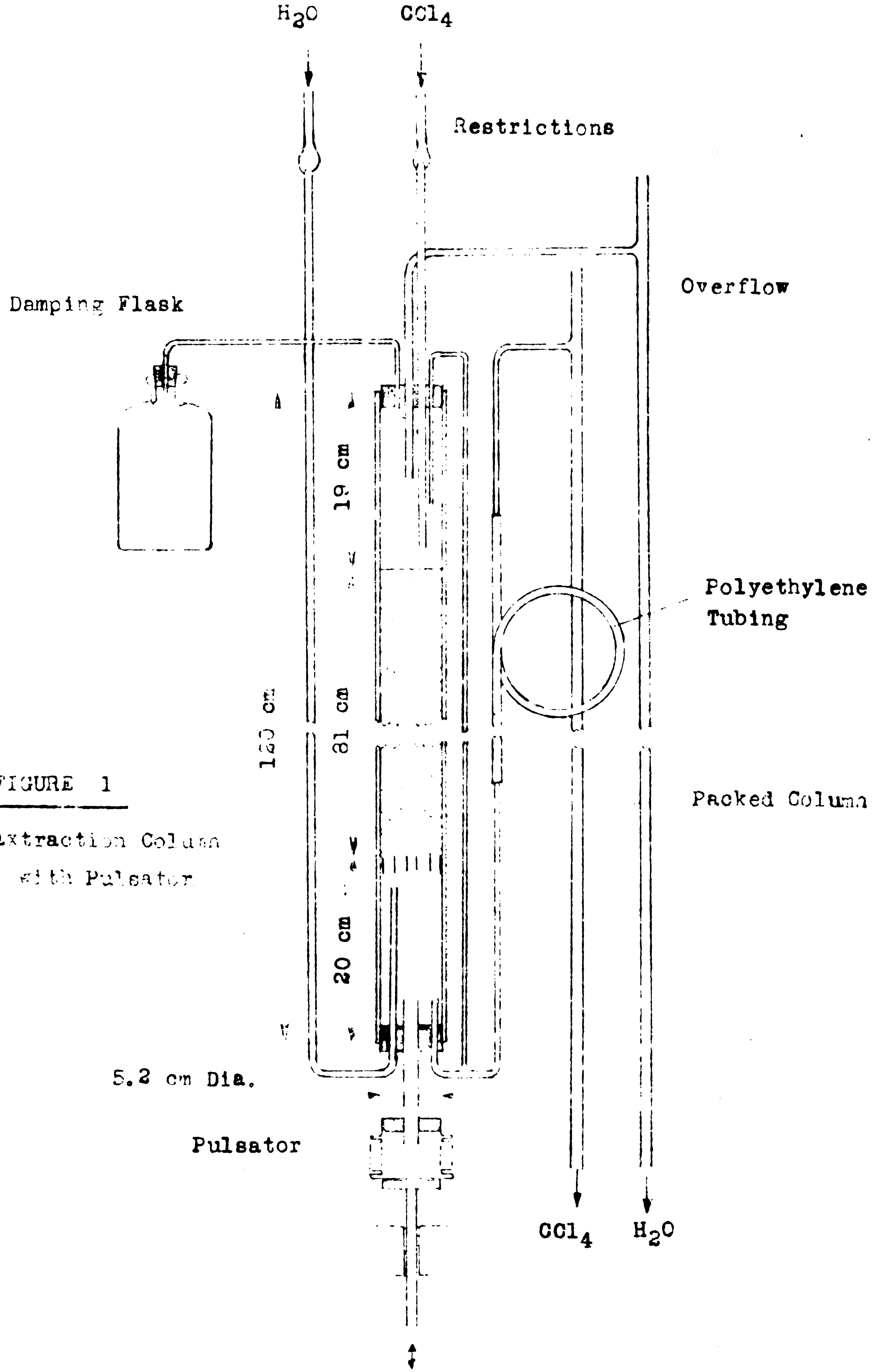


FIGURE 1

Extraction Column with Pulsator

was compressed by the eccentric. The bellows was closed with a rubber stopper which lasted three or four weeks. A glass tube with an inner diameter of 1.15 cm connected the bellows with the bottom of the column.

To get a relatively steady overflow of the liquids during pulsation, care was taken to have a free air space of 7 to 10 cm height between the rubber stopper and the liquid level at the top of the column. For additional damping the air space was connected by a small glass tube to a bottle having a volume of about two liters.

The two solvents to operate the column were stored overhead in two 5-gallon bottles. The height of the shelf for the two bottles, measured from the bottom of the column, was 270 cm.

For most of the experiments the flow of the two solvents was controlled by fixed restrictions in the form of little glass nozzles inside the tubing. The connections of the glass tubing were made with polyethylene tubing.

2. Investigated System.

The system acetone-water-carbon tetrachloride was selected because of convenience in handling, and because the distribution coefficient is relatively constant, in contradistinction to most dissociating compounds. Furthermore, the quantitative analysis of acetone is not too complicated.

The experiments were always started with a mixture of carbon tetrachloride (Dow, technical grade) and acetone (Merck C.P. reagent grade). The acetone was extracted with untreated tap water. The extact, after sampling, was discarded to the sewer, and after addition of more acetone the carbon tetrachloride was used in the next run. By means of this procedure no additional work for regeneration of the solvents was required.

3. Analysis of Acetone Content.

For the quantitative analysis of acetone several methods have been described in the literature; and Dehio and Schulz (10) gave an abstract for different methods 25 years ago.

The reaction of acetone with hydroxylamine hydrochloride, in which the amount of acetone is determined by titration of the hydrochloric acid formed, was considered the most convenient method. It is in frequent use for the determination of certain ketones, and was first proposed for acetone by Hoepner (11). In this analysis certain conditions, many of which were investigated by Marasco (12), have to be considered in order to obtain reliable and reproducible results.

The procedure preferred for the determination of acetone in the two solvents, water and carbon tetrachloride, followed in general the investigation of Bennett and Donovan (13).

A volume of 20 ml of the solution whose acetone content was to be determined was first neutralized with HCl, using methyl orange as an indicator. There was then added a volume of 10 ml of hydroxylamine hydrochloride solution of 1.00 normality, and finally a volume of 10 ml of potassium hydroxide of 0.79 normality. For completion of the reaction this mixture was then exposed for two hours at room temperature. After this time the mixture was titrated with hydrochloric acid, using methyl orange as an indicator. To prevent losses due to vaporization a 150-ml Erlenmeyer flask with a glass stopper was used. The procedure with carbon tetrachloride was exactly the same as with the aqueous solution, but because of the fact that the reaction probably proceeds only in the aqueous phase, the use of stirring was indicated. To contact the two liquids well without causing losses, the stoppered flask was mechanically rotated around its axis in an inclined plane. A 1.2 cm Raschig ring in the flask was intended to insure that the mixing would be turbulent.

The hydroxylamine method is reported by Koreman (14) as not giving satisfactory results in case very small amounts of acetone have to be determined. Therefore, some tests were undertaken to prove the method.

Four 10 ml samples of an 0.340 normal aqueous solution of acetone were analyzed; two in the presence of 20 ml of carbon tetrachloride and the other two alone. The samples with carbon tetrachloride were shaken thoroughly before the reagents (hydroxylamine hydrochloride and potassium hydroxide) were added. Titration with 0.392 normal acid required 8.65 and 8.67 ml of hydrochloric acid for the single phase aqueous solutions. For the samples in which carbon tetrachloride was present, 8.67 and 8.62 ml were required.

From these analyses the calculated acetone content is 3.39 millimols for a 10 ml sample.

Since the agreement was better than expected and far better than necessary, this analytical method appears satisfactory.

4. Scope of the Experimental Investigation.

The system under consideration, a packed pulsed extraction column, has a great number of process variables which determine its performance.

The process variables which might be taken into consideration are:

1. Kind of packing;
2. Diameter of packing;
3. Height of packing;
4. Degree of settling of packing;
5. Diameter of the extracting column;
6. Amplitude of pulsation;
7. Frequency of pulsation;
8. Shape of pulsation curve;
9. Flow rates of solvent;
10. The particular solvents and solute;
11. Choice of continuous phase in the column;
12. Direction of transfer of solute.

An investigation including all the process variables would require an extremely large number of test runs. The experimental work, therefore, could include only a very limited selection of this great number of possibilities.

For this investigation these variables have been cut down to only one kind of packing (0.78 Raschig rings), one height of packing (81 cm), one diameter of the column (5.2 cm), and water always as the continuous phase and as the extractant. The pulse frequency was kept constant and the motion of the pulses was always approximately a sine wave.

The test runs included the following investigation:

a) Flooding rates without pulsation and with different pulse amplitudes in a well settled packing;

b) Efficiency of the column at constant flow rates and different pulse amplitudes in a well settled packing;

c) Efficiency of the column at one given pulse amplitude and different flow rates of the solvents in a well settled packing;

d) Comparison of the efficiency of the column at given flow rates and pulse amplitudes for a well settled packing in one case and loose filled-in packing in the other case.

5. Operating Procedure.

One of the two feed bottles overhead was filled with tap water and the other with carbon tetrachloride containing approximately 1 percent acetone by weight. In most runs the flow to the column was fixed by nozzles in the lines. The pulse amplitude of the liquid was determined by measuring the total displacement of the liquid surface at the top of the column. The flow rates were determined by measuring the overflow from the column in a given time by means of graduated cylinders of one and two liters volume. The interface of the two liquids was maintained at a constant level by adjusting the height of the flexible overflow for the carbon tetrachloride. Samples were collected from each stream 35 or 40 minutes after getting the operating conditions (flow and level) constant. Since the total holdup in the column was only 2.5 liters, this flow rate was sufficient to replace the liquid in the column several times during each run.

IV. RESULTS

1. Distribution of Acetone in Water and Carbon Tetrachloride.

The data for the distribution of acetone in water and carbon tetrachloride were determined by shaking thoroughly nearly equal parts of the two solvents together with an amount of acetone in a separatory funnel. Samples were taken from the two layers and analyzed. The temperature was 30° C. within 0.5°.

The results are given in Table I.

TABLE I.

Distribution of Acetone in Water and Carbon Tetrachloride.

Concentration of acetone, millimols/liter

H_2O phase	CCl_4 phase	Distribution coefficient D.
178	83.0	2.145
283.5	134.3	2.110
150.8	71.0	2.125
66.7	34.4	2.135
38.7	17.8	2.175
13.52	6.47	2.09

As shown in the graph, Figure 2, the ratio of the concentration of acetone in the two solvents is essentially constant in the range under consideration. The average value for the distribution coefficient D is 2.135 on a volumetric basis for the solutions. By comparison, Seidell (17) reported distribution coefficients of 2.205 and 2.235 at a temperature of 25° C. for the range covered.

2. Flooding Rates for Unpulsed column.

The flooding rates have been determined for the unpulsed column under normal conditions with well settled packing. The data have no high degree of reproducibility, possibly because of a hysteresis which may be involved.

TABLE 2.

Flooding Rates in Unpulsed Column.

Flow rates in liters per hour	
H_2O phase	CCl_4 phase
32.3	11.83
11.5	24.9
14.2	24.2
24.1	19.0
20.2	21.2
18.0	21.6
14.0	22.9

The data are illustrated in Figure 3.

According to literature reported by Treybal (18), the sum of the square roots of the linear superficial liquid velocities (based of the empty tower cross section) at flooding should give a straight line for each system of liquids. It was furthermore shown that the sum of these two square roots is nearly a constant for each system.

In the graph, Figure 3, the sum of the square roots of these velocities is also shown. It appears nearly a horizontal line, as expected.

3. Flooding Rate for the Pulsed Column.

The data for the flooding rates for the pulsed column with well settled packing are given in Table 3. With pulsation the reported instabilities were even greater than in the unpulsed column. The results are correspondingly less reproducible. The pulse frequency was always 125 strokes per minute.

TABLE 3.

Flooding Rates in Pulsed Column.

Pulse Amplitude cm	Flow rate liters per hour H_2O	CCl_4
0	20.7	19.0
0.50	18.9	19.8
0.65	19.2	18.5
0.88	16.3	18.7
1.15	13.2	18.9

The data are plotted in Figure 4. The plot of the square roots of the velocities in this case appears to be a slightly curved line.

4. Variation of Performance with Pulse Amplitude.

The investigation was performed at constant flow rates and variable pulse amplitude. The frequency of the pulsation was 125 strokes per minute, the well settled packing consisted of 0.78 cm Raschig rings, the height of the packing was 81 cm, the continuous phase was water, which was also the extractant. The flow rate of the carbon tetrachloride was 18.9 liters, and the velocity of the water 13.2 liters per hour. The temperature was 30° C., constant within 2° C.

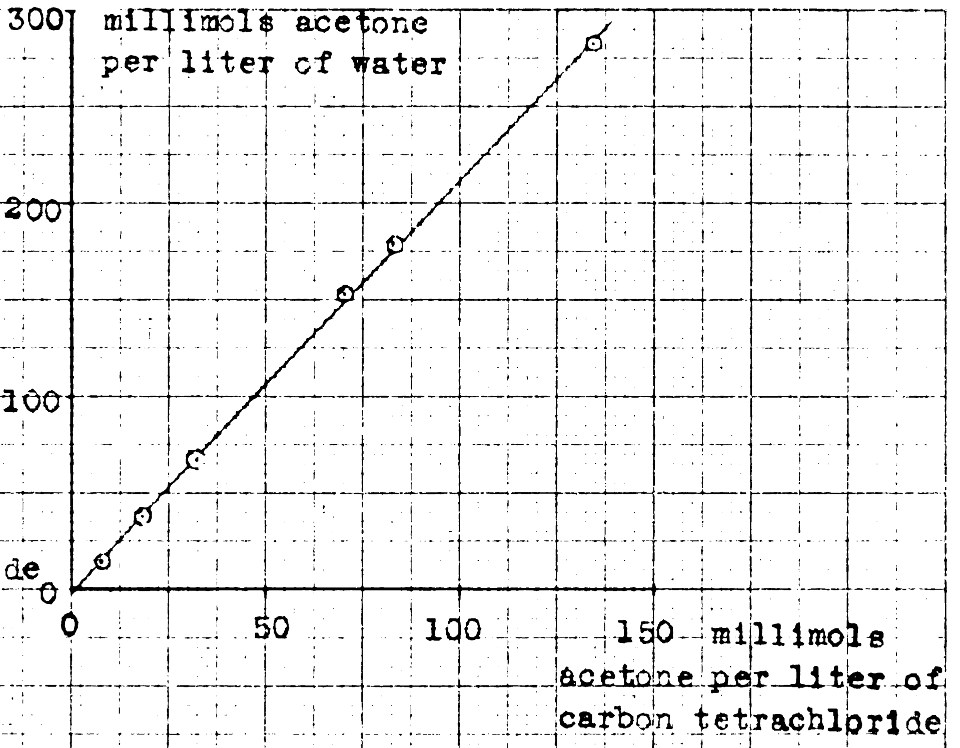
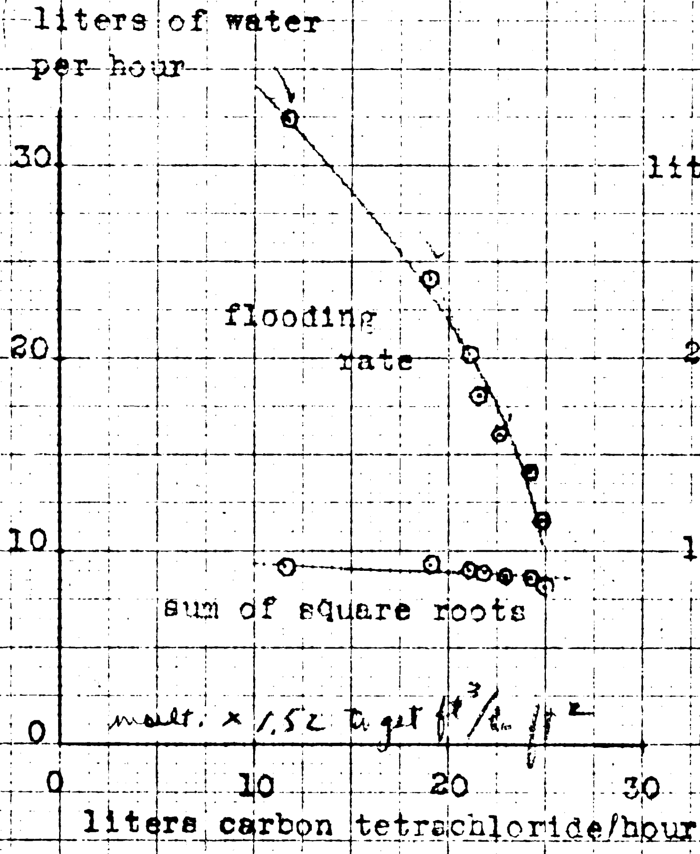


FIGURE 2

Distribution of Acetone in Water and Carbon Tetrachloride at 30° C.



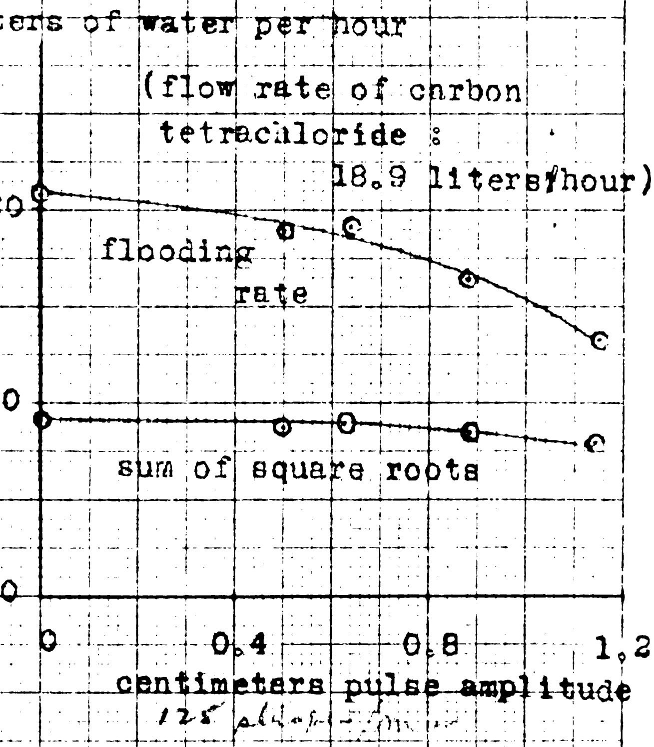


FIGURE 3

Flooding Rates for Unpulsed Column

FIGURE 4

Flooding Rates Pulsed Column

18.9 l CCl4
18.2 l H2O

packed height 81 cm
125 sec/min

cont phase H2O

used small packing

TABLE 4.

Variation with Pulse Amplitude.

Run No.	Pulse Amplitude cm	Acetone, millimols/liter in H2O in	out	in CCl4 in	out	Number of ideal stages	H.E.T.S. cm
1	0.00	0	214.5	218.8	70.0	1.33	60.8
2	0.28	0	241	220.5	53.3	1.77	45.7
3	0	0	233	238	75.9	1.327	61.0
4	0.43	0	276	235.5	47.1	2.13	38.0
5	0	0	243	214	77.3	1.34	60.4
6	0.48	0	259	217	44.9	2.25	36.0
7	0	0	199	204.5	66.1	1.31	61.8
8	0.60	0	252	208	33.5	2.51	32.2
9	0	0	179	188.5	61.0	1.31	61.8
10	0.82	0	242	185	16.98	3.62	22.4
11	0	0	192	195.8	62.5	1.32	60.8
12	0.98	0	256.5	188	9.98	4.94	16.7
13	0	0	204.5	208.5	67.4	1.31	61.8
14	1.15	0	318	231.5	11.15	5.01	16.1
15	0	0	203.5	209.4	66.1	1.34	60.4
16	1.30	0	307.5	224.5	11.37	4.93	16.4
17	0.90	0	312	227	20.1	4.28	18.9
18	0.90	0	274	198.1	14.57	4.10	19.7

The data are represented in the graph, figure 5. Flooding of the column started at the pulsation of about 1.1 cm. At the same amplitude the packing as a whole started to get lifted up by the pulsed liquid. For these reasons there is a break in the curve representing the number of ideal stages.

6. Effect of Flow Rates in Pulsed Column.

In this test the variation of the performance of the pulsed column with flow rate was investigated. The pulse amplitude was fixed at 0.90 cm. All other conditions were as given before in Table 4.

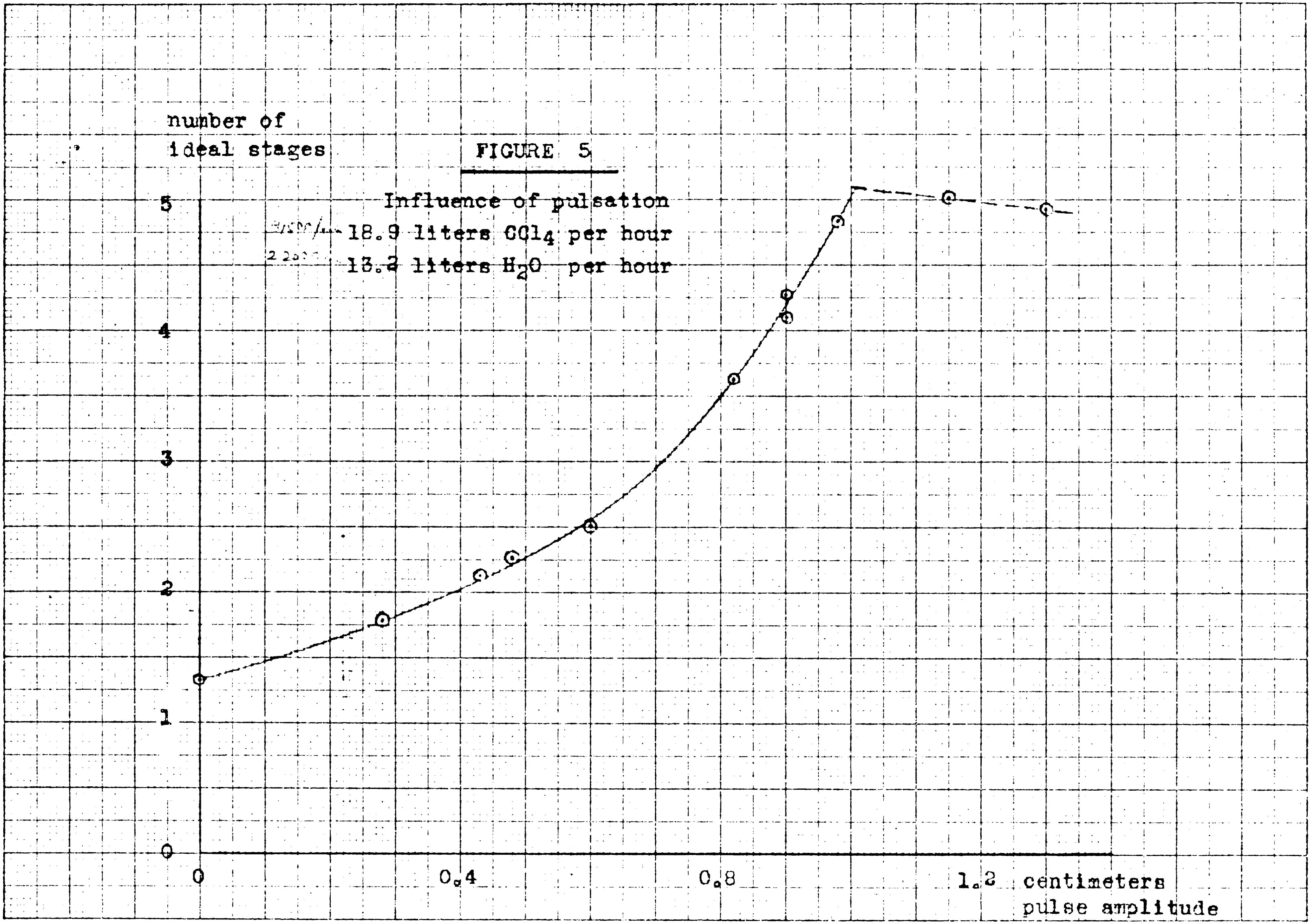


FIGURE 5

Influence of pulsation

18.9 liters CCl_4 per hour

13.2 liters H_2O per hour

TABLE 5

Pulsed Column with Variable Flow Rates.

Run No.	Flow rate liters/hour		Acetone, millimols/liter				Number of ideal stages	H.E.T.S. cm
			in H2O		in CCl4			
	H2O	CCl4	in	out	in	out		
	C	D						
19	9.00	18.9	0	303.0	191.4	47.0	3.08	26.3
20	6.58	19.3	0	316	175	68.1	2.82	28.7
21	2.86	19.1	0	327	192	14.29	1.16	69.8
7 — 22	3.27	18.0	0	323	181	122.2	1.51	- 53.6
23	13.10	18.8	0	234.5	198.1	14.57	4.12	19.6
24	12.55	8.80	0	174	257	7.55	2.80	28.9
25	13.27	4.28	0	149	457	6.00	2.23	36.3
26	12.57	4.77	0	141	402	4.63	2.49	32.5
27	12.12	5.09	0	159	455	4.8	2.66	30.4
28	12.60	5.77	0	126.5	260	3.1	2.78	29.1
8 — 29	11.88	10.43	0	224	265	10.3	3.02	- 26.4
9 — 30	13.33	3.20	0	67.2	282	6.87	1.66	- 48.4
31	13.1	18.8	0	319	297	20.1	4.28	18.9

The results of this table are plotted in Figures 6 and 7, together with more data.

A discontinuity at a flow rate of 5 to 6 liters per hour of solvent may be observed. Apparently at least two influences are involved in determining the performance of the column.

6. Effect of Flow Rates in Unpulsed Column.

The operating conditions of Table 5 have been reproduced with the exception that there was no pulsation and the flow rates were varied.

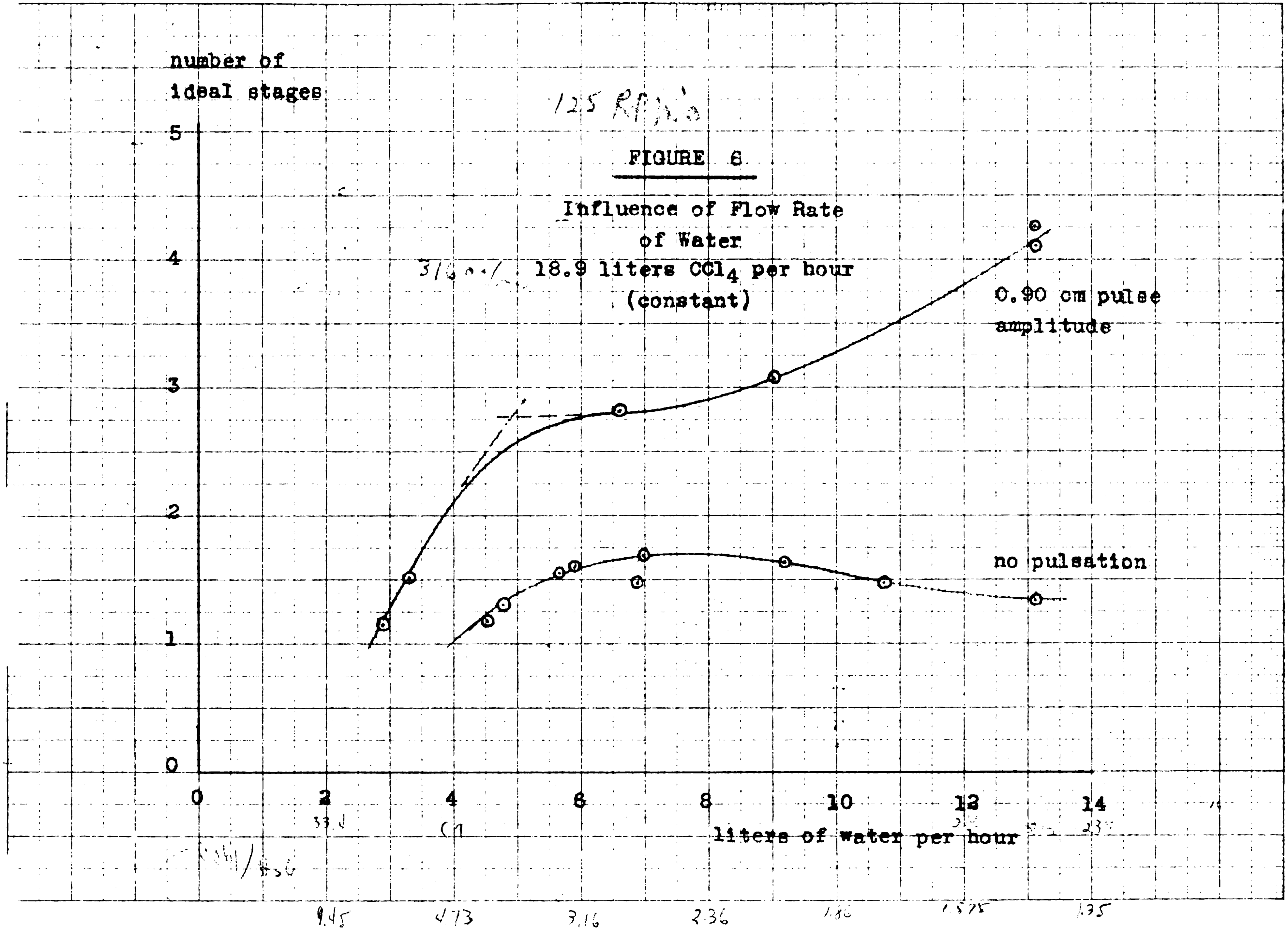


FIGURE 6

Influence of Flow Rate of Water

18.9 liters CCl_4 per hour (constant)

TABLE 6

Unpulsed Column with Variable Flow Rates.

Run No.	Flow rate liters/hour		Acetone, millimols/liter				Number of ideal stages	H.E.T.S. cm
			in H2O		in CCl4			
	H2O	CCl4	in	out	in	out		
32	12.85	5.79	0	87.1	223.0	33.1	1.10	73.6
33	12.2	2.89	0	44.5	227.5	24.7	0.96	84.3
– 34	13.2	13.2	0	189.8	250	60.4	1.29	– 62.8
– 35	12.6	3.03	0	23.4	131.3	20.6	0.98	– 82.7
36	13.1	5.7	0	64.4	172	26.5	1.06	76.4
37	13.1	18.8	0	293.5	209.4	66.1	1.33	60.9
38	6.87	18.55	0	345	212	114.0	1.49	54.3
39	10.7	18.0	0	278	244	81.4	1.49	54.3
40	6.93	18.2	0	275	185.1	80.8	1.69	47.9
41	5.87	18.7	0	233.5	149.3	76.1	1.61	50.3
42	5.63	18.0	0	231	150.0	77.8	1.55	52.2
43	9.16	18.9	0	302	236.5	90.2	1.65	49.1
44	4.75	18.9	0	230	148.3	90.6	1.30	62.3
12 – 45	4.51	18.0	0	277	146	91.2	1.10	– 68.6
46	13.1	13.6	0	243	250	78.5	1.35	60.0

The results of this table are plotted together with the data of Table 5 on Figures 6 and 7.

In case of a constant flow of water and a variable rate of carbon tetrachloride, the efficiency of the unpulsed column is slightly increased by increasing the rate of the carbon tetrachloride, which is the dispersed phase.

In the case of a constant flow of carbon tetrachloride and a variable rate for the water, which is the continuous phase, a maximum in the efficiency of the column could be found. At very low speeds there seems to be a very poor efficiency, probably because of mixing of the continuous phase. At high speeds there is a slight decrease in efficiency up to the point where flooding conditions are reached.

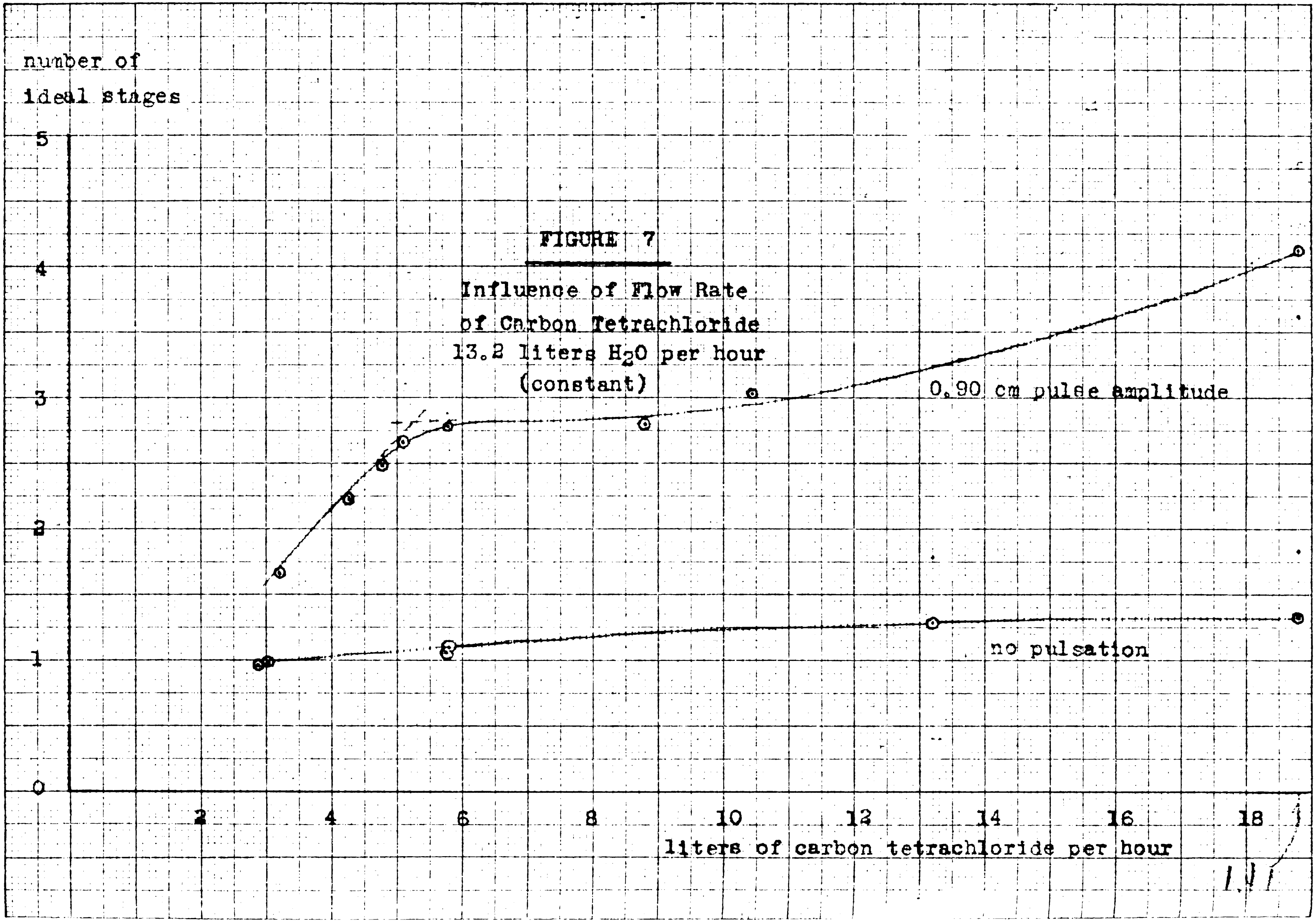


FIGURE 7

Influence of Flow Rate
of Carbon Tetrachloride
13.2 liters H_2O per hour
(constant)

7. Performance with loosely Filled-in Packing.

In the early stages of this investigation it was observed that some of the runs at low pulsation could not be reproduced after high pulsation runs had been made. This gave the impression that the difference had been caused by settling of the packing during pulsation of the liquid phase at high amplitudes.

It was found that the volume of the packing was reduced while pulsing the column with carbon tetrachloride. To obtain a proper packing the column was first filled with liquid and the Raschig rings were then poured into the liquid. Because of the densities of pyrex glass and carbon tetrachloride being not very different, the Raschig rings bounced at pulse amplitudes of about 1.0 cm, and by this process the packing as a whole lost in volume about 15 percent within one hour of pulsing.

To obtain a quantitative picture of this influence, some test runs were made. All conditions were the same as those used to obtain the data of Table 4, except that the packing was now prepared by simply pouring the Raschig rings into the water-filled column.

TABLE 7.

Run No.	Pulse Amplitude cm	Acetone, millimols/liter in H_2O in	out	in CCl_4 in	out	Number of ideal stages	H.E.T.S. cm
47	0	0	97.4	117.2	40.6	0.92	88.0
48	0.40	0	108.0	114.5	39.8	1.19	68.0
49	0	0	185.4	200.3	91.5	0.95	86.1
50	0.55	0	218.4	217	66.7	1.37	59.1
51	0	0	225	260	103.8	0.98	82.7
52	0.87	0	285	262	63.9	1.75	46.2
53	1.05	0	342	292.7	55.7	2.18	37.2
54	1.10	0	322	270	48.5	2.36	53.3

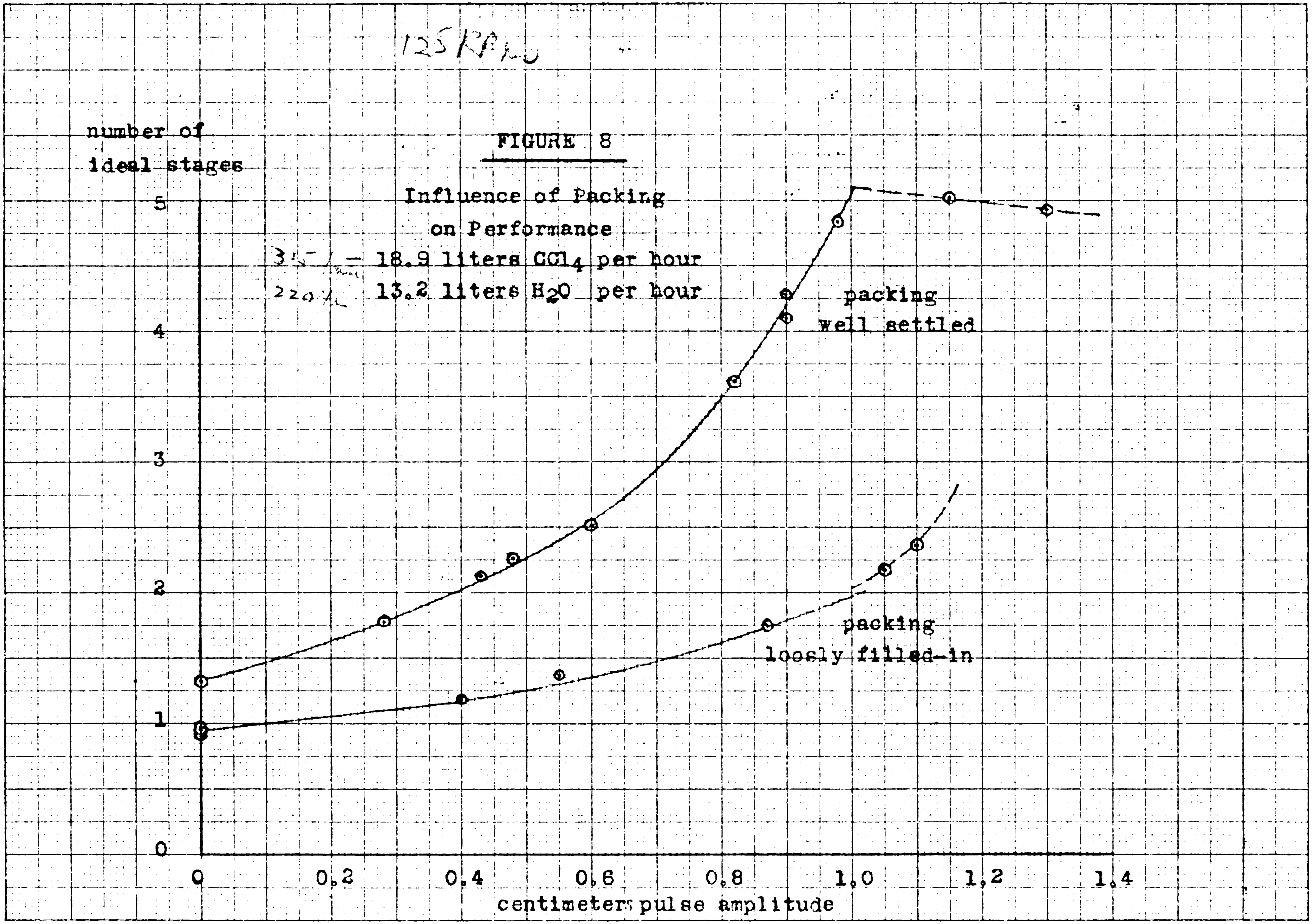


FIGURE 8

Influence of Packing
on Performance
35 l/hour — 18.9 liters CCl_4 per hour
220 l/h 13.2 liters H_2O per hour

The data are shown graphically in Figure 8, in which the data for Table 4 are represented again for comparison. The investigation could not be extended to higher pulse amplitudes because the packing started to settle at pulse amplitudes of about 1.0 cm.

V. DISCUSSION OF RESULTS

As pointed out in advance, this investigation could only cover a few of the tests required to explain the behavior of a pulse extraction column. These few tests show that pulsation in extraction columns might have a considerable economic interest.

In the data reported, the number of ideal stages was increased by pulsation up to 3.5 times the number obtained without pulsation under the same conditions. Greater improvements can be expected after further investigation of operating conditions such as pulse frequency, pulse amplitude, or type of packing in the column.

The efficiency of the column was steadily increased with increasing pulse amplitudes at given pulse frequency and given flow rates of the solvents up to the point at which flooding occurred. The height equivalent of an ideal stage was reduced, due to pulsation, from 60 cm (23.6 in.) down to 16 cm (6.3 in.) at flooding. The flow rates of the solvents under these conditions were 18.9 liters per hour for carbon tetrachloride and 13.2 liters per hour for water (890 cm or 29.2 ft. per hour velocity for the carbon tetrachloride in the empty tower and 622 cm or 20.4 ft. per hour for the water which was the continuous phase). Decreasing the flow rate of water at a constant rate for carbon tetrachloride of 18.9 liters per hour also decreased the efficiency of the tower in case of 0.9 cm pulse amplitude. In case of no pulsation there was a maximum efficiency of the column near 8 liters water per hour.

In case the amount of carbon tetrachloride was decreased at a constant speed of 13.2 liters water per hour, there was a decrease in the number of ideal stages in both cases with 0.9 cm pulsation and also without pulsation. At a flow rate of 5 to 6 liters carbon tetrachloride per hour and with 0.9 cm pulsation there was a discontinuity in performance as shown in Figure 6, which reminds that more than one influence controls the efficiency of the column.

Feick and Anderson (8) report an improvement due to pulsation up to a factor of 15, but where such improvements were observed, the height of a transfer unit without pulsation was of the order of 10 ft. However, where the initial height of a transfer unit was similar to the values in this report, the improvement due to pulsation was also similar to the improvements of this investigation.

It seems that with pulsation a bigger diameter of packing can be used without losing too much efficiency.

A comparison of the data of Tables 4 and 7, which are reproduced in Figure 8, shows clearly the effect of settling of the packing on efficiency. A loose packing gives a considerably poorer performance than a packing which has been settled well by pulsation.

This may partly explain why data reported in the literature about the performance of packed columns are often in such a considerable disagreement.

The increase of efficiency of the column due to pulsation might be attributed to more than one phenomenon. It was easy to see differences in the appearance of the column. Without pulsation big droplets streamed down the column, but with pulsation, especially at high amplitude, these droplets were several times smaller, and these smaller droplets bounced up and down in the pulsed continuous phase. While many theories might be advanced on the basis of this observation, it is not felt that any of these could be substantiated without further experimentation.

VI. SUMMARY

As the first step, to a more thorough study of the performance of packed pulsed columns, an apparatus was designed, consisting mainly of a column and a pulsator.

The column had a diameter of 5.2 cm (2 in.), and the length of the packing was 81 cm (32 in.), consisting of Raschig rings with 0.78 cm diameter. The pulsator consisted of a cam-driven bellows, which applied the pulsating motion to the liquid phase.

In this investigation only to a few of the interesting variables could be given attention, and the rest had to be left for future studies.

The results showed that the efficiency of the column was increased with increasing pulse amplitude, up to the point where flooding occurred. The height equivalent to a theoretical stage was reduced due to pulsation from 60 cm (23.6 in.) to 16 cm (6.3 in.) at a pulse amplitude of 1.0 cm and at superficial velocities of 890 cm (29.2 ft.)

per hour for carbon tetrachloride and 622 cm (20.4 ft.) per hour for water.

The efficiency of the pulsed column increased in every case when the flow of either solvent was increased. This was also true for the unpulsed column except at high throughput where a slight decrease in efficiency occurred with increasing water rate.

Pulsation reduced the capacity of the column slightly. At high pulse amplitude the flow rates at which flooding occurred were about 85 percent of those at which flooding occurred without pulsation.

The condition of the packing had a considerable influence on performance. The well settled packing gave more theoretical stages for the same height than a loosely filled-in packing. The ratio was 1.24 : 1 without pulsation and 2.5 : 1 at high pulse amplitudes.

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