CHAPTER 5

CONSTRUCTION AND LEAK-TESTING OF REACTOR SET

In March and April 1983 , Dr Wiwut Tanthapanichakoon went to Kyoto University under the sponsorship of The Asahi Glass Foundation for Industrial Technology to learn the know-how of assembling a high-pressure through-flow tubular reactor for methanol synthesis. After his return, a similar reactor set was constructed in his laboratory at the Department of Chemical Engineering, Chulalongkorn University. The maximum design pressure and temperature were 50 atg and 450 ^{o}C respectively (1 atg =1 kg_f/cm^2 gauge). The materials of construction were all stainless steel. Swagelok unions, joints, and reducers, etc. were used because of their high reliability.

Figure 5.1 shows a schematic diagram of the constructed high-pressure through-flow tubular reactor for methanol synthesis. Syngas (premixed CO and H_2) was the starting material for the synthesis $CO + 2H_2 \rightleftharpoons CH_3OH$. N_2 was used to purge the system after experiment. Valves SV-1 and SV-2 belonged to the N_2 and pre-mixed CO/H_2 cylinders, respectively. Pressure control valve PVC was used to regulate and maintain the pressure in this system as required. PI-1 and PI-2 indicated the inlet and outlet pressures of the PVC. Needle valve NV-1 served to roughly adjust the flow rate and the inlet pressure indicated by PI-3, whereas NV-2 was used to fine adjust the flow rate indicated by the wet-test meter FM and to reduce the outlet pressure to nearly atmospheric. A safety valve was provided to release any accidental build up to pressure that might result in an explosion.

The reactor tube (1/2" O.D.) was inserted through a uniformly heating furnace. Three sets of thermocouple were used to measure the temperatures at the inlet, the mid-section and the outlet of the catalyst bed in the reactor tube. SP-1, SP-2 were sampling ports. Gaseous samples withdrawn at SP-1 contained the desired product (methanol) and such by-products as H_2O, CO_2, CH_4, C_2H_4, C_3H_8 etc., and the unreacted CO and H_2. Condenser unit CU used ice to condense methanol, hydrocarbons, and H_2O. The condensate was separated and accumulated in a receiver pot. After completion of an experiment, stop valve SV-3 might be opened to drain out the condensate. Gaseous samples withdrawn at SP-2 were "dry", that is, without methanol, H_2O, or any condensible compounds. Wet-test meter FM measured the actual flow during an experiment.

The reactor set was supported on an angular-steel framework (H180 cm x W70 cm x D40 cm) (see Figure 5.1).To prevent methanol condensation in the line between the ractor and SP-1 the line was wrapped with a tape heater and insulated, so that inside temperature might be kept above the boiling point of methanol.

Because of the high inflammability of H_2 and the toxicity of CO, test for leaks was first carried out using N_2 at 1 atg, 2 atg, 5 atg, and 10 atg, respectively. Next He gas was used in place of N_2 gas to 20 atg, 30 atg , and 40 atg, reapectively. In each test. a soapy solution was squirted onto all areas susceptible to leakage, such as unions, joints, valve handles, etc. Appropriate measures were then taken to correct all detected leakages. In constructing the high-pressure reactor set and in correcting leaks, it was impottant not to turn any joints tighter than necessary, since that could lead to irreparable damage.

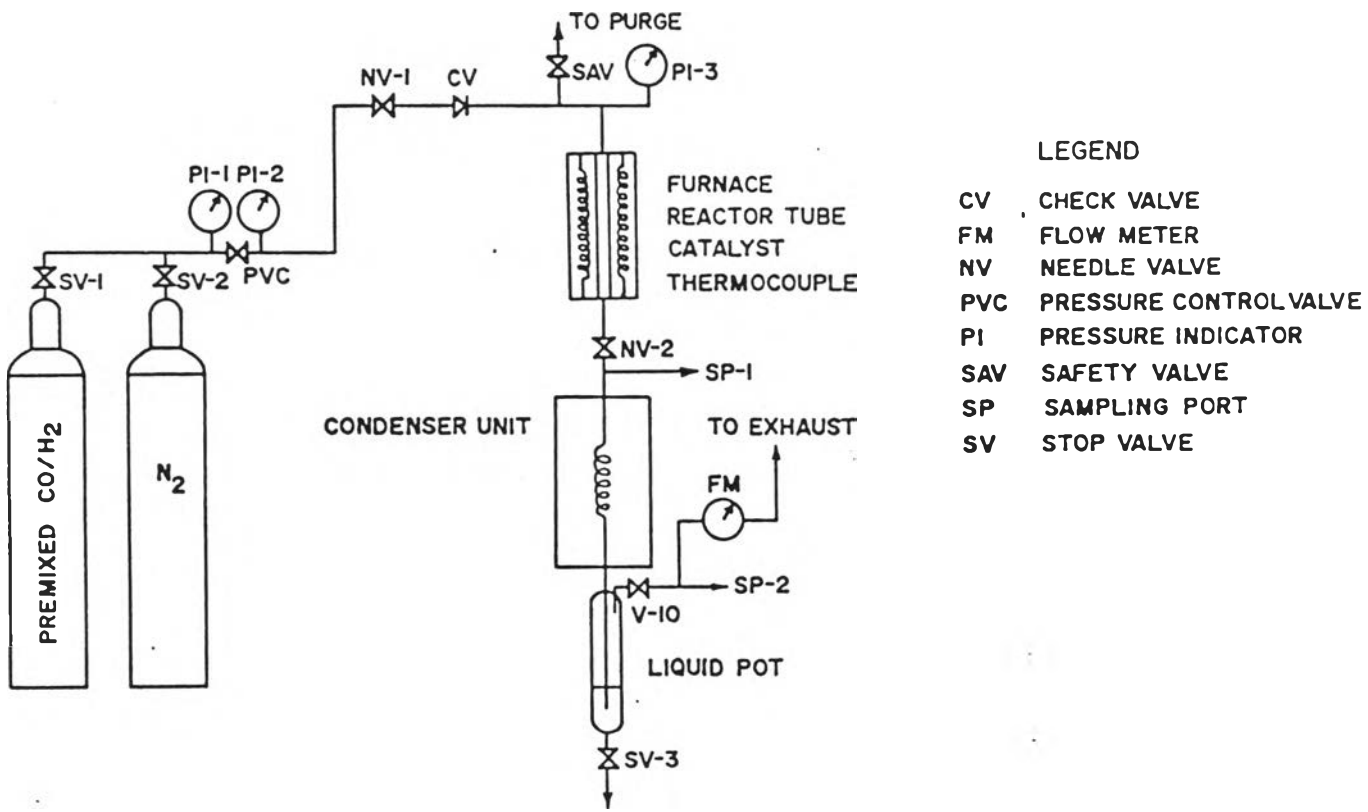
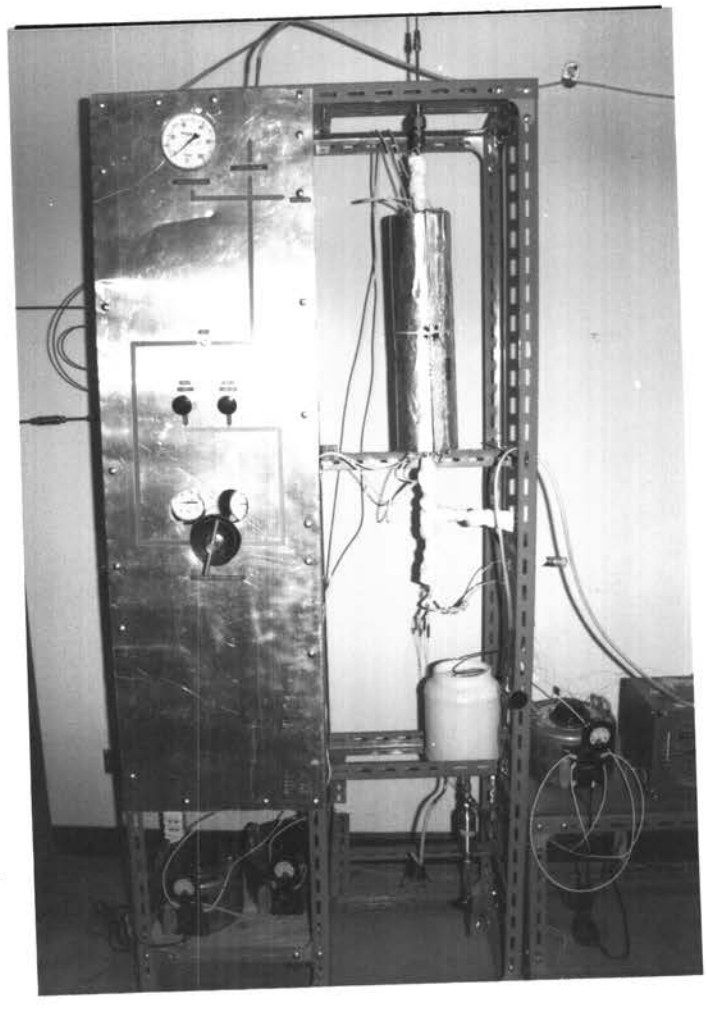


Figure 5.1 Schematic Diagram of the Construced High Pressure Through-Flow Tubular MeOH Synthesis Reactor



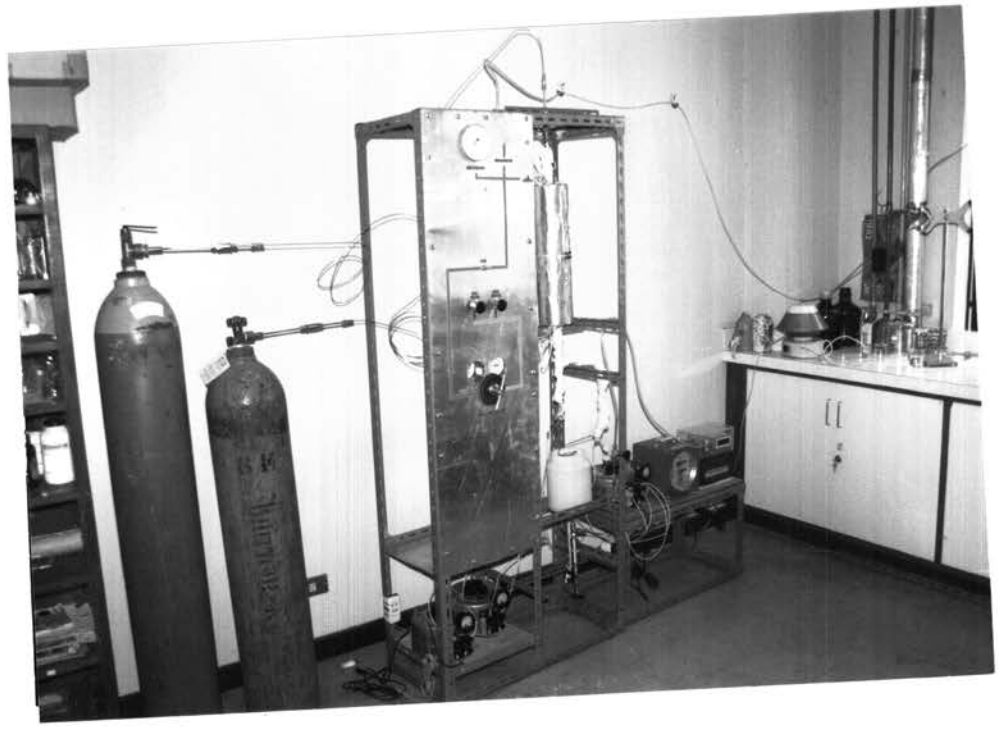


Figure 5.2 Set of Methanol Synthesis Reactor

Construction of Electric Furnace and Measurement of its Axial Temperature Distributions

The furnace of methanol synthesis was constructed from refractory brick block. The farnace was made by carving out 4 refractory brick blocks, inserting electrical wires inside grooves, and wrapping each semicircular section of the furnace in an aluminium-tin sheet. To control the heating temperature, the electric current in the wires was adjusted via two separate slidaces (see Fig. 5.2 and 5.3) one for the upper half and one for the lower half of the furnace length

To ensure uniform axial temperature distributions along the middle section of the constructed furnace, we measured the axial temperature distributions and adjusted the electric wires, so that an axial temperature variation within $\pm$ 3°C was achieved over the entire middle section of the furnace (see Fig. 5.3)

Temperature measurement was made by inserting a thermocouple into the reactor tube within the furnace and adjusting the heating rate via slidaces. After the whole furnace had reach steady state, the axial temperature distribution was then measured with the CA (chromel-alumel) thermocouple. Base on the observed temperature distribution, short portions of the electrical wires were stretched and compressed accordingly (by trial and error) until the maximum axial temperature variation within the middle 20-cm region of the furnace was less than $\pm$ 3°C. The same procedure was repeated at various temperatures (200°C, 300°C and 400°C) to ensure uniform axial temperature distributions under these circumstance. Fig 5.4 shows the obtained axial temperature distributions for the above furnace under no-gas-flow conditions

Figure 5.3 The Inside of the Reactor Furnace

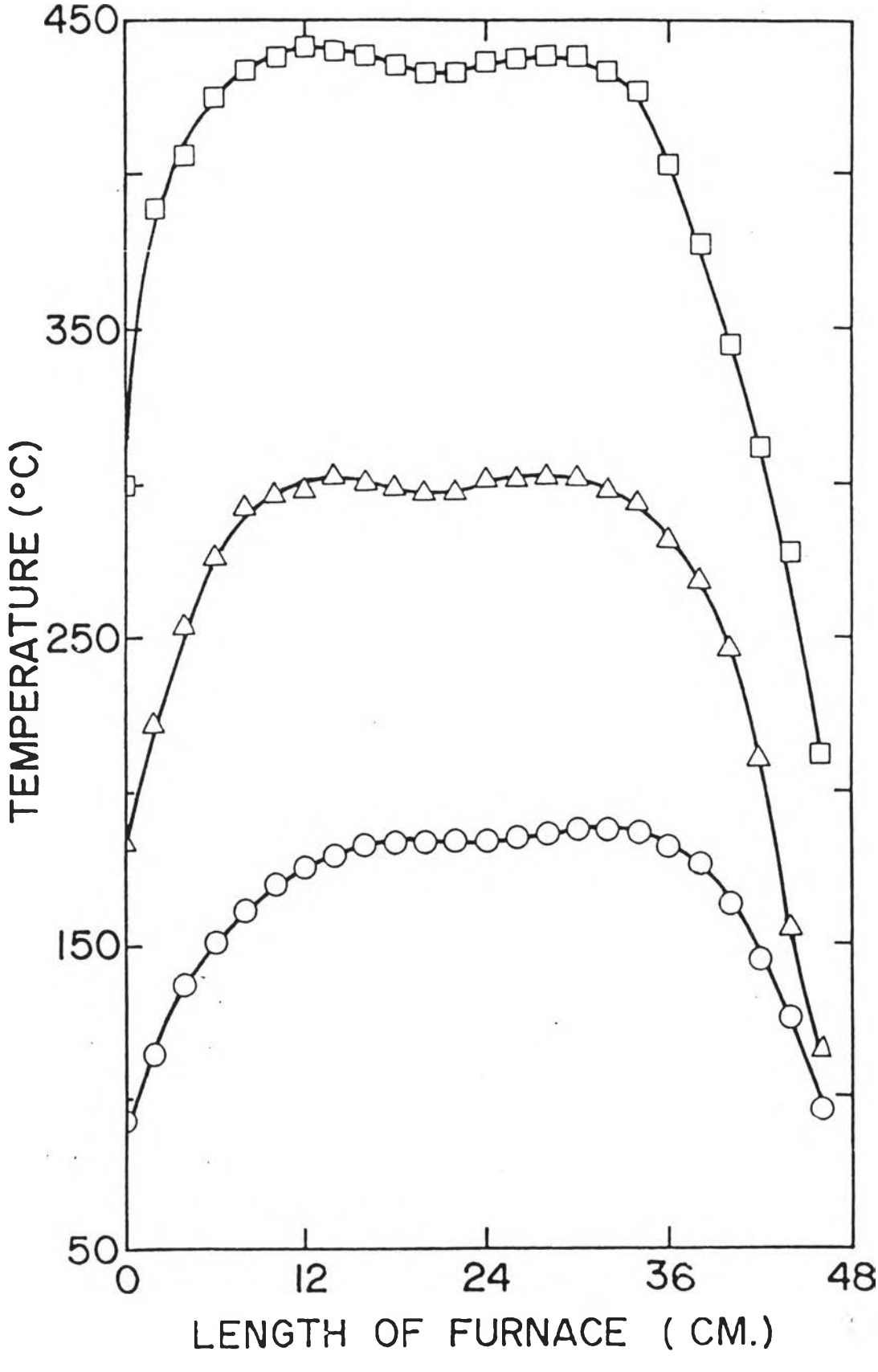


Figure 5.4 Axial Temperature Distribution within the Reactor Tube Inside the Electric Furnace