CHAPTER 5

RESEARCH METHODOLOGY

5.1 Apparatus

The schematic flow diagram is shown on Fig.9 on page 34. The experimental apparatus consisted of

5.1.1 Packing specimens

5.1.2 Column.

5.1.3 Measuring and Auxiliary equipments

5.1.1 Packing specimens : The packing materials used were raschig-type rings of 1/2-in.size. Two types of materials were selected each with different wetting characteristics as given in Appendix D ; these were stainless steel and plastic tubing cut into rings with length equal to the diameter. The plastic rings were added to the empty column and held in place by themselves as no buoyancy effectswere noticed. The plastic rings appeared to be unaffected by the carbon tetrachloride even after considerable use. The stainless steel rings were dumped into the column which had previously been filled with water. See fig. 10 and 11.

5.1.2 Column : The pulsed extraction column shown schematically in Fig.9 shows the device used for measurement of flow rates and for level interface control. The column consisted of a 4-ft. section of 2-in. standard glass pipe fitted with a PVC tee at both ends as disengaging sections. Plexiglas flanges were used to join

the 2-ft. tubes together (see Fig.12). Figure 13, 14, and 15 show the pulsing mechanism provided by a pneumatic pulser using compressed air. The stoke and frequency of the pulser could be easily controlled.

5.1.3 Measuring and Auxiliary equipments :

1) Rotameters : A rotameter was used to measure flow rates of dispersed phase (carbon tetrachloride) and continuous phase (water). For the dispersed phase, a rotameter with a ruby ball float inside the measuring tube was used. Flow range of this rotameter could be varied from 0 to 4.0 liter/hr. at room temperature. For the continuous phase, a rotameter with a glass ball float was used with flow ranges between 0 and 60 liter/hr. also at room temperature.

2) Pulser : The pulser was a pneumatic type pulser manufactured by L'Equipment Industriel en Verres Speciaux. Frequencies ranges between 15 to 85.7 pulse per minute. See Fig.16.

3) Pumps : An Arno pump type MC 20 was used to pump the continuous phase to the head tank with the following characteristics: 220 volts, 50 Hz ., 1.7 A , 0.35 HP For the dispersed phase, a magnetic pump of 0.25 HP was used.

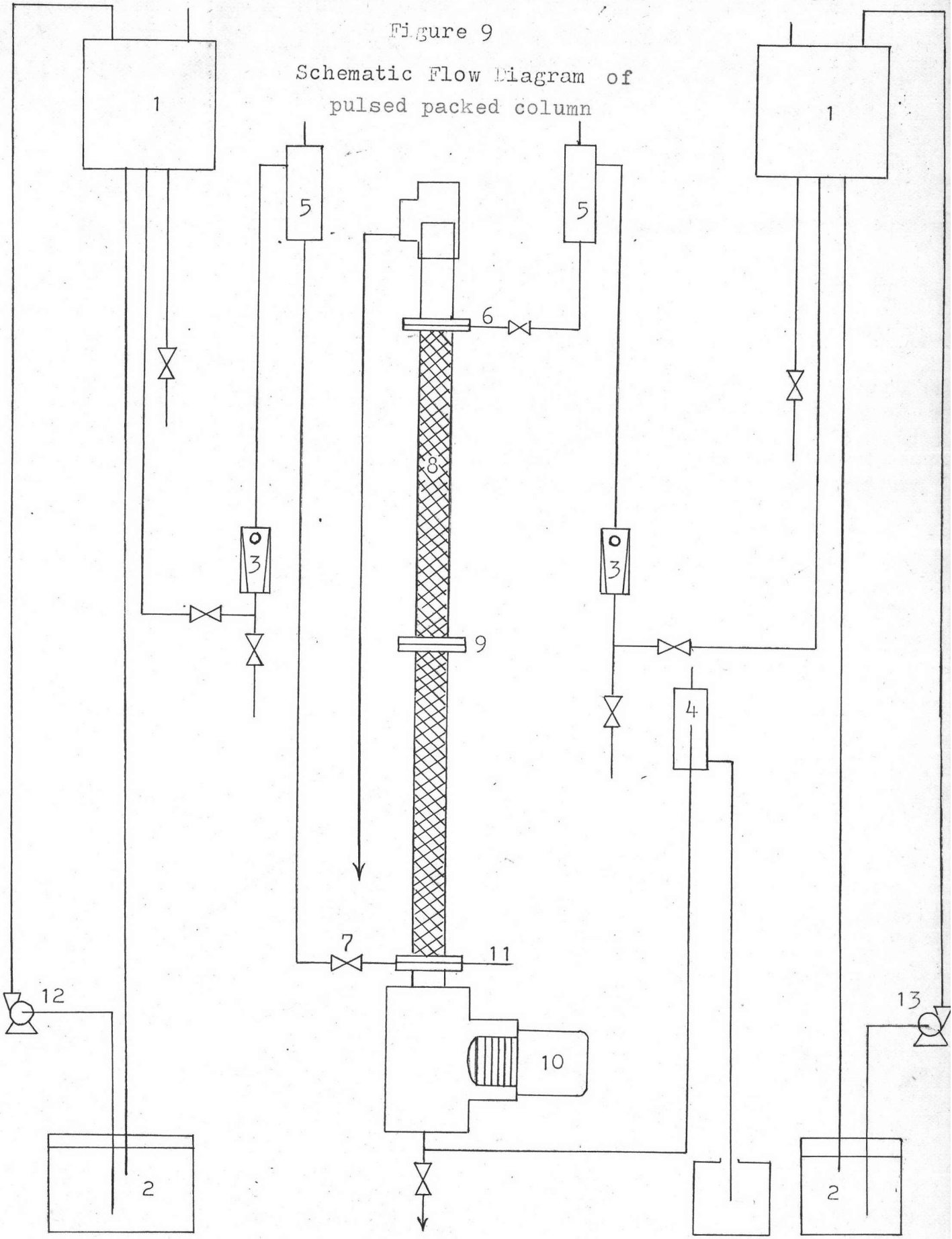
4) Compressor : The air compressor used was a CS-490N, SU type. This is a single stage compressor utilizing an automatic unloader. This type of compressor

was suited where continuous operation of the compressor is necessary. The automatic unloader automatically operates to open the intake valve of the compressor, thus causing the compressor to run idle, while the compressor contunues revolving and the automatic unloader causes the intake valve of the compressor to close and the compressor continues to function normally. Pressure supply from this compressor is 90 psig and can be reduced to the desired pressure by using a pressure regulator (oxygen pressure gauge type).

5) Titration apparatus : The titration apparatus consisted of a burette and a microburette containing sodium thiosulfate (0.1N), volumetric cylinders, starch solution as indicator and flasks.

Figure 9

Schematic Flow Diagram of
pulsed packed column



Flow diagram description

1. Head tanks
2. Storage tanks
3. Rotameters
4. Interfacial level control
5. Feed control tubes
6. Carbon tetrachloride feed pipe
7. Water feed pipe
8. Transfer section with packings
9. Plexiglas flanges
10. Pulser
11. Sample lines
12. Centrifugal pump
13. Magnetic pump

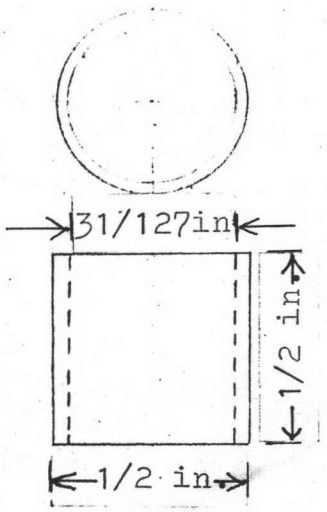


Figure 10 Dimension of Raschig ring packings

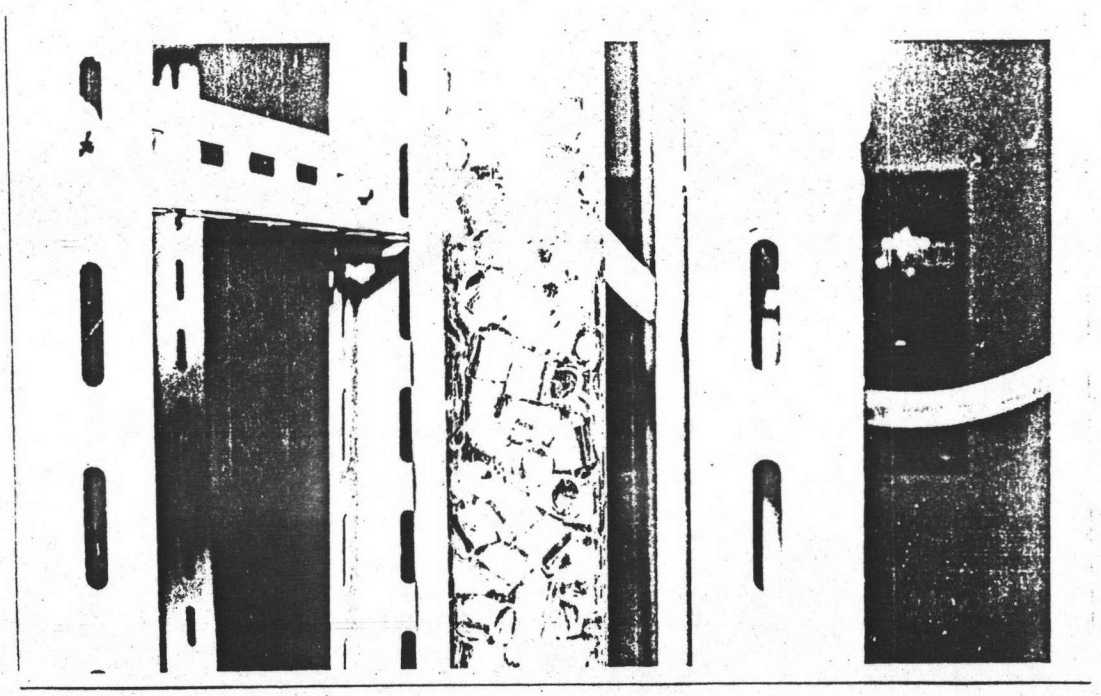


Figure 11 Stainless steel packings inside column

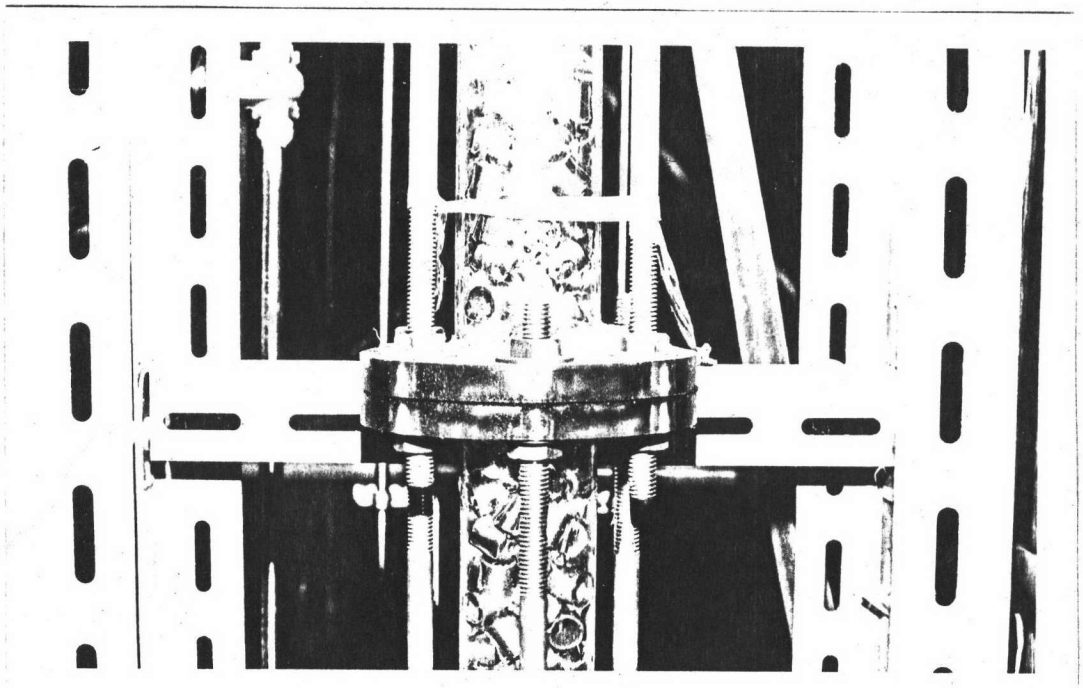


Figure 12 Plexiglas flanges

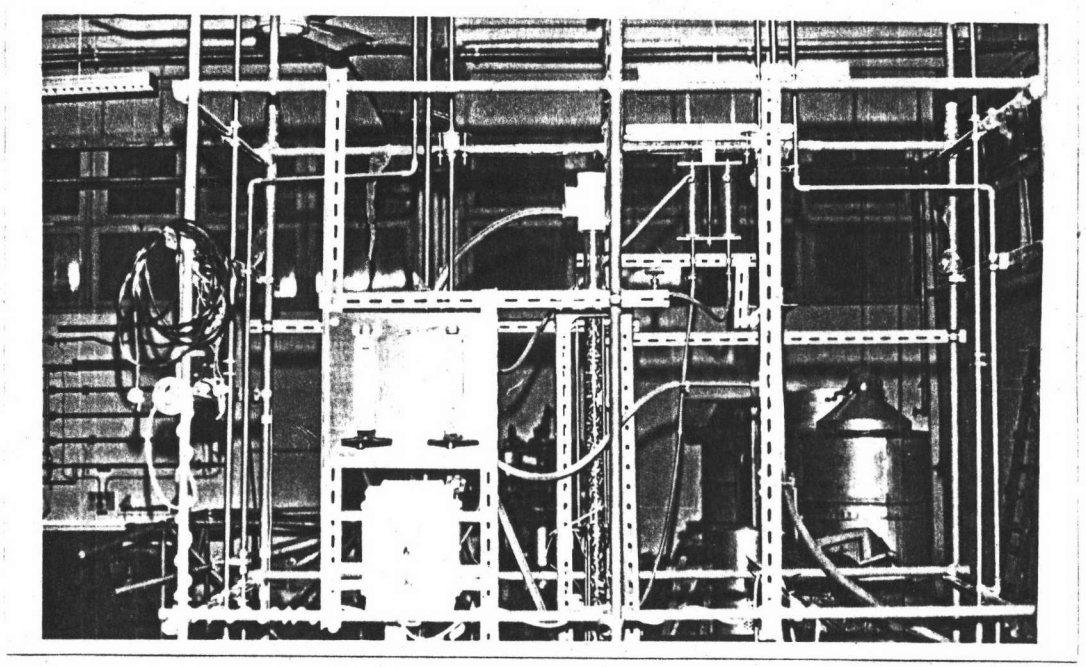


Figure 13 The upper parts of column

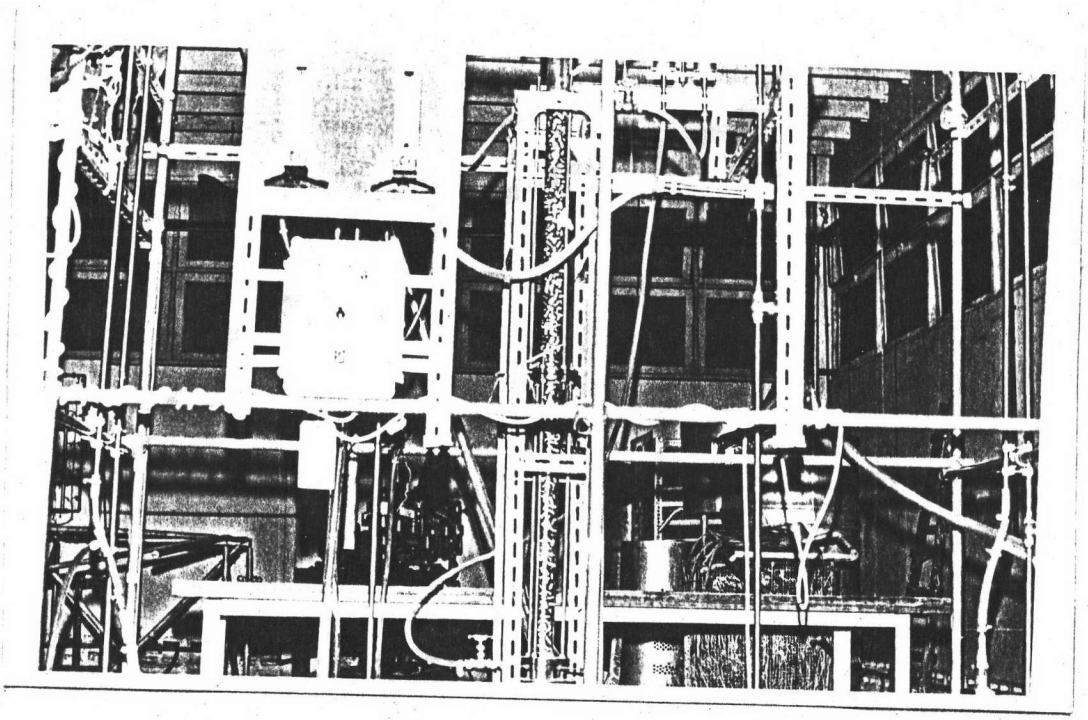


Figure 14 The transfer section

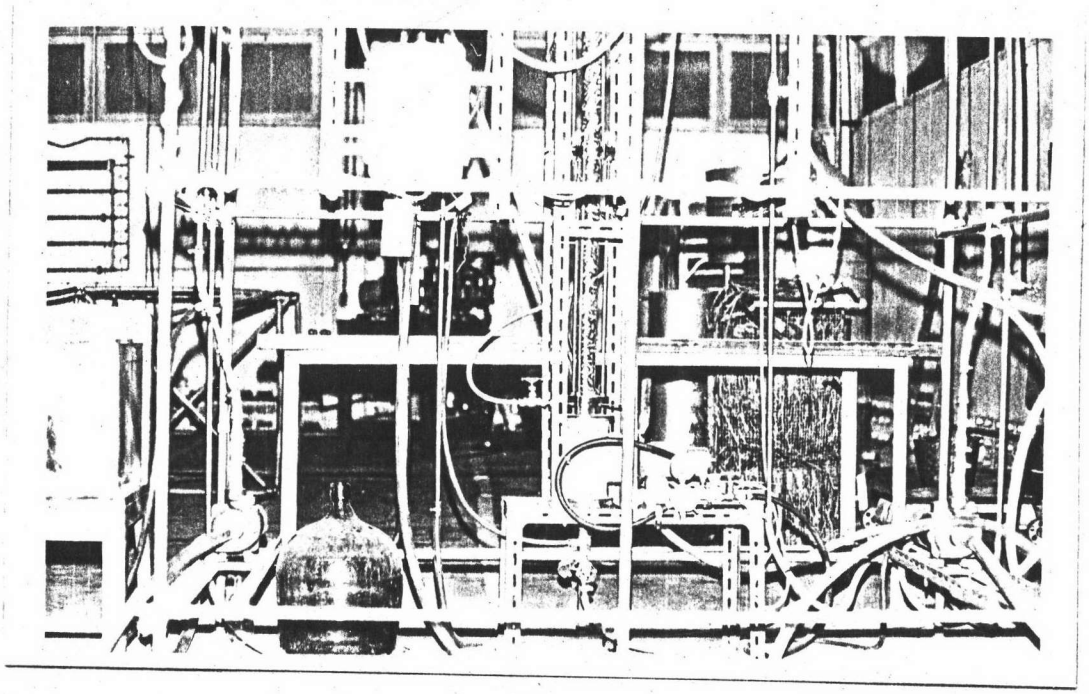


Figure 15 The bottom parts of column showing pneumatic pulser and air regulator.

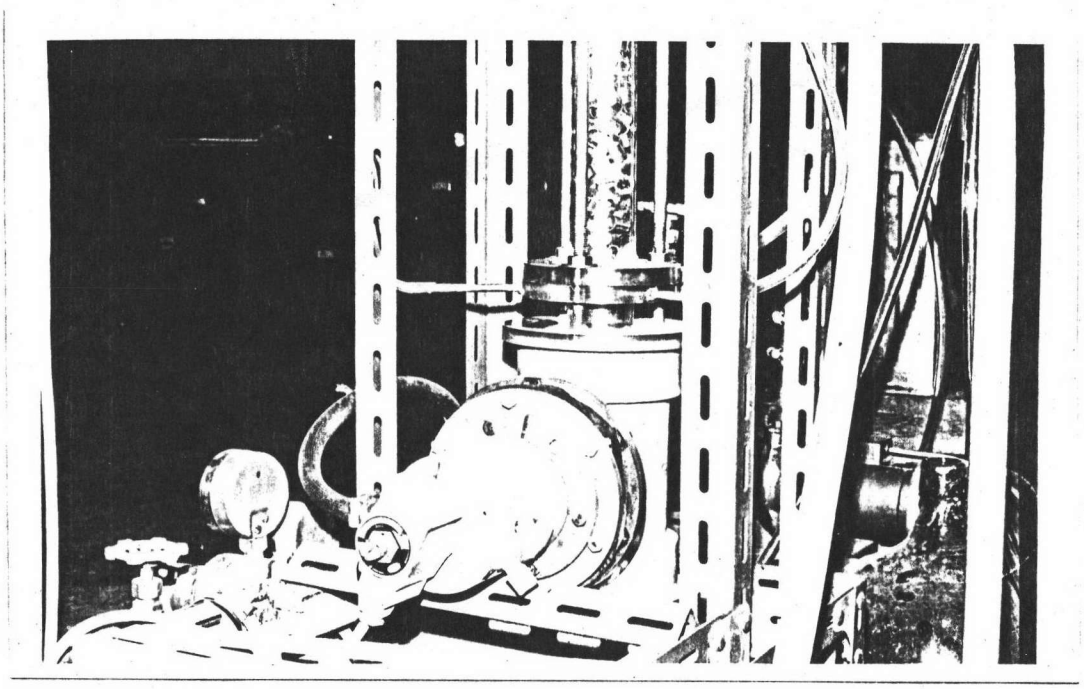


Figure 16 Pneumatic pulser

5.2 Procedure

1/2-in. stainless steel and plastics were selected as packing for the determination of flooding capacities and efficiencies of the pulsed packed liquid-liquid extraction column.

The water-carbon tetrachloride system was used for the determination of flooding capacities. The column was first filled up with water which enters from the bottom part of the column and carbon tetrachloride is fed at the top. In this case carbon tetrachloride flows as a dispersed phase and falls in the column. Drop characteristics depend upon the influence of pulsation energy provided to the column. Random packing effect was first determined for flooding characteristics by using stainless steel rings at amplitude(a) = 1 cm., varying frequency(f) from 22 to 85.7 pulse per minute or the af values in the range of 0.37 to 1.43 cm/sec. About 630 pieces of stainless steel rings were used in the entire column.

The flooding capacities of stainless steel and plastic rings were determined for a=2, 3 and 4 cm. and f from 22 to 85.7 pulse per minute or af values in the range of 0.73 to 5.71 cm/sec. The flow rates of the continuous phase(water) used was 12.6 liter/hr. and the dispersed phase flow rates (carbon tetrachloride) was varied until flooding occurred.

The water-iodine-carbon tetrachloride system was used for determining the column efficiencies using the same packings. Aqueous iodine solution was prepared by dissolving iodine flakes into water until the desired concentration is obtained (see Appendix C) at 270 mg/l . Aqueous iodine solution as continuous phase was injected into the column instead of water. Carbon tetrachloride was fed at the top of column. The pulser was turned on, the amplitude and frequency adjusted to the desired value. The system was left to reach the steady state after about 30-45 min. then samples were taken and titrated with 0.1N $Na_2S_2O_3$ in the presence of starch solution as indicator.

Concerning techniques of the efficiency measurements,[31] to calculate the efficiency of a column, it is necessary to know the concentration difference between the phases in and out. The flow ratesof aqueous iodine solution and carbon tetrachloride chosen affect the efficiency measurement of the column. The concentration of iodine in the aqueous phase is 270 mg/l .and the carbon tetrachloride employed was a 99.5% technical grade. Sampling of the aqueous phase was made in the part of the intering tube and in the section of the raffinate output (at the entering level of carbon tetrachloride). The concentration of iodine in the aqueous phase was determined by titration with a solution of 0.1N $Na_2S_2O_3$ using the titration equipment. The preparation of 0.1N $Na_2S_2O_3$ is presented in Appendix C.

The efficiency determination was made at a=2, 3 and 4 cm., f from 22 to 85.7 pulse per minute or af values in the range of 0.73 to 5.71 cm/sec. The continuous phase flow rate 12.6 liter/hr., and the dispersed phase flow rates0.5, 1.0 and 2.0 liter/hr. were given.

The sum of the flow rate of both liquids are given at 50-65% of the flooding limits by the Spaay[15] suggestion which put column operation in the emulsification regime where efficient results are obtained. The extraction factors (E) were determined for a continuous phase flow rate (F_c) of 12.6 liter/hr. and by varying dispersed phase flow rate (F_d) from 0.5 to 2.0 liter/hr. The extraction factors used were 3.56, 7.11 and 14.22 respectively.