CHAPTER 4

APPARATUS FOR DETERMINATION OF ADSORPTION ISOTHERMS AND ADSORPTION RATE CONSTANTS

4.1 Flow diagram of apparatus.

The apparatus used for the determination of adsorption rate constants and adsorption isotherms consists of five parts as follows.

1. An adsorption column
2. A gas flow meter
3. An air bath system
4. A gas analysis system
5. A column regeneration system

The adsorption column

The adsorption column used in this study is a 1/4 inch diameter 24 inches long copper tube packed with 3.8316 g. of molecular sieve carbon 5A. The molecular sieve was obtained from the Takeda Chemical Co. Ltd. of Japan free of charge. Three particle sizes were used : 1.34796 , 1.88014 , and 2.40724 mm in diameter. The ratios between the particle diameters and the column diameter used in this study are in similar ranges as those used in various references [5],[9],

[10],[11],[12],[13].

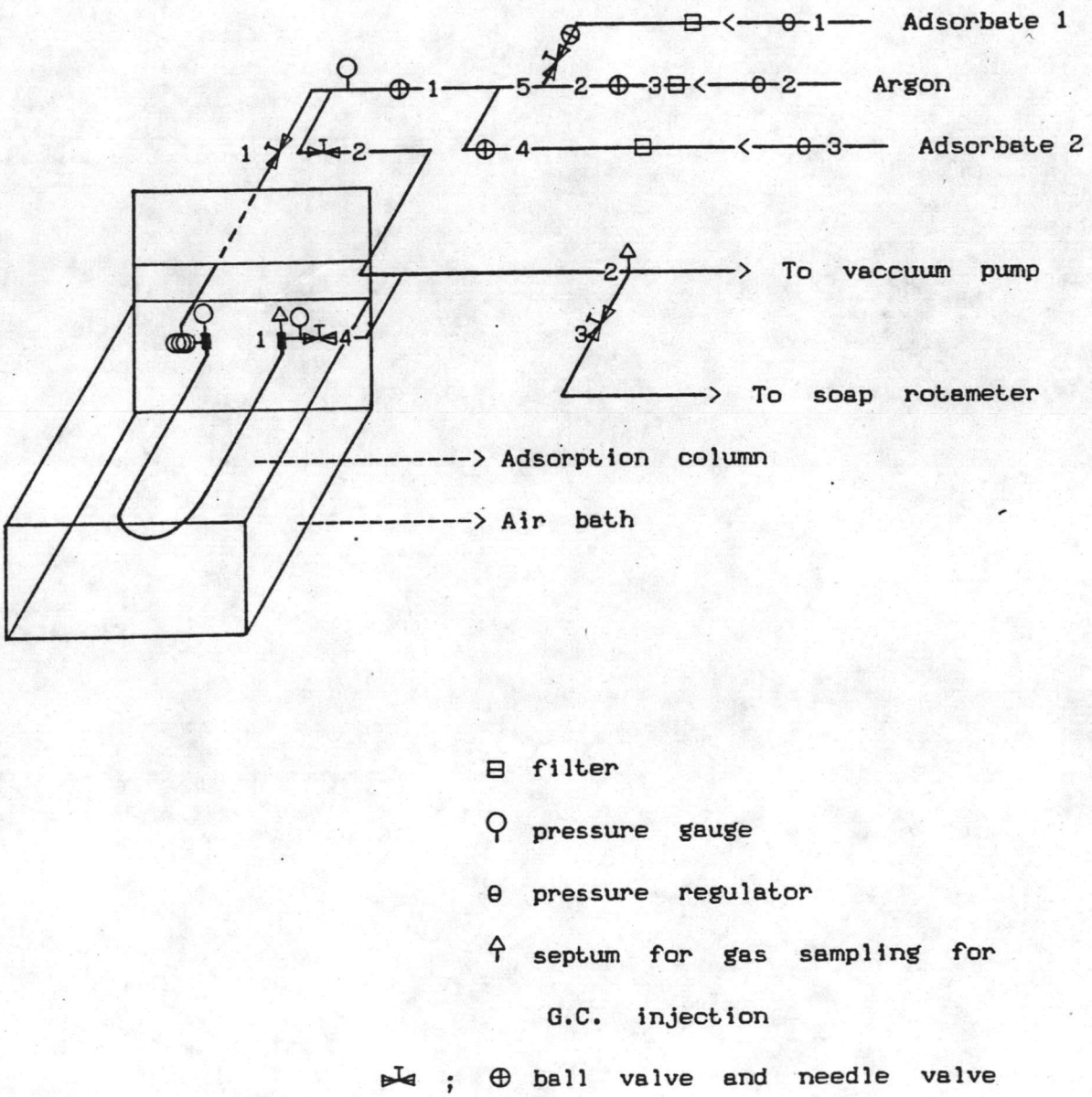

Figure 4.1 Flow diagram for the determination of adsorption rate constants and adsorption isotherms.

The gas flow meter

A soap gas flowmeter was built from a 50 cc burette and used to measure the gas flow rate in this study. The construction procedure involves removing the ordinary glass valve at the tip of the burette and replacing it with a three way glass fitting. One end is the entrance point for the gas to be metered and at the other end is attached a rubber syringe ball containing a small amount of soap water to form the soap bubbles needed for flow rate reading.

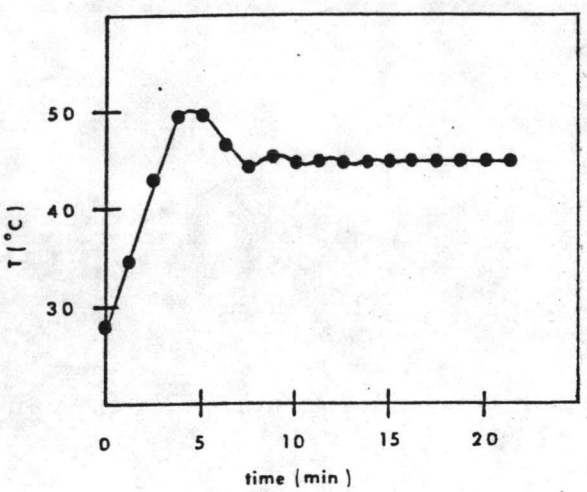

Figure 4.2 Performance curve for the temperature control system.

The air bath system

A constant temperature air bath chamber was made up

of plastic sheets into a box of size 2x1.5x1.5 feet. Inside the bath is a heater and two fans for heating and recirculating the air. Temperature is controlled by a PID controller and set at 45 ° C with an accuracy of ± 0.1 ° as shown in the performance curve presented in figure 4.2. The PID controllers were set at 10 , 2 , and 1 for proportional , integral , and differential modes respectively.

The gas analysis system

A VARIAN 3400 gas chromatograph with a thermal conductivity detector was used to determine the gas concentrations both in the inlet and in the outlet of the adsorption column.

The operating conditions of the gas chromatograph are presented as follows.

Argon flow rate (carrier gas)	20 cc/min
Injector temperature	90 ° C
Column temperature	100 ° C
Detector temperature	120 ° C
Filament current	110 mA
Column packed	Chromosorb P , A/W
	Length : 9 m

The column regeneration system

The regeneration system is composed of a specially made electric oven and a vaccuum pump. The oven is used to heat the packed column to 300 ° C and the vaccuum pump is used to reduce pressure in the packed column down to 0.5 mbar. The construction of the regeneration system are shown in figure 4.3.

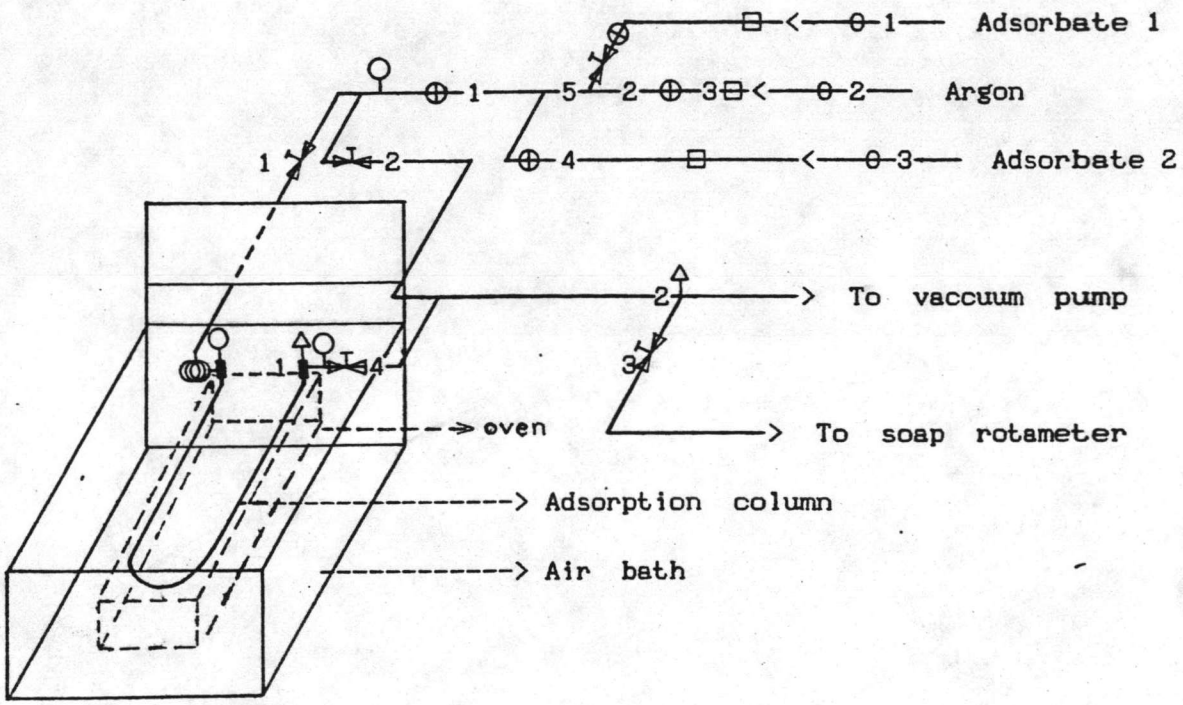

Figure 4.3 The construction of the regeneration system.

4.2 Materials

The adsorbent used in this study is MSC-5A manufactured by Takeda Chemical Ind. Co. Ltd. This molecular sieve carbon has micropores of $5*10^{-10}$ m in slit open , and

has a micropore volume of $1.76*10^{-4}$ m^3 per kilogram of adsorbent.

The gases used in the adsorption experiments were carbondioxide , methane , propane , and argon of high purity grade , properties of which are shown in table III.

Table III

Properties of gases used in this investigation.

gas	purity(%)	manufacturer
carbondioxide	99.9	Thai industrial gases Ltd.
methane	99.5	"
propane	99.8	"
argon	99.9	"

4.3 The experimental procedure

The various experiments performed in this study are as follows

1 Regeneration of the adsorption column.

2 Determination of the chromatographic curves.

3 Determination of adsorption isotherms of single components.

4 Determination of adsorption isotherms of mixtures.

The details of each experimental procedure used is presented as follows

1 The regeneration of the adsorption column.

1.1 Close all valves in the system.

1.2 Open ball valves No. 1,3,4.

1.3 Open needle valves No. 1,3 and pressure regulator No. 2.

1.4 Adjust needle valves No. 1,3 until the argon flows through the adsorption column at a flow rate of about 40 ml/min. Then connect the oven to the adsorption column as shown in figure 4.3 and heat the column to about 300 ° C.

1.5 One hour later close ball valves No. 1,3 and connect the vacuum pump to the adsorption column to reduce the pressure down to about 0.5 millibar for five hours.

1.6 Repeat procedure 1.1-1.3

2 The determination of the chromatographic curves.

2.1 After column regeneration close ball valves No. 1,4 and switch on the temperature controller for an air bath setting at 45 ° C.

2.2 Open ball valves No. 2,3,5 and needle valves No. 1,2,3. Then adjust the needle valves and sampling the gas from septum No. 2 for analysis until the flow rate and the composition of the gas is constant at the desired point.

2.3 Close ball valves No. 2,5 and open ball valves No. 1,4 to let the gases pass through the adsorption column.

2.4 Sample and analyse of the gas from septum No.1 every one minute. After five minutes (for carbondioxide and ten minutes for methane and propane) close ball valve No. 5

while continuing the gas analysis every one minute until no more adsorbates come out in the effluent stream.

3 The determination of adsorption isotherms for single component.

3.1 Follow procedure 1.1 to 2.2.

3.2 Open ball valves No. 1,4 and sampling the gases every one minute until the composition of effluent gases become constant.

4 The determination of the adsorption isotherm of mixtures.

4.1 Follow procedure 1.

4.2 Open ball valves No. 2,3,5 and all needle valves. Adjust needles valves until the flow rate and composition of the gases are constant at the setting point.

4.3 Close ball valve No. 2 and open ball valves No. 1,3,4.

4.4 Analyse the effluent gases every one minute until the composition of the effluent gases are constant.