CHAPTER V

CONSTRUCTION AND TEST RUN OF A HIGH-PRESSURE THROUGH FLOW TUBULAR REACTOR

Most petrochemical processes require stringent precautions. These systems involve heterogeneous catalytic reactions under high pressure. So increasingly it has become evident that laboratory studies on catalytic petrochemical reactions should be carried out under high pressure using, for example, a through-flow tubular reactors. Operating at high pressures is very hazardous. The main dangers come from flying fragments, due to the failure of some component of the equipment; and from the release of large volume of gas, which is generally toxic and inflammable. In the case of methanol synthesis, the know-how of constructing a reactor set that can be operated at a maximum pressure of 50 atm and a maximum temperature of 450°C has recently been reported by W. Tanthapanichakoon (17).

5.1 Construction of a Heating Furnace and Measurement of Its Axial Temperature Distribution

A heating furnace for the methanol reactor set was made by carving out 4 refractory brick blocks, inserting 4 pieces of electrical heating wire in parallel circuit, and enveloping the furnace in two thin aluminium sheets. The electric power to the heating wires on the lower and upper halves of the furnace can be adjusted separately via two slidacs (see Figure 5.1)

To ensure uniform heating along the middle section of the constructed furnace, it was necessary to measure its axial temperature distribution under no-flow condition and to adjust the heating wires accordingly, so that an axial temperature variation within $\pm$ 3°C was achieved over the middle section of the furnace.

Temperature measurement was made within an inserted stainless steel tube inside the furnace by setting the heating rate via the slidacs. After the whole furnace has reached steady state, a CA (chromel - alumel) thermocouple was inserted into the stainless steel tube to measure the axial temperature distribution . According the result of temperature distribution measurement, portions of the heating wires were stretched and compressed accordingly (by trial and error) until a satisfactorily uniform axial temperature distribution in the middle section of the tube was obtained. The same procedure was carried out repeatedly to ensure uniform axial temperature distributions around 200°C, 300°C, and 400°C , respectively, under no-flow condition. Figure 5.2 shows the measured axial temperature distributions around 200°C, 300°C and 400°C.

5.2 Construction of a High Pressure Through-Flow Tubular Reactor Set

The maximum design pressure and temperature of the constructed high - pressure through - flow tubular reactor set were 50 atg and 450°C, respectively. (1 atg = 1 kg_f/cm^2 guage). The materials of construction were all stainless steel. Swagelok fittings were used because of their high reliability. A diagram of the reactor system is shown in Figure 5.3. Such a system was equipped with 3 sets of thermocouples, a wet-gas flow meter, and 3 slidacs.

A 180 - cm high, 80 - cm wide and 40 - cm deep main frame was assembled with angular steels and the whole reactor set was fabricated on this frame. Figure 5.4 is a photograph of the constructed reactor set. The reactor tube ($\frac{1}{2}$ - in.O.D) was 60 cm. long. The system was connected together by various swagelok reducing fittings to accommodate $\frac{1}{8}$ - in- O.D inlet and outlet tubings. The reactor tube was heated by placing it in the above - mentioned heating furnace. The reaction temperature was measured by inserting three sets of CA thermocouple into a thermowell at the center of the reactor tube. The volumetric flow rate of the outlet gas (at room temperature, 1 atm) was measured with a wet - gas flow meter and controlled by adjusting by the needle valves. Pressure in reactor was regulated by using a pressure control valve. For safety, a pressure relief valve was installed. The spring-loaded swagelok relief valve was designed to close, once the excess pressure had been removed, so as to retain part of the contents of the system.

5.3 Testing the Reactor Set for Leakage

First Nitrogen and then helium were used to test for possible leakage of the whole reactor set. Nitrogen was used to test leaks at 1 atg, 2 atg, 5 atg and 10 atg, respectively. At each test pressure, a soapy solution was squirted onto all areas susceptible to leakage, such as unions, joints and valve handles. Appropriate measures were then taken to correct all found leakages. It was important not to turn any joints tighter than necessary during the construction stage and the anti-leak tightening stage. Otherwise, it could lead to irreparable damage. Next helium was used in place of nitrogen to test for leakage at 20 atg, 30 atg and 40 atg, respectively.

Only when the testing for leakage has successfully been completed, were we ready to test-run the reactor set.

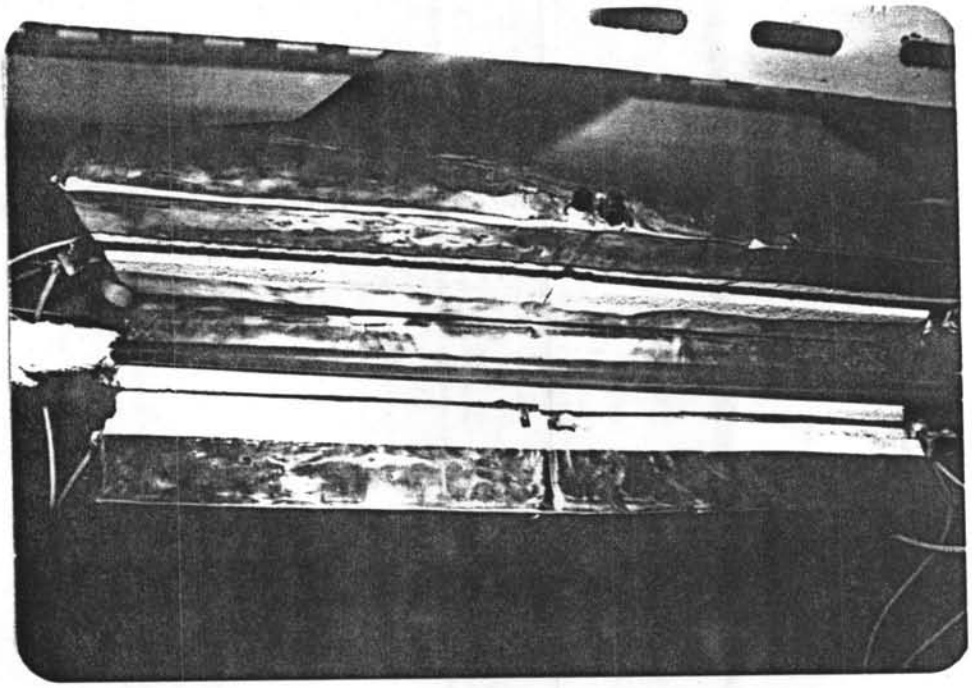

Figure 5.1 Heating Furnace

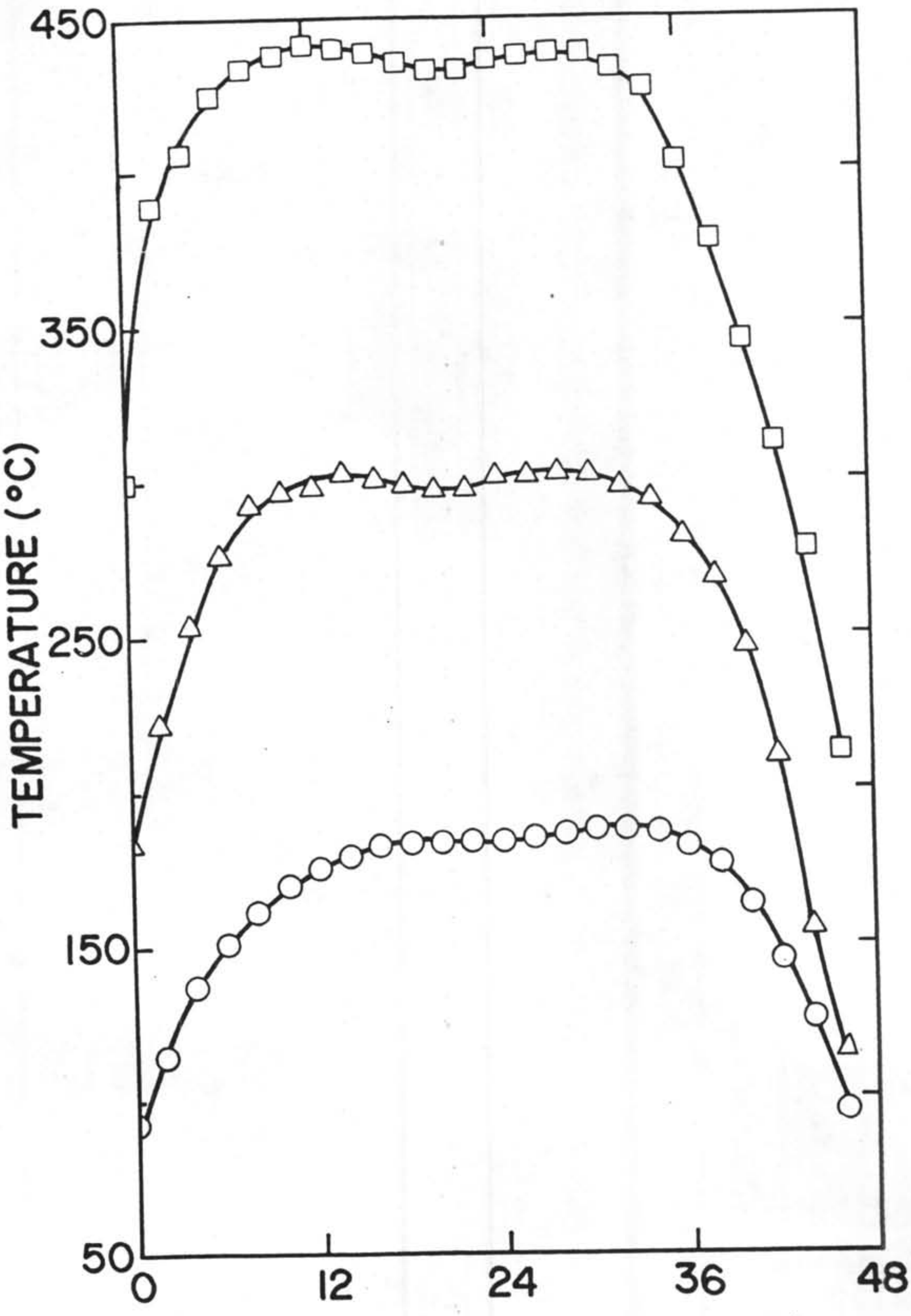

Figure 5.2 Observe Axial Temperature Distribution in Furnace

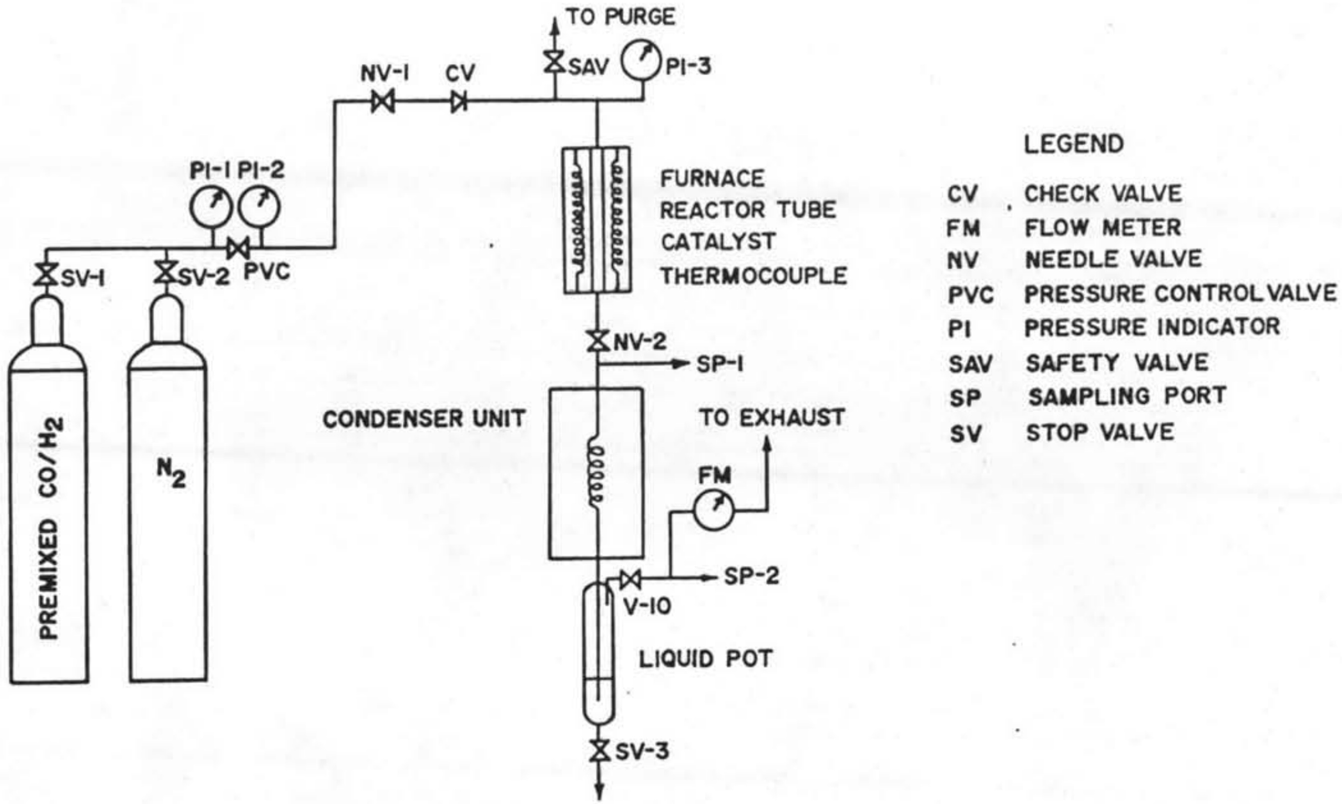

Figure 5.3 Schematic Diagram of the Constructed High-Pressure Through-Flow Tubular Reactor

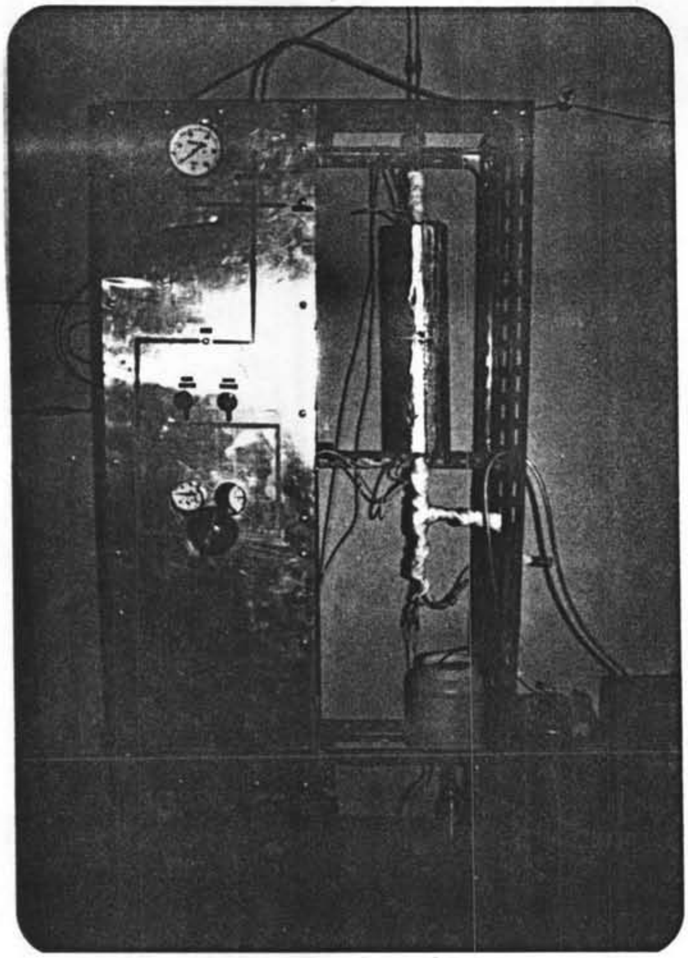

Figure 5.4 Photograph of the High Pressure Through-Flow Tubular Reactor Set