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ASSEMBLY, COMMISSIONING AND OPERATING PROCEDURE FOR THE HIGH PRESSURE THERMOBALANCE REACTOR

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INTRODUCTION

The purpose of this report is to describe the assembly, commissioning and operation of the high pressure thermobalance reactor in the Coal Gasification Section. The thermobalance reactor will provide quanitative correlations to describe residual char gasification (ref. 1) under a wide range of conditions.

The thermobalance reactor is a pressure balance reactor where up to 100 grams of coal can be gasified at temperatures up to 1000°C and 2000 psi. The thermobalance is a modified version of the one at the Institute of Gas Technology in Chicago, Ill., and it was purchased from Autoclave Engineers, of Erie, Penn.

A schematic diagram of the thermobalance and added accessories is shown in Figure 1; a schematic diagram of the pressure balance module and its relation to the thermobalance is shown in Figure 2; and a block diagram of the thermobalance flow system is shown in Figure 3.

PRINCIPAL PARTS OF THE THERMOBALANCE REACTOR

The integral parts of the thermobalance reactor and a short description of each is as follows:

1. Pressure Vessel: The pressure vessel is a mild steel, water cooled vessel with the following dimentions - O.D-16 inches; I.D.-13 inches; and length - 56 inches.

2. Furnace/Insulation Section: This section consists of a three zone; 15kw (5kw per zone) 220 volt, single phase Kanthal heating element and cast refactory cement thermal barrier (ref. 3).

3. Reaction Vessel: The Reaction Vessel is made of 316 stainless steel and capable of withstanding 1000°C. Its dimentions are O.D.-5.56 inches; I.D.-4.81 inches and length - 47.50 inches.

4. Specimen handling and Observation section: This section consists of a flanged spindle with an observation port, a specimen handling vessel with a crank assembly (magnadrive) and a load cell which has a capacity of 100 grams.

5. Pressure Balance System: This section consists of an operating module with one back pressure regulator, one forward pressure regulator, two pressure gauges, four manually operated valves, one air operated valve for emergency vent, one air operated valve for the high limit shut off, one differential pressure transmitter, one differential pressure controller, and four solenoid operated air valves (ref. 3). The system is factory adjusted and calibrated to maintain the pressure balance system approximately 20 psi higher than the reactor section pressure. The process gas serves as the control gas for both pressure regulators. A pressure increase in the reaction section simultaneously opens the forward pressure regulator and closes the back pressure regulator - the reverse occurs if the reaction section pressure decreases. Two high limits are provided for each gas loop. If the pressure differential deviates from zero psig in excess of the first preset limit the pressure controller activates the solenoid valve associated with the high pressure, and causes the corresponding air operated valve to close. The closed valve reopens when the pressure differential returns to normal limits. If the pressure deviation continues a second preset limit actuates in a similar manner the valve pair associated with the high pressure gas. The valves return to normal operation when an acceptable pressure balance is reached (Fig. 2).

6. System Monitoring Panel: This section consists of three digital turbo flowmeters, four pressure gauges, three high pressure metering valves, three liquid flow indicators, four high pressure manual valves, and three manual liquid valves. This panel monitors the reaction and balance gas pressures which come from the gas manifold system. It provides an instantaneous visual pressure reading on the operational side of the system. It controls and monitors all flow rates, by means of the turbo meters, into the pressure balance system. It also provides manual and visual control of the cooling water in the system.

7. Operational Gas Manifold System: This section provides all the gases required for the operation of the system. It consists of five high pressure gas-in and high pressure gas-out regulator, four gas manifolding high pressure manual valves, and two gas cylinders(per operating gas)for helium, oxygen, nitrogen and air. Hydrogen is supplied from an independent source manufactured and located in another building complex.

8. <u>Electrical and Temperature Control Panel:</u> This section consists of three (one-zone) temperature controllers; three silicon controlled rectifier power controllers and three power transformers. The panel also includes a strip chart recorder for recording the weight loss of the sample, a digital temperature indicator which monitors all the temperatures and a lockable "Kill Switch" which when activated, in the "on" position, turns everything on or when activated in the "off" position, turns everything off.

9. <u>The Rider Guide:</u> The rider guide consists of a 48" long 10 carat gold chain, a wire connector, a set of 316 stainless steel sister hooks, a high temperature nicron wire, a $\frac{1}{4}$ X 5" long basket made of 316 stainless steel screen, an aluminum pully which works off the magnadrive, a free running cable made of a malable wire and an aluminum spindle or guide which is machined with a highly polished finish to approx. 1" diameter less than the internal diameter of the sample handling section spindle. A suitable hole is drilled in this guide to allow the high temperature wire to hang through it but small enough to catch the wire connector and cause the gold chain to pile on top of the rider guide as the basket is lifted or unpile as the basket is lowered. The gold chain is attached to the weighing transducer at one end and to one side of the wire connector on the other end. The high temperature wire is attached to the under side of the wire connector at one end and the sample basket is attached to the other end. The cable is attached to the pully on the magnadrive at one end and to the side of the aluminum guide on the other end. When the magnadrive is rotated the cable begins to wind on the pully causing the aluminum guide to rise. When it hits the wire connector the gold chain piles on top of the guide. The sister hooks provide an easy means of attaching the basket to the end of the high temperature wire. This is how the basket is raised and lowered.

10. <u>Accessories:</u> The main accessory meeded for the operation of the thermobalance is a means to analyze the product gas produced. This was done by incorporating a mass spectometer or if needed a gas chromatograph in the exit gas line.

INSTALLATION OF THE THERMOBALANCE

To accomodate the thermobalance reactor it was necessary to build a structure to support it (approximately ~ 3500 lbs) and some operating accessories such as a leveling device, a safety barrier and a work and accessability area. The means chosen to accomodate this was a small mezzanine floor (Fig. 4). The thermobalance could be suspended from the middle of the mezzanine floor low enough for an operator to work comfortably at the sample handling section yet the base of the thermobalance could be far enough off the ground floor to allow gas and electrical connecitons to be easily installed and serviced. A steel ring was installed from the top of the mezzanine floor to provide support for leveling screws. These leveling screws only level the system and do not support any weight. All weight is supported from the base of the unit.

The area chosen for the mezzanine floor was the north-west corner of Building #6. Here some support for the mezzanine could be utilized from the building wall ledge. The dimenisons of the mezzanine floor are 8 ft. wide, 14 ft. long (including a 2 ft. wide stairway) and 9 ft. high. It is constructed of a welded 6 inch I beam steel frame placed on the wall ledge on two sides and a 6 inch I beam on the outside corner. The floor is made of $\frac{1}{4}$ inch steel plate and a protection railing is surrounding the outer side A 24 inch diameter hole was cut for the thermobalance to stand in, at a spot that could be serviced by the overhead crane. A 24 inch square sub-frame made of 6 inch I beam was welded onto the main floor frame directly under the floor hole. Four 6 inch I beams 8 ft. high were welded on each corner of the sub-frame as support posts. A cradle constructed of welded 6 inch angle iron was bolted 21 inches down from the floor of the mezzanine onto the four support posts (fig. 8). A 23 inch I.D. diameter ring 5 inches wide was mounted 43 inches high on two 6 inch I beam posts for leveling purposes.

The position of the sample basket in relation to the reaction zone had to be established so that the five interior thermocouples could be set in the proper position to record temperatures as close as possible to the sample basket. The top cover of the thermobalance was placed in an upright position onto the leveling ring supported by three leveling brackets. The specimen handling and observation section was then mounted in the correct position on top of the top cover. The internal thermocouple guide was installed from the bottom of the top cover. A fishing line was suspended from the weighing transducer at the top of the specimen handling and observation section to the middle of the reaction section with a sample basket attached. The length of the fishing line was noted to be 81½ inches. A 10 K gold chain 16 inches long with suitable size linkage for free piling ability was ordered. The five reaction temperature monitoring thermocouples were installed through the top cover and through the thermocouple guide with the tips placed around the circumference of the sample basket 1 inch in height apart over the 5 inch length of the basket. The fishing line and basket was then removed. The specimem handling and observation section was removed. The inner reaction vessel was installed to the underside of the top cover. The top cover and attached reaction vessel with the thermocouples in place was removed and put aside while the main portion of the thermobalance was assembled. The rest of the thermobalance was assembled on the ground floor. The furnace leads were attached to the electrical pass through leads of the bottom cover. Then the furnace pedestal was bolted to the bottom cover. The main body and water jacket which came pre-assembled was then lowered over the furnace and bolted to the bottom cover. Because of height restrictions a special chain sling had to be made to lift the main body, water jacket, furnace, and bottom cover up to the mezzanine floor. Then the main assembly was lifted to the mezzanine and dropped through the ring to be supported by the cradle. The top cover was lowered with the reaction vessel positioned inside the furnace and the cover bolted to the main assembly, after three 7/8 inch diameter leveling screws were installed through the leveling brackets.

The specimen handling and observation section was mounted on the top cover and bolted down. Explosion walls were then installed around the main body of the thermobalance. A steel cubicle was made for the top portion and wall bolted to the existing mezzanine frame closed in the bottom portion. The walls are ¼ inch steel plate.

The gas manifold system, control panel and gas monitoring panel were assembled and all ancillary piping and electrical work completed (Fig 7). High pressure 1/4 and 3/8 tubing valves and fittings with appropriate rupture discs were installed on all gas inles lines in pressure areas. All water lines are 3/8 copper tubing. On the outlet side of all the water lines a flow control switch is installed so that if the flow is reduced to an unacceptable limit or fails the whole thermobalabce system shuts off automatically i.e. - electric power to the furnace shuts off, and the thermobalance depressurizes.

A gas exit line was installed at the sample handling and observation area where the gas outlet is located on the top cover (Fig 1). When the gas exits it passes through a high pressure filter, a depressurizing regulator, a dry test meter and then through the mass spectrometer (Fig. 5). Also if required, samples of the gas can be automatically taken at predetermined intervals and later analyzed with the gas chromatograph.

OPERATING PROCEDURES

The operating procedure to perform an experiment is as follows:

1. Turn on the water to keep the top and bottom covers and main body water jacket cool during operation.
2. Turn on the main power switch on the control panel. Turn on both recorders and the digital readout.
3. Set the temperature desired on each furnace controller and turn them on. (Fig. 6) To heat the furnace up to 1000°C takes approximately four hours.
4. Turn on the helium flow (Fig.1). Set the helium gas manifold system to the desired run pressure (which is the same as the pressure to be set for the balance gas and process gas in point #9).The gas metering valve which is located below the helium turbo flow meter on the gas monitoring panel,is opened slightly so that a small gas flow is permitted over the transducer to keep it cool while the furnace is heating up the reaction chamber.
5. Weigh the empty sample basket.
6. Place the desired charge in the sample basket and weigh.
7. Load the charged basket by hooking it to the end of the high temperature wire at the observation port on the thermobalance.
8. Seal the thermobalance by torquing down the observation port cover as per torquing instructions (Fig. 9).
9. Pressurize the thermobalance to the desired pressure. Set the outlet pressure of the process gas and balance gas at the gas cylinder manifolding section to the desired pressure. The process gas in and balance gas in manual valves are then opened simultaneously to permit gas flow. The

solenoid switches and air operated valves (Fig. 2) then automatically raise the pressure in the inner vessel and the outer vessel to the pre-set pressure of the gas manifolding section. The manual valves are normally open throughout the run. During pressurization the helium flow must be gradually turned higher as the pressure in the system rises so that a constant atmosphere of helium is around the transducer.

10. After the desired pressure has been reached set the digital turbo flowmeters (Fig. 1) to the desired flow of process gas through the reaction chamber.

11. The system must then reach equilibrium. The temperature profile must level off at the desired temperature and the process gas and helium coolant gas must be the same.

12. Turn the mass spectometer to "run" and check calibration of apparatus by plugging the gasline quick connect into the calibration station. If the calibration is correct switch the valve to "system flow" and allow the exit gas to pass through the mass spectrometer. Turn on the eight pen recorder on the apparatus to provide a hard copy of the gas analysis.

13. Drop the sample basket into the hot zone of the thermobalance by rotating the magnadrive manually twenty-two full turns in a clockwise direction. The transducer recorder should now be recording a full load.

14. Gasification should now be taking place. Run sheets should be provided all changes noted and all information recorded.

15 After the experiment is completed the sample basket is removed from the hot zone and returned to the starting position by turning the magna-drive manually twenty-two full turns in a counter-clockwise direction.

16. Turn off the three furnace controllers on the control panel. Cooling of the system now begins. (If a second run is desired omit this step and continue to the next step.)

17. The pressure in the reaction vessel and pressure vessel are returned to atmosphere by closing simultaneously the process gas "in" and "balance gas in" valves and then opening the "process gas out" and "balance gas out" valves simultaneously. The solenoid switches and air operated valves then take over automatically, to lower the pressure in the reactor vessel and the balance vessel to atmospheric pressure. When the system reaches atmospheric pressure close the process-gas out and balance gas-out valves. As the pressure in the system is dropping, the helium metering valve should be

slowly closed co-incidentallly with the drop in pressure until it is returned to the same status as reached in step #4. If a second run is desired, leave the helium flow flowing at a small rate and proceed to the next step. If however, a second run is not desired, wait until the system has cooled and then shut off the helium flow.

18. Shut the power off from the system by pushing the off button at the kill switch on the control panel. (Omit this step if a second run is desired).

19. Open the observation port by unbolting the cover plate.

20. Remove the sample basket from the end of the high temperature wire.

21. Weigh the sample basket.

22. Remove the high temperature wire from the end of the gold chain and replace it with a new wire of the same length. (ref. 2) (Note: this step must be done after every run)

23. If a second run is desired, return to step #5 and continue. If no further runs are desired, shut off the mass spectrometer and close all gas cylinders.

SHAKE DOWN TRIALS

The first trials were to pressurize the system to 200 psi and test for leaks, and then to repeat this until a reasonable operating pressure (1000 psi) was reached. A problem surfaced which was as follows: pressure could not be maintained in the reaction vessel, which behaviour at first was thought to be caused by leaks. When no leaks were found, and it was still not possible to maintain pressure in the reactor vessel it was thought that the normally closed valve (Fig. 2) was not properly adjusted at the factory or possibly it was faulty. A check of the exhaust line after the valve indicated that gas was leaking through when it should not have. After a check of this part of the system indicated that the valve was operating properly, attention was directed to the forward pressure regulator. This was removed, examined and tested using cylinder gas. This regulator was designed to operate in such a manner that pressure in the reactor vessel was maintained 20 psi lower than the balance vessel. In fact the forward pressure regulator did the opposite. Autoclave Engineers was then notified that it was suspected that they had installed the wrong forward pressure regulator. After checking, Autoclave Engineers confirmed this and they agreed to exchange the old one for the correct one. The replacement forward pressure regulator is expected by August 3, 1981.

REFERENCES

1. Nandi, S.P., and Johnson,J.L."Catalytic Gasification of Lignite Chars" paper presented at the Coal Gasification Symposium, American Chemical Society, Fuel Chemistry Division, Washington, D.C., Sept 9-16, 1979.

2. P. S. Soutar
   Visit to the Institute of Gas Technology
   26th & 27th March 1980, Trip Report

3. Autoclave Engineers Ltd.,
   Thermobalance Reactor Engineering Data Book
   Erie, Penn. USA

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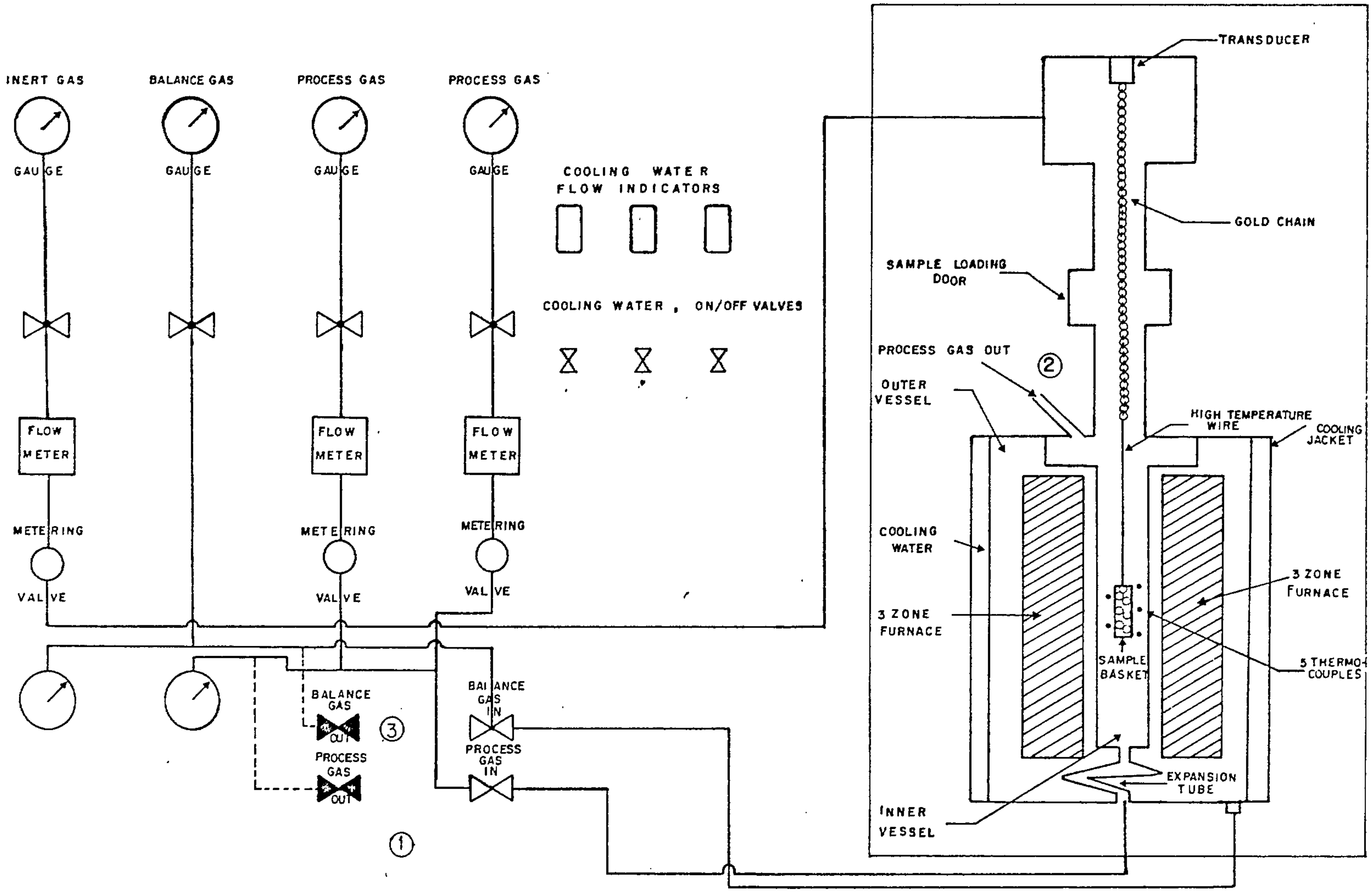

Fig. 1 - Schematic diagram of the thermobalance and added accesories

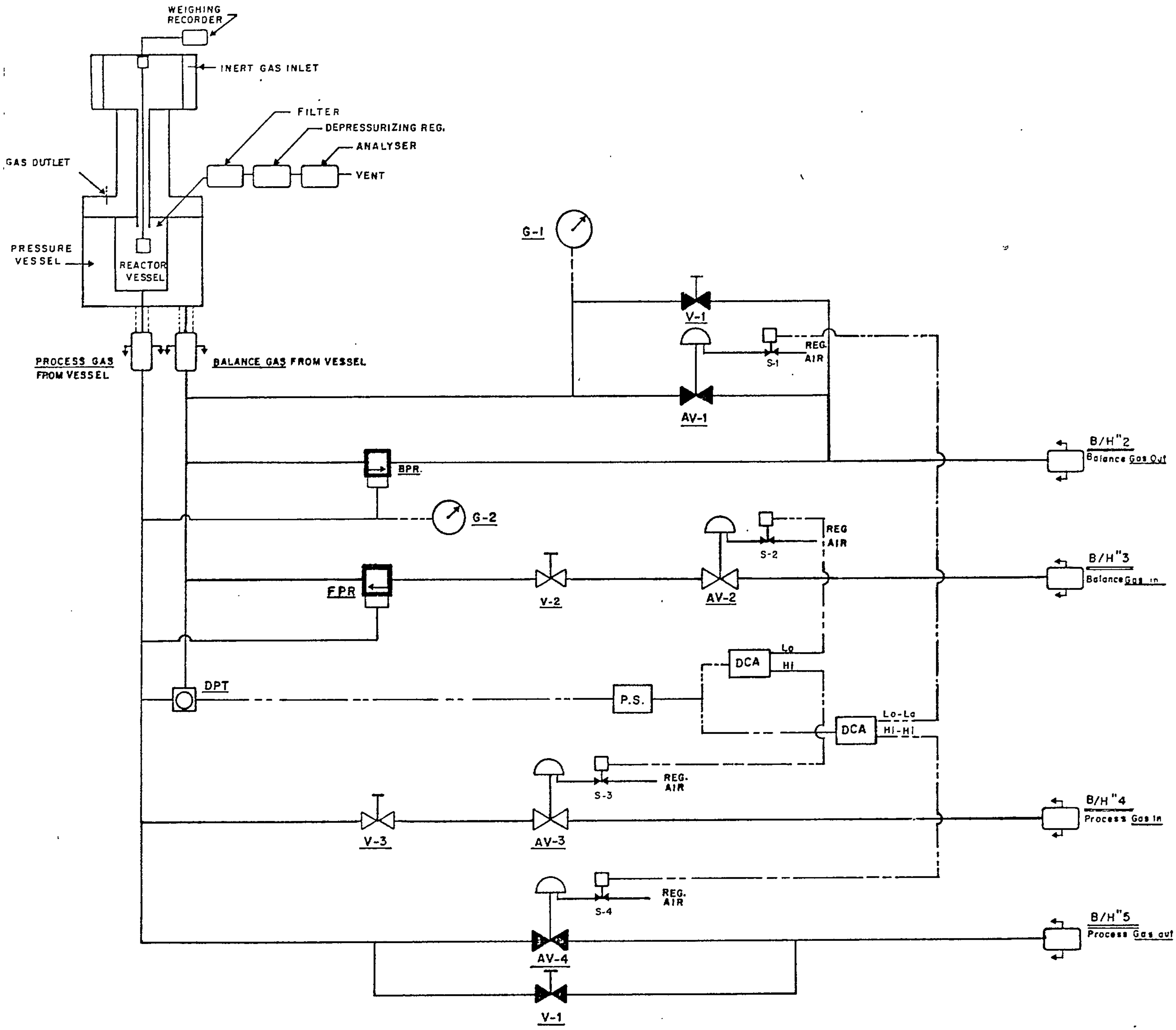

Fig. 2 - Schematic diagram of the pressure balance module and its relation to the thermobalance.

LEGEND

FPR	–	Forward Pressure Regulator
BPR	–	Back Pressure Regulator
	–	Normally Open Valve
	–	Normally Closed Valve
DPT	–	Differential Pressure Transmitter
AV	–	Air Operated Valve
V	–	Manual Valve
G	–	Gauge
DCA	–	Direct Current Alarm
S	–	Solenoid

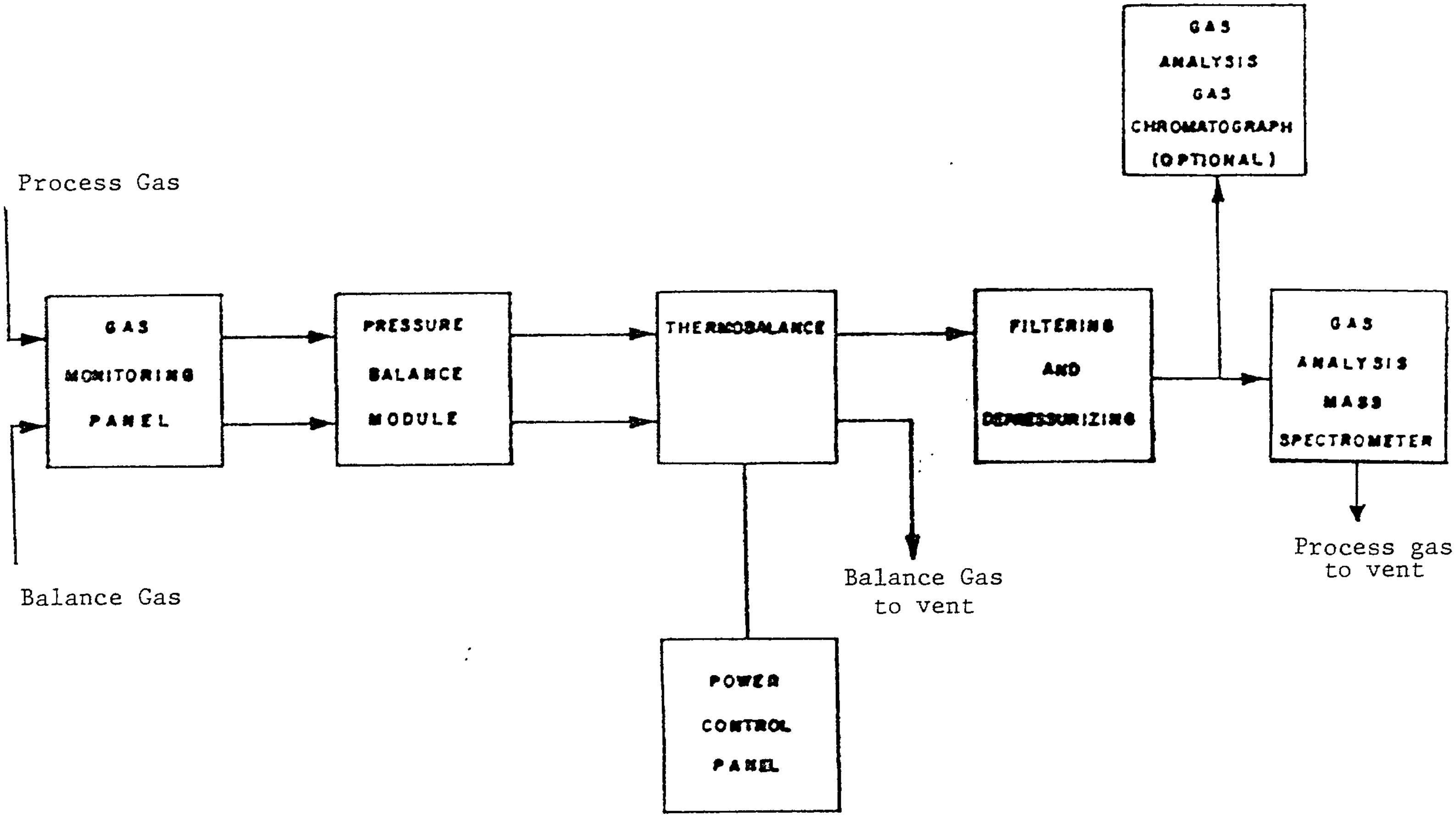

Fig. 3 - Block diagram of the thermobalance flow system.

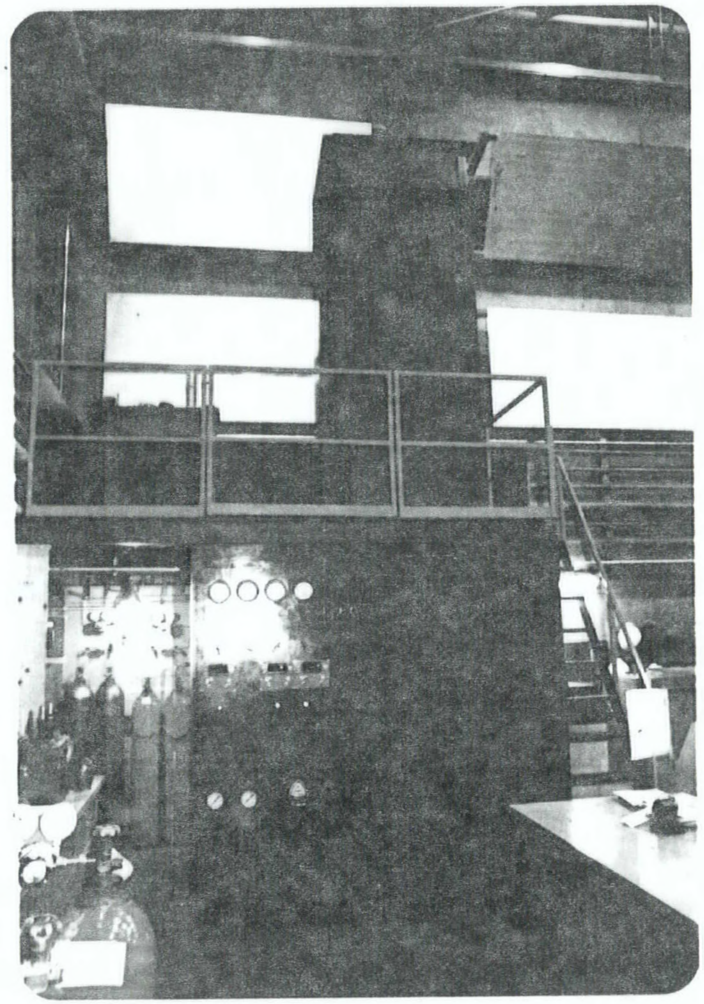

Fig. 4 - Overall view of thermobalance system.

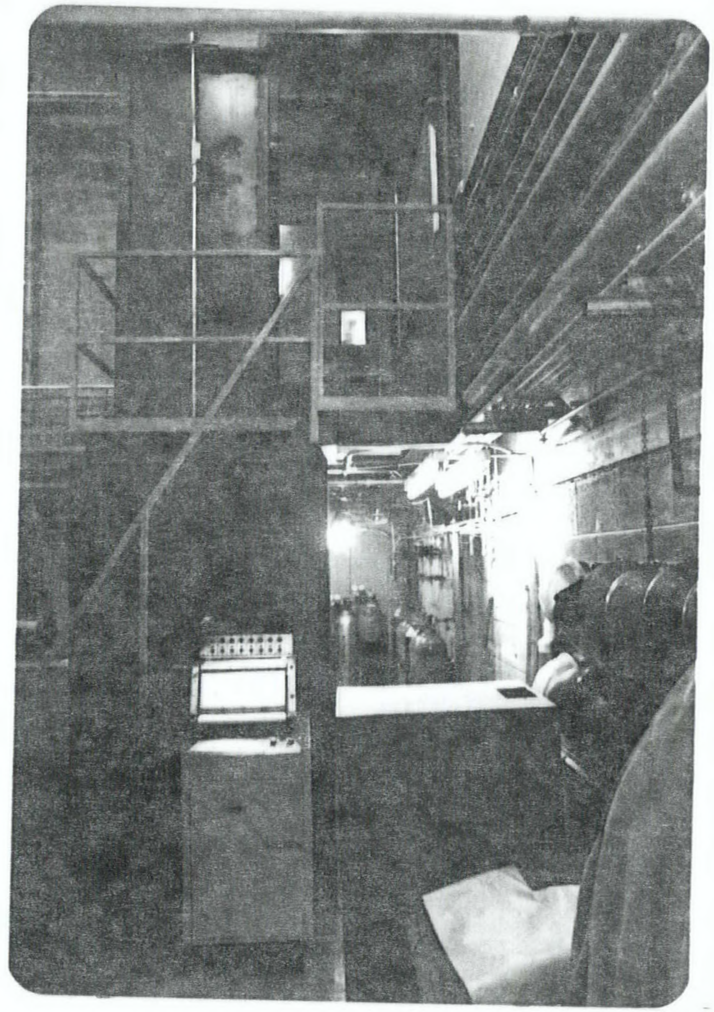

Fig. 5 - Side view of overall thermobalance system.

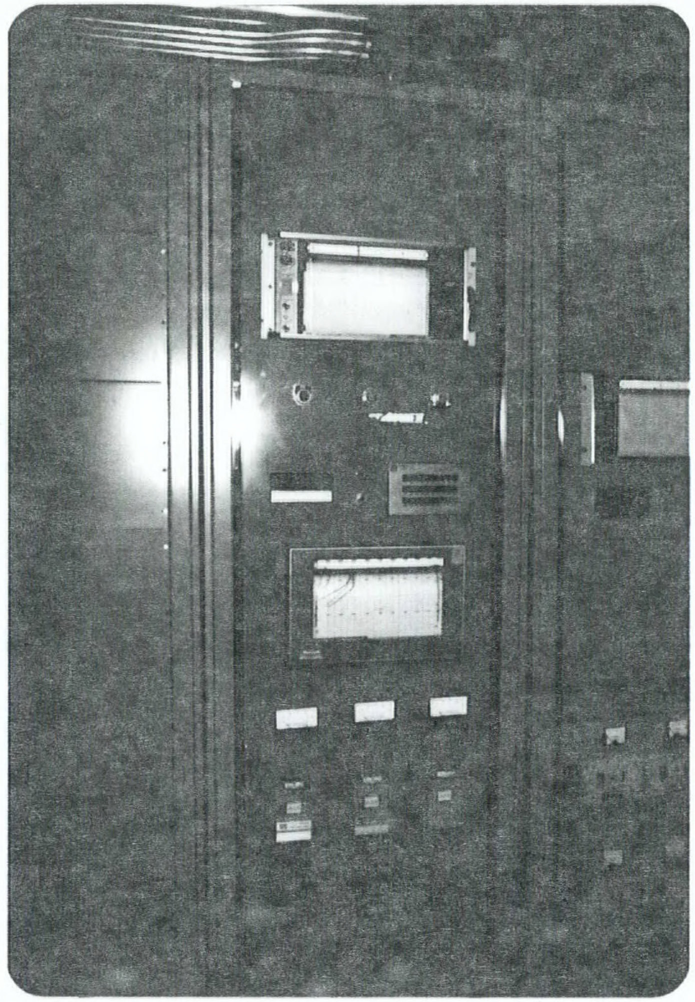

Fig. 6 - Control Panel

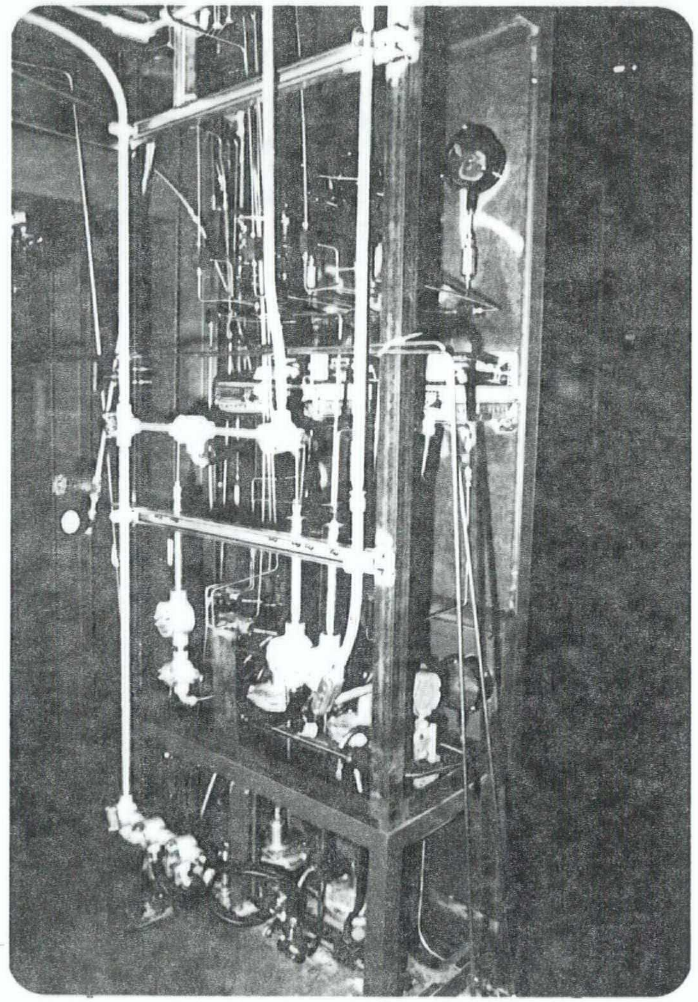

Fig. 7 - Rear view of pressure balance and gas monitoring systems.

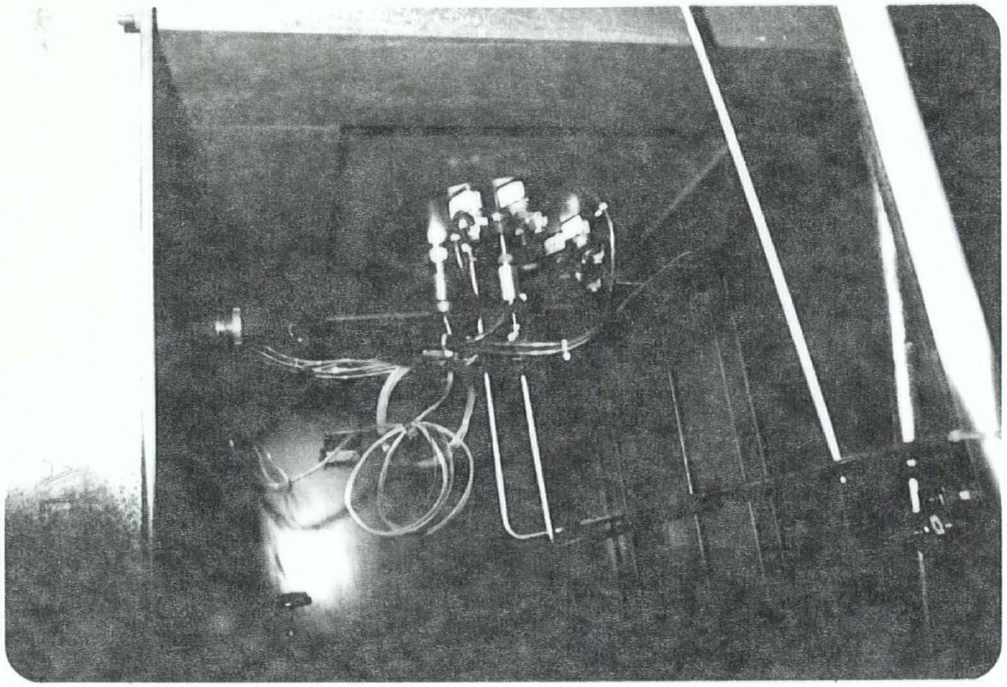

Fig. 8- Thermobalance reactor bottom view.

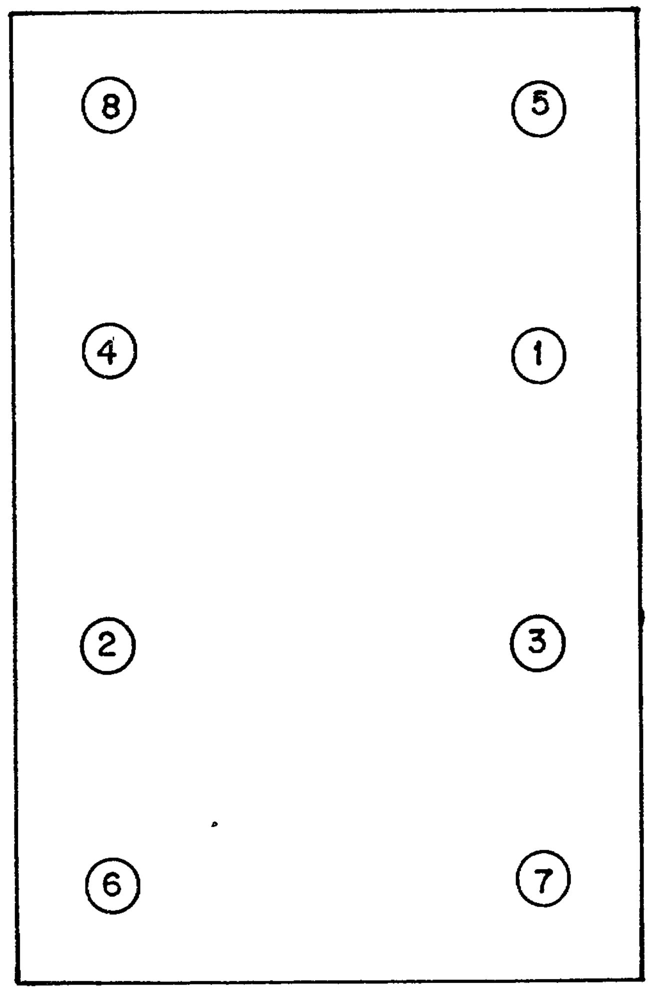

Fig. 9 - Sample port torque sequence.