CHAPTER III

EXPERIMENTAL EQUIPMENTS AND PROCEDURE.

3.1 The Small Mass Spectrometer.

The instrument could be divided into two parts, the body and the removable cover. They were both made of brass. The heating unit, slits, the ion collector and the pressure indicator were located on the cylindrical part of the main body of which the dimensions were 10 cm. in diameter, 0.3 cm. in thickness and 2.5 cm. in height, whereas the connecting tube was 28 cm. in length and 1.8 cm. in diameter, bending at an obtuse angle. The rubber tube was used for connecting it with the adaptor of the diffusion pump. There were two flat surfaces of the device, the fixed wall and the removable cover. Both of them were squares in shape. Around the rim of the circular hole of the opening, there was the circularly annular groove. There were eight bolts positioning in a circle around the groove in which an O - ring was placed in contact with the cover for preventing the leaks. About 49 c.c. of volume of air at one atmosphere was to be pumped out. Shellac in alcohol and epoxy-resin were used as the semi-permanet sealings. The plan and the side view of the instrument were shown in Fig. 3 and Fig.4 respectively.

The heating unit, slits, the ion collector and the pressure indicator were now considered. Two capillary glass tubes, having narrow holes inserted with the copper wires and filled with epoxy resin were provided for the heating unit. Both of them were fixed to the body by epoxy-resin. One end of the glass tube was outside

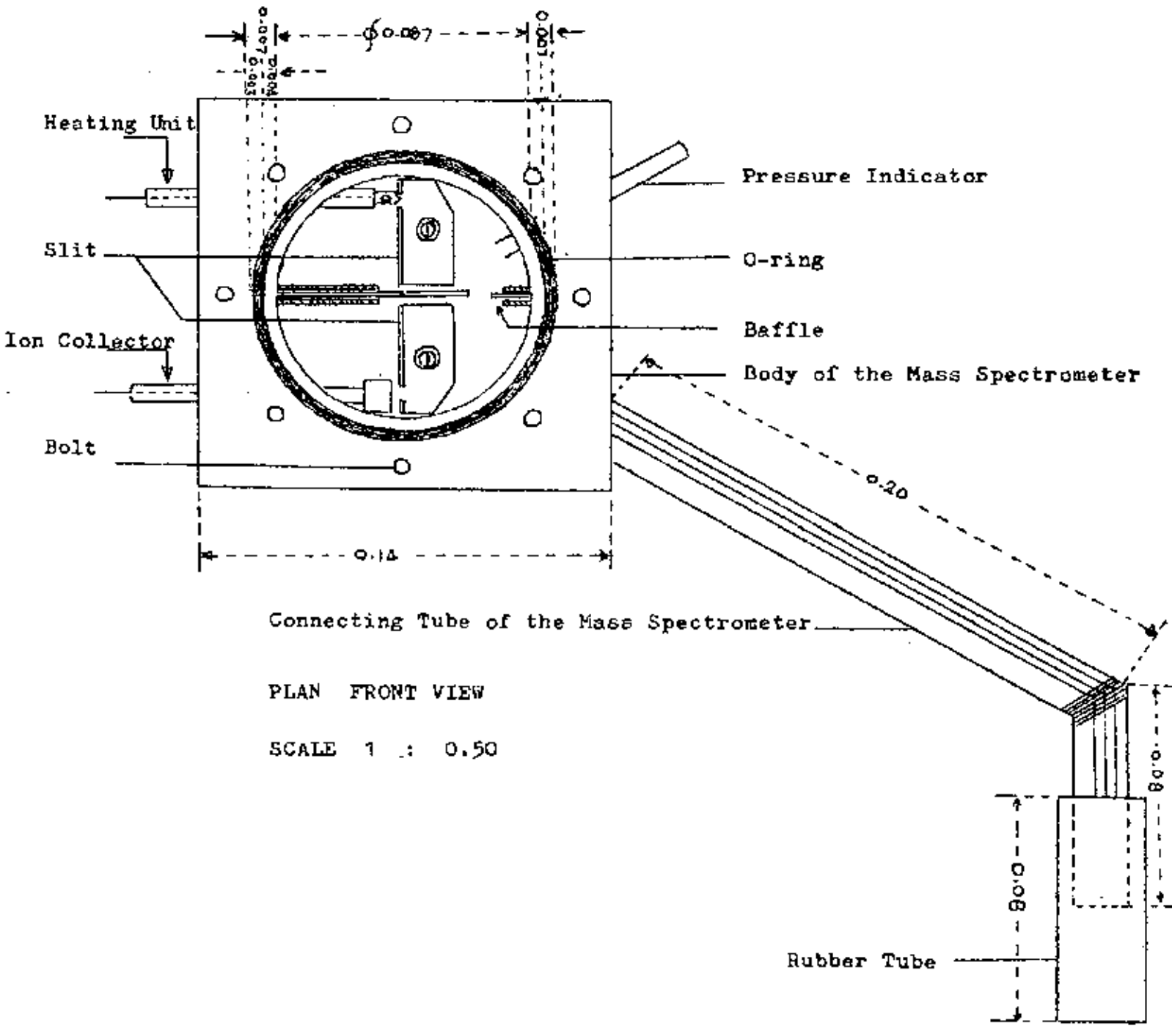


Fig.3 The Front View of the Mass Spectrometer

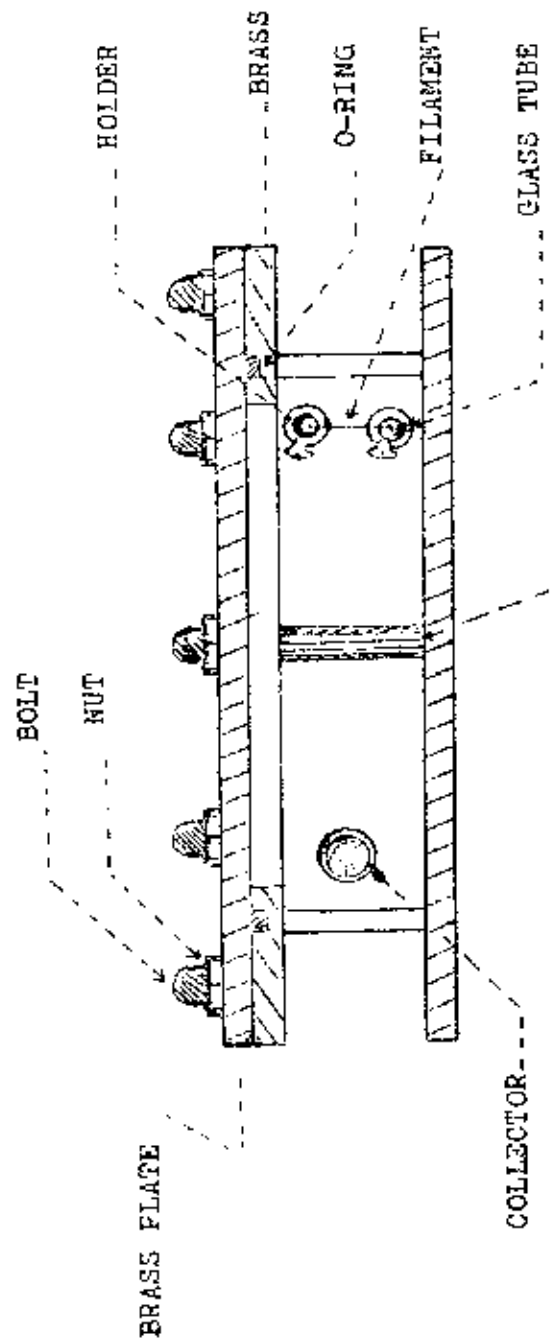


Fig.4 The Side View of the Mass Spectrometer

the instrument and the other end was inside. Tungsten wire was passed through the small circular holes of the needles attached to the inside ends of the copper wires by the electrical connectors.

The ion collector was made of brass in the form of a cylindrical box with one side opened to collect the ions. It was joined by the copper wire inserted through the narrow hole of the capillary glass tube at the end lying inside the instrument. The glass tube was also fixed to the body by epoxy-resin.

The slits S_1 and S_3 were made of thin sheet of brass folded perpendicularly in two planes. One plane could be attached to the base wall of the body by means of screws, the other plane was cut into a slit as it was needed. The slit S_2 or baffle was also made of brass. There was a socket for the baffle, through which it could be easily shifted according to the desired width. The brass socket itself was fixed to the body by soldering.

The pressure indicator was a pyrex glass tube, fixed to the body by epoxy-resin with the opening end inside the instrument and the closed end at the outside.

The slit and the heating unit were shown in Fig.5 and Fig.6 respectively.

3.2 The Megavac and the Metrovac Vacuum Pumps.

The rotary pump was the Cenco Magavac vacuum pump having the serial number 968 and catalogue number 92003. It was operated at 600 r.p.m. and was guaranteed to produce an absolute pressure of 0.0001 m.m. of mercury. The rubber tube for the connection with

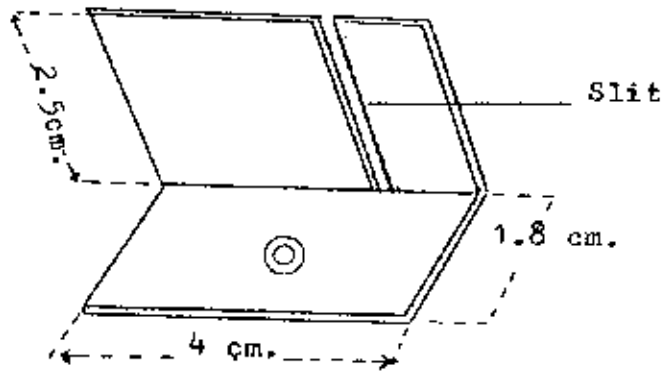


Fig.5 The Slits for the Ion Source and the Detector.

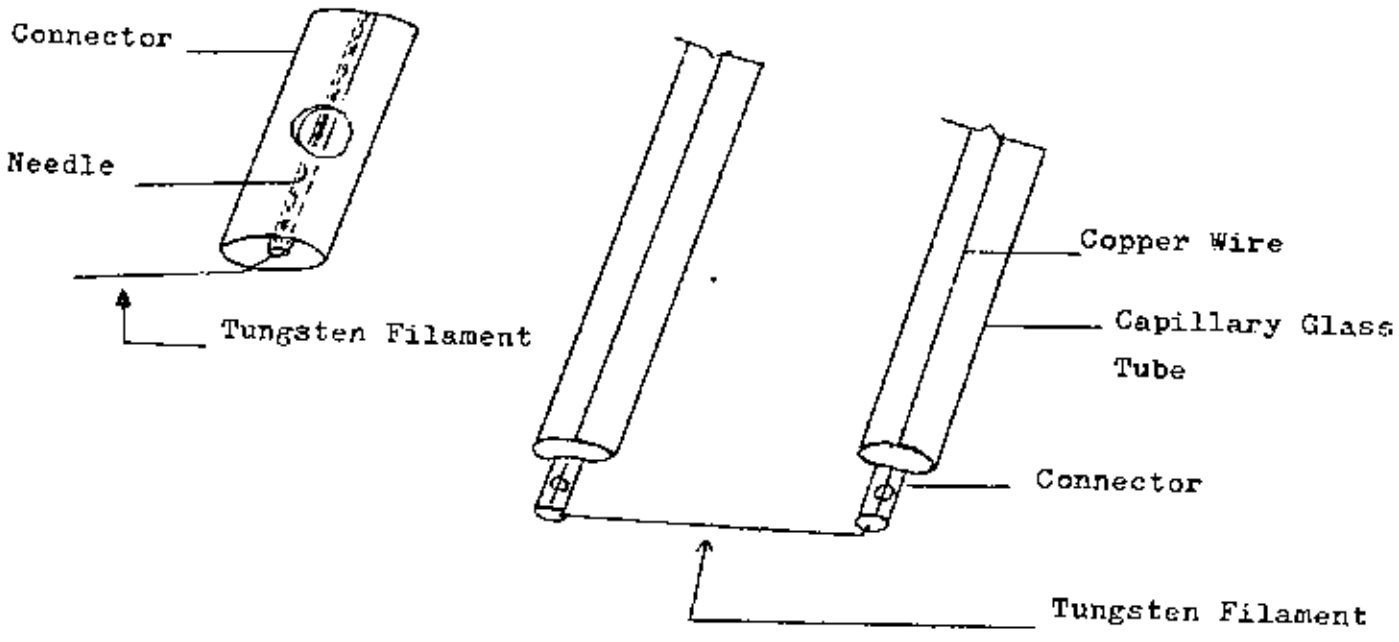


Fig.6 The Heating Unit.

the diffusion pump was 40 cm. in length and 4 cm. in diameter.

The diffusion pump was the Metrovac vacuum pump type 03B manufactured by W. Edwards & Co. (London) Ltd. It was the two-stage oil diffusion pump and was supposed to produce a pressure below 10^{-6} m.m. of mercury. The working fluid of the diffusion pump was silicone oil No. 704. It was found to resist to both cracking and oxidation when exposed to air at high temperature.

3.3 The High Frequency Oscillator.

It was the push - pull negative resistance oscillator and it was constructed on a wooden frame by Mr. Bhiyayo Panyarjun. Its circuit was shown in Fig.7. The output voltage was 600 volts and the oscillation frequency obtained was 32 Mc/s.

The high frequency electrical energy was coupled to the pressure indicator electrostatically, by wrapping two pieces of copper wire around the outside end of the glass tube about 3.5 cm. apart. Then the two copper wires were connected directly to opposite ends of the oscillator coil of the oscillator.

3.4 The Heater, the Accelerating Voltage Supply and the Measuring Amplifier.

The Variac Cenco, Cat. No. 80297 A, the filament transformer and the a.c. ammeter Welch Cat. No. 3081 F were provided for the heater. The a.c. current for heating the tungsten filament was not more than 3 amperes, otherwise the filament got quickly burnt out.

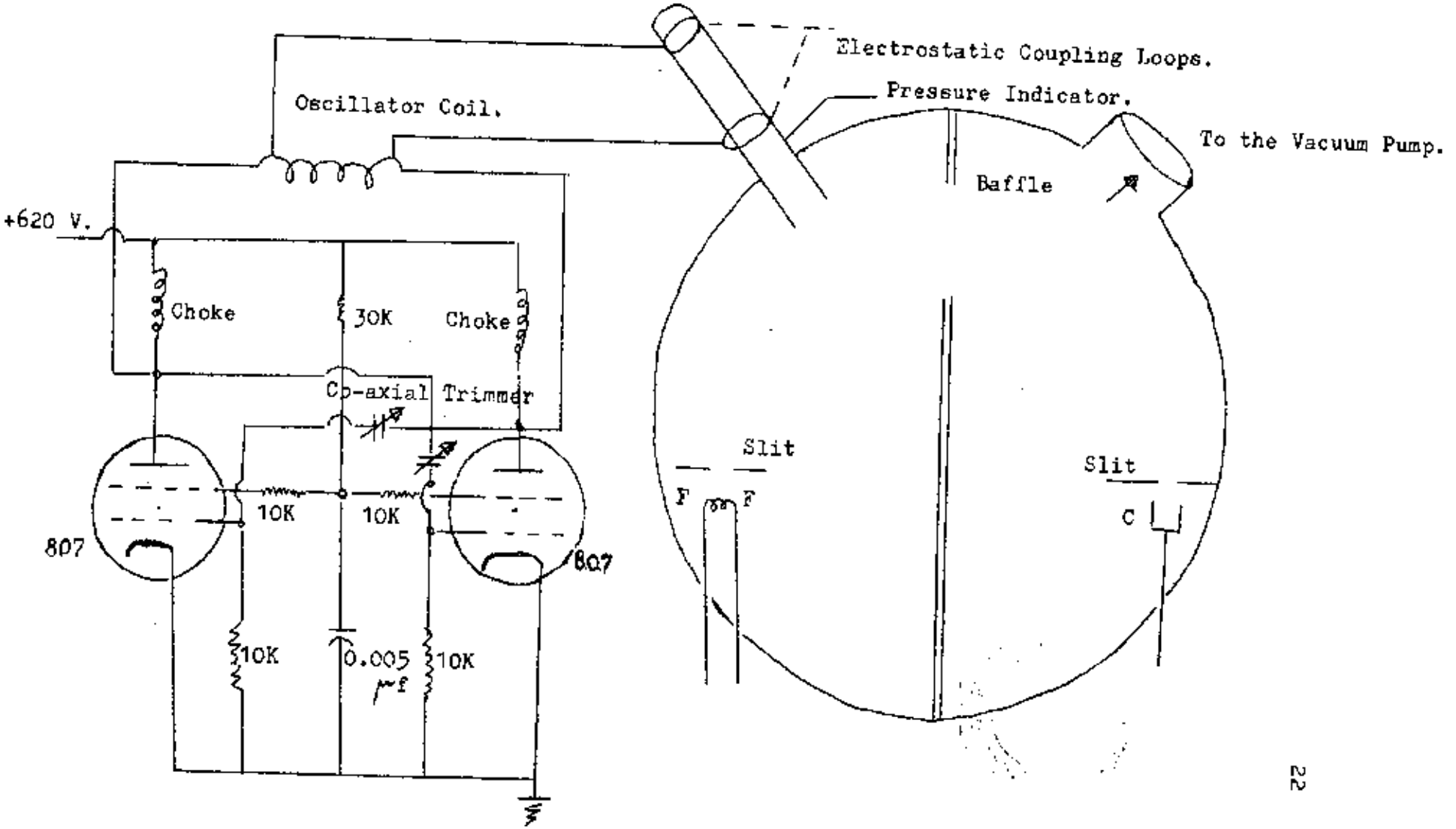


Fig.7 The Oscillator Circuit.

The power supply was electronically stabilized and the range was from 0 to 400 volts. The accelerating voltage was measured by Knight V.T.V.M. manufactured by Allied Radio Corp. Chicago, Illinois.

The measuring amplifier was Leybold Cat. No. 53201 of E. Leybold's Nachfolger. It was used to measure small currents. Its characteristic feature was a very high input resistance. The Universal Avometer Model 8 of the Automatic Coil Winder and Electrical Equipment Company Limited and the resistance box Cat. No. 4775 by Leeds & Northrup Co. were connected in parallel to the output of the measuring amplifier for detecting ion currents after positive ions' impinging on the ion collector. The measuring circuit was shown in Fig.8.

The measuring amplifier has five current measuring ranges shown on the left side of the range control switch 30×10^{-8} A, 30×10^{-9} A, 30×10^{-10} A, 30×10^{-11} A and 30×10^{-12} A. The factor 30 corresponded to the 100 scale divisions referring to full scale deflection on the Universal Avometer by which the resistance box setting at 550 ohms was connected in parallel.

The diagram of the heater, accelerating volage supply and the measuring amplifier were shown in Fig.9.

3.5 The Electromagnet.

For this experiment, the electromagnet constructed at the Physics Department many years ago, was used. The electromagnet has 10 cm. pole face diameter. The resistance of the coils in series

Fig.8 The Measuring Circuit.

F-F The Filament
C The Ion Collector
P The Regulated Power Supply
E The Measuring Amplifier

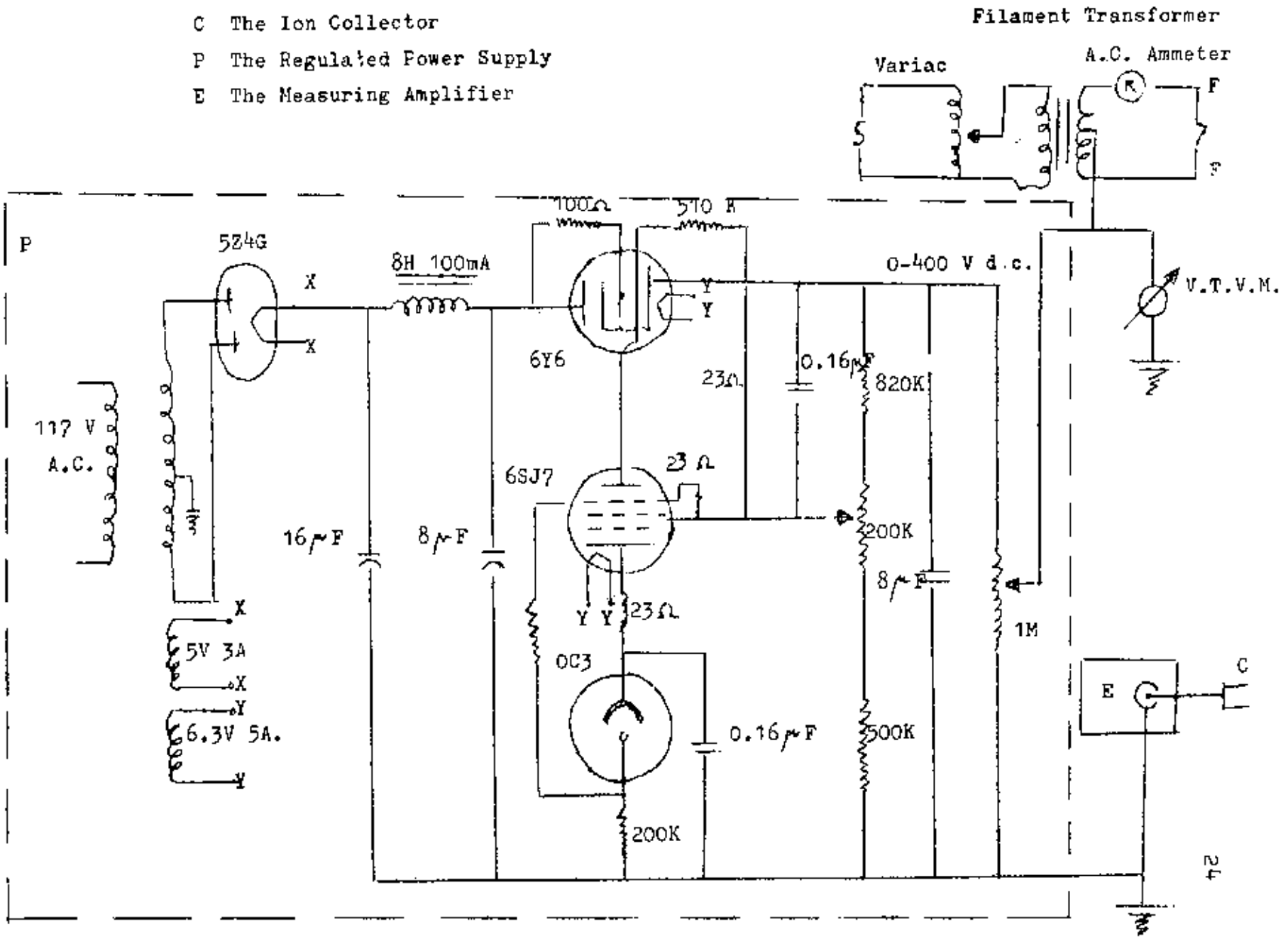
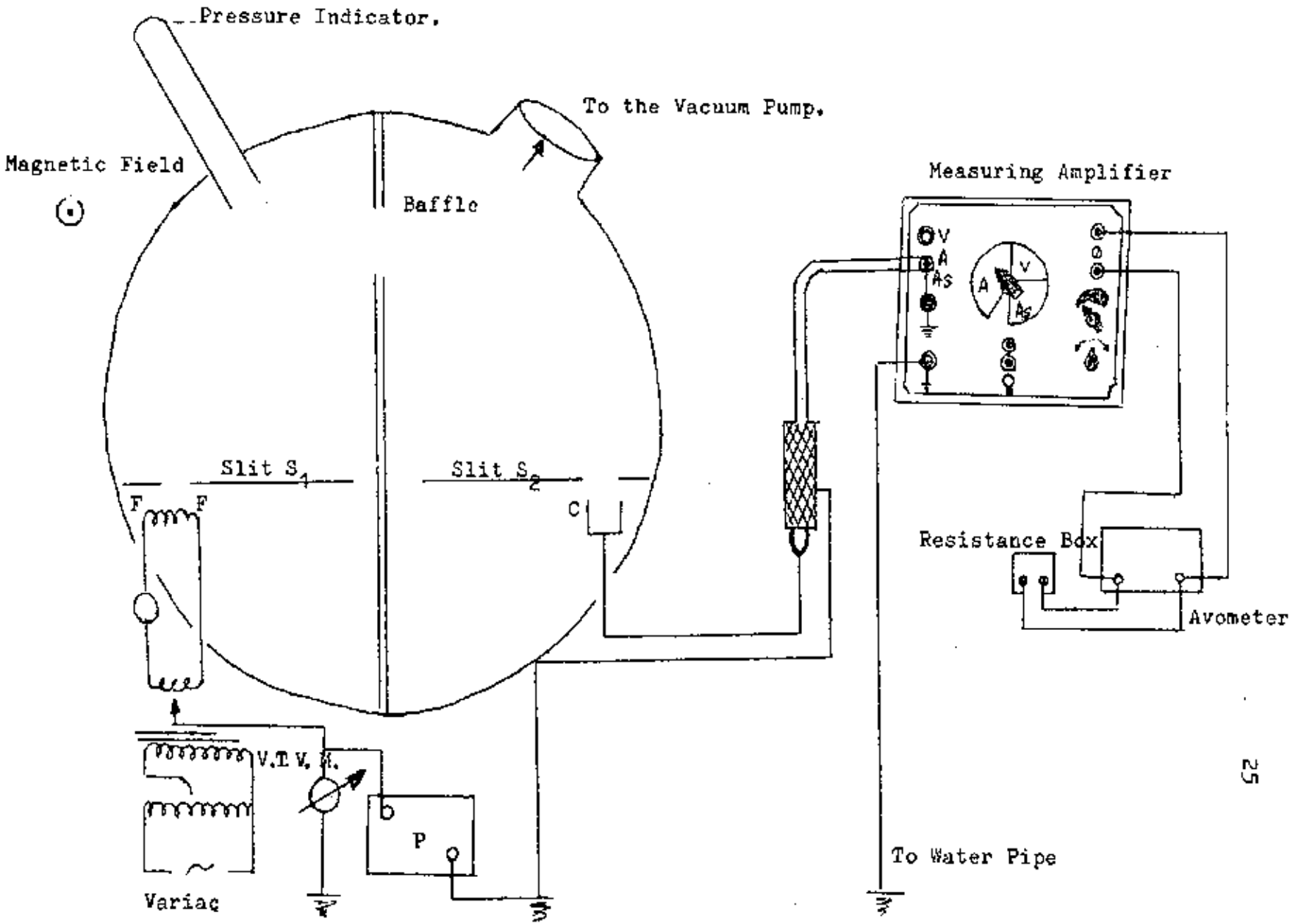


Fig.9 The Diagram Showing the Heater, the Accelerating Voltage Supply and the Measuring Instrument.



is 1 ohm and the total number of turns of the copper coils is 2424. The pole gap of the electromagnet can be adjusted by means of screwing the cores with respect to the yoke. The current for the electromagnet was supplied by the Bridge Selenium Rectifier A.E.G Leybold No. 523. The maximum input voltage of the rectifier was 20 volts, and the powerstat Cenco. Cat. 80298 A was used. Consequently the magnetizing current could be varied smoothly from 0 to 10 amperes by reading from the d.c. ammeter Model 45, No. 37521 of the Western Electrical Instrument Corp.

3.6 Experimental Procedure.

The apparatus was shown in Fig. 10. Sequence was important and it was as follows :-

1. Cleaning Procedure, Ion Source and Slits Preparation.

First of all, the inside of the mass spectrometer must be kept clean without dust by rubbing with a cotton pad soaked in benzene A short length of 0.01 cm. diameter tungsten wire was used as the filament. In order to separate the isotopes of K^{39}, K^{41}, anhydrous KCl in powder was mixed with 2 - 3 drops of pure water. Then it was coated directly on the tungsten filament and was allowed to be absolutely dry by means of the heat from the electric lamp. The slit S_1, having 0.02 cm. slit width was set in its position. The baffle or slit S_2 was arranged for having the width of 1.38 cm. as calculated, and the slit S_3 of 0.12 cm. slit width was positioned.

A small amount of apiezon grease L was smoothly coated as a thin film on the O-ring. Thereafter the cover was placed directly

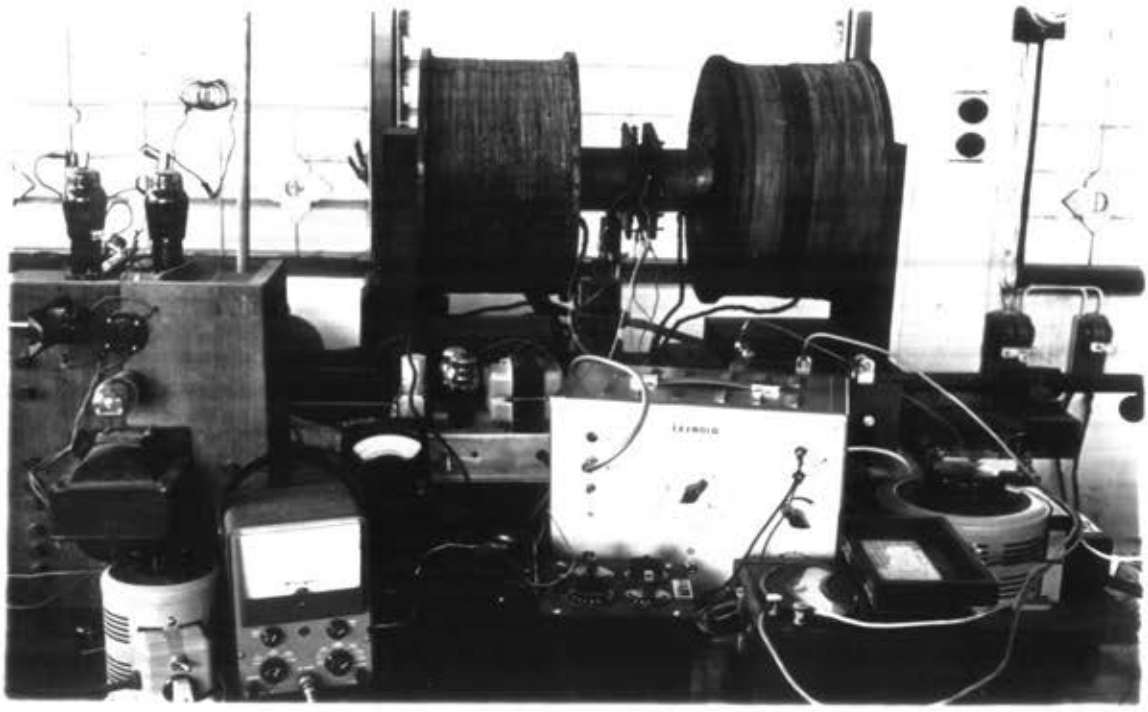
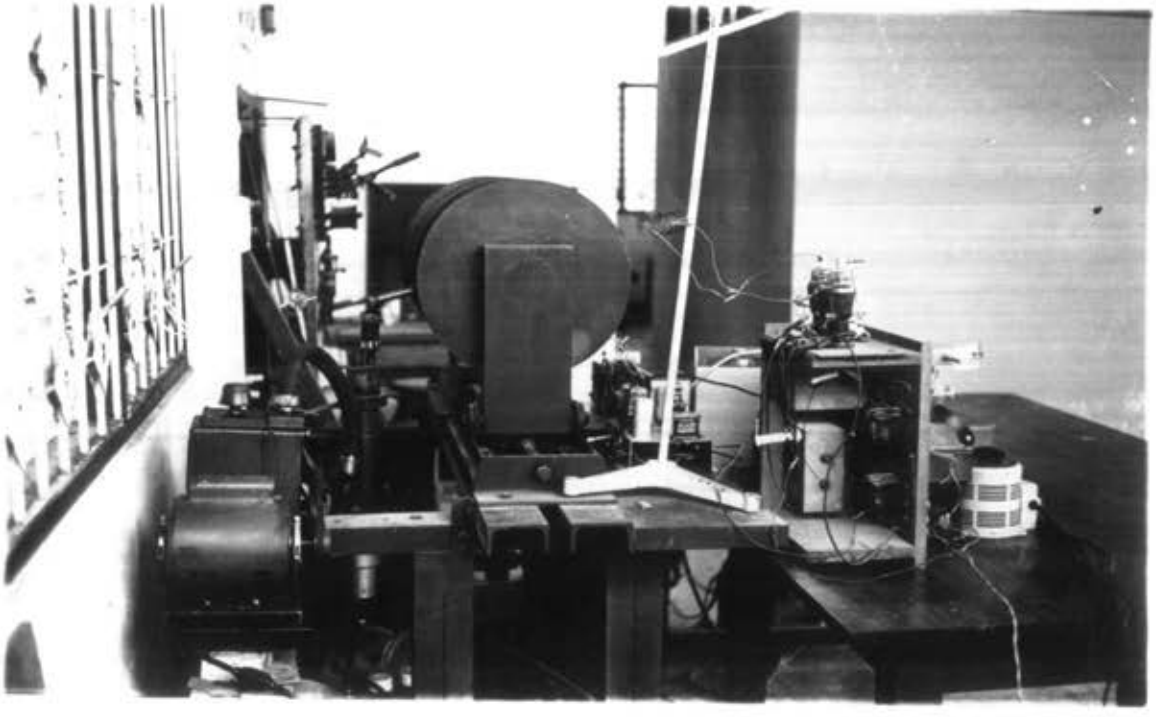


Fig. 10 The Apparatus. (Front View)



(Side View)

on the O-ring and the eight nuts were tightly driven. Then the mass spectrometer was placed in the air gap closely touching the pole faces of the electromagnet perpendicularly to the direction of the magnetic field whereas its connecting tube was connected to the adaptor of the diffusion pump. It was assured that the junctions were vacuum tight by the clamps. The two pieces of copper wire from the oscillator coil of the high frequency oscillator, were attached to the pressure indicator about 3.5 cm. apart.

2. Vacuum System

The rotary pump was started after making sure that the air admittance valve for venting the system to the atmosphere had been closed. Progress of the pumping was followed by turning on the high frequency oscillator. At first, red glow was observed due to the band spectrum of nitrogen, and twenty minutes later, the colour of the glow changed to light purple. While passing water through the cooling coil of the diffusion pump, its heater was switched on. Half an hour later, no glow was obtained if there was no leak. If there were some leaks in the system, glow would persist on. The leaks would be found out by tracing with the cotton wool pad soaked in benzene on the mass spectrometer. Either epoxy-resin or shellac in alcohol was sealed at the leaks. In practice, after three hours of the working of the diffusion pump, a good vacuum system with a pressure below 10^{-5} m.m. of mercury, was obtained.

3. Magnet Operation.

By using a search coil and its accessories, the magnetic field strength was determined. It was found that the field varied with

the magnetizing current. For the separation of the isotopes K^{39}, K^{41}, the magnetic field strength was 3100 gauss when the magnetizing current was 9.6 amperes.

4. The Measuring Amplifier, the Heater and the Power Supply.

The ion collector was connected to the current input socket and the case of the mass spectrometer was joined to the earthing socket connected directly to the casing of the measuring amplifier. At any rate, the earthing socket during operation, must be connected to a good earth conductor for instance a water pipe. For this experiment, the measuring amplifier was used with main supply of 220 volts. After a warming period of half an hour, the knob of the gain control of the device was turned up fully. The set-zero knob was then used to adjust zero deflection on the Avometer.

When the measuring amplifier was set ready to detect small currents. The heating current of about 2 amperes was passed through the filament. When the filament was hot enough, red glows were observed at the ends of the two glass tubes of the heating unit. The power supply was turned on and the accelerating voltage was varied from 150 to 230 volts for the separation of the isotopes of K^{39}, K^{41}. As the accelerating voltage was varied, ion currents were read from the Avometer instantaneously.

5. Shut Down Procedure

After detecting the ion currents, the electromagnet was turned off. The power supply, the heater and the measuring amplifier were then turned off in sequence. The diffusion pump was

switched off. Cooling water was left on until the heater of the diffusion pump had been off for at least fifteen minutes. Then the rotary pump was switched off and at the same time the air admittance valve was opened for admitting air to the system.

For repeating the experiment or changing the new alkali salts, the procedure was carried out in proper sequence as mentioned.