



CHAPTER 5

CONSTRUCTION OF EXPERIMENTAL APPARATUS

5.1 Apparatus construction

As shown in Fig. 5.1, nearly all of the main lines outside the gas chromatograph were constructed from 1/8 inch o.d. copper tubes and various Swagelok brass valves and fittings, whereas seamless stainless steel tubes and fittings were used to connect with the joints in the six-way sampling valve and inside the chromatographic oven. A 1/16 inch o.d. stainless steel tube was used between the sampling valve and the column as well as between the column and the detector to reduce the "dead" volumes existing in the connecting lines. The adsorption column was made of 3/16 inch o.d. stainless steel tube (internal cross sectional area = 0.0876 cm^2). In this study, the Shimadzu 9-APF gas chromatograph with a flame ionization detector was used, and its output signal was calculated with the aid of a Shimadzu Chromatopac 6A integrator.

5.2 Adsorbent

The NaY and Offretite/Erionite zeolite samples employed in this study were obtained from the Toyo Soda Manufacturing Co., Ltd. and carried the designations, TSZ-320NAA and TSZ-410KOA (see Table 5.1). The original zeolite powder was pilled without binder, crushed, and sieved to the desired mesh size. The properties of the crushed pellets are given in Table 5.2.

Table 5.1 Chemical composition of NaY and Offretite/Erionite zeolites

| Zeolite Chemical Composition | NaY | Offretite/Erionite |
|---------------------------------------------------------------|------------|--------------------|
| | TSZ-33ONAA | TSZ-410KOA |
| SiO ₂ (wt%) dry basis | 66.7 | 70.8 |
| AlO ₂ (wt%) dry basis | 20.3 | 15.6 |
| Na ₂ O (wt%) dry basis | 12.3 | 2.36 |
| K ₂ O (wt%) dry basis | — | 11.2 |
| SiO ₂ /Al ₂ O ₃ (mole ratio) | 5.6 | 7.7 |
| Na ₂ O/Al ₂ O ₃ (mole ratio) | — | 0.25 |
| K ₂ O/Al ₂ O ₃ (mole ratio) | — | 0.78 |
| LOT NO. | 55Y-D1020 | GZ-131-1,2 |

Table 5.2 Properties of zeolite pellets

| Zeolites Properties | NaY | Offretite/Erionite |
|-----------------------------------|--------|--------------------|
| Pellet density, g/cm ³ | 0.9682 | 0.5018 |
| Intrapellet void fraction, - | 0.338 | 0.3702 |

Table 5.3 Characteristics of Packed Columns

| Properties | NaY column | | | Offretite/Erionite column | | |
|----------------------------|------------|-------|-------|---------------------------|--------|--------|
| | 1 | 2 | 3 | 4 | 5 | 6 |
| Weight (g) | 0.6504 | 0.638 | 0.643 | 0.3124 | 0.3031 | 0.3126 |
| Length (cm) | 12.45 | 12.00 | 11.79 | 12.38 | 11.97 | 12.08 |
| Mesh size | 40-50 | 50-60 | 60-80 | 40-50 | 50-60 | 60-80 |
| Average particle size (mm) | 0.558 | 0.425 | 0.339 | 0.558 | 0.425 | 0.339 |
| Bed void fraction | 0.384 | 0.373 | 0.357 | 0.426 | 0.424 | 0.411 |
| Temperature range (°C) | 100 | 150 | 150 | 200 | 250 | 250 |
| | 150 | | | 225 | | |
| | 200 | | | 250 | | |

5.3 Gases

The gases used are helium of 99.995% purity from the Thai Industrial Gas Company and n-butane of 99.5% purity from the Union Carbide Company. Both gases were dried before use by passing them through the molecular sieve dryers.

5.4 Experimental procedures

For the chromatographic studies, 0.5 cm³ pulse size was selected to attach to the 6-way sampling valve to introduce the pulse. To ensure the linearity of the system, the concentration in the pulse injected was up to 0.5 mole% of n-butane in helium. The pulse was introduced into beds of particles packed in a 3/16 inch o.d. copper tube. The characteristics of various columns, including the conditions for which chromatographs were measured, are shown in Table 5.3. The temperature ranges were 100-200 °C for the adsorption of n-butane in NaY zeolite and 200-250 °C for the adsorption in Offretite/Erionite zeolite, all at atmospheric pressure. Pretreatment consisted of passing helium (at 20-25 cm³/min) through the packed column for 8 hours at 350 °C and then bringing it to the desired flow rate and temperature.

Adsorption of n-butane on a packed bed of zeolites was studied in the apparatus as follows (See Fig. 5.1). Helium and n-butane gases from the gas cylinders flowed through the pressure regulators and were dried over molecular sieve filter (F). Helium was next separated into two streams, the carrier gas stream and the diluent gas stream. The flow rate of the carrier gas stream was maintained constant at the rotameter (R) by adjusting a needle valve (NV) and periodically measured with a soap bubble flowmeter (B) via the three-way ball valve (3V). Then the carrier gas passed through

the six-way sampling valve (charge position in Fig. 5.2) into the adsorption column.

Similarly, the flow rates of the diluent gas stream and the n-butane stream were maintained constant and periodically measured. The concentration of n-butane in the combined stream was varied by adjusting the flow rate of each merging stream. The gas mixture with the desired concentration of n-butane was then passed through the sampling loop of the six-way valve and discharged. After steady-state condition had been obtained, the sampling valve was switched to the discharge position (Fig. 5.2) so that the carrier gas swept the known amount of n-butane gas mixture to the adsorption column and onto the FID detector. The output signal from the detector was then calculated and printed out on the chart paper by the integrator.

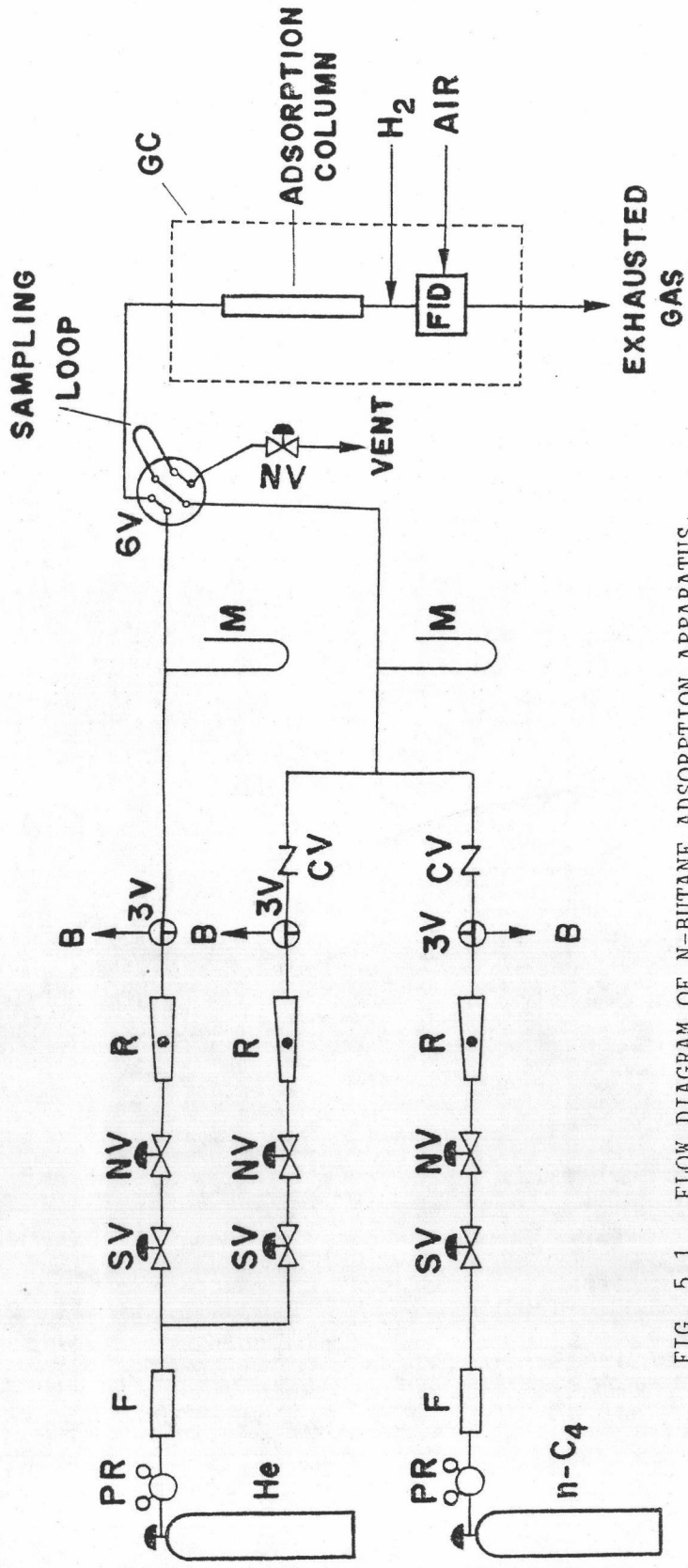


FIG. 5.1 FLOW DIAGRAM OF N-BUTANE ADSORPTION APPARATUS.

- B - BUBBLE FLOWMETER
- CV - CHECK VALVE
- F - MOLECULAR-SIEVE FILTER
- M - MANOMETER
- NV - NEEDLE VALVE
- PR - PRESSURE REGULATOR
- R - ROTAMETER
- SV - STOP VALVE
- 3V - THREE-WAY VALVE
- 6V - SIX-WAY SAMPLING VALVE
- He - HELIUM GAS
- nC₄ - N-BUTANE GAS
- GC - GAS CHROMATOGRAPH
- FID - FLAME IONIZATION DETECTOR

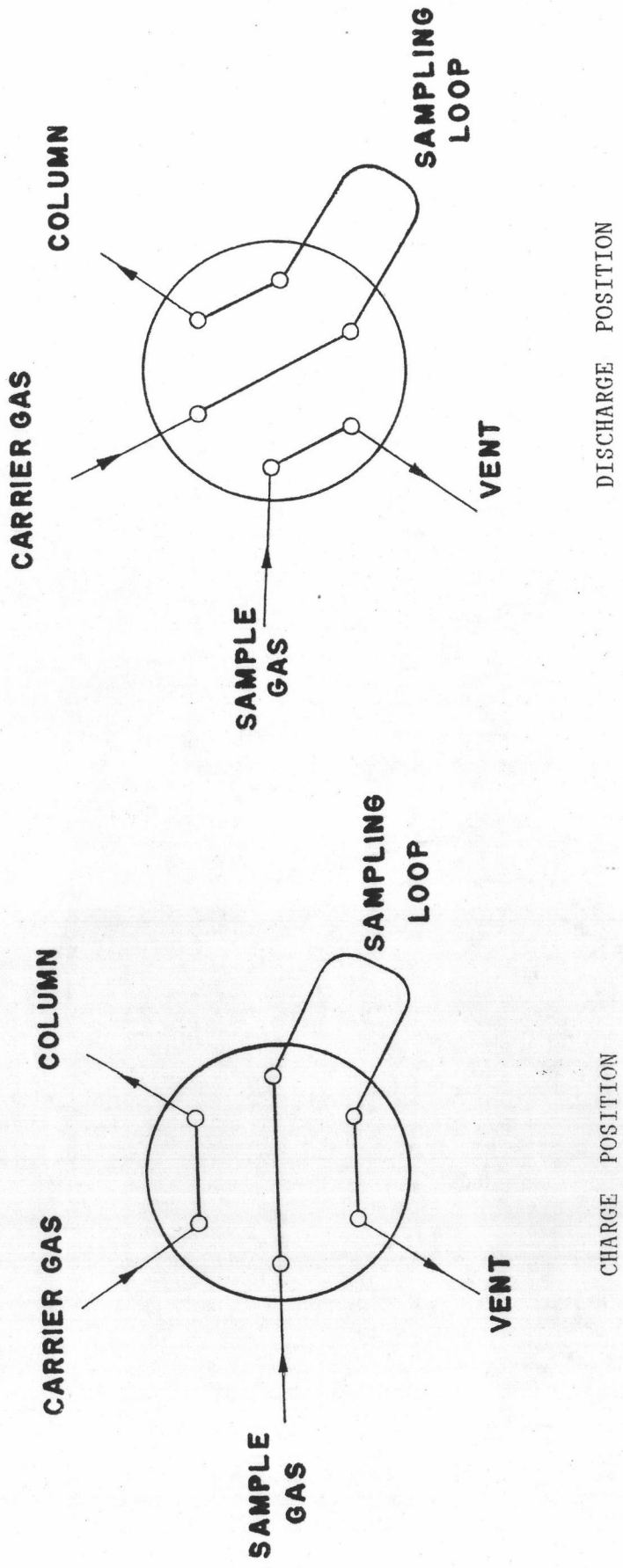


Fig. 5.2 Diagram showing charge and discharge positions of the six-way sampling valve.

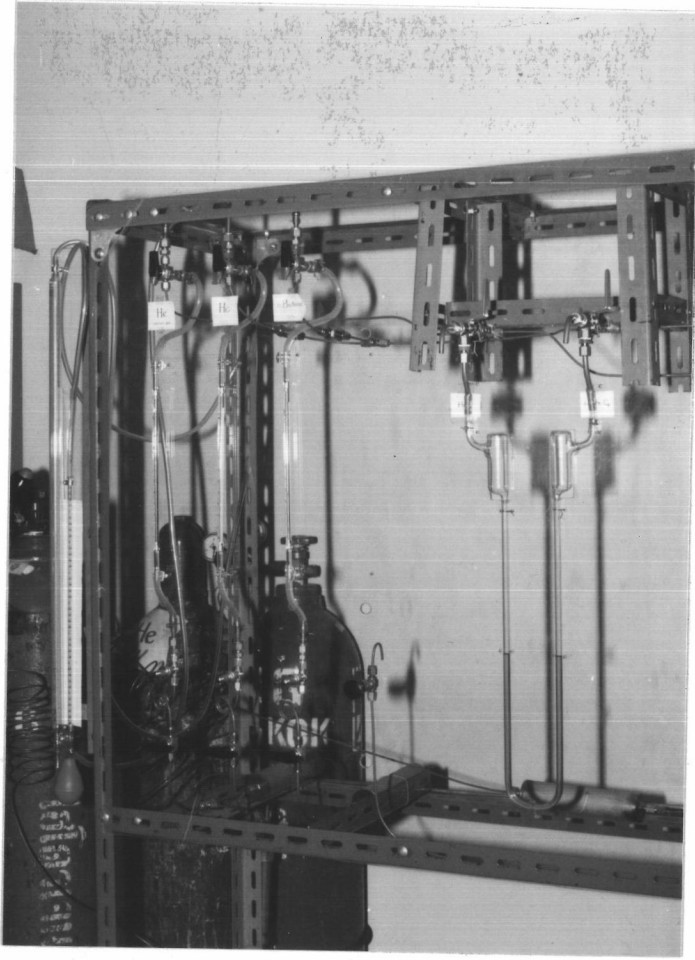


Fig. 5.3 Photograph of the n-Butane adsorption apparatus.

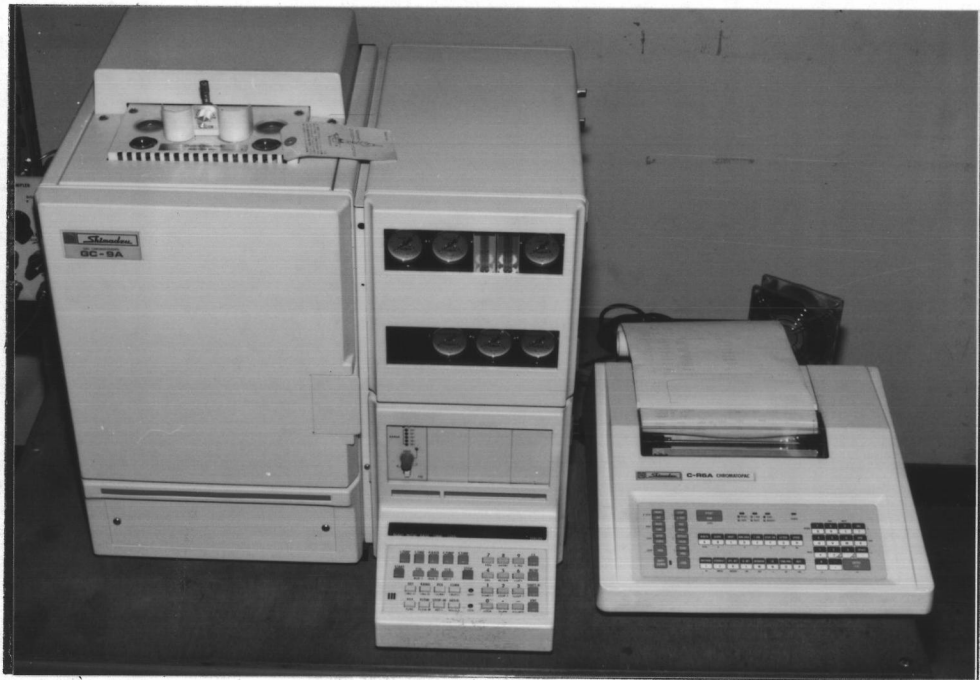


Fig. 5.4 Photograph of the gas chromatograph and the integrator.