CHAPTER 3

DESIGN AND CONSTRUCTION OF EXPERIMENTAL UNIT

3.1 Design of Experimental Unit

3.1.1 Requirements of Experimental Unit

In this study the working pressure ranges from 40 atg to 100 atg and the temperature from 200°C to 350°C. The chemical feed may contain both methanol and syngas, or only syngas. It should noted that the syngas requires great attention because it is composed of H_2 and CO. The CO gas is extremely toxic, and its inhalation can easily lead to death. H_2 is explosive and flammable. So it is imperative to prevent leakage from the experimental unit. A pressure release valve must be installed to ensure that excessive pressure will not build up. Furthermore, the experimental unit should be easy to refabricate or modify. Thus the corresponding fittings should be interchangeable. **Fig. 3.1** shows the diagram of the experimental unit.

3.1.2 Materials of Construction

Since the working pressure is about 100 atg, all parts of the unit must be able to withstand pressures up to 150-200 atg. Also they must be leak-proof and trouble-free upon connection and reconnection. In this regard, all of the tubes, tube-fittings, connectors, stop valves, safety valves and metering valves, etc. are made of stainless steel and their sealing material must resist corrosive materials and tolerate an elevated temperature. Swagelok tube fittings and valves, a patented, precision machined, quality tube fitting employing two ferrules which swage the tubing to obtain the ultimate in a reliable, leaktight tube fitting connection, were chosen. **Fig. 3.3** show longtitudal cross section of a swagelok tube fitting. The two ferrules and nuts can provide leak tightness up to its design pressure if the fittings or valves are fabricated in a proper manner. Sealing materials are Kel-F and Viton. Kel-F is a thermoplastic polymer with excellent chemical inertness and a temperature range of $-320^{\circ}F$ to $+300^{\circ}F$. Viton is a fluorocarbon rubber used as an O-ring material, especially good for hard vaccum service and a temperature range of -40° to $450^{\circ}F$. In the needle valves the O-rings are made of Viton.

3.2 Fabrication of Experimental Unit

3.2.1 Preparation

After all the parts and components have been purchased, keep them in a safe place. Be careful not to let the tube surface be scratched because such defects may cause leakage at high pressures (So the tubes should be stored vertically). 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.

3.2.2 Fabrication

In the fabrication of the experimental unit, 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 eg. curves, angles, coils. The minimum radius of curvature is shown in **Appendix A.2.** The procedure to connect a tube to the fittings and valves is as follows:

1. Insert the tube into the nut and ferrule 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 ferrule 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, and one and a quarter rounds the case of 1/4 inch O.D. tube and larger. (see **Figs. 3.4-3.5.**)

3. When connecting a fitting or valve with its own nut to the same tube, insert the ferruled 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 (see **Fig. 3.6**). Poor wrenches or improperly adjusted wrenches will damage and distort nuts, often ruin the fitting and, if they slip off, can result in injury.

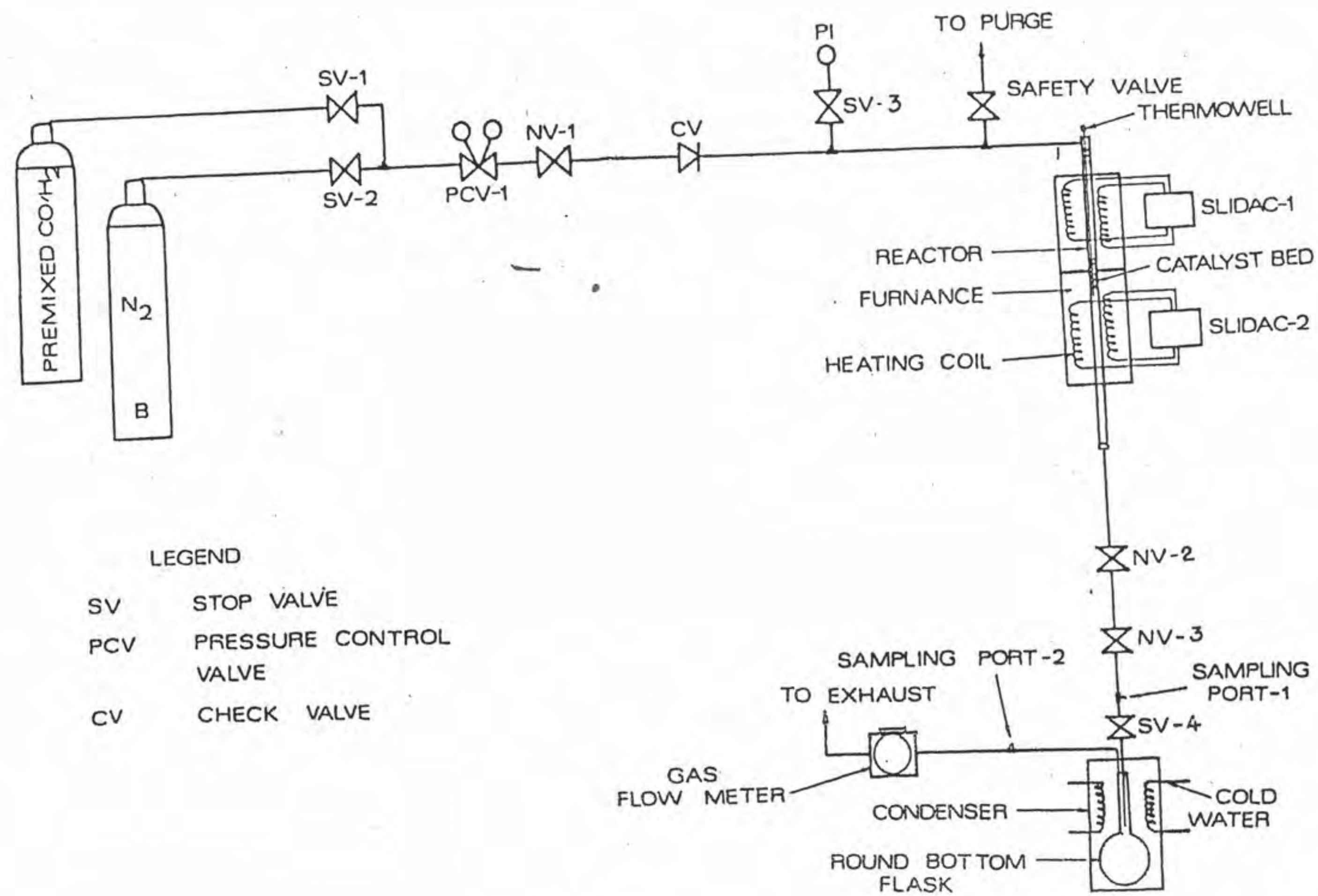


Fig. 3.1 Diagram of The Experimental Apparatus

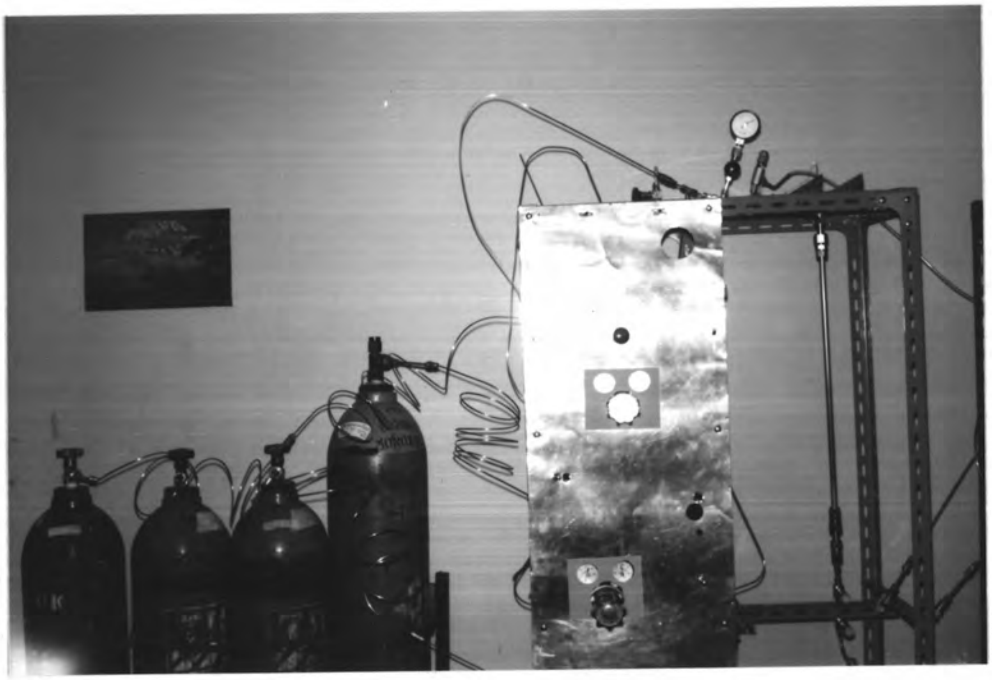


Fig. 3.2 Photograph of The Experiment Apparatus

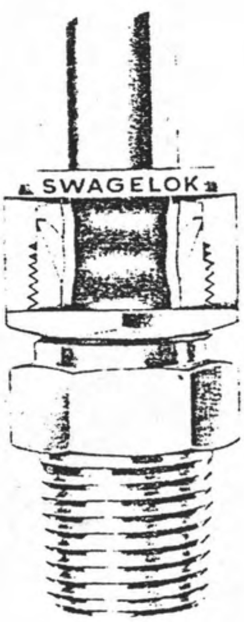
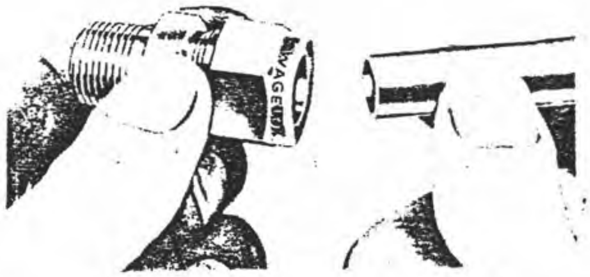
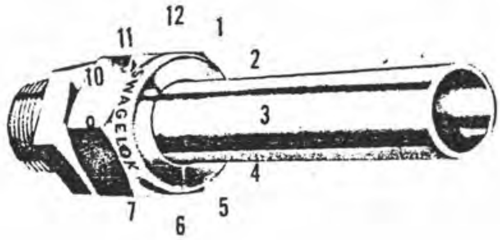


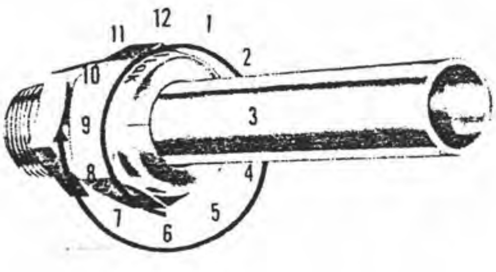
Fig. 3.3 Longtitudal Cross Section of a Swagelok



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.

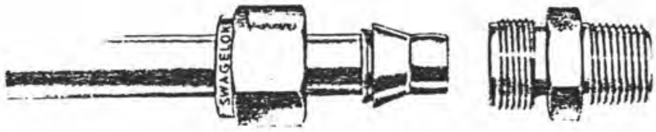


Step 2. Before tightening the SWAGELOK nut, scribe the nut at the 6:00 o'clock position.

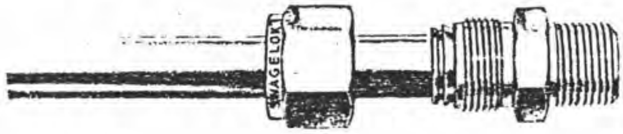


Step 3. Now, while holding the fitting body steady with a backup wrench or vise, tighten the nut one-and-one-quarter turns.* Watching the scribe mark, make one complete revolution and continue to the 9:00 o'clock position.

Fig. 3.4 Installation Procedure of a New Swagelok Tube Fitting



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.

Fig. 3.5 Procedure of Retightening Swagelok.

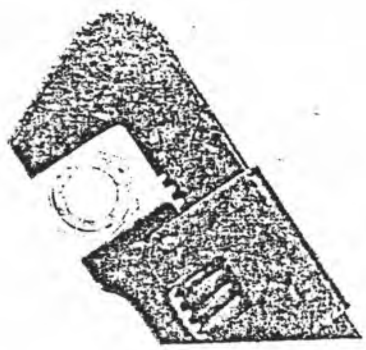
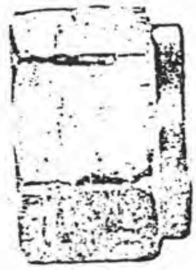


Fig. 14
Poor wrench used improperly. Notice contact only on corners of nut due to loose fit of improperly adjusted wrench.



Damage to nut caused by use of improper wrenches.

Fig. 3.6 Poor Wrenches or Inproperly Adjusted Wrenches.

3.3 Furnace Fabrication and Testing of Furnace

Fabrication of the reactor furnace and checking for uniformity of temperature profile are explained here.

The furnace for ethanol synthesis was constructed of refractory brick blocks. It was made by carving out 4 refractory brick blocks, inserting heating wires inside the prepared grooves and wrapping the semicircular blocks of the furnace together in aluminium-tin sheets. To control the heating temperature, the electric current in the wires is adjusted via two separate slidacs, one for the upper half and one for the lower half of the furnace.

To ensure uniform heating along the middle section of the constructed furnace, axial temperature distributions were measured and the electric wires were stretched or compressed locally so that axial temperature variation within $\pm 3^{\circ}C$ was achieved over the entire middle section of the furnace. Temperature measurement was made by inserting a thin-wire thermocouple into a blank reactor tube placed within the furnace. The desired steady-state temperature was obtained by adjusting the slidacs. After the whole furnace had reached the steady state, the axial temperature distribution was then measured with a CA (chromel-alumel) thermocouple. Based on the observed temperature distribution, specific portions of the electrical wires were stretched or compressed accordingly (by trial and error) until the maximum axial temperature variation along the middle 20-cm. region of the furnace was less than $\pm 3^{\circ}C$. The same procedure was repeated at various temperatures (250, and $350^{\circ}C$) to ensure uniform axial temperature distributions under all such circumstances. **Fig. 3.7** shows the observed axial temperature distributions of the above furnace under no-flow conditions.

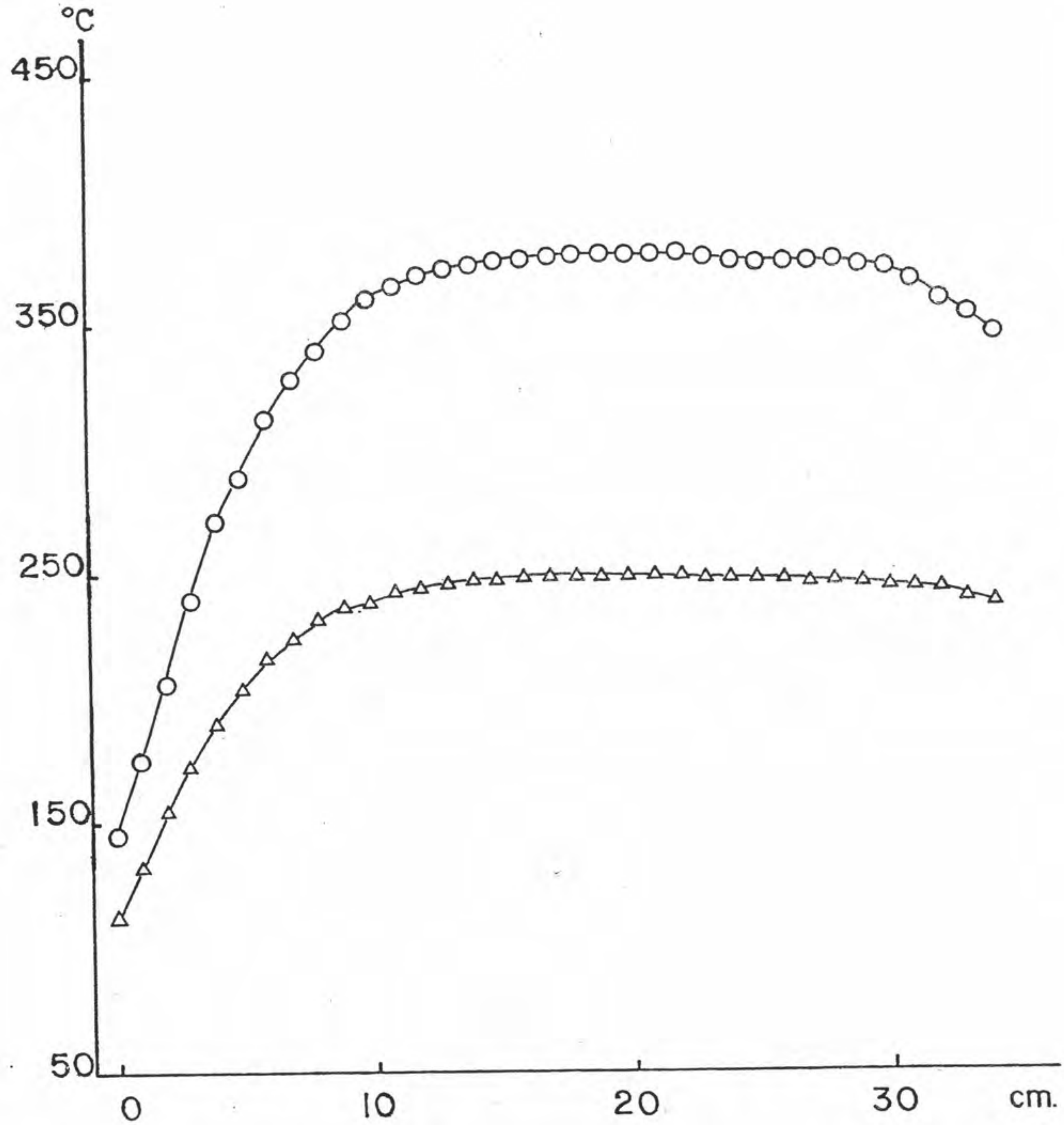


Fig. 3.7 The Observed Axial Temperature Distribution of The Furnace under No-Flow Conditions.

3.4 Inspection and Leak Test

Because of the inflammability and explosivity of hydrogen gas, the high toxicity of carbon monoxide gas and the rather severe operating conditions, leak test must be carried out at all points susceptible to leakage, such as unions, T joints, valve handles, etc. In the first stage, nitrogen gas was used to test for leakage in the experimental apparatus and after that it was replaced with H_2 gas.

3.4.1 Inspection of Leakage

1. Introduce N_2 gas into the reactor set while the outlet valve is shut off.

2. Increase the pressure at an interval of 10 atg. starting from 10 atg. until the pressure in the system reaches 50 atg.

3. At each pressure interval, squirt soap solution onto each leak-susceptible point.

4. Inspect for bubbles. If tiny bubbles are observed, this means that there is leakage at the point. So turn the nut more tightly and squirt more soap solution onto the same point. If there are no more bubbles, the leak has been eliminated.

5. After the pressure has reached 50 atg., use H_2 gas instead of N_2 gas. Repeat steps 3 and 4 until the final pressure reaches 120 atg.

3.4.2 Pneumatic Pressure Test

1. After the inspection for leakage has been completed, set the pressure in the reactor set at the highest pressure. Let it stand at that pressure for 1 week, and record

the pressure reading daily. Be sure that all valves have been shut off.

2. If the pressure gradually decreases as the time goes on, it means that H_2 gas is leaking from certain points assuming there is no change in temperature. In this case, inspection for leakage with bubble solution, especially at such points as stems of valves, and connectors close to the reactor, which are susceptible to high torques upon installing and retightening, must be carried out carefully again.