

CHAPTER 4

APPARATUS FOR STYRENE SYNTHESIS

4.1 Flow diagram for styrene synthesis

The apparatus for styrene synthesis can be divided into 4 parts as follows.

1. Gas flow meter
2. Reactant feed system
3. Reactor and salt bath
4. Analysis system

The apparatus for styrene synthesis is represented as shown in figure 4.1

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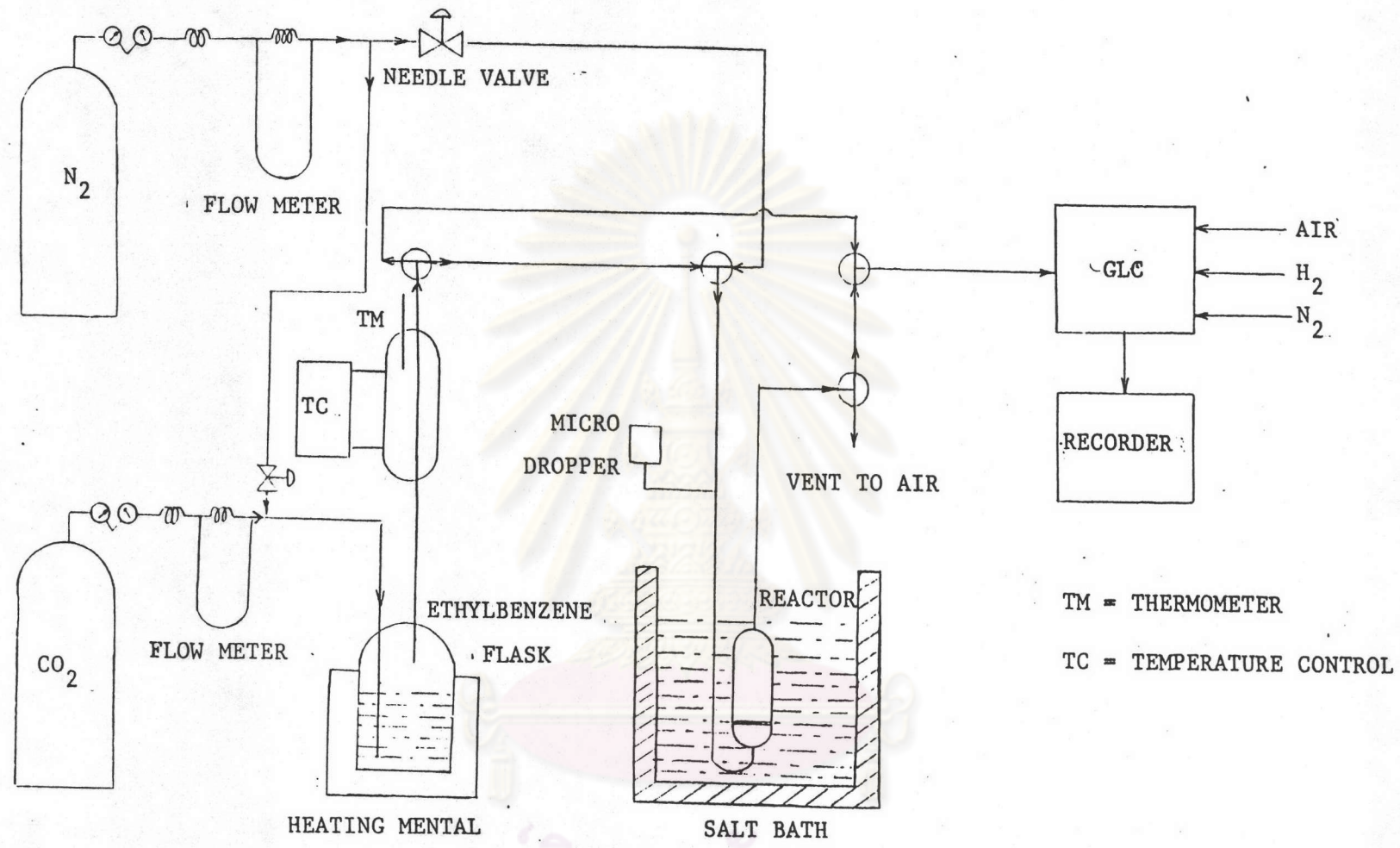


FIG 4.1 FLOW DIAGRAM OF STYRENE SYNTHESIS

TM = THERMOMETER
 TC = TEMPERATURE CONTROL

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4.2 Gas flow meter

The gas flow meter is built to use for CO_2 and N_2 , by using gas flow through the sand packed bed and using the manometer to measure the pressure drop. The gas flow meter is shown in figure 4.2

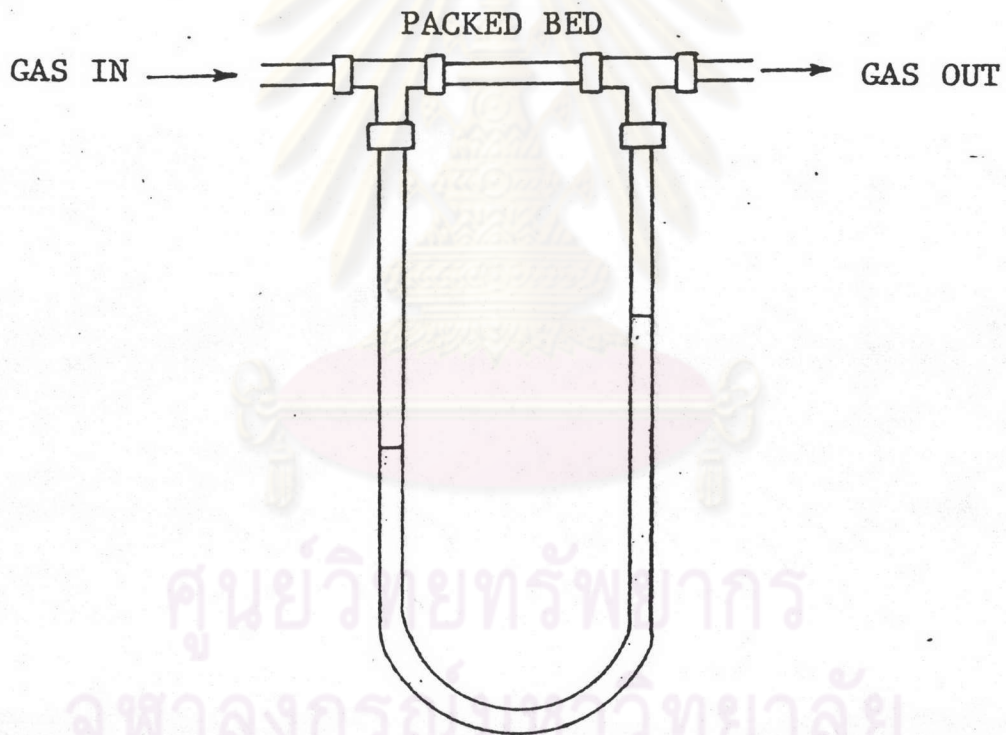


FIG 4.2 GAS FLOW METER

4.2.1 The theory of the gas flow meter

Ergun(5) has found that the pressure drop through fixed bed of uniform sized solid can be correlated by using the equation

$$\frac{\Delta P}{L} g_c = \frac{150(1-\epsilon_m)^2}{\epsilon_m^3} \frac{\mu U_o}{(\phi_s d_p)^2} + \frac{1.75(1-\epsilon_m)}{\epsilon_m^3} \frac{\rho_g U_o^2}{\phi_s d_p} \quad (1)$$

The pressure drop in above equation is represented by 2 factors, the viscous and the kinetic energy losses. At low Reynolds numbers the viscous loss predominate and the equation is simplified to

$$\frac{\Delta P}{L} g_c = \frac{150(1-\epsilon_m)^2}{\epsilon_m^3} \frac{\mu U_o}{(\phi_s d_p)} \quad Re_p = \frac{d_p \rho_g U_o}{\mu} < 20 \quad (2)$$

At high Reynolds numbers only the kinetic energy losses can be considered from the equation which is simplified to

$$\frac{\Delta P}{L} g_c = \frac{1.75(1-\epsilon_m)}{\epsilon_m^3} \frac{\rho_g U_o^2}{\phi_s d_p} \quad Re_p = \frac{d_p \rho_g U_o}{\mu} > 1000 \quad (3)$$

From $\frac{\Delta P}{L} g_c = \frac{150(1-\epsilon_m)^2}{\epsilon_m^3} \frac{\mu U_o}{\phi_s d_p}$ and for fix bed which L,

$\epsilon_m, \mu, \phi_s, d_p$ constant, it can be seen that the pressure drop varies with superficial velocity or flow rate in the range of $Re_p < 20$. For the gas flow rate range 0-10 lit/hr, sand is used as a bed material packing in the pipe of 4 mm diameter. the gaseous properties are fixed.

FLOW RATE (cc/min)

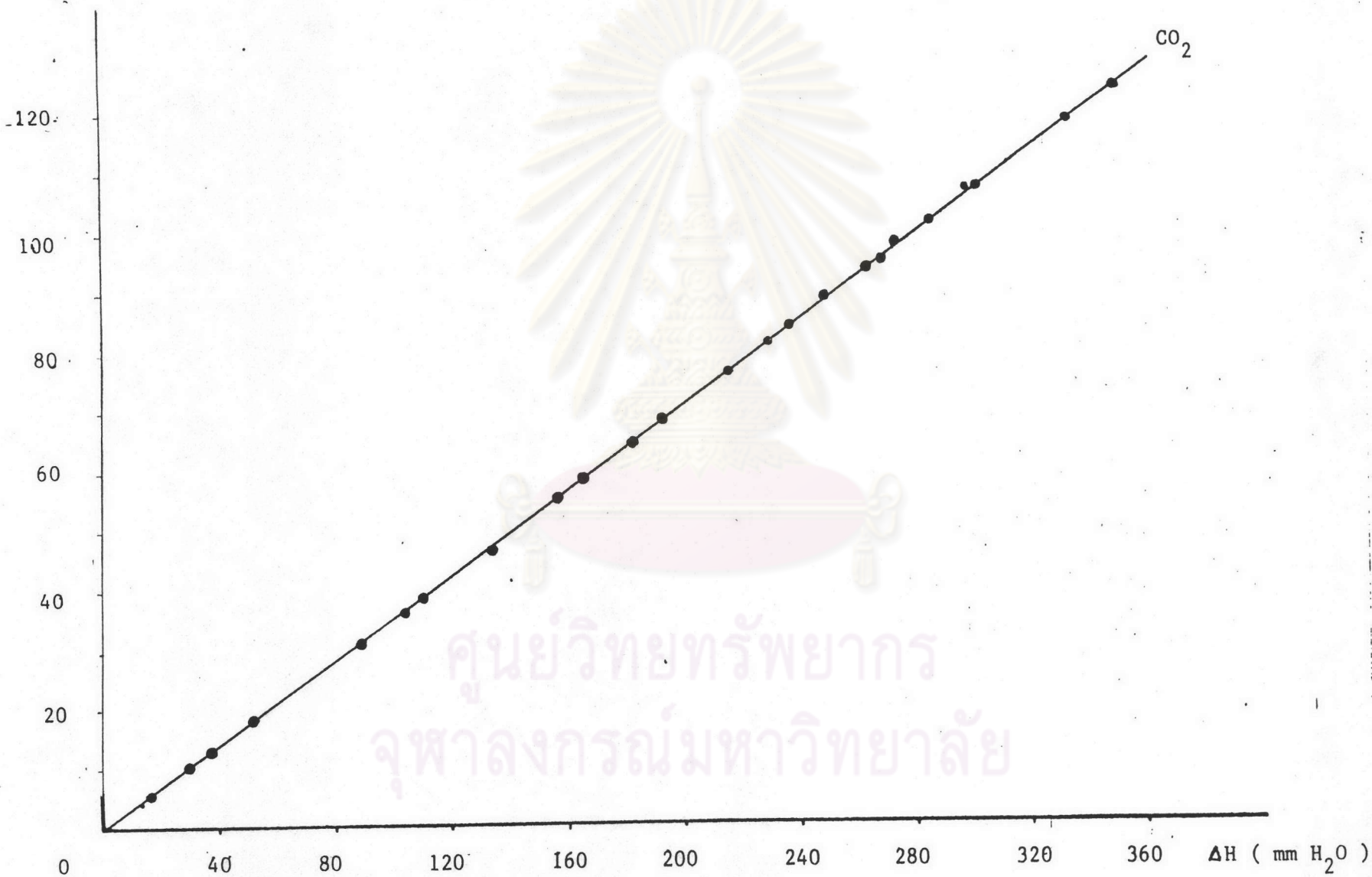


FIG 4.3 THE CALIBRATION CURVE OF CO₂ GAS FLOW METER

FLOW RATE (cc/min)

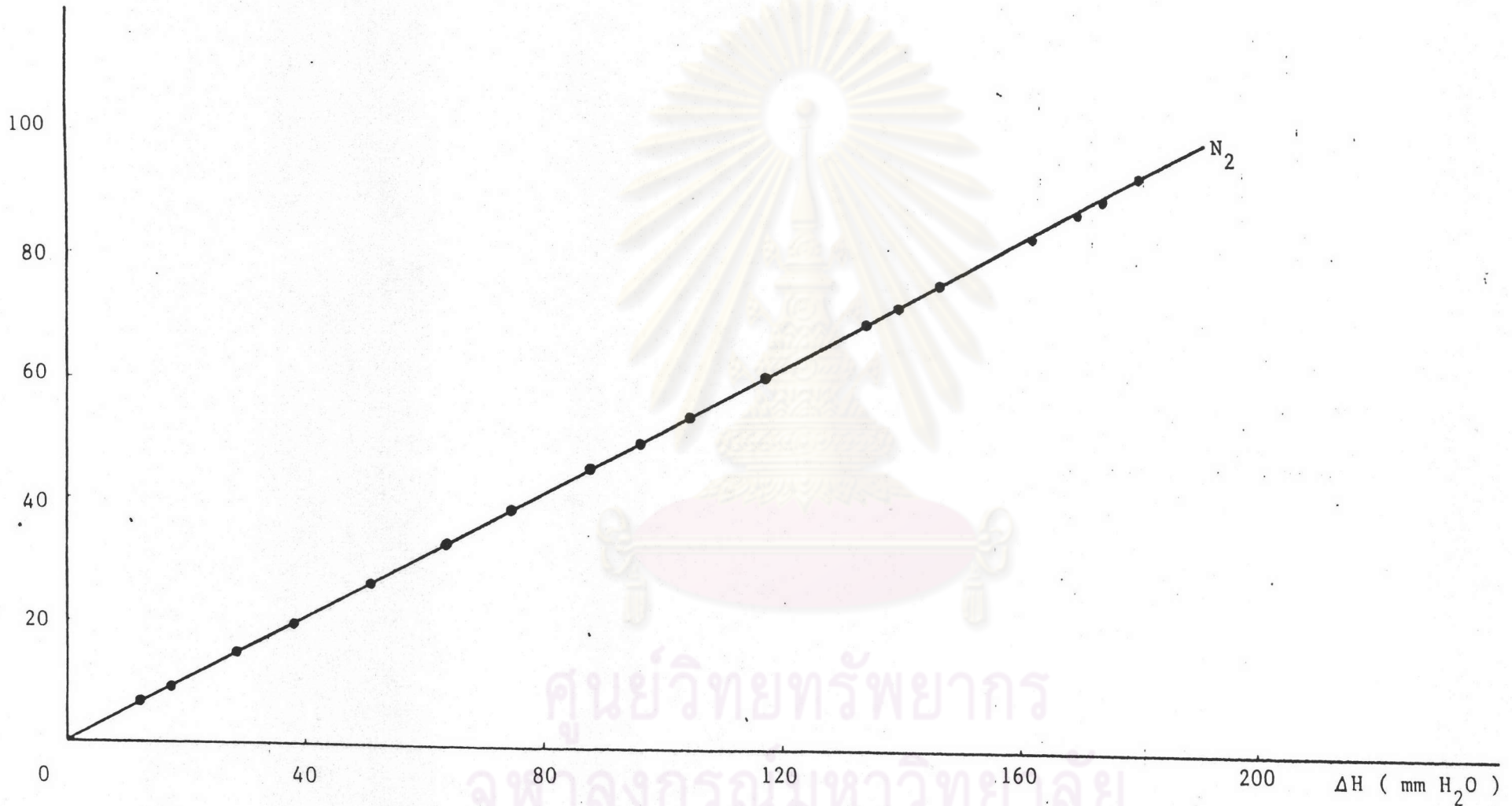


FIG 4.4 THE CALIBRATION CURVE OF N₂ GAS FLOW METER

4.3 Reactant feed system

In the differential reactor, the flow rate is very small, hence the method of saturated vapor for feeding ethylbenzene is more appropriate.

In order to ensure the saturated CO_2 gas is passed through the ethylbenzene flask and then passed through a glass packing condenser in which the temperature is controlled to approximately $3-5^\circ\text{C}$ below the temperature of ethylbenzene in the flask. Based on the saturation curve for CO_2 -ethylbenzene, the composition of ethylbenzene is known. The flow rate of CO_2 can be known by the CO_2 gas flow meter, hence the exact flow rate of ethylbenzene in the reactor is determined.

The saturation curve of CO_2 -ethylbenzene is shown in figure 4.5

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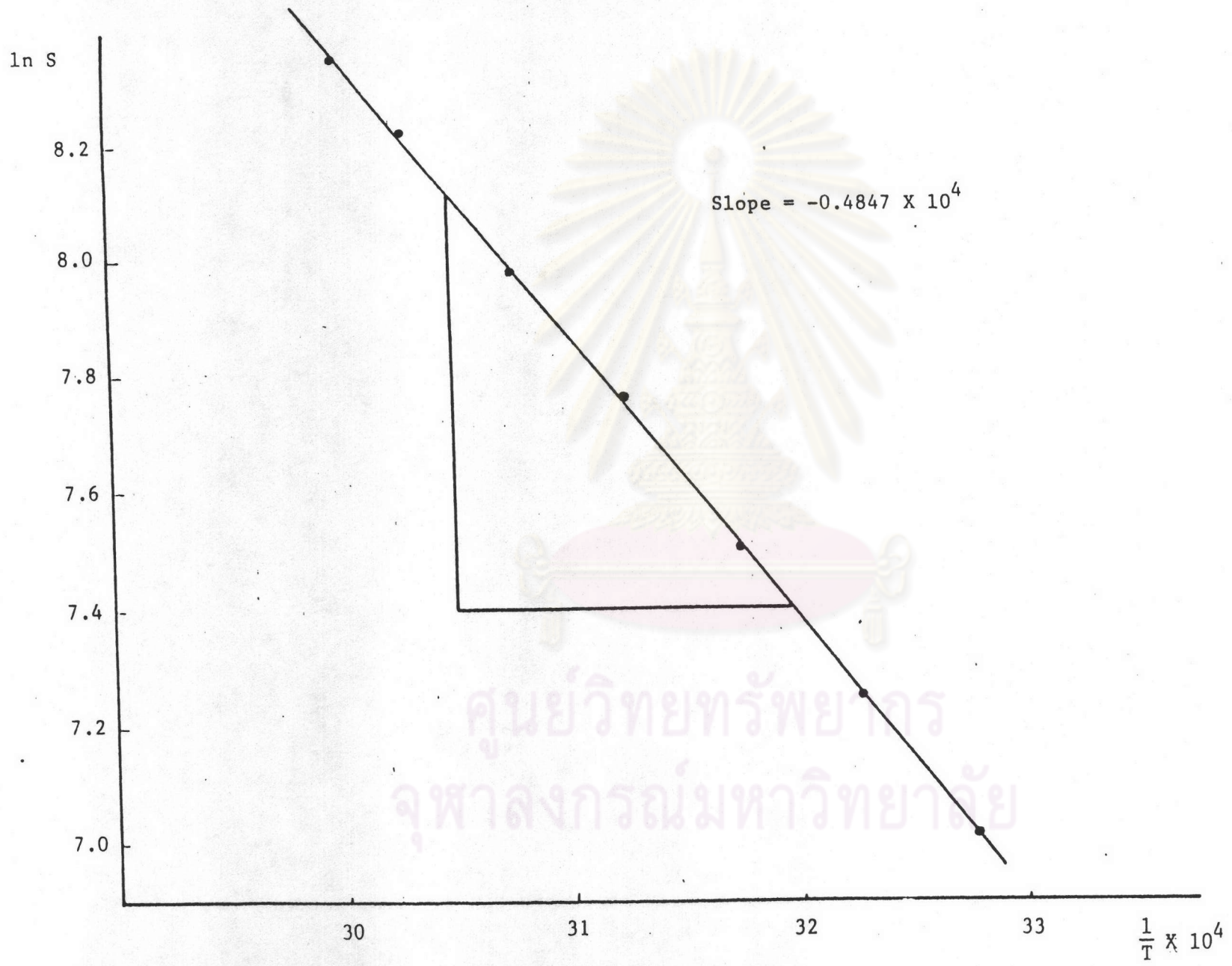


FIG 4.5 SATURATION CURVE OF CO₂-ETHYLBENZENE

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From the saturation curve of CO₂-ethylbenzene, the heat of vaporization of ethylbenzene is calculated to 0.91 kcal/gm. The value of heat of vaporization from handbook is 0.95 kcal/gm at 25°C.

4.3.1 Theory of the saturation curve of CO₂-ethylbenzene

Applying the Clausius-Clapeyron(15) equation, we obtain

$$d(\ln P) = \frac{\Delta H_v d(1/T)}{R}$$

where ΔH_v = heat of vaporization

P = partial pressure of substance

R = gas constant

T = absolute temperature

From the above equation, the correlation between $\ln P$ and $1/T$ is linear and the slope is $\Delta H_v/R$. Thus from the slope of the graph $\ln P$ VS $1/T$ we can calculate to determine the ΔH_v .

4.4 Reactor and salt bath

The principle element is a tubular reactor of differential type. The form of reactor is represented in figure 4.6

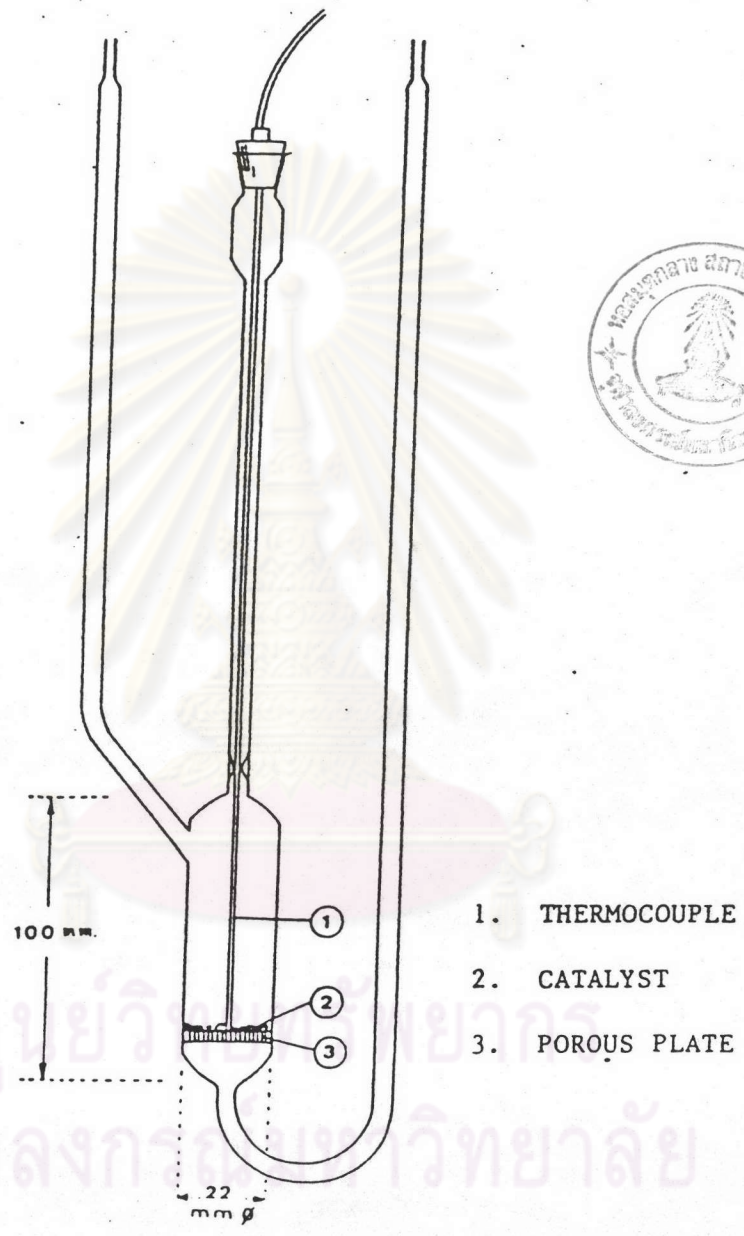


FIG 4.6 REACTOR

The reactor is a Pyrex tube of 22 mm in diameter and the catalyst is deposited on the upper surface of a no. 2 porous plate. A thermocouple is placed inside the close-end glass tube located at the center of the reactor. The tip of thermocouple is close to the catalyst bed, enabling the operator to measure the temperature of the reaction. The thin layer of catalyst and the large surface area of the porous plate permit good heat transfer and isothermal condition.

The reactor is immersed in the salt bath which composes of 53% KNO_3 , 40% NaNO_2 , 7% NaNO_3 (16). A 2 kw heater is applied for heating up the salt bath and an agitator is employed to ensure well distribution of temperature.

The temperature of salt bath is maintained constant by a controller type PF-9684C-M series Re 96 as shown in figure 4.7

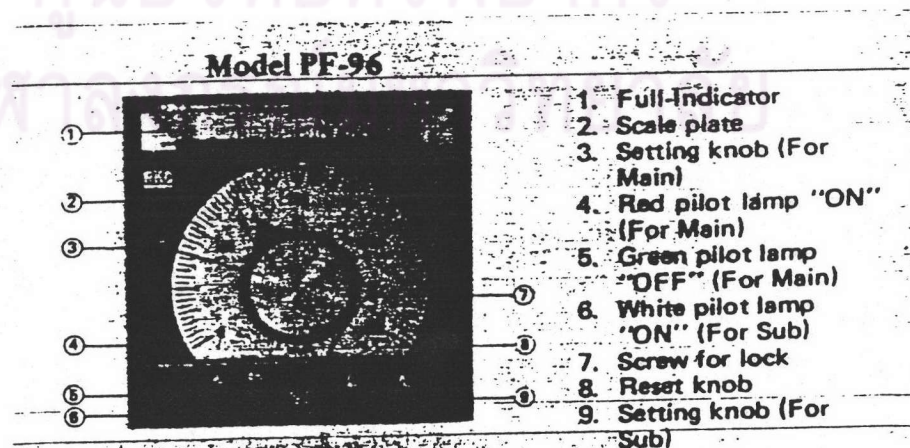


FIG 4.7 CONTROLLER

4.5 Analytical system

There are many methods used for quantitative analysis of styrene such as

1. Common chemistry
2. Infrared
3. Refractrometer
4. Gas chromatography

The gas chromatography method is found to be the most useful one.

In general, the exit gas mixture can be separated into 3 parts.

1. Condensable hydrocarbon: styrene, benzene, toluene, ethylbenzene.
2. Non-condensable gases: CH_4 , H_2 , C_2H_6 , CO , CO_2 ,
 C_2H_4
3. Water phase (when steam is used as diluent)

The sample is analysed by carried out in the column of gas chromatography. The gas chromatography used in this analysis is GOW-MAC standard FID series 750 as shown in figure 4.8.



FIG 4.8 GAS LIQUID CHROMATOGRAPHY

4.5.1 Operating condition of gas chromatography

Air flow rate	240	cc/min
H ₂ flow rate	20	cc/min
N ₂ flow rate	30	cc/min
Column temperature	110	°C
Injector temperature	160	°C
Carrier gas	N ₂	
Detector	FID	
Column	3 m stainless steel	
Column packed	Dinonyl Phthalate 25%	
	Chromosorb W 80-100 mesh	



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