CHAPTER IV

EXPERIMENTAL METHODS

4.1 Fabrication of Experimental Unit (59)

In the fabrication of the experimental unit, it is important to estimate the correct length of the tube to cut, and always use a tube cutter for the job. Before connecting the tube with tube fittings, all cut edges must be deblurred and the mouths enlarged with a rattle file. These instructions are illustrated and explained in the swagelok tube fitting and installation manual.

Tube sections have to be fitted to unions, valves, pressure control valve, tees, etc.. The tubes are cut at required lengths, and may have to be bent into desired shapes eq. curves, angles, coils. The minimun radius of curvature is shown in the Table 4.1. The procedure to connect a tube to the fittings and valves is as follows :

1) Insert the tube into the nut and furrule of the fitting or valve until the tube end rests firmly on the shoulder of it, then tighten the nut by hand, to fasten the furrule onto the tube. Make a mark across the tube and nut.

2) Turn the nut clockwise with a wrench for 3/4 rounds in the case of an 1/8 inch O.D. tube or smaller size (following step to step in the Figure 4.1)

3) When connecting a fitting or valve with its own nut to the same tube, insert the furruled tube until the end contact the inner wall and tighten the nut by hand. Then turn the nut with a wrench until it feels tight. The nut must be turned in a lesser degree than the first time of connection.

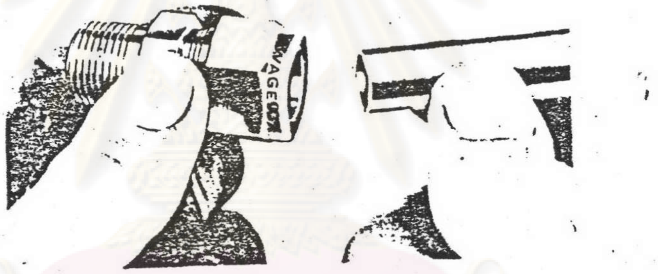
N.B.

1. Be sure to insert the tube to rest firmly on the shoulder of a fitting or valve before tightening the nut by hand and with a wrench ; otherwise the parts will be damaged.

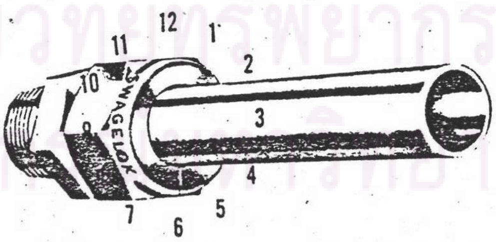
2. To connect tube fittings to tubing, use standard wrenches. Adjustable wrenches should be of good quality and adjusted so that there is no play on the nut or body hex. If poor wrenches are used, or if they are adjusted improperly there is danger of slipping off the fitting or nut. Poor wrenches or improperly adjusted wrenches will damage and distort nuts, often ruin the fitting and, if they slip off, can result in injury. (see in Figure 4.2)

Table 4.1 The minimum radius of curvature (59)

Tube O.D. Diameter (inches)	A Minimum Tube Bend Radius Optimum Conditions (inches)	B Minimum Tube Bend Radius Without Mandrel (inches)
1/8"	.250"	.500"
1/4"	.500"	1.000"
3/8"	.750"	1.500" to 2.000"
1/2"	1.000"	1.500" to 2.000"
5/8"	1.250"	1.500" to 2.000"
3/4"	1.500"	3.000"
7/8"	1.750"	3.500"
1"	2.000"	4.000"
1 1/4"	2.500"	2.500"
1 1/2"	3.000"	3.000"
2"	4.000"	4.000"

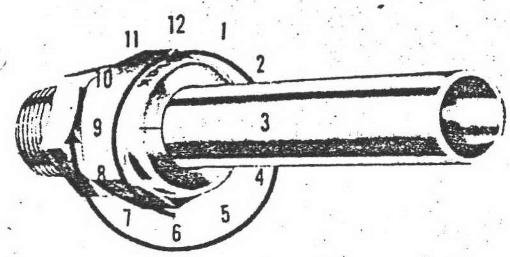


Step. 1. Simply insert the tubing into the SWAGELOK Tube Fitting. Make sure that the tubing rests firmly on the shoulder of the fitting and that the nut is finger-tight.

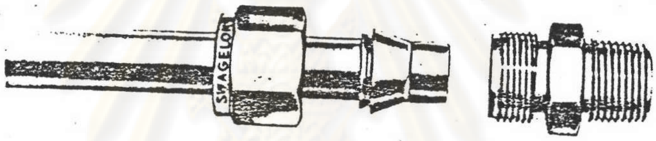


Step 2. Before tightening the SWAGELOK nut, scribe the nut at the 6:00 o'clock position.

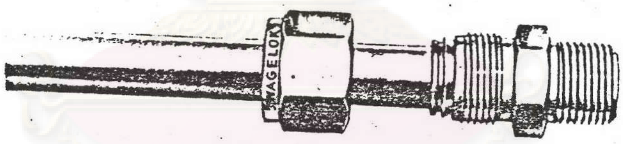
Figure 4.1 The procedure connects a tube to the fittings (59)



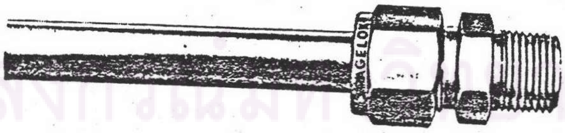
Step 3. Now, while holding the fitting body steady with a backup wrench or vise, tighten the nut one-and-one-quarter turns.* Watching the scribe mark, make one complete revolution and continue to the 9:00 o'clock position.



Fitting shown in disconnected position.

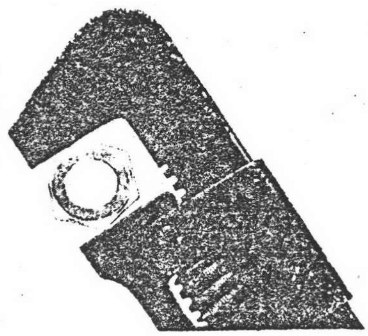


Tubing with pre-swaged ferrules inserted into the fitting until front ferrule sits in fitting and the tubing is bottomed against the shoulder of the body.

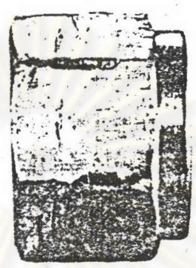


Tighten nut by hand. Rotate nut about one quarter turn with wrench (or to original position) then snug slightly with wrench.

Figure 4.1 The procedure connects a tube to the fittings (59)



Poor wrench used improperly. Notice contact only on corners of nut due to loose fit of improperly adjusted wrench.



Damage to nut caused by use of improper wrenches.

Figure 4.2 Improperly adjusted wrenches will damage nuts (59)

Fabrication of the reactor furnace and checking for uniformity of temperature profile are explained in here. The furnace for the comparative study of reactivity of alcohols over zeolite catalysts was constructed of refractory material and insert heating wires inside the refractory material.

To ensure uniform heating along the middle section of the constructed furnace, axial temperature distributions were measured. Temperature measurement was made by inserting a thin-wire thermocouple into a blanck reactor tube placed within the furnace. The desired steady-state temperature was obtained by adjusting the slide control. After the whole furnace had reached the steady-state, the axial temperature distribution was then measured with a CA (chromel-alumel) thermocouple. Base on the observed temperature distribution, the same procedure was repeated at various temperatures (200°C and 350°C) to ensure uniform axial temperature distributions under all such circumstance. Figure 4.3 shows the observed axial temperature distributions of the reactor funace under no-flow conditions.

4.2 Preparation of the Catalyst for the Reaction Test

Catalysts used for comparative study of conversion of alcohols (C_1^{OH} - C_4^{OH}) to light olefins can be shown in Table 4.2.

Table 4.2 Catalysts used for comparative study of conversion of alcohols to light olefins

No.	1	2
Type	JRC-Z-HM10	JRC-Z-HY5.6
SiO_2, wt.%	83.7	72.8
Al_2O_3, wt.%	14.3	22.0
Na_2O, wt.%	0.12	3.5
Fe, wt.%	-	-
SiO_2/Al_2O_3	9.9	5.6
Ion Exchange, times	2	2
Rate of Ion Exchange, %	98	72

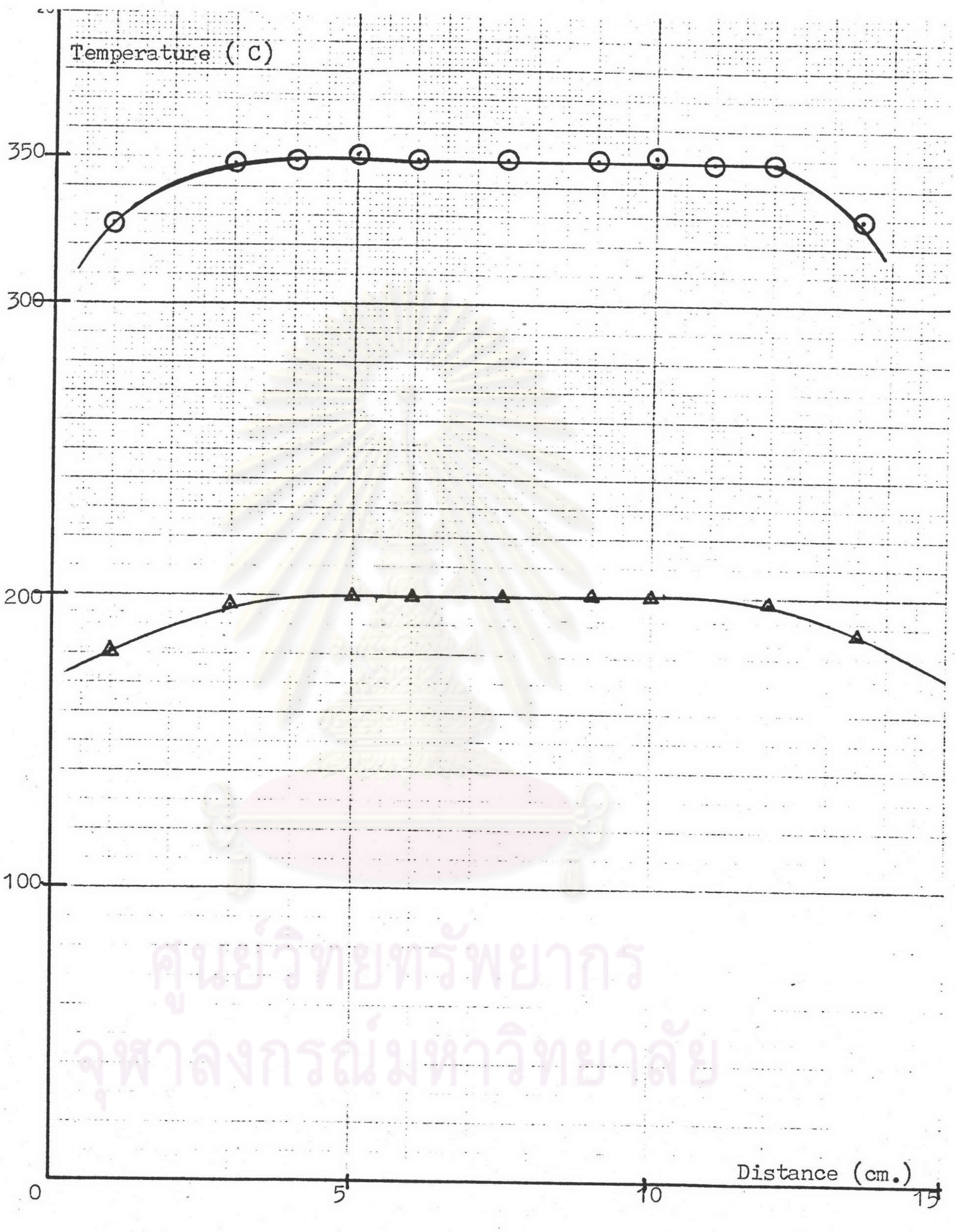


Figure 4.3 The Observed Axial Temperature Distribution of the Pulsed Microcatalytic Reactor Furnace under No-Flow Conditions.

The catalysts were obtained from Catalytic Research Laboratory of Prof. Hiroo Niiyama, Department of Chemical Engineering, Tokyo Institute of Technology, in Japan.

To use in the experiment, the fine powder crystal of desired zeolite catalyst was weighted and then compressed to obtain a pellet. The pellet was next cut up and screened to select fragments of size between 20-40 mesh. Then classifiled fragments were packed into the middle section of a stainless steel-microtube reactor (I.D. 5 mm.) for the reaction test. The height of catalyst bed was then measured.

4.3 Reaction Engineering Test

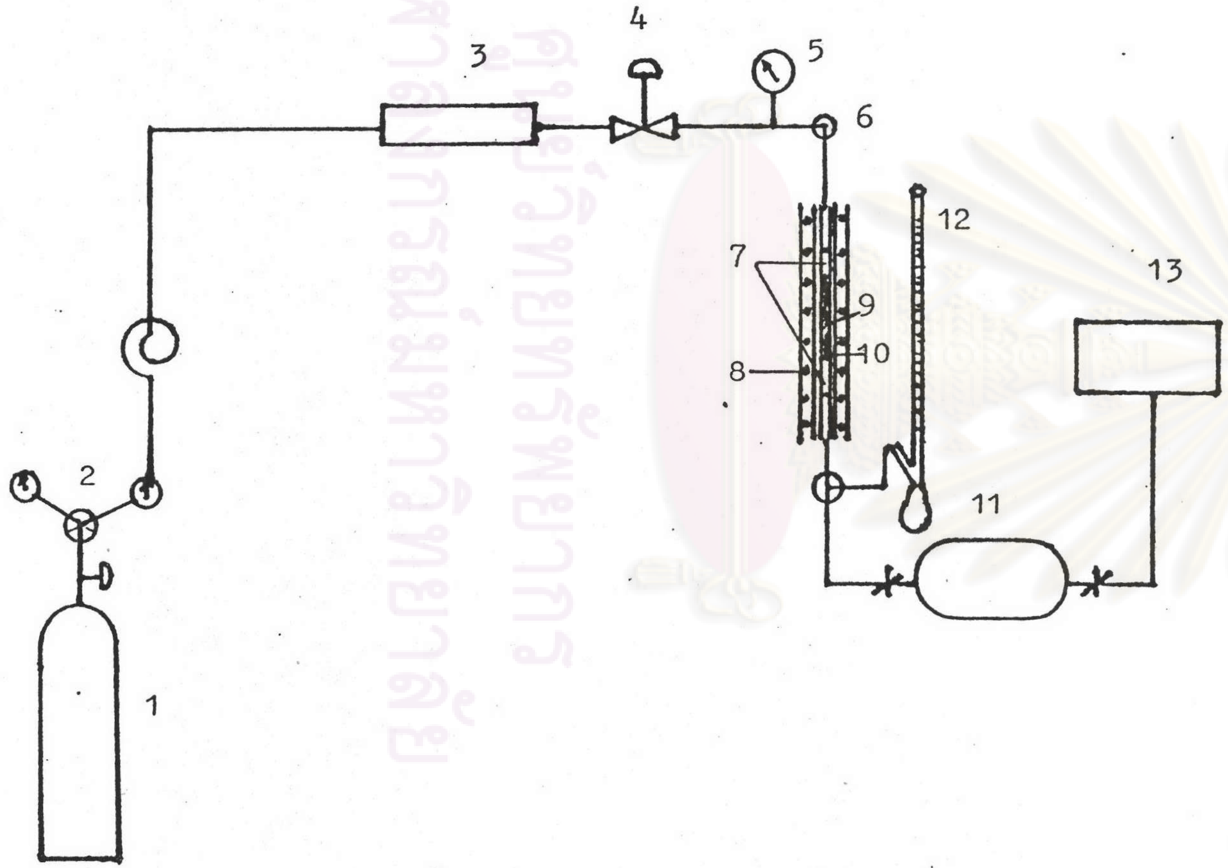
4.3.1 Reaction Method

The reaction is carried out in the reactor as shown in Figure 4.4 . The procedure used to operate this reactor is as follows :

(1) Adjust the outlet pressure of He gas to 10 Psig, and allow the gas to flow through a filter, fine metering valve, and pressure gauge.

(2) Adjust the fine metering valve to allow gas to pass through the reactor and measure the outlet gas flowrate by using a bubble flowmeter.

(3) Heat up the reactor by raising the temperature as follow :



1. He Gas
2. Pressure Regulator
3. Filter
4. Fine Metering Valve
5. Pressure Gauge
6. Injection Port
7. Quartz Wool
8. Furnace & Insulator
9. Catalyst Bed
10. Microreactor
11. Gas Sampling Bulb
12. Soapfilm Flowmeter
13. G.C.

Figure 4.4 Schematic diagram of the reaction experimental unit.

Temperature = 400°C

(hold constant 30 min.)

from room temperature → Adjust to the required reaction temperature

and wait until the required reaction temperature becomes constant (the reactor temperature was controlled by a proportional derivative controller [PD controller])

(4) At the same time switch on the heating transfer line and injection port.

(5) Start to run the reaction by injecting 4 ul of the C_1OH alcohol pulse size at injection port, and allow. He gas to carry the C_1OH vapour to the reaction zone.

(6) At that time adjust three way valve to allow the production gas to collect in the gas sampling bulb by substibution the water.

(7) Take samples to analyse on the gas sampling bulb and the liquid=reciever, the products and the reactant are analysed by the gas chromatograph.

(8) Repeat the same procedures (step 1-7) for the others alcohols (C_2OH - C_4OH), the other zeolite, and others operating conditions.

4.3.2 Analysis of Products from the Reactor

Analytical determination of the reaction gas composition was carried out using gas chromatograph where :

(1) Ethylene and Propylene were analysed by using (3 mm. O.D. x 6 m.) 5% SE-30 on Supelcoport 80/100 mesh,

and the gas chromatograph used for this analysis was a Shimadzu model GC-9APF. (see in Figure 4.5) Setting condition of G.C. shows in Appendix D.2

(2) C_1OH - C_4OH alcohols were analysed by using (3 mm. O.D. x 6 m.) 10% PEG-20 M on chromasorb P-AW 60-80 mesh, and the gas chromatograph uses for this analysis was a Shimadzu model GC-9APF. (see in Figure 4.5) Setting condition of G.C. shows in Appendix D.2

The recorder connected to a Shimadzu C-R6A chromatopac, where their program condition of the recorder were

ANALYSIS PARAMETER FILE 0

WIDTH	5	SLOPE	40	METHOD $	241
DRIFT	0	MIN.AREA	10	SPL.MT	100
T.DBL	0	STOP.TM	655	FORMAT $	0
ATTEN	0	SPEED	5	IS.WT	1

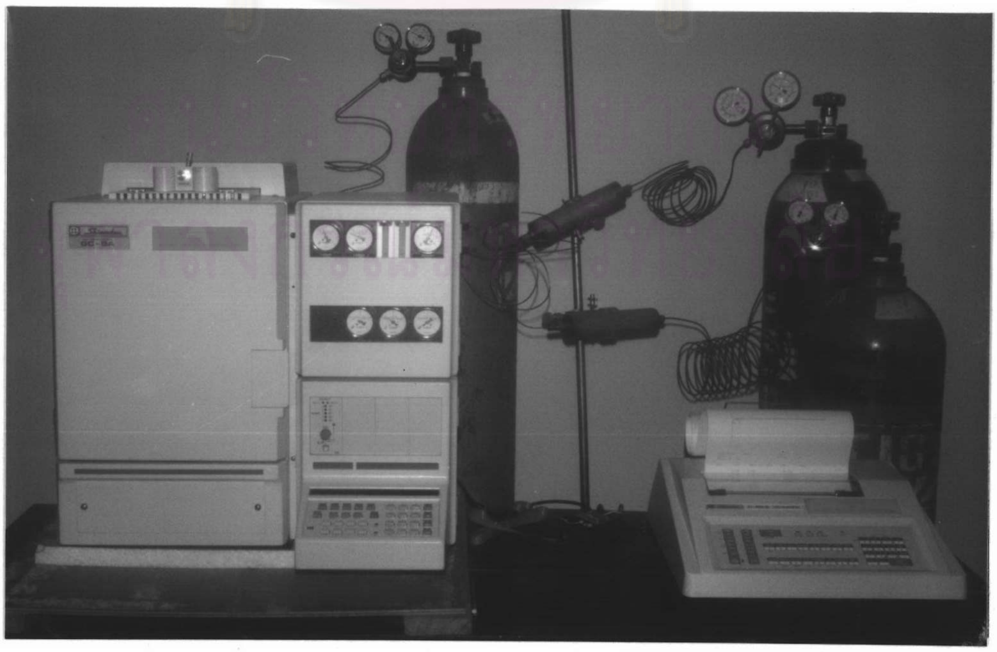


Figure 4.5 Shimadzu FID Gas Chromatograph (Model GC-9APF)

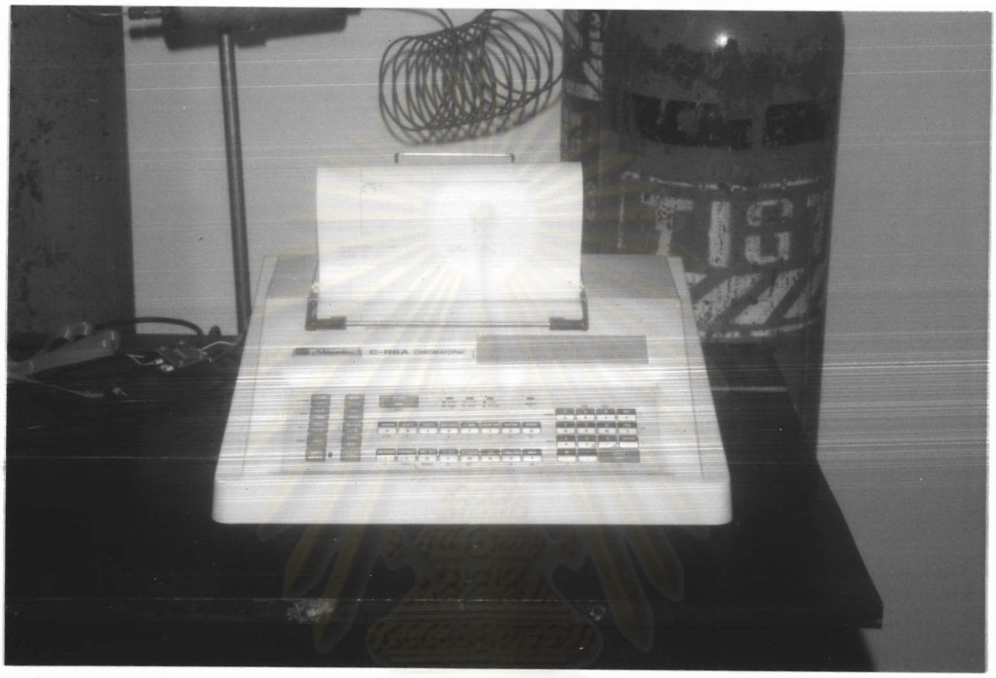


Figure 4.6 Shimadzu Area Integrator Recorder (Model C-R6A chromatopac)

4.3.3 Study of Optimum Operating Condition

i) Influence of Alcohols ($C_1\underline{OH}$ - $C_4\underline{OH}$) on the Reactivity: To study this effect, the alcohol reactants, methanol, ethanol, n-propanol, and n-butanol were used. The reaction conditions for this purpose were space velocity = 1500 - 5000 hr^{-1}, reaction temperature = 200-350°C, and the alcohol pulse size was 4 μl.

ii) Effect of Reaction Temperature.: To study this effects, the HM has the Si/Al ratio 10 and HY-5.6 zeolites were used, where the reaction conditions were space velocity = 1500 - 5000 hr^{-1}, alcohols = C_1OH - C_4OH and the reaction temperature varied at 200 , 250 , 300 , and 350 °C.

iii) Effect of Space Velocity.: The HM and HY zeolite catalysts were chosen for this study. The reactants were alcohols (C_1OH - C_4OH). The reaction conditions for this purpose were the reaction temperature = 200 - 350°C, and the space velocity varied at 1500 , 3000 , and 5000 hr^{-1}.

iv) Influence of Types of Zeolites.: To study this effect, the two types of zeolites were the HM-10 and HY-5.6 were selected. The reaction conditions for this purpose were the reaction temperature = 200 - 350°C, and space velocity = 1500 - 5000 hr^{-1}. The reactants were the C_1OH - C_4OH alcohols.

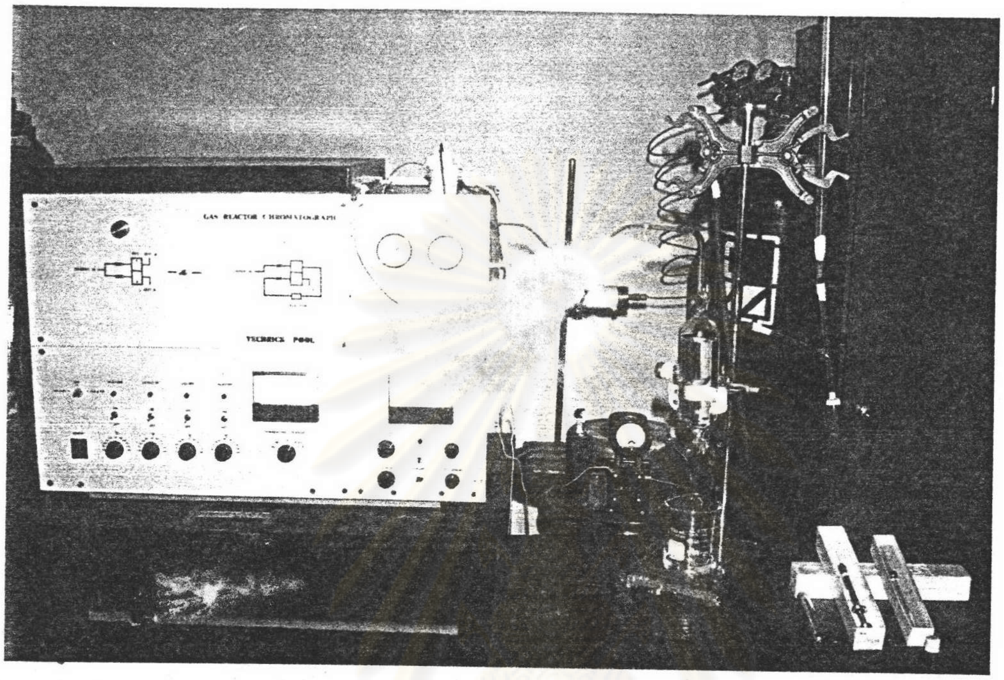


Figure 4.7 The Reaction Experimental Unit for Comparative Study of Conversion of Alcohols to light Olefins