

A THESIS
PRESENTED TO THE FACULTY OF ARTS
IN PARTIAL FULFILMENT OF THE REQUIREMENTS
FOR THE DEGREE OF
MASTER OF SCIENCE
BY

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"ON THE DESIGN AND CONSTRUCTION OF
A MASS SPECTROMETER"

Introduction

Long before the term Mass Spectrograph or Mass Spectrometer was invented, experiments had been carried out in an effort to learn something of the nature of positive and negative rays. The experiments of Sir J. J. Thomson and W. Wien marked a great step forward in our understanding of atomic physics. By the application of electric and magnetic fields to fast flying particles in a discharge tube, these experimenters were able to measure the velocities as well as the ratio e/m of the charge to mass both of the cathode rays, which turned out to have always the same value of e/m , and some of the positive particles, for which e/m was not always the same.

In 1919 Thomson (1) was lead to conclude from the results of certain positive ray experiments that the element neon was capable of existing in two forms with masses 20 to 22 respectively, and shortly thereafter Aston (2,3) constructed an improved apparatus for studying these rays. Because of the close analogy of this apparatus with the optical spectrograph, and the similar "spectrum" of sharp lines obtained on a photographic plate when exposed to the positive rays issuing from it, Aston called the instrument a "mass spectrograph".

As early as 1921, Dempster (4) constructed a mass spectrometer

which was employed in the investigation of the metallic ions, and with which he demonstrated the isotopes of lithium, calcium, magnesium and zinc. The first quantitative determinations of packing fractions were begun by Costa (5) who designed a mass spectrograph in 1925 capable of greater accuracy than any instrument in existence at that time. In 1930, Bainbridge (6) constructed a mass spectrometer applying an original principle of velocity focussing, and on account of the high resolving power, he was able to make a critical search for unknown isotopes.

Smythe and Mattauch (7) in 1932 devised a mass spectrometer in which the mass rays were analysed without the use of a magnetic field. A mass spectrograph of resolving power 10,000 was constructed by Bainbridge at Harvard in 1936 (8) and was used in demonstrating isobars. In 1936 Nier described a new mass spectrometer of the 180° type (9), improved it in 1937 (10) and made abundance measurements upon the isotopes of several elements. In 1940 Nier described a 60° type spectrometer (11) intended for routine abundance measurements upon the lighter elements.

The mass spectrometer is indeed an instrument of many uses some idea of which may be gained by reference to the following:

- 1) Used in the determination of absolute atomic masses.

- 2) Used in the discovery and identification of isotopes.
- 3) Applied to relative abundance measurements of isotopes
 - i) in biochemical tracer work.
 - ii) in isotope exchange investigations.
 - iii) in following the course of reaction mechanisms and chemical kinetic studies.
- 4) Used in gas analysis
 - i) Where only a minute sample is available.
 - ii) For distinguishing between isomers in a mixture.
 - iii) For identifying soil gases in petroleum prospecting.
 - iv) In operational control work in the refinery.
 - v) In the study of reaction kinetics in gaseous systems (12).

The mass spectrometer to be described is a modified Nier 180° type. The modifications devised by us emphasize the use of precision methods in construction, and several new features of design have been originated.

Assembly of the Tube.

The mass spectrometer tube was built entirely upon a jig. As may be gathered from the accompanying photographs, the jig was constructed from a piece of steel 1 inch thick, approximately 18 inches square, and with a surface milled flat. The purpose of the jig is to have a permanent, substantial support upon which all the necessarily critical lining-up may be done. In order to support the glass envelope of the tube, metal "V" blocks were cut, machined flat and dowelled so as to rest perfectly plane upon the heavy base of the jig. The dowels were prepared so as to form a tight "press fit" when the blocks containing them were laid over the holes drilled in the base to receive them. As a further means toward permanence, $\frac{1}{8}$ " cap screws were used to fasten the blocks to the base. An extension consisting of a perfectly flat sheet of $\frac{1}{8}$ " steel was bolted to the base, and an additional "V" support erected upon this to steady the "U" section of the tube. The outlines of the envelope and the proposed course of the ion beam were now accurately scratched upon the base as well as the 180° and 183° base lines which serve to check the alignment of the D and S_3 plates in the vertical plane. This feature of the jig is shown in Figure 1. The glass envelope of the tube was prepared according to the blueprint herein included.

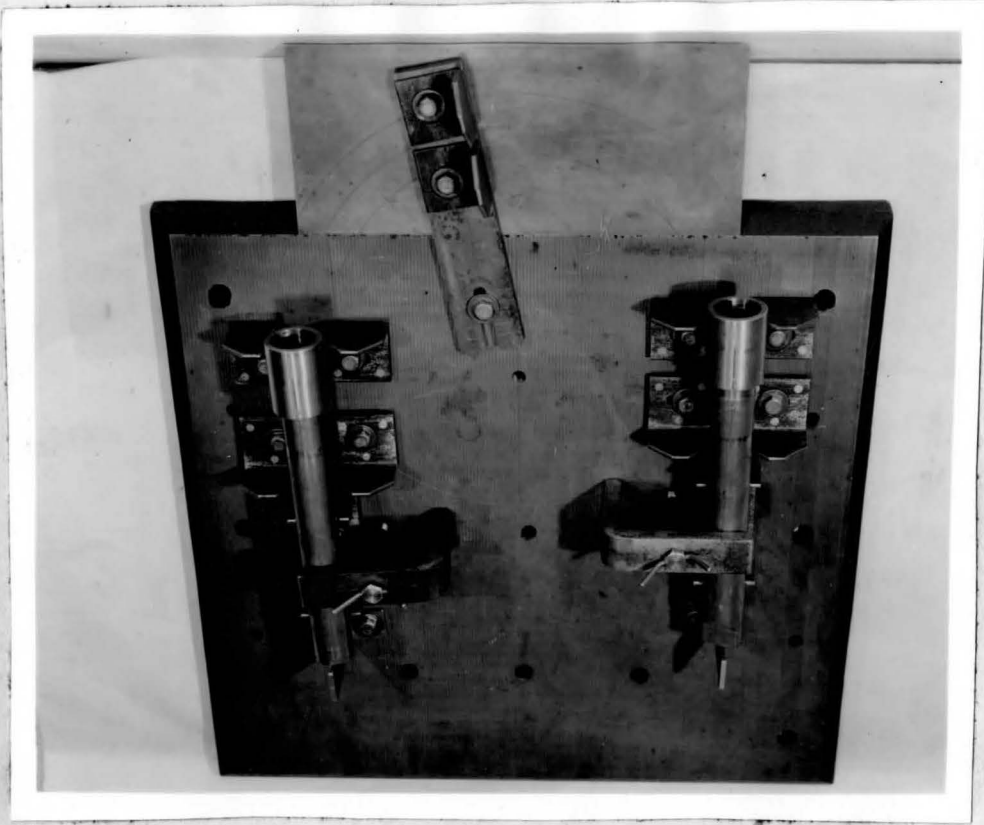


Figure 1.

As a support upon which to construct the plate assemblies, two rods were cut from machine steel and accurately turned to 1.025" and 1.030" diameter respectively, the outside diameters of the inner glass seals. Cylinders were then machined, slightly tapered on the inside at one end, so as to fit snugly over the ends of the above rods. The cylinders were made sufficiently long so as to allow approximately 1 inch to protrude beyond the end of the rod. This combination of rod and cylinder was then accurately placed in the "V" blocks and tightly clamped in place. The glass envelope was now slipped into the "V" block bed and the inner glass seals slid carefully into the protruding portions of the cylinders. The tube was thus held firmly yet accurately in place while the shielding was prepared and slipped into the tube prior to sealing the two halves together. This is illustrated in Figure 2.

The method of shielding presented a rather difficult problem. Although other spectrometer tubes have been shielded by providing a copper analyzer tube, we felt that the weight of such a body would be too great to allow suspension inside the inner seals. Moreover it is difficult to keep such a tube accurately centred by means of spacers inside the curved envelope. We were hesitant about trying to plate a shield inside the tube, for although this method works well for sometime, the plating tends to flake away from the glass wall, leaving a minute pinhole unshielded, and since this causes serious fluctuations in the

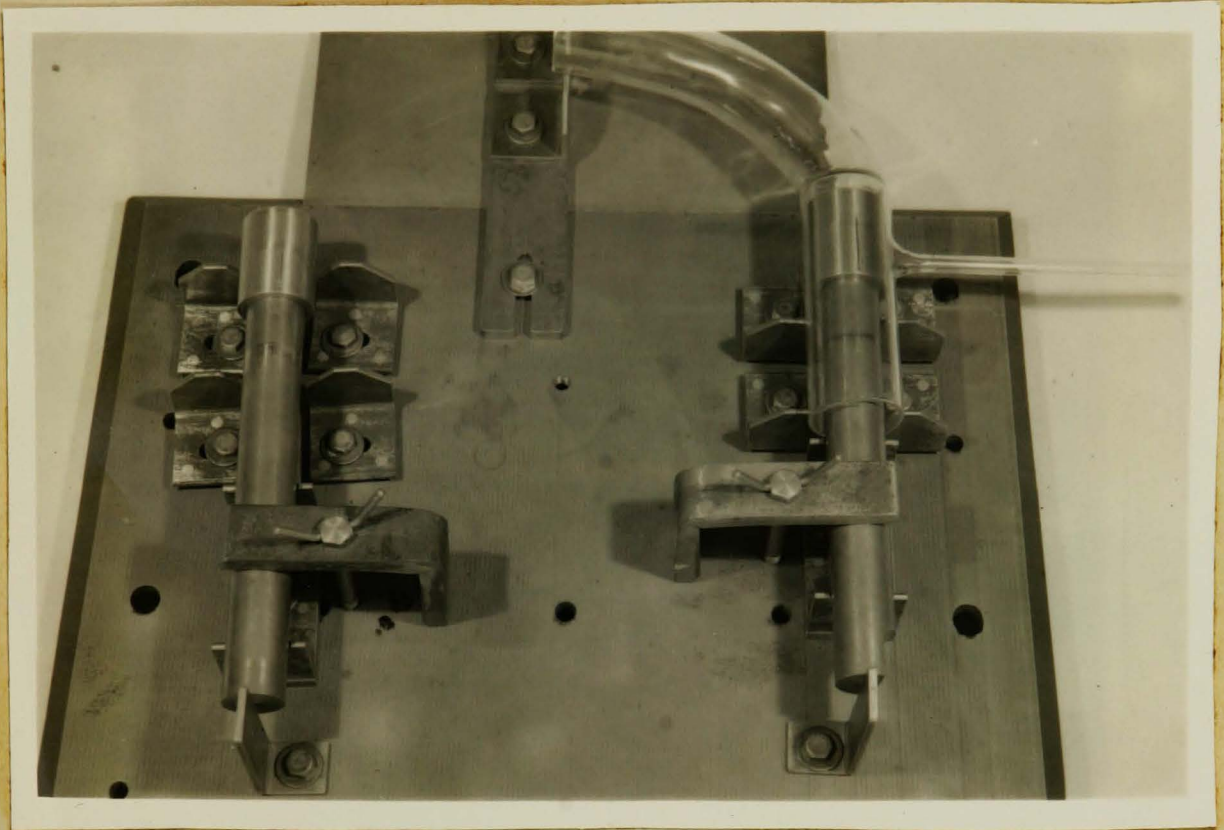


Figure 2.

ion beam, this expedient was abandoned, temporarily, at least. We finally arrived at a solution to the shielding problem by constructing a series of small "stovepipe" sections slightly tapered (see Figure 3.) so that one would fit well into the adjacent member. These components were fashioned from 0.0004" Nichrome "V" sheet, and if made sufficiently small can be caused to round any bend very neatly. The only restriction imposed is that the telescoping sections shall be cut off as short as possible so as not to extend into the path of the ion beam.

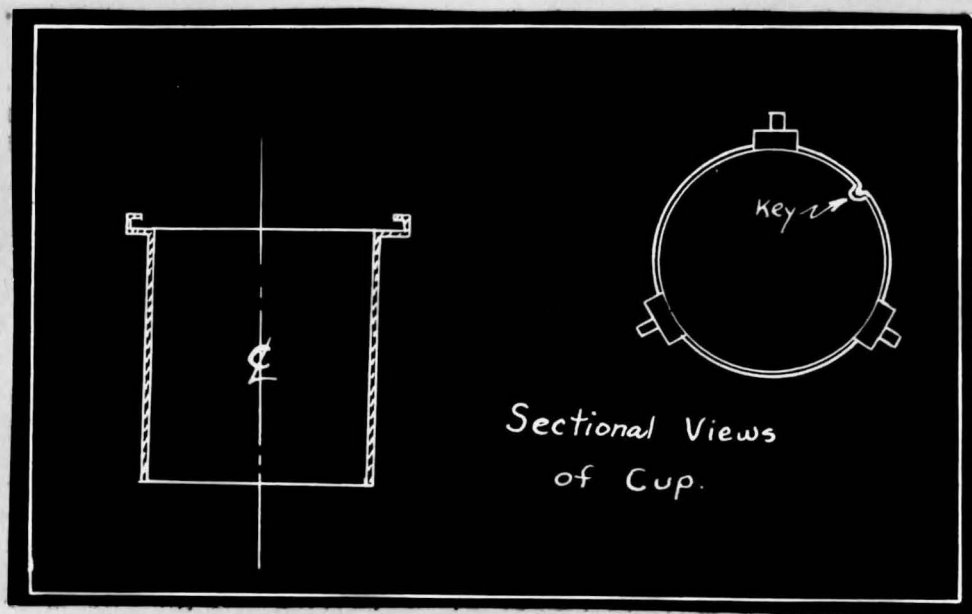


Figure 3.

The nichrome shield was grounded by spotwelding on a piece of #26 B. and S. Nichrome wire and running this up through the side pumping lead to a tungsten seal. Before insertion, each section of the shield was given treatment by dipping in hot concentrated aqua regia followed by thorough washings with distilled water, and drying. In this manner a bright, metallic surface was exposed and this provided good electrical contact from one section to the next. As soon as the shield had been completed and inserted, the tube was placed upon the jig and the two halves were sealed together.

The plate assemblies were now constructed. Seamless cylindrical cups were made and ground so as to fit snugly over each of the inner glass seals. It is well at this point to have the cups slightly larger than the outside diameter of the seals and to grind a slight taper inside the cups so that the farther down they are pressed upon the seals, the tighter they grip, yet may be readily removed. Equally spaced about the circumference of the cups were three slot-springs which may be adjusted to cause the cups to slip on more tightly or loosely as desired. Also provided upon the cups at one end were three sectional flanges bent at right angles to the cups; the purpose of these is to provide a base upon which the entrance and exit slit plates may be securely spot-welded. In order to allow good shielding, it is necessary that these cups be fashioned in such a way that the top surfaces of these

flanges may be ground even with the end of the cup so that when placed upon a perfectly flat surface, no light may be seen issuing through the crack between the end of the cup and the flat surface. A small indentation in the form of a key was hammered into the top of each cup, its purpose being to slip into a corresponding groove ground into each of the inner glass seals. With this arrangement, the cups may be taken off the seals and later replaced with the assurance that they will be in exactly the same position as before. Figure 3 shows the essential features of the cups.

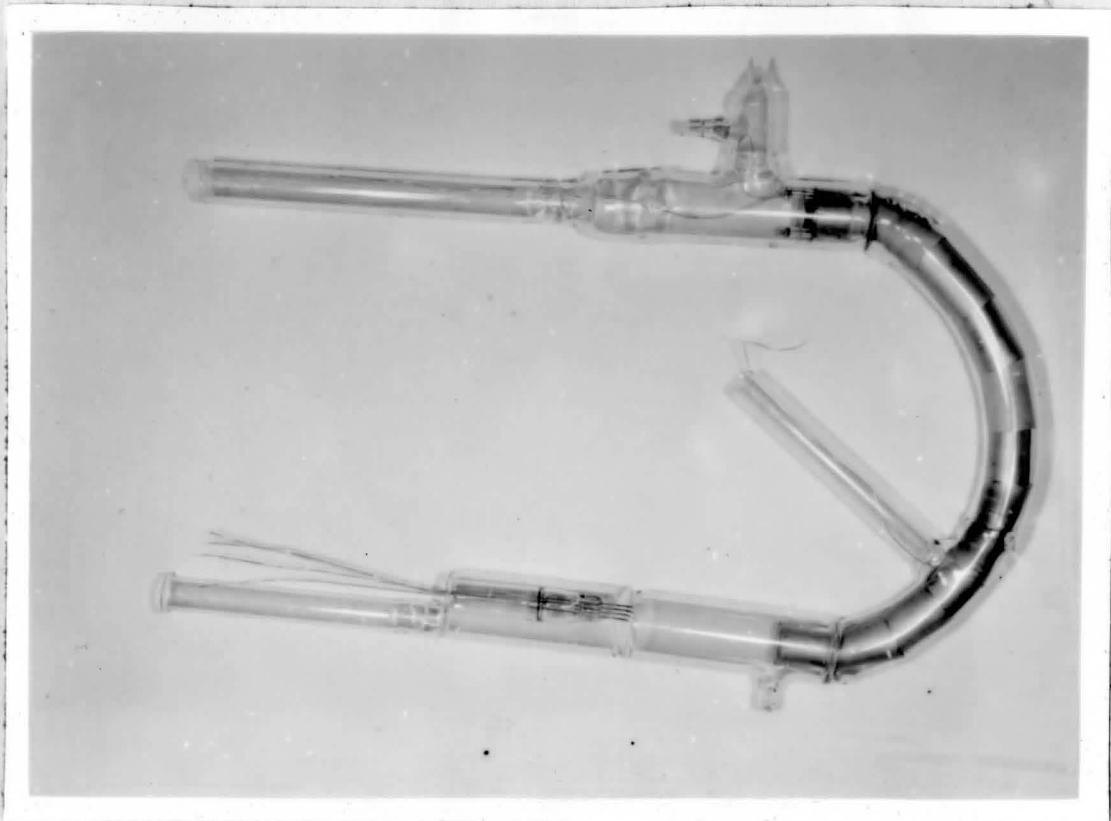


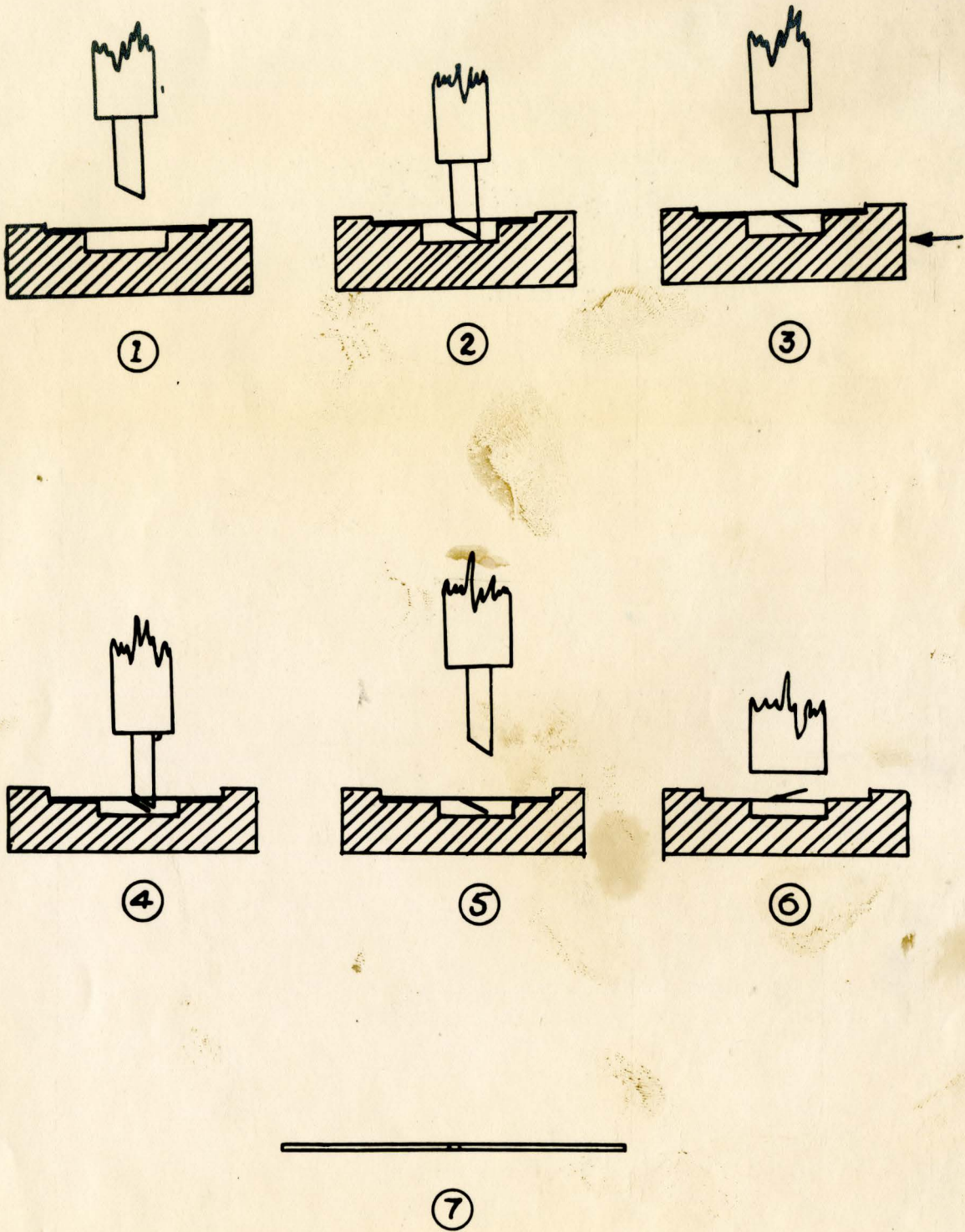
Figure 4.

In the matter of producing accurately cut circular plates 0.025" thick, we have made a die 3.5 cm. in diameter with which the discs may be quickly stamped out. It is perhaps well here to add that the Nichrome sheet is exceedingly tough and difficult to work, so that the die method of producing plates or other parts is probably the most convenient. Upon a circle 2.7 cm. diameter four holes 3 mm. in diameter were drilled into each plate to receive the supporting studs. The A plate further was provided with two stud holes for the B. plate supports, and one for the trap. In the centre of the A plate was drilled a 1/16" hole through which the gases to be analysed diffuse into the region of the electron beam. A rectangular section 13.5 mm. long and 8 mm. deep was cut from the A plate to serve as an opening into the electron box. In order that all drilling could be done in a precise manner, a drill jig was constructed, so that it was merely necessary to clamp the jig over the plate and make the drill holes.

A very neat method of accurately cutting slits in the plates was developed by the machinist. A rectangular die with a slanted cutting edge is set into a press (see Figure 5, #1), and the plate about to receive the slit is accurately placed and clamped over a corresponding recess in a tool upon a micrometer adjusted bed. The die is fixed in such a manner as to be able to penetrate the plate only to about $\frac{1}{8}$ " (Figure 5, #2), thus producing a sort of tongue in the plate. The plate is now shifted laterally by the micrometer adjustment an amount equal to the width of the slit

(Figure 5, #3) and the die brought down once more (Figure 5, #4). This cuts out a piece of metal equal to the thickness of the slit, (Figure 5, #5). The plate is then turned over and the tongue hammered back into flat coincidence with the plate (Figure 5, #6). The finished slit (Figure 5 #7) may be turned out in a few moments and the entire process is illustrated in Figure 5.

Figure 5.



A rectangular plate 11 mm. x 14 mm. was cut from 0.025" sheet Nichrome and drilled for two studs; this comprised the B plate. A die was made with which we could stamp out the filament sheet from 0.025" stock. From the 0.008" sheet were cut the forms which were bent into the electron and trap boxes respectively. The trap plate and collector were also cut from the 0.008" stock and bent to shape. Figure 6 and Figure 7 illustrate the detail of the plates and boxes

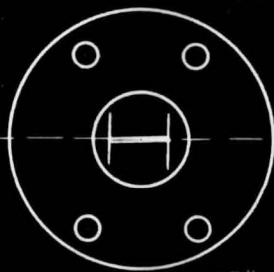


Plate D
Slit = 0.25mm.

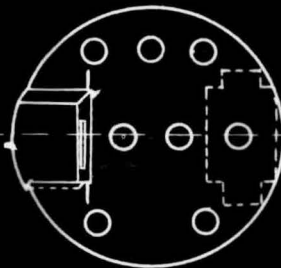


Plate A

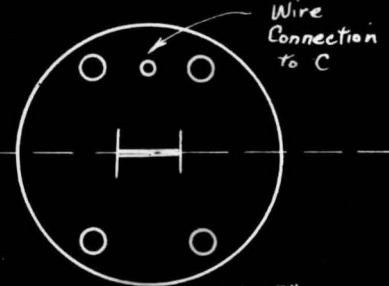
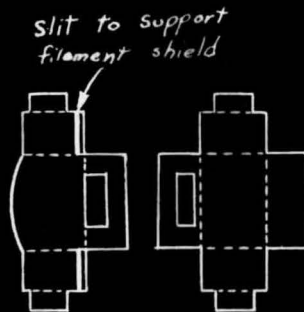


Plate C
Slit 0.38mm.

All parts Actual Size,
of Nichrome V



Plate B



Details of Ion Source

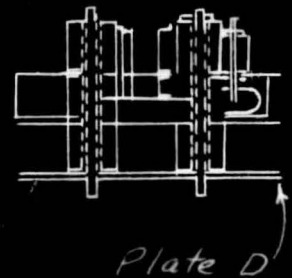


Figure 6.

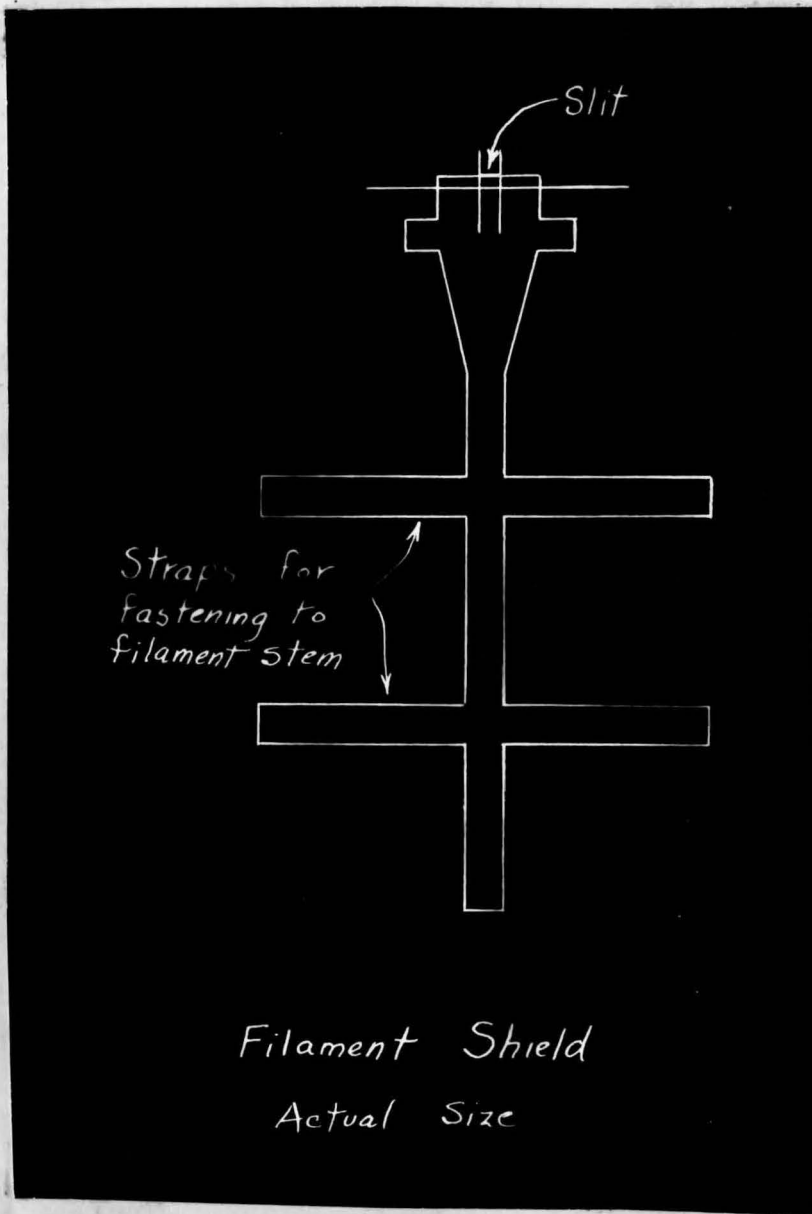


Figure 7.

For the rather tedious process of making small nuts, a die was made by which several hundred nuts could be blanked out in an hour. The nuts were made from the 0.025" nichrome stock and were drilled and tapped to fit the studs. The studs were fashioned from #15 Nichrome wire cut to length and threaded to receive the nuts. The stud lengths were as follows:

4 studs 26 mm. long for the A, C and D plates.

4 studs 18 mm. long for the S₃ and S₅ plates.

3 studs 15 mm. long for the B plate and trap.

The glass spacers used to separate the plates were made from Pyrex capillary tubing 6.0 mm. O.D. x 2.5 mm. bore, in the following lengths.

12 spacers 6 mm. long between the A, C and D plates; and between the S₃ and S₅ plates.

11 spacers 4 mm. long between the A plate and locknuts; and between the S₅ plate and locknuts.

3 spacers 2 mm. long between the B and A plates; and between the trap and A plate.

The glass spacers were made for us by the University of Minnesota glassblower and were ground flat at both ends accurately to the lengths required. In order to insulate the plates one from another as the studs went through them, internal spacers were made by pulling down to capillary size ordinary 8 mm. tubing of such a diameter that they slipped over the studs and would fit inside the 6 mm. spacers. The inner spacers on the source side were 1.64 cm. long, while those on the analyser assembly were 1.25 cm.

Before the final assembly, the D plate was clamped upon one of the cups by means of the sectional flanges. By twisting hard one was able to rotate the plate slightly in the clamped position. The cups were now placed upon the glass seals and the tube was clamped in the jig. Into the end of one of the accurately machined rods mentioned previously, was set a knife blade with a tapered surface that the blade would slip accurately and tightly into the slit in the D plate. At the opposite end of the rod a flat surface was ground in the same vertical plane as that of the knife blade. Thus by applying a square to the flat surface and getting it at right angles to the base of the jig, we were certain that the knife blade was likewise perpendicular to the base. See Figure 8.



Figure 8.

The blade was now run into the tube (still upon the jig) by sliding the rod carrying the blade in its "V" blocks and the D plate was rotated until the blade was able to fit accurately into the slit. Thus we were sure that the slit was perpendicular to the base of the jig. The whole process was repeated using the S₅ plate and the other cup, and after this simple procedure we were certain that the D slit and S₅ slit were absolutely parallel with each other.

Having all the foregoing parts available, the assemblies were then completed. The D plate was spotwelded to one of the cups and the S₅ plate to the other. Into each of these, 4 studs were threaded and the ends riveted in place. The inner glass spacers were placed above the studs and then 4- 6 mm. spacers were inserted. The C plate was now placed upon the spacers above the D plate and the S₃ plate above the S₅ plate. A further set of 4- 6 mm. spacers each were added and the S₃ plate assembly was completed by locknuts. Over the C plate was placed the A plate to which were spotwelded both the electron and trap boxes, while the B plate and trap plate were fixed to it by means of short studs, 2 mm. spacers and locknuts. An additional 4 spacers completed the assembly and the whole was firmly fixed together by locknuts. To the C plate was spotwelded a short piece of #26 Nichrome wire as a lead, and similar wires were spotted to

the A plate and to the D plate. In order to connect to the B plate and trap plate, wires were fastened between the locknuts upon the appropriate studs. The completed assembly is shown in Figure 9, and is the source side assembly looking through the electron box toward the trap.

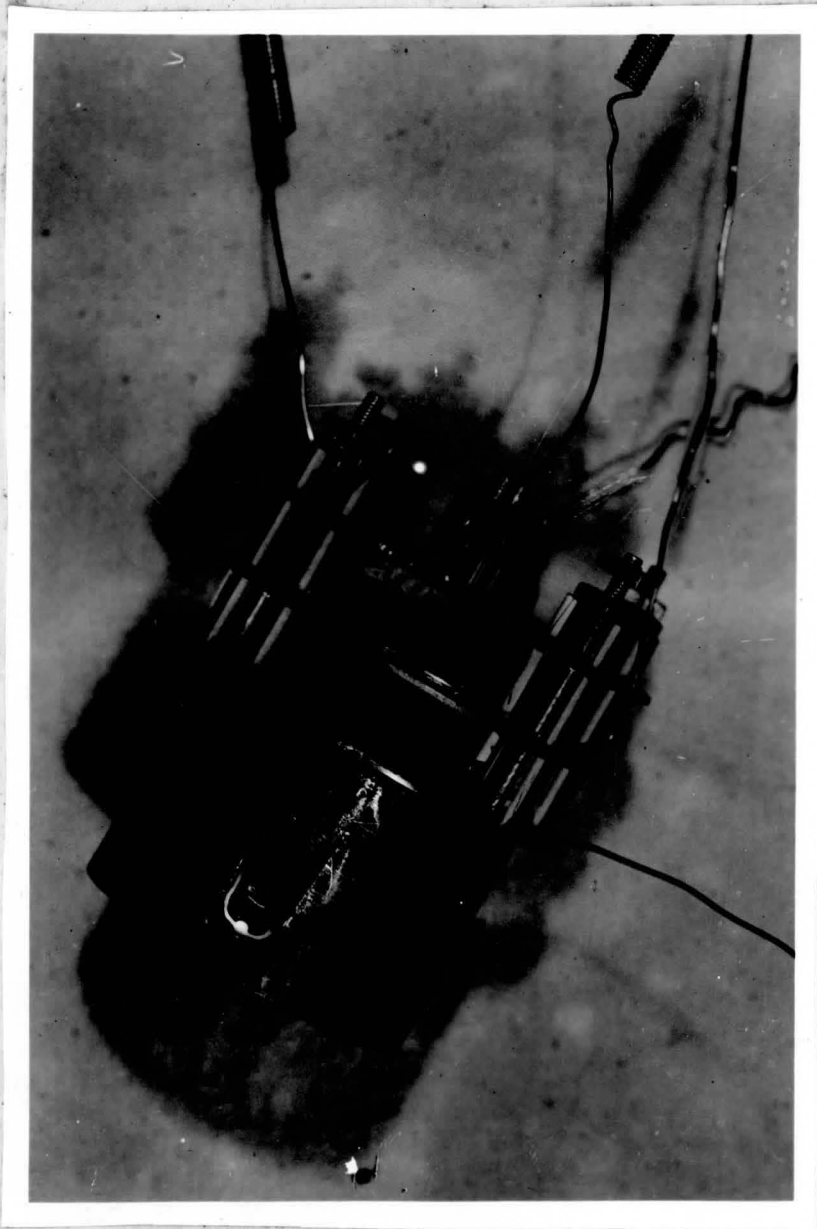


Figure 9.

The completed assemblies were inserted into the tube upon the inner glass seals, care being exercised that the keys on the cups slipped accurately into the keyways upon the glass seals. The wire leading to the nichrome shield was lead out through a hole in the side of the pumping lead, spot welded to a tungsten seal, and the latter sealed into the lead. A similar procedure was carried out with the wire leading from the D plate and also with that from the S_3 plate. A #26 nichrome wire was spot - welded to the S_3 plate and lead out through a tungsten seal in the FP-54 lead. The leads to the A, B, C, and trap plates were spotwelded through nickel junctions to the four 40 mil tungsten wires in the press seal after being mutually insulated by pyrex raschig rings.

The collector box prepared according to the plan was silver soldered to a heavy tungsten seal and whole sealed to the spectrometer envelope. The four wire press seal was fused in place and the pumping lead completed. After spotwelding a tungsten filament 0.025 mm. x 1 mm. x 10 mm. in place, the filament stem was sealed in place and the entire mass spectrometer tube was tested for leaks. Several leaks were found and repaired and subsequent tests revealed the fact that the tube was evidently leak-free.

The mass spectrometer tube was now wound with 0.005" aluminium sheet followed by a loose layer of #18 B. and S. tinned copper wire which served to ground the aluminium layer throughout, after which a layer of wet sheet asbestos was applied. After allowing the asbestos to dry overnight, it was given a coat of sodium silicate solution and while still moist the furnaces were wound. The Number 1 furnace consisted of 550 cm. of #26 Nichrome wire wound from the area of the D plate around the curve to the region of the S₅ plate; connection leads were run out and insulated. The furnace was then covered with a layer of wet asbestos and connected to the 110 volt supply in order to dry quickly. When dry, sodium silicate was applied and the whole re-dried. This process was repeated three times so that the furnace received in all, 4 coats of water glass. The Number 2 furnace was wound in the same manner, around the pumping lead and FP-54 exit, and consisted of 600 cm. of #26 Nichrome. A third furnace was prepared by winding upon a tin cylinder 6 inches long x 46 mm. inside diameter, a resistance winding in the same manner as that described above. This furnace was intended as a removeable unit to be slipped around the ion source. In ordinary baking out procedure, furnaces #1 and #2 were connected in series and kept the tube at approximately 225° C. For more drastic baking out, the furnaces were connected in parallel and heated the tube up to approximately 375° C.

The mass spectrometer was then sealed in place between the pole faces of the electromagnet and connected to the vacuum system through a one inch glass pumping lead controlled by a 15 mm. bore stopper. In order to remove as much strain as possible from the pumping leads, the tube was supported by a semicircular wooden cradle lined with asbestos. The vacuum systems on the mass spectrometer tube side and on the sample line side were constructed according to the plan illustrated in Figures 10 and 11.

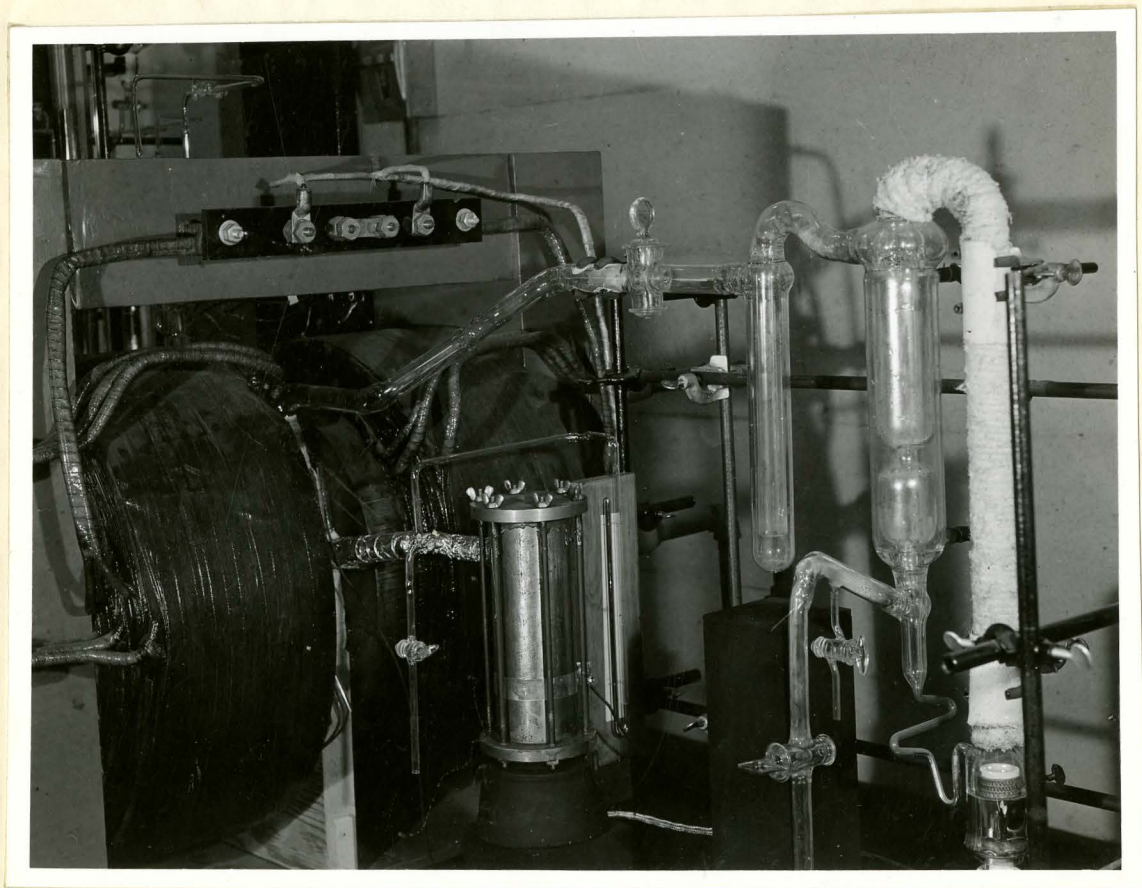


Figure 10.

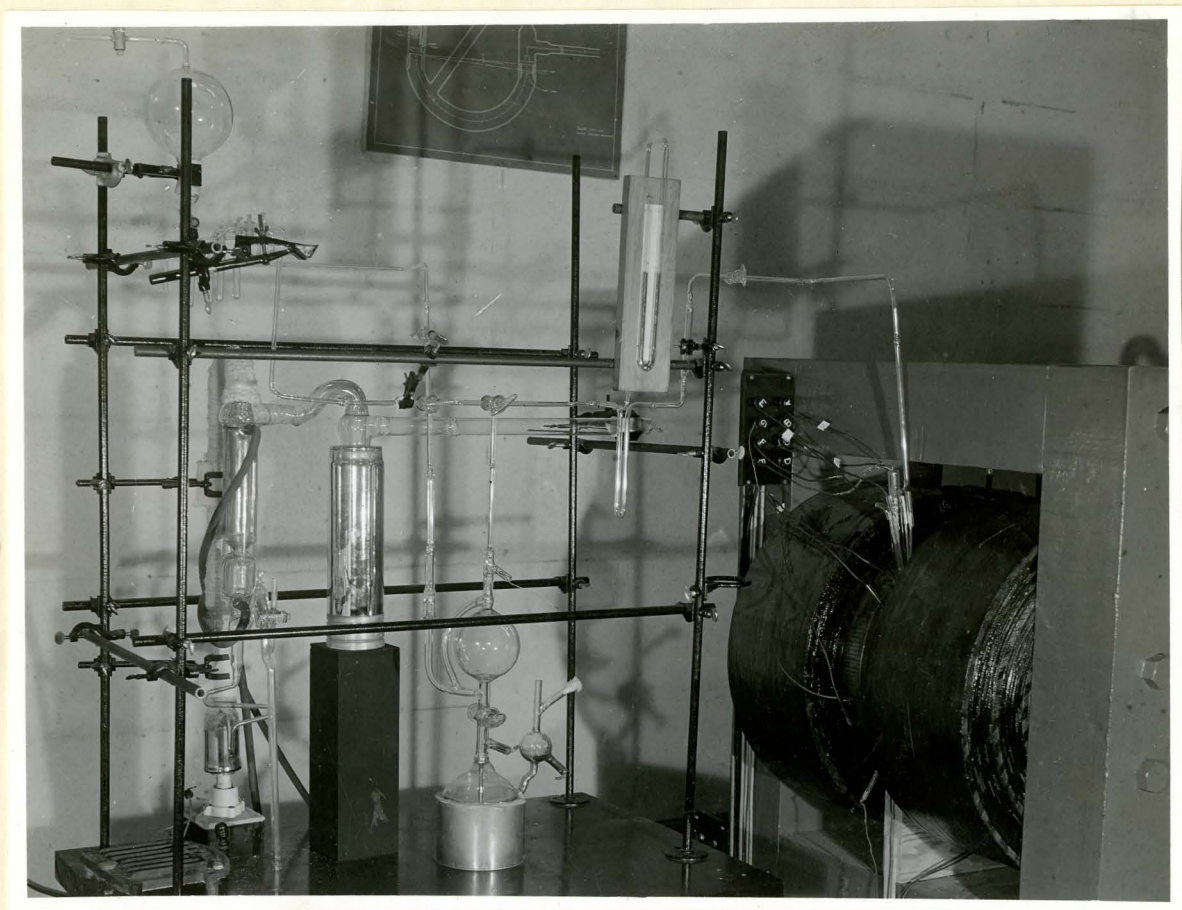
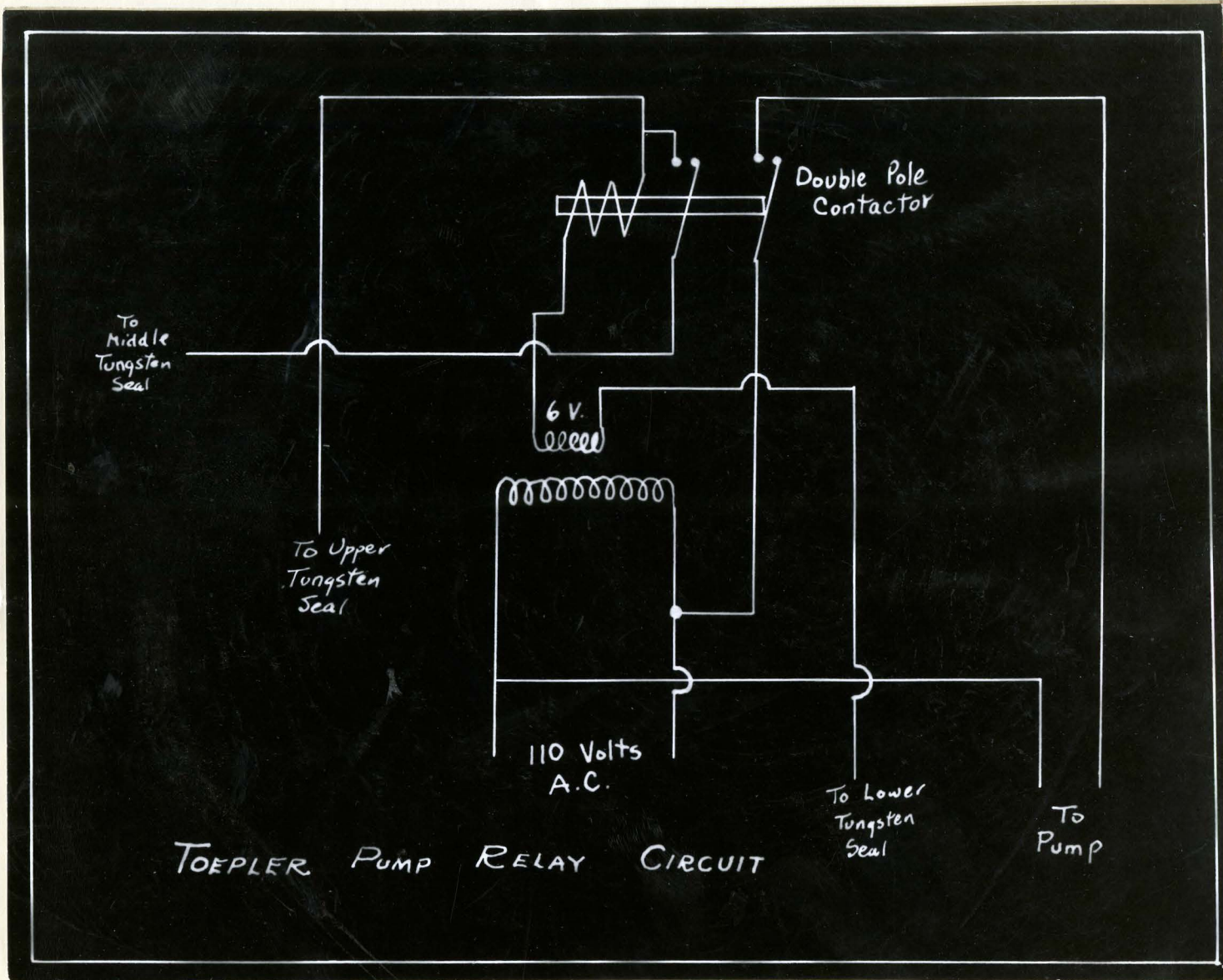


Figure 11.

In order to have as small a volume as possible, the entire sample line was constructed of 6 mm. O.D. pyrex capillary tubing with 3 mm. bore. For obtaining the whole of a very small sample for analysis, a Toeppler pump was provided; it was connected electrically through tungsten seals with a relay system so as to operate automatically. The relay system is illustrated in Figure 12.



N.D.G. - it won't work

Figure 12.

The Magnet Base

Inasmuch as the concrete floor of the mass spectrometer room is covered with a rather thick layer of mastic it was felt the placement of the 3000 lb. magnet directly upon the floor would eventually result in a gradual sinking of the heavy yoke into the flooring. Further, since the magnet stands but 34 inches high it was felt that a base upon which it might rest would add to convenience in routine repairs and operations. A base 1 foot high was constructed of heavy timbers of British Columbia Fir 6 inches thick, 12 inches wide and 4 feet long. In order to add strength to the base, it was constructed of two thicknesses placed cross-grain to each other. To lend greater permanence to the base, the upper edges were protected by two inch angle iron placed about the circumference. The timbers of the base are, in addition to the weight of the magnet held together by 5/8" cold rolled steel tie-rods threaded at each end and provided with locknuts and washers. The overall area of the base is 8 square feet; thus the weight distribution is approximately 450 pounds per square foot upon the mastic flooring, well within the limits. A plan of the base is given in Figure 13.

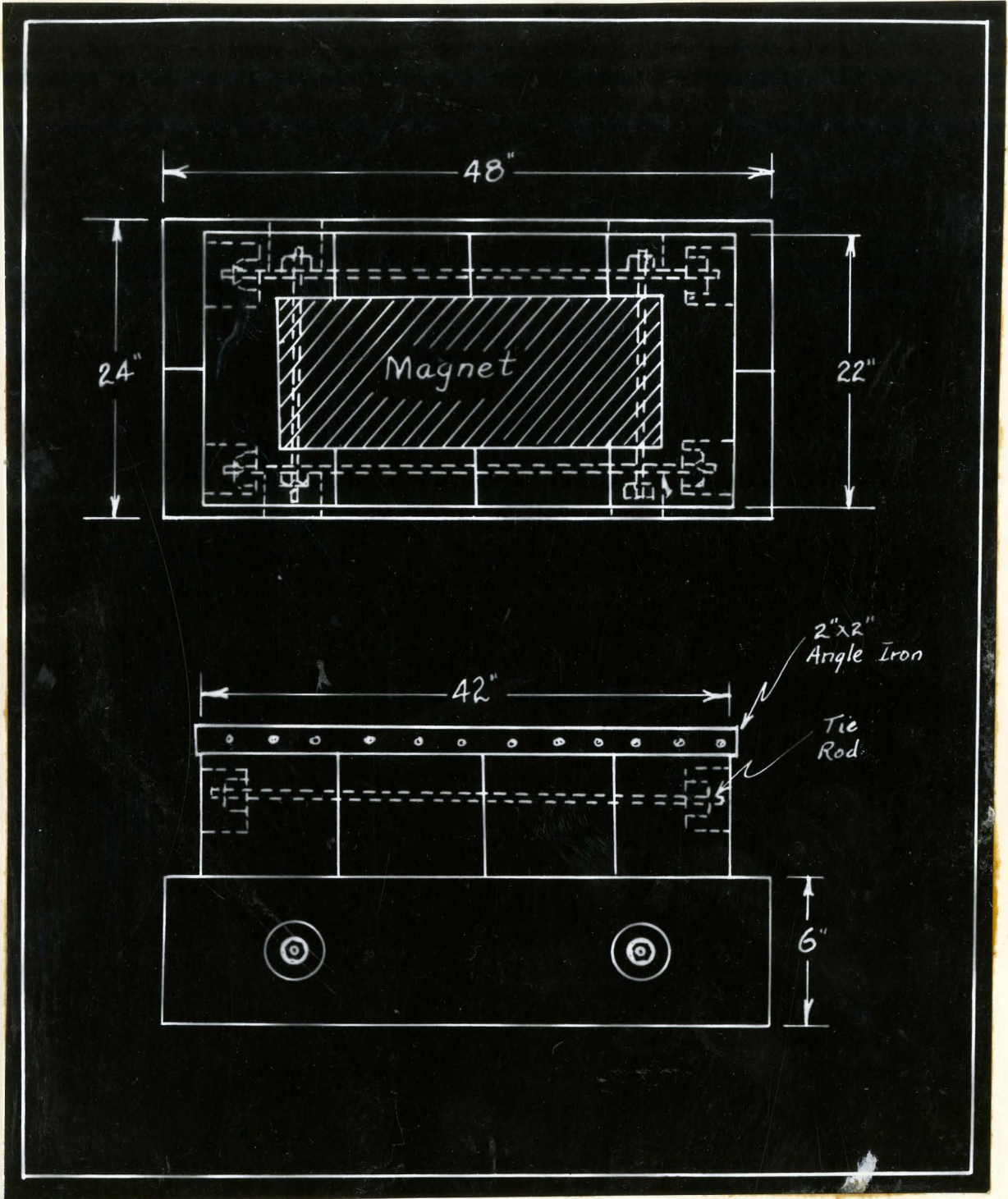


Figure 13.

The Electromagnet

The electromagnet for the mass spectrometer was constructed by the Canadian Ferranti Electric Company at Toronto, according to specifications. The following data describe the design of the electromagnet illustrated in Figure ¹⁴ M.

A field of 6500 gauss is required in the air gap,

therefore $B = 6500 \times 6.45 = 42000$ lines per square inch.

Total flux in gap = $202 \times 42000 = 8,460,000$ lines.

Total flux in pole shoe = $1.36 \times 8.46 \times 10^6$ lines.

B in pole shoe = $\frac{1.36 \times 8.46 \times 10^6}{12^2} = 75,000$ lines per square inch.

Total flux in pole $1.63 \times 8.46 \times 10^6$ lines.

Area of pole $\pi \times 6^2 = 113.2$ square inches.

B in pole $\frac{1.63 \times 8.46 \times 10^6}{113.2} = 122,000$ lines per square inch.

Total flux in yoke = $1.63 \times 8.46 \times 10^6$ lines.

Area of yokes (two in parallel) = $12 \times 5.5 \times 2 = 132$ square inches.

B in yokes $\frac{1.63 \times 8.46 \times 10^6}{132} = 104,500$ lines per square inch.

Gap length	2 inches	B	42,000 lines/sq. in.
Pole shoe	4 inches	B	75,000 lines/sq. in.
Pole length	17 inches	B	122,000 lines/sq. in.
Yoke length	54 inches	B	104,000 lines/sq. in.

If the electromagnet were made of Cast Steel the ampere turns required would be:

2 x 0.313 x 4200	26,300
4 x 26	104
17 x 500	8,500
54 x 110	<u>5,950</u>

Total: 40,854 ampere turns.

The flux densities in the yokes are too high and blow holes in the casting would cause saturation, therefore cast steel is unsuitable.

For a magnet of SAE 1010 steel the ampere turns required would be:

2 x 0.313 x 42000	26,300
4 x 22	88
17 x 350	5,950
54 x 80	<u>4,320</u>

Total: 36,658 Ampere turns

Since the flux densities are more reasonable, the magnet was therefore cast of SAE 1010 steel and annealed at 1300°F. for 1 hr.

The coils must be good for 40,000 ampere turns, and since there are two coils, this means 20,000 turns per coil. Number 10 square Delta Beston copper wire was employed for the coils, each coil consisting of 1450 turns wound in 23 layers of 63 turns per layer.

Dimensions of coils:

Width	Thickness
Thickness of wire 0.102"	Thickness of wire 0.102"
Insulation 0.020"	Insulation 0.020"
Width per turn 0.122"	Thickness/turn 0.122"
Turns per layer 65.5	No. layers 23
Width per layer 8.00"	Thickness 2.81"
Plus end checks 0.50"	plus 22 layers
Total width 8.50"	of insulation 0.264"
	Total thickness 3.074"

Since there are 20,000 ampere turns per coil, the current per coil will be $20,000/1450 = 13.8$ amperes,

and the current density will be $13.8/0.1159 = 11.9$ Amp./ sq.in.

Mean turn length of coil = $\pi \times 16.32 = 51.4'' = 4.28$ ft.
 Total length = $2 \times 1450 \times 4.28 = 12,400$ ft.
 Total weight (bare) of coils = $12.4 \times 37.93 = 470$ lbs.
 Total weight, insulated = 500 lbs.
 Resistance at 75°C . (2 coils) = 12.4 ohms.
 RI^2 at 75°C . (2 coils) = $13.8^2 \times 12.4 = 2360$ watts.

The coils are wound on insulated cores, so that the heat will go into the core and into the air. Core insulation 0.05" mica.

Drop through 50 mils = $0.050 \times 250 \times W_s = 12.5 W_s$

Surface drop = $100 W_s$ (average value).

Practically all the heat will be conducted into the core.

Surface of the core outside coils = 3970 sq. in.

Core rise 34°C .

Temp. rise of coil above core = 51.7°C .

Total temp. rise of coil $34 + 51.7 = 85.7^{\circ}\text{C}$.

Cooling coils of copper IPS 0.27" I.D. x 0.405" O.D.

Diameter of coil (bare) per layer	0.405"
Insulation	<u>0.100"</u>
Total, per turn	0.505"
For 16 turns	<u>16</u>
Total:	8.08"

Three cooling coils per magnet coil, one next to core, one in middle and one on outside.

16 turns per section, 48 turns per pole, or 96 turns for two poles.

Mean turn length 51.4" = 4.28 ft.
Total length 96 x 4.28 + 10 = 420.8 ft.
Weight (bare) 0.29 x 420.8 = 122 lbs.

RI^2 loss at 75°C. = 2360 watts

Water rate = 2360 / 3.2 = 0.738 gallons per minute to dissipate 2360 watts.

Cubic inches per second $\frac{0.738 \times 277.2}{60} = 3.41$ cu. in per sec.

Area of pipe (I.D) $0.27^2 \times \frac{\pi}{4} = 0.0572$ sq. in.

Therefore flow = 3.41 / 0.0572 = 59.6 in./ sec = 298 ft. / min.

All nine cooling sections were connected in parallel to reduce rate of flow.

Total weight of electromagnet 3893 lbs.

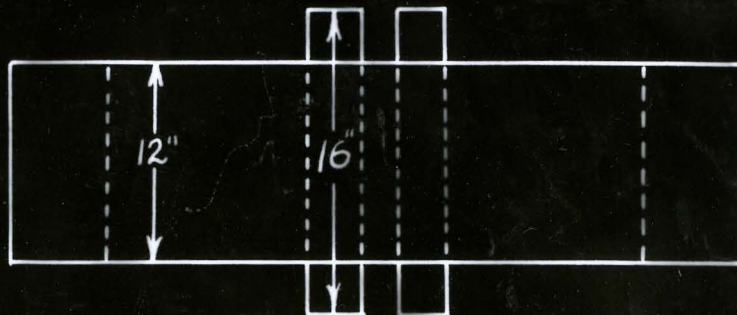
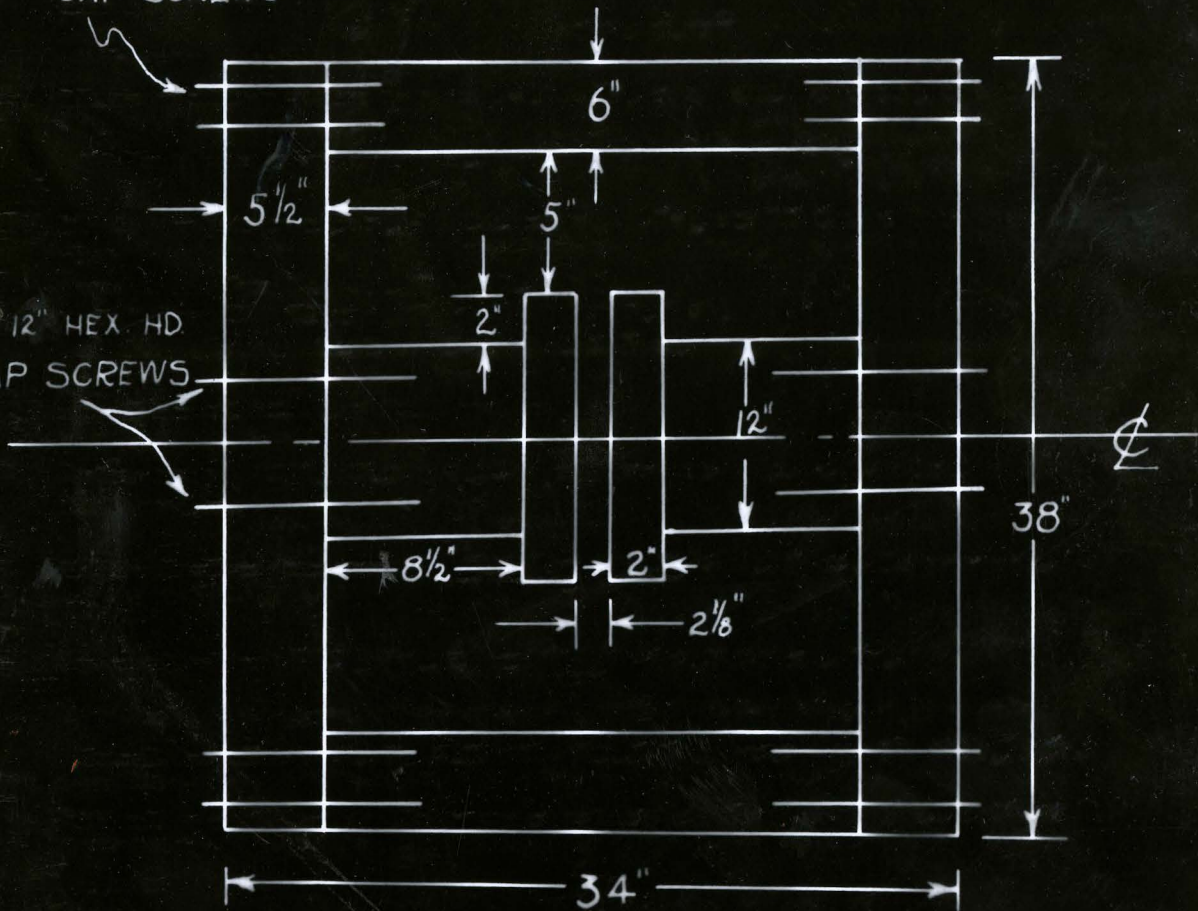
The plan of the magnet is given in Figure 14, and several photographs in Figure 15.

Figure 14

SAE 1010 STEEL

2- 12" HEX. HD.
CAP SCREWS

4- 12" HEX. HD.
CAP SCREWS



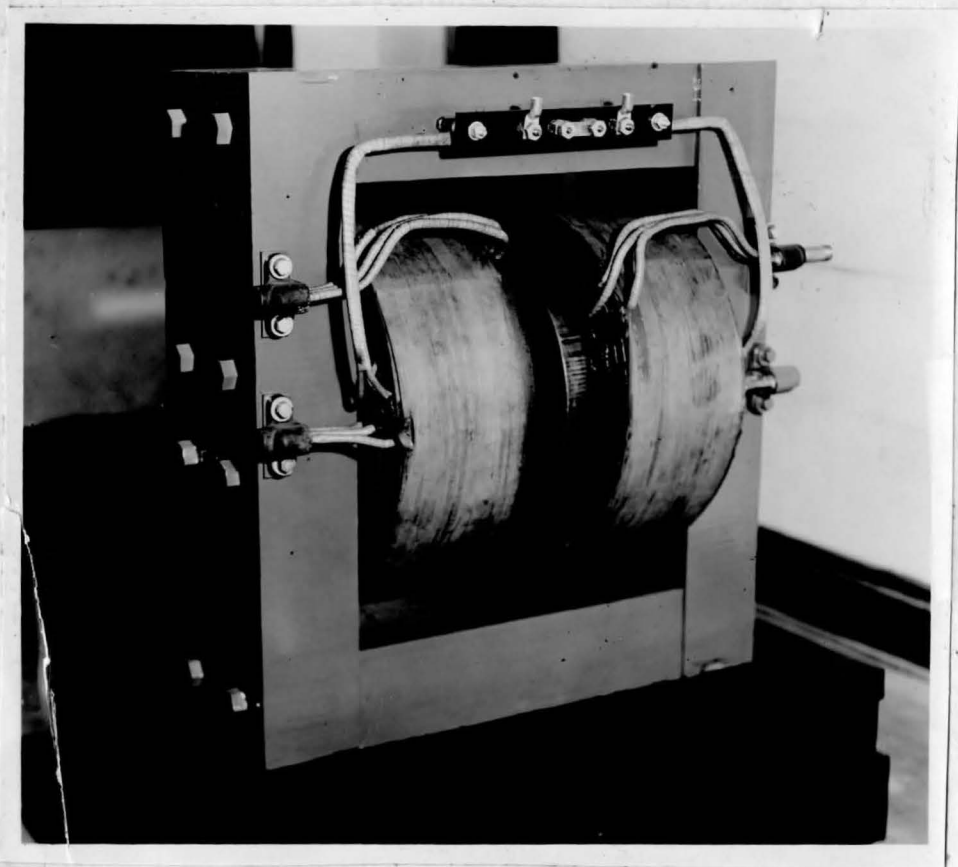
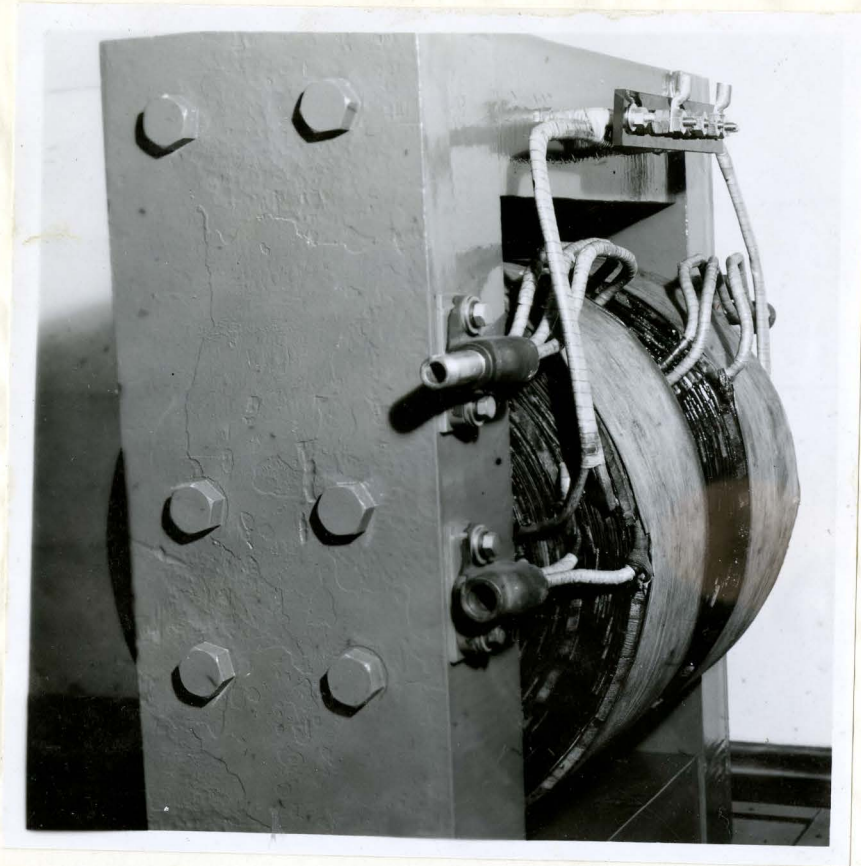
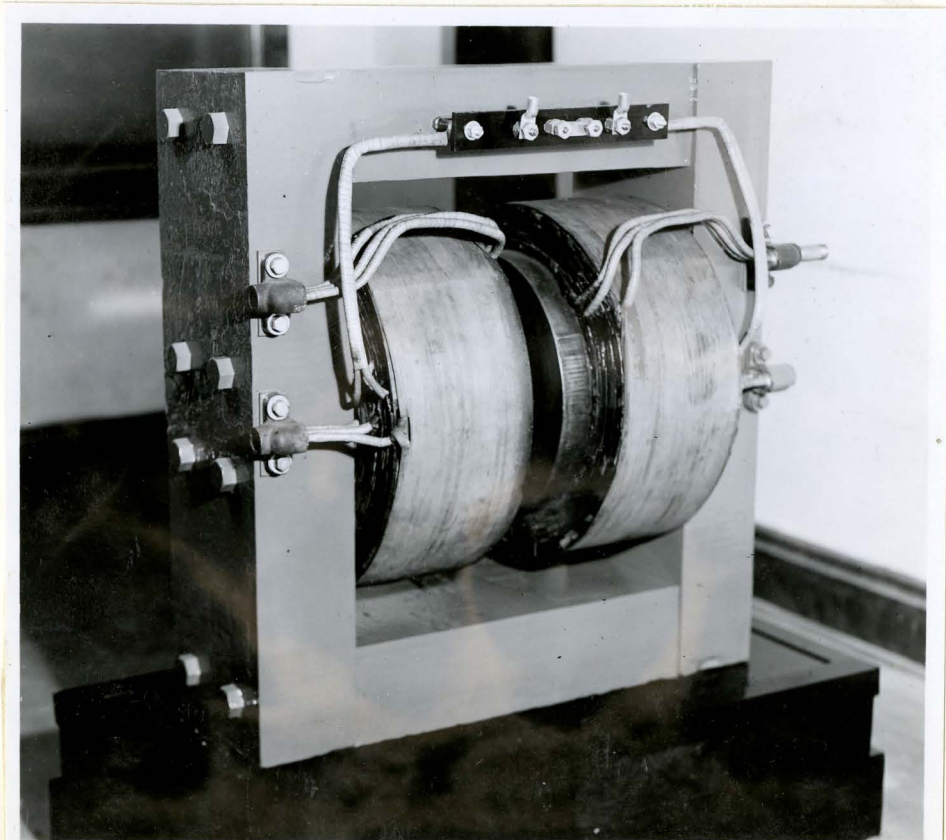


Figure 15.

Figure 15



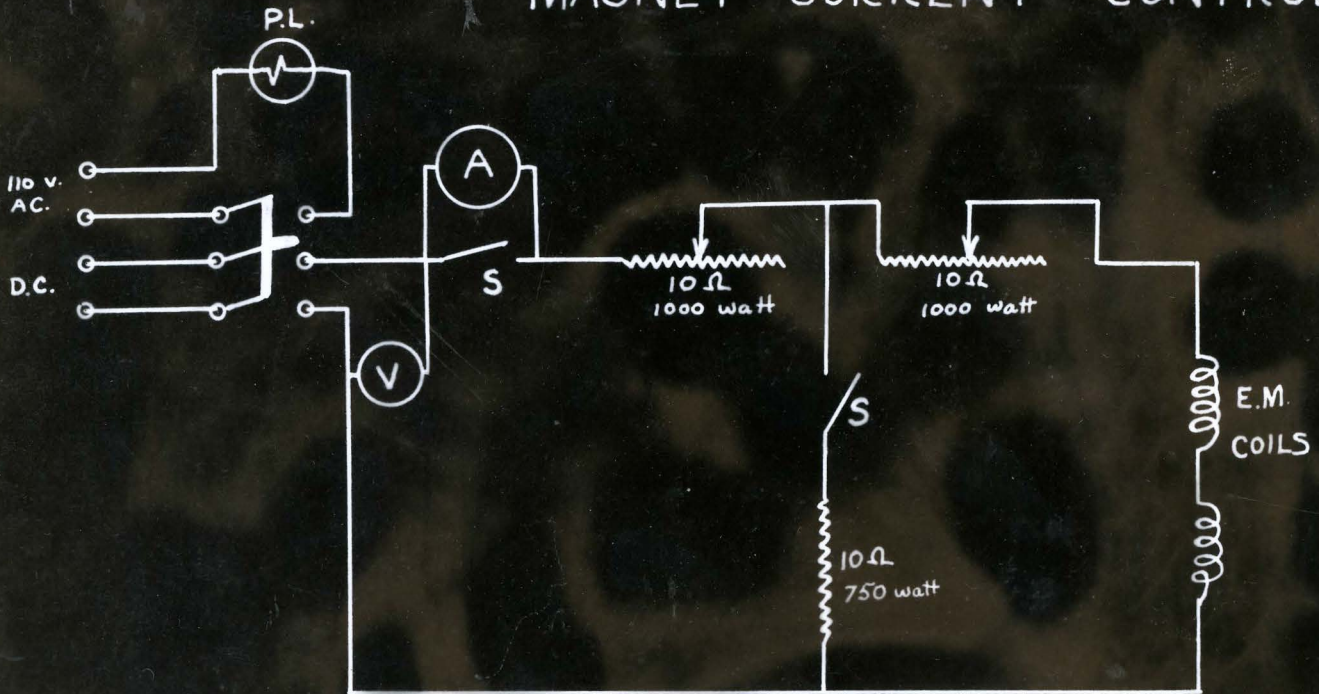
(b)



(c)

For the present, the magnet is to be energised by means of a high capacity battery supply of ~~20~~³⁰ volts which will be described later. The energising current is supplied to a master switch fused at 15 amperes after which it travels to the control panel through a triple pole single throw switch in a pilot light indicator circuit. Two 1000 watt, 10 ohm Ohmite vitreous enamelled variable resistors in series are used to control the energising current. The energising circuit is illustrated in Figure 16.

MAGNET CURRENT CONTROL



It will be seen that the circuit contains two shorting switches; S_1 is for the purpose of protecting the ammeter, especially from the surge developed by back EMF when the energising current is abolished; S_2 is for the purpose of providing an extra 10 ohm, 750 watt resistance into which this back EMF may be safely dissipated.

In practice, S_1 is usually kept closed; it is opened momentarily only to obtain an indication of the magnitude of the energising current. Switch S_2 is always kept open. In turning off the electromagnet, however, first S_2 is closed then the triple pole switch is opened. In this manner, the extra ohm resistance is provided for back EMF dissipation and arcing at the main switch terminals is reduced to a minimum.

For interesting discussions on the construction of powerful electromagnets, the reader is referred to the literature (13, 14, 15).

Magnet Current Supply

During the course of operation of a mass spectrometer the prime requisite is an unvarying, homogeneous magnetic field. It is apparent that by judicious design one may obtain homogeneity of field, and while constancy of magnetic field is in some measure dependent upon the physical properties of the magnet, the most important consideration is the source of energising current. There are two types available for consideration, and we hope ultimately to employ them both if the occasion demands. For routine abundance measurements of the lighter elements (those from hydrogen to sulphur), relatively smaller magnetic fields are required than those necessary for measurements, say, upon the lead isotopes. We employ then, very high capacity storage batteries for energising the magnet coils when we are only examining those lighter isotopes. For the heavier isotopic examinations, resort must be had to much higher field densities, and while this might conceivably be attained by using sufficient number of batteries in series-parallel, the cost would be out of the question. We must therefore employ a source of 120 volts D.C. such as that supplied by a generator. Unfortunately the current from this source is far from satisfactory due to variations in output which would, of course, cause corresponding variations in the magnetic field density. To circumvent this difficulty, a circuit has been

devised which automatically compensates these variations by controlling the field current in the generator. This unit has not been built to date, so that further remarks are not in order.

In lieu of using the DC generator for a magnet current supply, we were able through the generosity of the Physics Department to obtain 11 high capacity Exide 2-volt storage cells, and this stable 22 volt supply will be quite adequate for the lighter isotopic investigations. In practice, we operate the magnet at 2 amperes current, and with this low drain the batteries remain quite stable for a considerable period.

The batteries are contained in a special two-compartment battery chamber in another room. The photograph in Figure 17 will illustrate the manner of housing. The batteries are connected in series with 1/8 inch x 1 inch copper bus bar jumpers, and the supply line into the mass spectrometer room consists of number 12 type R rubber covered solid copper wire shielded in 1/2 inch conduit, fused at 15 amperes in a Bulldog type 1122 double pole single throw enclosed switch. From the switch a number 12 type R solid copper two conductor line leads to the control panel master switch. For convenience in charging a two conductor line was run to the charging room where special outlet taps were made available on the panel.



Figure 17.

The Amplifier Unit

Since the methods of mass spectroscopy demand an efficient means for accurately measuring the values of ion currents which are extremely minute, it is well to justify our choice of the FP-54 amplifier tube for this purpose. The FP-54 Plotron is a tetrode designed particularly for amplification of direct currents smaller than about 10^{-9} ampere. This tube is a convenient and sturdy substitute for a sensitive galvanometer or electrometer in any circuit involving the measurement or detection of small currents.

The FP-54 may be used in any of the standard types of circuits designed for DC amplification (16). The circuit which is being used in this amplifier is a modified Barth type. For measurements of very small currents a steady high resistance unit is required to obtain sufficient voltage drop to operate the tube. For measurements of plate current changes, a sensitive galvanometer, with an arrangement for balancing out the initial plate current is required.

The surface of the tube must be kept free from all sources of surface leakage. Before the tube is placed in the apparatus the bulb must be thoroughly washed in order to remove traces of foreign matter which might provide a conducting path from control grid to ground. In our apparatus, the tube is kept inside a steel chamber which shields the tube magnetically and electrostatically. In operation, the changes in grid voltage are measured by the

changes in plate current. As mentioned above, this may be accomplished with a sensitive galvanometer with the initial plate current balanced out. By this method it is possible to obtain an overall sensitivity of 250,000 ma. per volt. Another method, of course would be to use an amplifying tube with a high mutual conductance in place of the galvanometer; The plate current of the amplifying tube can then be measured with a less sensitive galvanometer.

In our apparatus we employ a Leeds and Northrup type R galvanometer with a sensitivity of 0.0005 microamperes per ma. and there is therefore no necessity for employing a second amplifying tube. Since the plate current will remain constant with no tendency to drift if the supply voltages are held constant, two high capacity storage batteries in series are used as supply. In order to obtain the greatest sensitivity with only one tube and for making measurements over a long period of time we have built the amplifier unit according to the circuit recommended by DuBridge and Brown (17). This circuit can be adjusted so that the changes in battery voltage as well as drift in plate current have no effect on the balance of the galvanometer.

The following parts are required for the amplifier unit.

- 1- IRC 500 ohm all metal power rheostat, 25 watt.
- 2- IRC 5,000 ohm all metal power rheostats, 25 watt.
- 1- IRC 75 ohm all metal power rheostat, 25 watt.
- 2- IRC 50 ohm all metal power rheostats, 25 watt.

- 1- IRC 2,500 ohm all metal power rheostat, 25 watt.
- 1- IRC 4,000 ohm wire wound fixed resistor, 2 watt.
- 1- IRC 30 ohm wire wound fixed resistor, 2 watt.
- 1- S.S. White Dental molded resistor, type 65X, 900 megohms.
- 1- 6.3 volt pilot bulb, minute socket type.
- 1- 1/2" Jewel bracket.
- 1- Jewel for pilot light.
- 1- DPST toggle switch.
- 1- 4 pole, 2 position (single gang) Yaxley switch, #1312.
- 1- Etched Yaxley dial plate marked 1-2.
- 7- Etched Yaxley dial plates marked 0-100.
- 1- Triplet D.C. Milliammeter, range 0-100 Ma.
- 1- Triplet D.C. Microammeter, range 0-500 Microamperes.
- 8- Rheostat knobs.
- 1- Hammond steel panel 1/16" x 12 $\frac{1}{2}$ " x 19".
- 1- 1/8 Ampere Littlefuse and holder.
- 1- General Electric type EP-54 Pilotron tube.
- 30 feet 4 conductor Belden shielded cable.
- 60 feet 2 conductor Belden shielded cable.
- solder, lugs, connecting wire.

In Figure 18 is given the circuit diagram for the amplifier.

In operation, one adjusts the circuit by setting the filament current to obtain approximately the rated value (90 Ma) and bringing the galvanometer to zero by adjusting R_5 and R_4 . Then by varying R_3 a point should be found where the rate of change of galvanometer deflection with filament current is zero. If this point cannot be found within a range of plus or minus 3 or 4 percent of filament current, the setting of R_2 is changed and the operation repeated. The galvanometer is then balanced to zero with R_4 and R_5 . After a period of operation of 20 or 30 minutes it may be necessary to readjust R_4 and R_5 for zero balance.

FP-54 Tube Unit

As mentioned previously in the description of the amplifier unit, the FP-54 tube itself is housed in a steel chamber. In this manner the tube is shielded both electrostatically and magnetically moreover the air in the chamber may be kept perfectly dry by the use of a drying agent, thus maintaining a non-conducting atmosphere. Some workers advocate maintaining the tube in an evacuated chamber, while still others (18) recommend coating the envelope of the tube to within 1 inch of the control grid cap with a wax such as natural Ceresin which has very high dielectric properties. The inside of the chamber and the lead through plug are also coated with ceresin and the chamber maintained at 0.1 mm. pressure.

The FP-54 housing is illustrated in Figure 19.



The High Potential Supply

The DC high potential supply is a unit designed especially after the requirements of the mass spectrometer for a unit capable of delivering a stabilised high-voltage output of approximately 1500 volts under constant load, low current characteristics. An excellent review of the subject of electronic voltage stabilisers is given in a paper by Hunt and Hickman (19). The type of circuit used by us is described as "an S circuit with a U circuit arranged to provide the biasing voltage"; for further discussion of the theory of operation of such a unit, the reader is referred to the original paper.

In the construction of the unit several changes in design have been made from that used by Keevil (18). The rectifier circuit has been fundamentally altered by the use of two type 88⁶ tubes in place of a single type '80. It was felt that the type 88⁶ tube was better suited due to its higher inverse peak voltage, thereby offering less possibility of breakdown by flashing across the base. To smooth all but approximately 1% of the inherent ripple, we inserted a heavy duty 30 Henry choke in the output circuit, and provided a radio-frequency choke in the plate circuit of each 88⁶ tube. Similarly a 15 Henry choke was put in the output circuit of the '80 tube. In place of the paper condensers originally used, 2000 volt, 1 microfarad oil-filled transmitting condensers

were supplied. By the provision of S_4 the danger of tube breakdowns has been eliminated due to prevention of high voltage surges to the plate before the filaments have been adequately warmed.

Neither side of the output is grounded and the whole stabilized high potential is fed across a 510,000 ohm potentiometer which is described in the voltage divider section. In this manner we have constant load characteristics and the accelerating voltages are derived by drawing off potentiometrically fractions of the total output and supplying this to the plates of the mass spectrometer tube. The accelerating voltage applied to the ions is such that the drop between plates B and C is always a given fraction of that between B and D. As pointed out by Bleakney (20), this is a desirable feature, as then all the ions regardless of m/e value will have travelled the same path before entering the analyzer.

By actual measurement, our high potential supply is capable of providing a stabilized source of 1300 volts DC, quite adequate for any analysis which might be required of our instrument. The circuit is illustrated in Figure 20.

The following equipment is used in the construction of the high potential supply.

- 1- Hammond steel panel 19" x 12 $\frac{1}{2}$ " x 1/16", rack unit #1487.
 - 1- Hammond steel chassis 17" x 7" x 3".
 - 1- 2 megohm Yaxley volume control.
 - 1- 20,000 ohm Yaxley volume control.
 - 4- SPST toggle switches.
 - 4- $\frac{1}{8}$ " jewelled brackets, miniature socket type.
 - 2- Jewels for pilot lights, red, $\frac{1}{8}$ ".
 - 2- Jewels for pilot lights, green, $\frac{1}{8}$ ".
 - 2- Jewels for pilot lights, clear, $\frac{1}{8}$ ".
 - 1- Thordarson T19P56 transformer, 900 volts each side of centre tap.
 - 1- Thordarson T13R 11 transformer.
 - 1- Filament transformer, Pri. 110 v. 25 cycle, sec. 5.0 volts.
 - 1- Filament transformer, Pri. 110 v. 25 cycle, sec. 6.3 volts.
 - 3- 4 prong amphenol tube sockets.
 - 2- 8 prong amphenol tube sockets.
 - 1- Type 6SJ7 tube.
 - 1- Type 6J7 tube.
 - 1- Type '80 tube.
 - 2- Type 885 Tubes.
 - 5- G.E. type T-2, 1/25th watt neon bulbs with resistances removed.
 - 2- 2000 volt, 1.0 mfd. transmitting condensers.
 - 4- 450 volt, 8 mfd. electrolytic condensers.
 - 4- 6.3 volt pilot light bulbs.
 - 1- 50,000 ohm fixed resistor, 2 watt.
 - 1- 250,000 ohm resistor, 2 watt.
 - 1- 25,000 ohm resistor, 2 watt.
 - 1- 4 megohm resistor, 2 watt.
 - 1- 1 megohm resistor, 1 watt.
 - 1- 300,000 ohm resistor, 2 watt.
 - 1- 80,000 ohm resistor, 2 watt.
 - 1- 500,000 ohm resistor, 2 watt.
 - 1- 15 Henry Choke.
 - 1- 30 Henry Heavy Duty Choke.
- solder, wire, cambric tubing, lugs, etc.

The unit is housed in the control rack and the high potential leads run to the control panel via feed through insulators. In operation, one turns on switches S_1 , S_2 , and S_3 in order, and after a brief period, S_4 is closed. It is well to allow the unit to run for about 30 minutes before an analysis in order to allow fully stabilised conditions to set in.

The PP-54 Amplifier unit, High Potential supply, Generator field control, spectrometer tube meters, and various "B" and "C" batteries are all housed in a Hammond all-steel control rack, a photograph of which follows in Figure 22. The meters in the spectrometer tube circuit include a 0-10 ampere ammeter for measuring the filament current, a 0-500 microampere meter for the A plate current, and a 0-100 microampere meter for the trap current. The "B" batteries are used for energising the electrons emitted from the filament and the circuit is illustrated in Figure 21.

PLATE CONNECTIONS

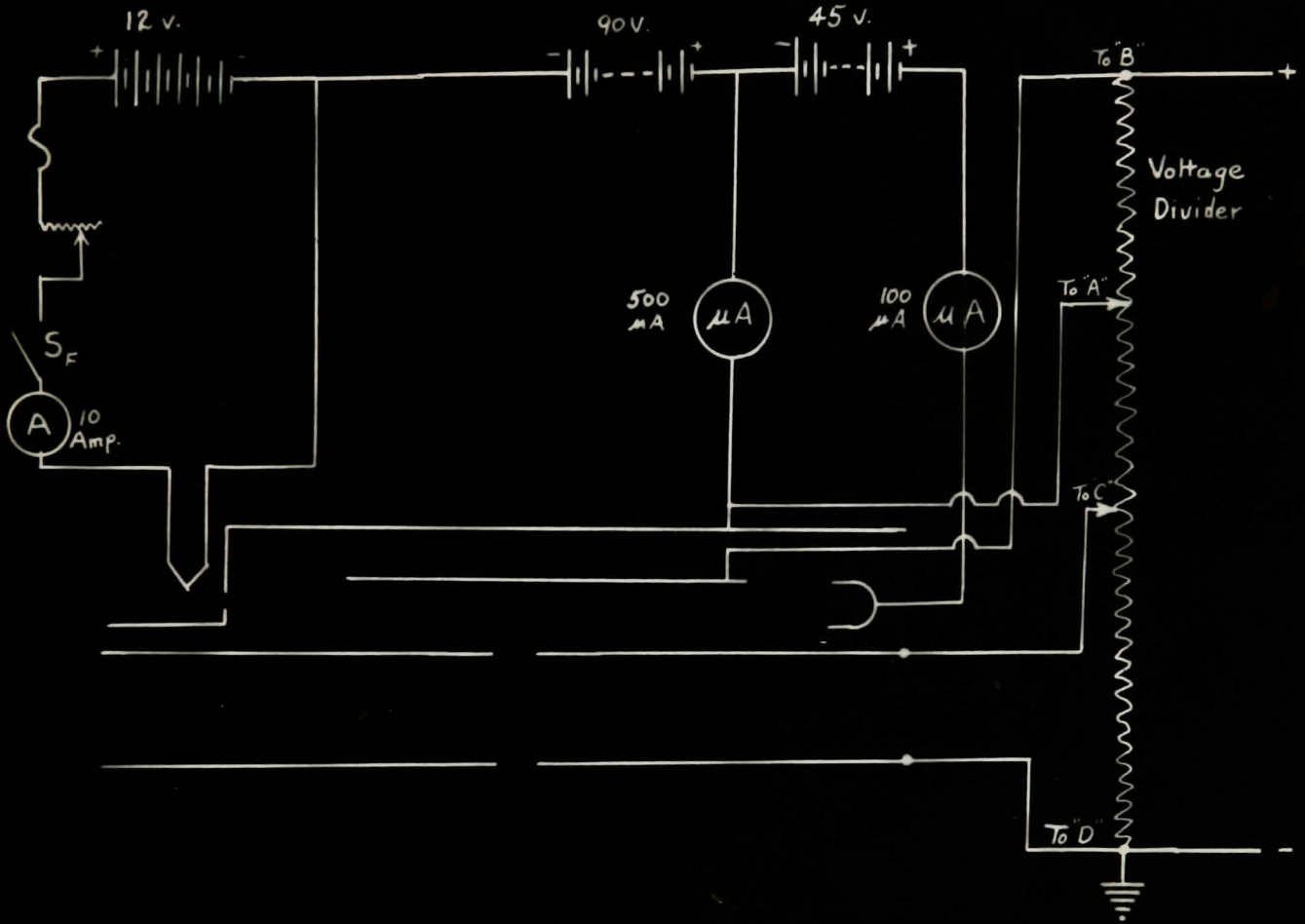


Figure 21.

The Control Panel.

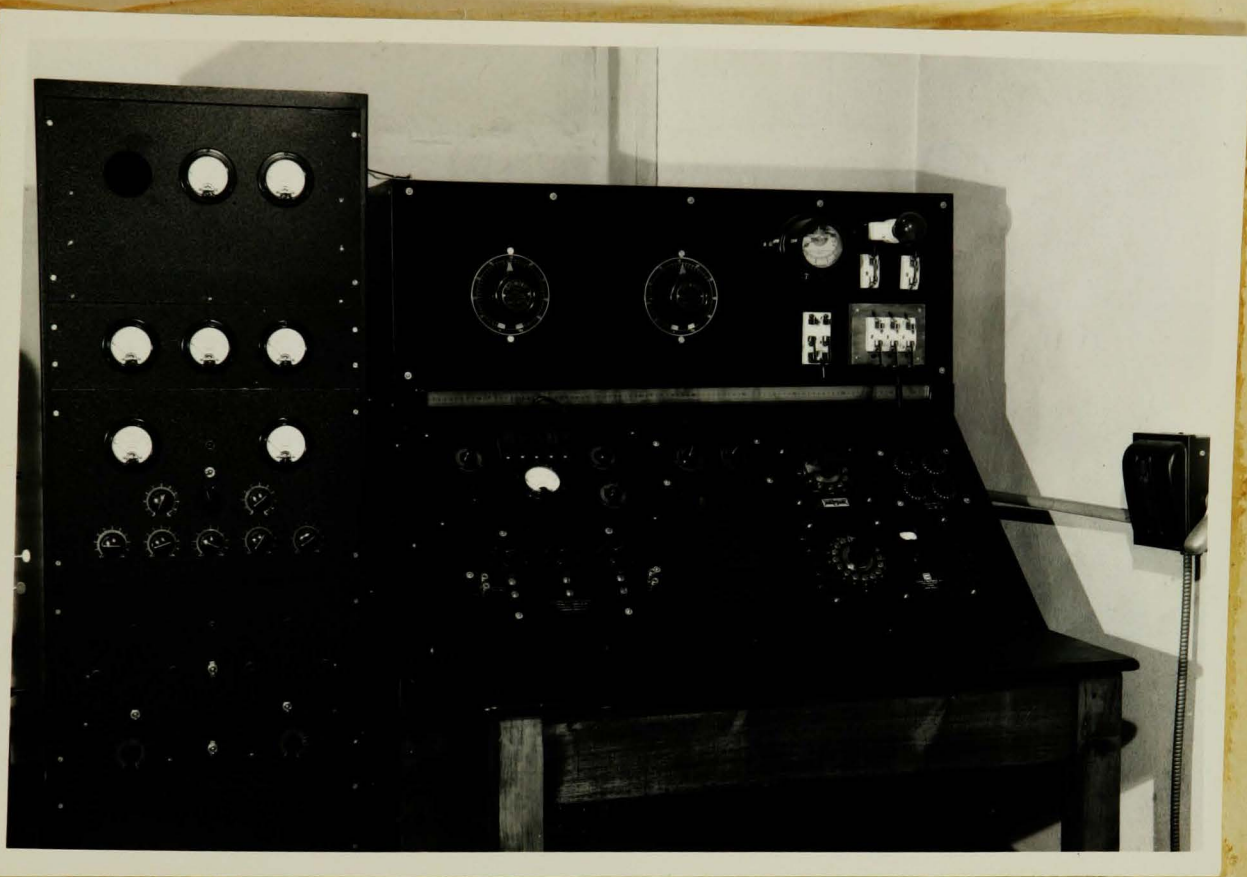
An essential of every large piece of apparatus is the central control system. In the mass spectrometer, no less important than any of the other features is the control panel. The panel was designed for practicability; thought was given to neat arrangement of the various appliances, as well as to convenience of arrangement from the point of view of the operator. The panel was constructed upon a 2 inch square spruce framework, and was built integrally with the operator's table. The sides and top of the panel were covered with 16 gauge sheet steel, as well as the vertical portion of the front of the panel. The sloping portion of the control board consists of $\frac{3}{4}$ " 5 ply birch plywood, into which the appliances were set. The panel together with the control rack are shown in Figure 22.

Figure 22.



Since the panel is completely covered, it provides shielding for the various instruments contained therein. The vertical portion of the panel contains the master control switch for the magnet, the pilot light, shorting switches, magnet current rheostats, and ammeter for measuring currents in the furnaces about the mass spectrometer tube. Immediately below

Figure 22.



this is the galvanometer scale upon which the intensities of the ion and electron beams are indicated by the respective galvanometer beams. The sloped portion of the panel supports the voltage divider circuit, the filament rheostats, the potentiometer unit, and the bank of 12 toggle switches which are used as on-off switches for the various pumps, furnaces, galvanometer lamps, and filament.

Voltage Divider and Potentiometer Circuits.

The voltage divider circuit consists of a fabricated high resistance unit in series with a Decade type 654-A Voltage Divider. The circuit is illustrated in Figure 23.

In constructing the voltage divider, the following equipment is necessary.

- 1- Bakelite panel 3" x 15" x $\frac{1}{4}$ ".
- 4- IRC Precision wire wound resistors type WW-4, 100,000 ohms. 0.1%.
- 10- IRC Precision wire wound resistors type WW-4, 10,000 ohms. 0.1%.
- 1- 4 pole 2 position Yaxley switch #1312.
- 1- single circuit 11 position Yaxley switch #1211.
- 1- 1.0 megohm fixed resistor, 1 watt.
- 2- 20,000 Yaxley wire wound volume controls.
- 1- Etched Yaxley dial plate marked 1-2.
- 3- Etched Yaxley dial plates marked 0-10.
- 1- Triplett DC voltmeter, range 0-2000 volts.
- 1- General Radio type 654-A decade voltage divider.
- 5- General Radio double plugs, type 274-M.
- 10- General Radio Binding posts, Type 138-VD.
solder, hook-up wire, cambric tubing, lugs, etc.

In operation the high potential supply provides a maximum stabilised voltage of 1300 volts. By suitable adjustments of the resistors on the voltage divider, any fractional voltage from 1300 volts to zero can be accurately secured and maintained.

The purpose of the potentiometer unit is to enable one to determine peak intensities with greater accuracy than that attainable from observation of the extent of galvanometer deflection. The deflection of the beam does not vary linearly with scale distance, due to the fact that the galvanometer scale lies in a single plane rather than being curved about the mirror as centre; thus

while readings in the mid portion of the scale probably are linear, those on either side of the median are not.

The potentiometer, then, will be used when a peak is reached, indicated by the sudden deflection of the galvanometer. Knowing the zero setting for the galvanometer, the potentiometer is adjusted so as to apply a reverse EMF thus bringing the galvanometer back to the zero position. The reading of the potentiometer is then assumed to be a measure of the intensity of the peak. The circuit diagram for the potentiometer unit is given in Figure 24.

Figure 23.

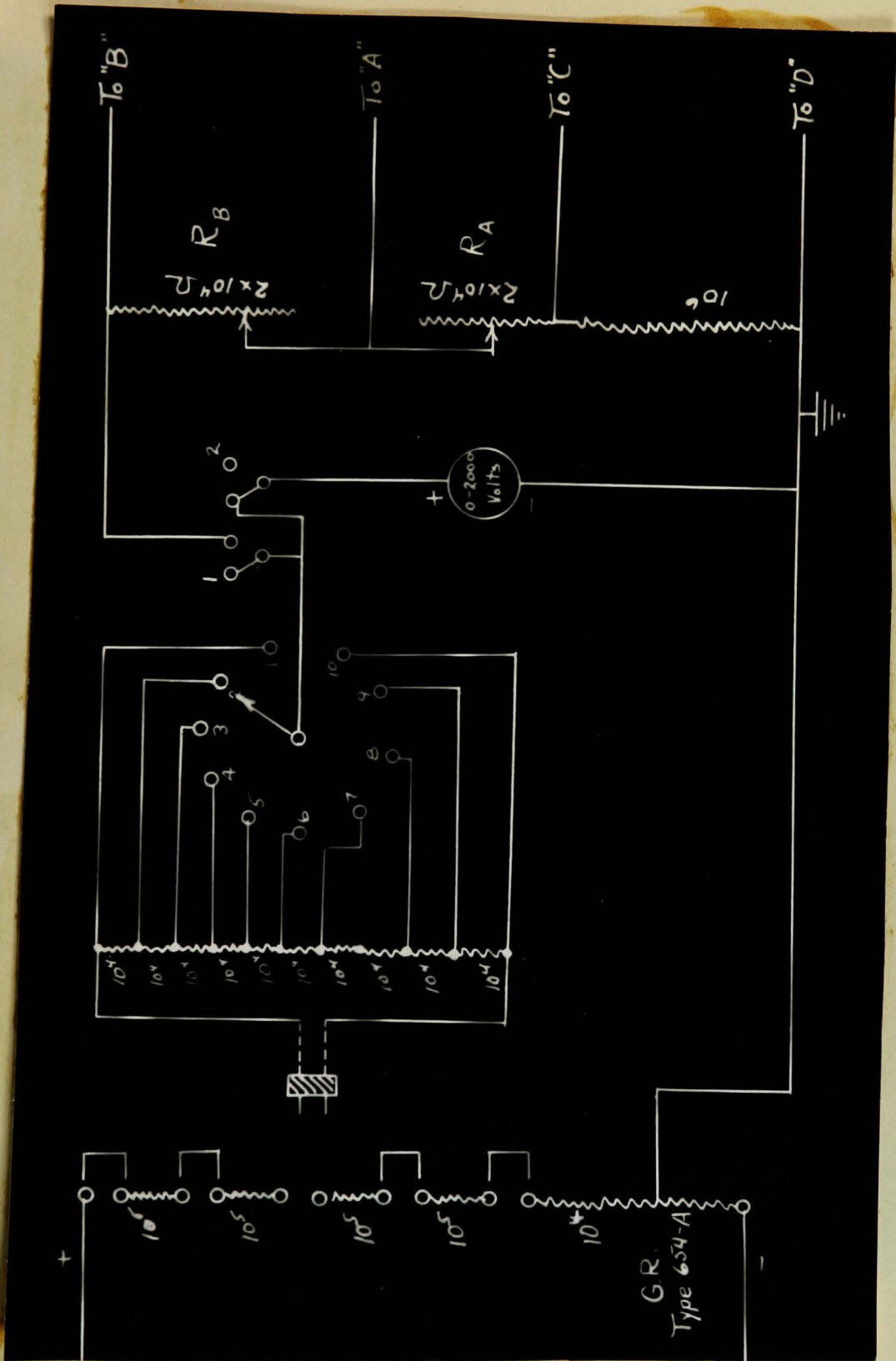
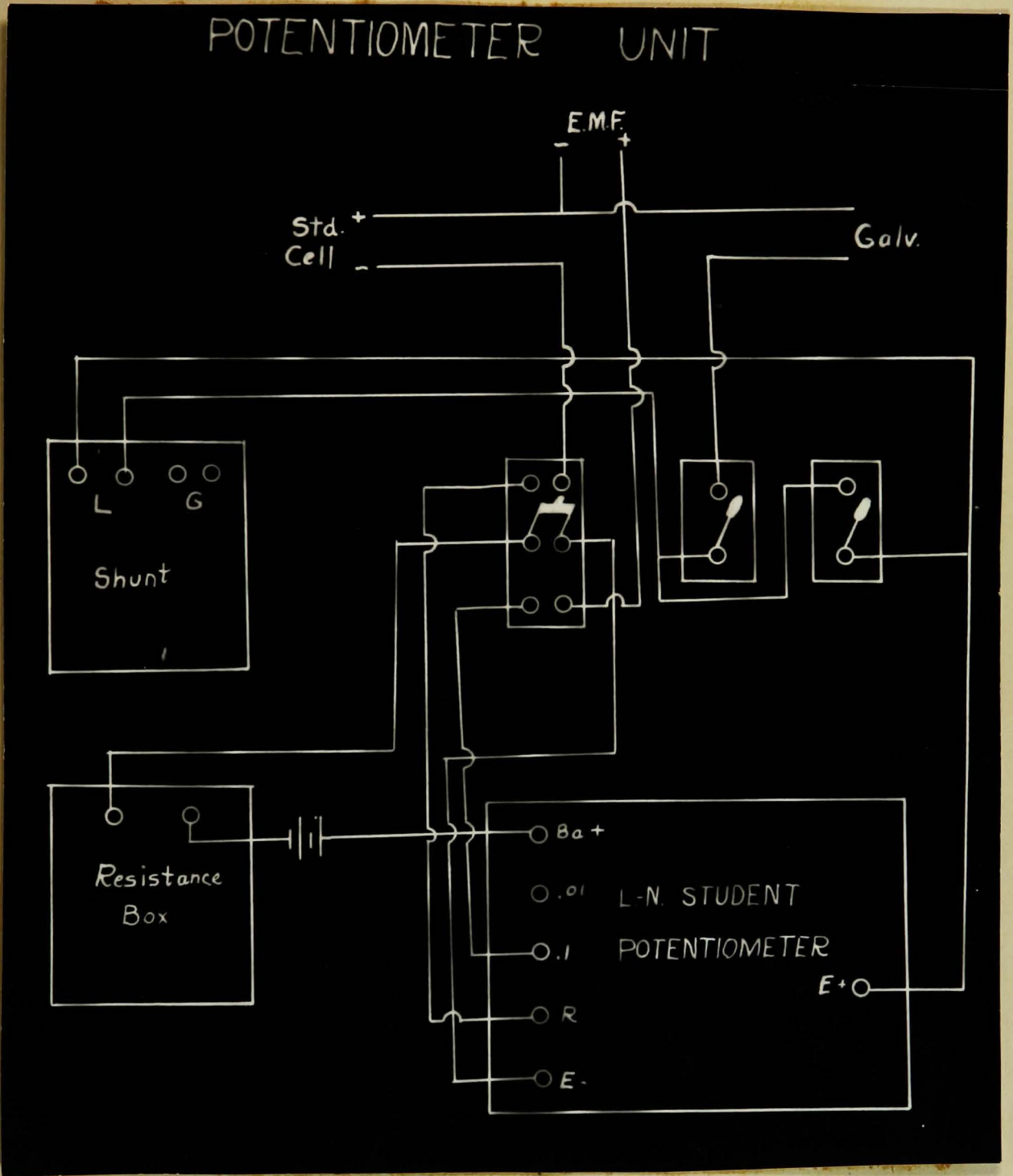


Figure 24.



Terminal Boards.

For convenience in connecting the leads to the mass spectrometer tube carrying the various accelerating voltages, filament current, and trap current outlets, a Bakelite terminal board was fastened to the side of the magnet nearest the ion source. The board contains 8 binding posts for the following supplies: "A", "B", "C", "D" voltages, trap current (E), ground connection, and two filament terminals. A schematic diagram in Figure 25, illustrates the arrangement. A similar terminal board was provided for the leads to the three furnaces around the tube. All leads to the board exclusive of the filament and ground wires consist of ignition cable insulated for 2000 volts. The filament and ground leads were run in with number 12B. and 8. guage type R solid copper conductor, with 700 volt insulation.

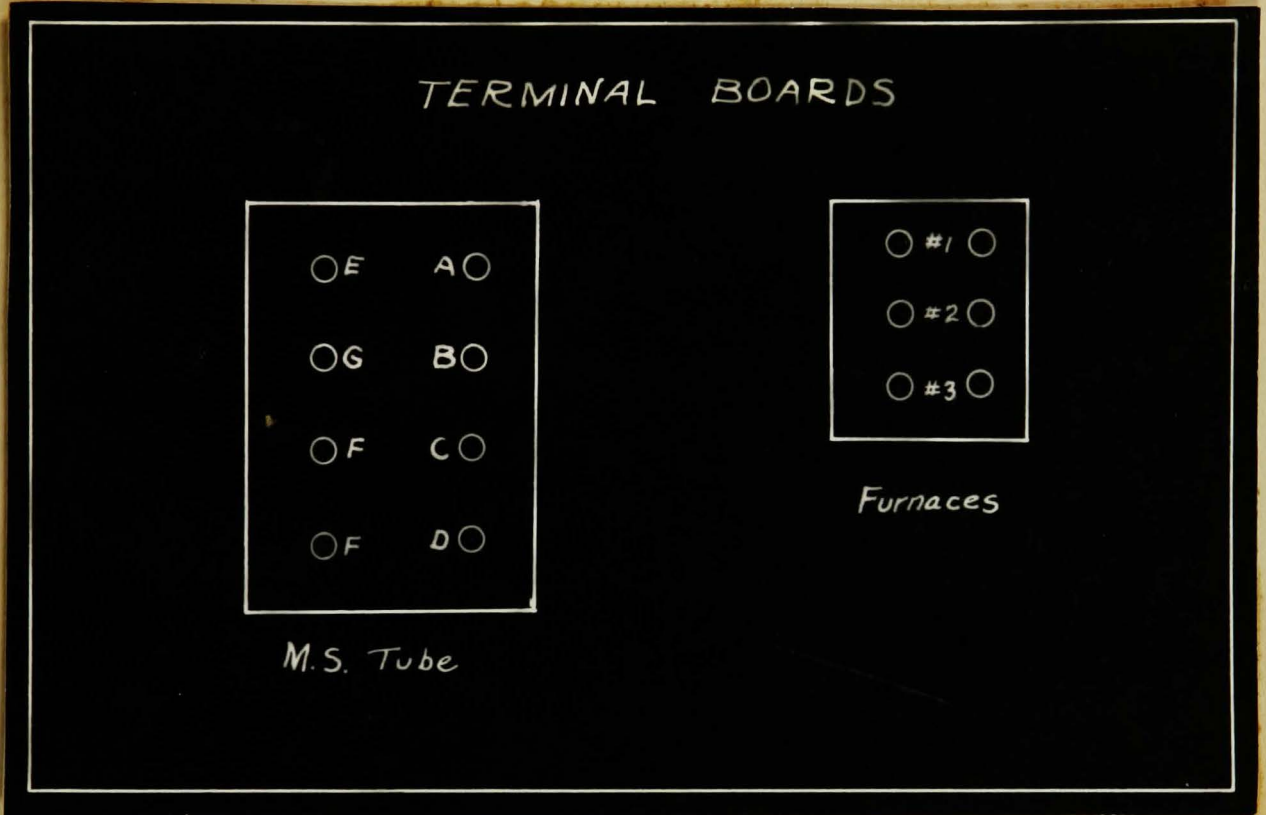


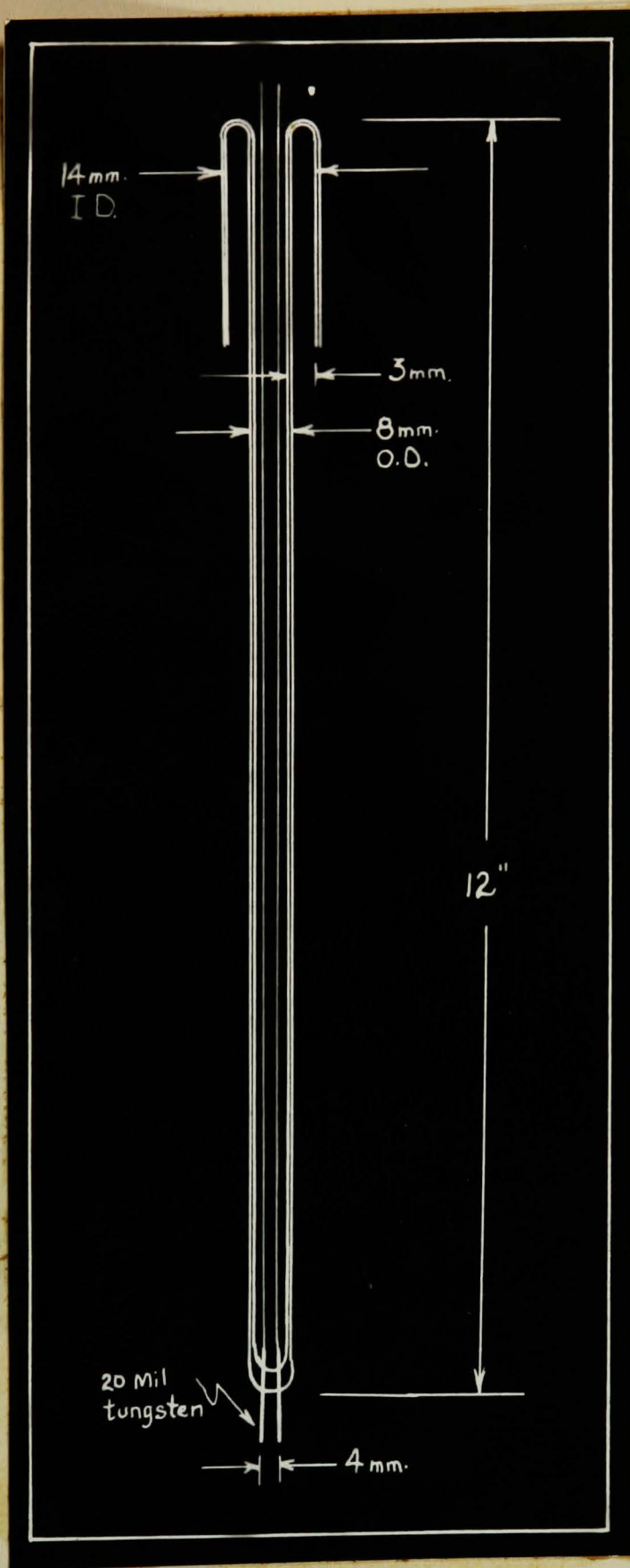
Figure 25.

The Filament and Filament Control.

The mass spectrometer filament consists of a piece of tungsten ribbon 1mm. wide, 0.025 mm. thick and 10 mm. long, bent into the shape of a horseshoe somewhat flattened. The filament is spotwelded by means of tungsten-nichrome inter seals to heavy tungsten supports of 30 mil tungsten wire in a press seal. The press seal is made in the form of a long stem of 8 mm. O.D. pyrex joined by a ring seal to the outer filament stem support of 17 mm. O.D. pyrex. The filament stem is illustrated in Figure 26. In order to limit the ionizing beam of electrons, a shield is fastened to the end of the stem, in front of the filament. This shield contains a slit of 0.2 mm. x 3.5 mm. through which the electron beam passes.

The resistance of the tungsten filament is approximately 1 ohm, and this small value necessitates the provision of power rheostats for adjusting the filament current to within tolerable limits. In the filament circuit we have placed in series, one 2.5 ohm IRC 50 watt power rheostat and one 0.5 ohm IRC 50 watt power rheostat. The former is for coarse adjustment of the current and the latter for fine adjustments during the course of a run. It is always evident, during a run, that the filament

Figure 26.



current tends to decrease, and periodic adjustment of the 0.5 ohm resistor enables one to set upon and hold the filament at any desired value. It is of interest to add that during the preparation of the manuscript, a paper by Leifer and Urey (12) disclosed that Nier of the University of Minnesota had devised a circuit for heating the filament by a stabilised source of AC; we intend to investigate this further. Our circuit, run on 12 volts from high capacity storage batteries is fused at 10 amperes, and is controlled by a master toggle switch at the control panel. A diagram of the circuit is given in Figure 27.

It must be remembered above all, however, that at the present time, there is not one substance suitable as a filament material which has proved satisfactory for all types of mass spectrometric analyses. For example, the tungsten filament is decidedly unsuitable for examining the oxygen isotopes because of the rapidity with which incandescent tungsten combines with gaseous oxygen. Moreover, investigations of the type involving the examination of products of ionisation of such substances as naphthalene for example, invariably lead to thermal decomposition of the compound upon the hot filament and a minute surface layer of tungsten carbide is formed on the filament. This acts in such a manner that although the energising filament current remains steady, the electron emission drops tremendously, and the only remedy for increasing the emission is to increase the filament current. Needless to say this often results in a burned-out filament. With regard to coated type filaments, only very general remarks can at this time be made. If a platinum filament is used in routine abundance measurements of the isotopes of oxygen, difficulty is experienced in obtaining sufficient electron current for ionising the oxygen. If resort is had to a barium-strontium oxide coating, tremendous emission is attained on one had, but this soon becomes "poisoned" by the oxygen and the emission drops. It is possible that this poisoning is similar to catalytic poisoning and is due probably to surface absorption.

The graph given in Figure 28 is a typical series of curves showing the relation between the filament current and the plate current, and between the filament current and the trap current, in the mass spectrometer tube.

The curves given in Figures 29 and 30 are typical curves showing the mass spectrums of ordinary Nitrogen and ordinary carbon dioxide respectively. The conditions existing at the time the analyses were made are described upon each curve.

Calculations on the Resolving Power.

From the geometry of the tube it is possible to predict what resolving power to expect. It has been shown that the angle focussing is independent of the angle through which the ions are deflected, and hence a 60° tube may be treated in the same manner as a 180° or 90° type. It is therefore, sufficient for a narrowly collimated beam such as is produced by slits in the C and D plates to calculate the resolving power by assuming that the image of the entrance slit upon the exit slit is the same width as the entrance slit.

The resolution is given by (21):

$$\frac{m}{m} = \frac{r}{2r}$$

since r is given approximately by

$$r = r_0 - r \frac{\text{width entrance slit} - \text{width exit slit}}{2}$$

and in this case, the entrance slit is 0.25 mm or 10 mils.

while the exit slit is 0.30 mm or 11 mils.

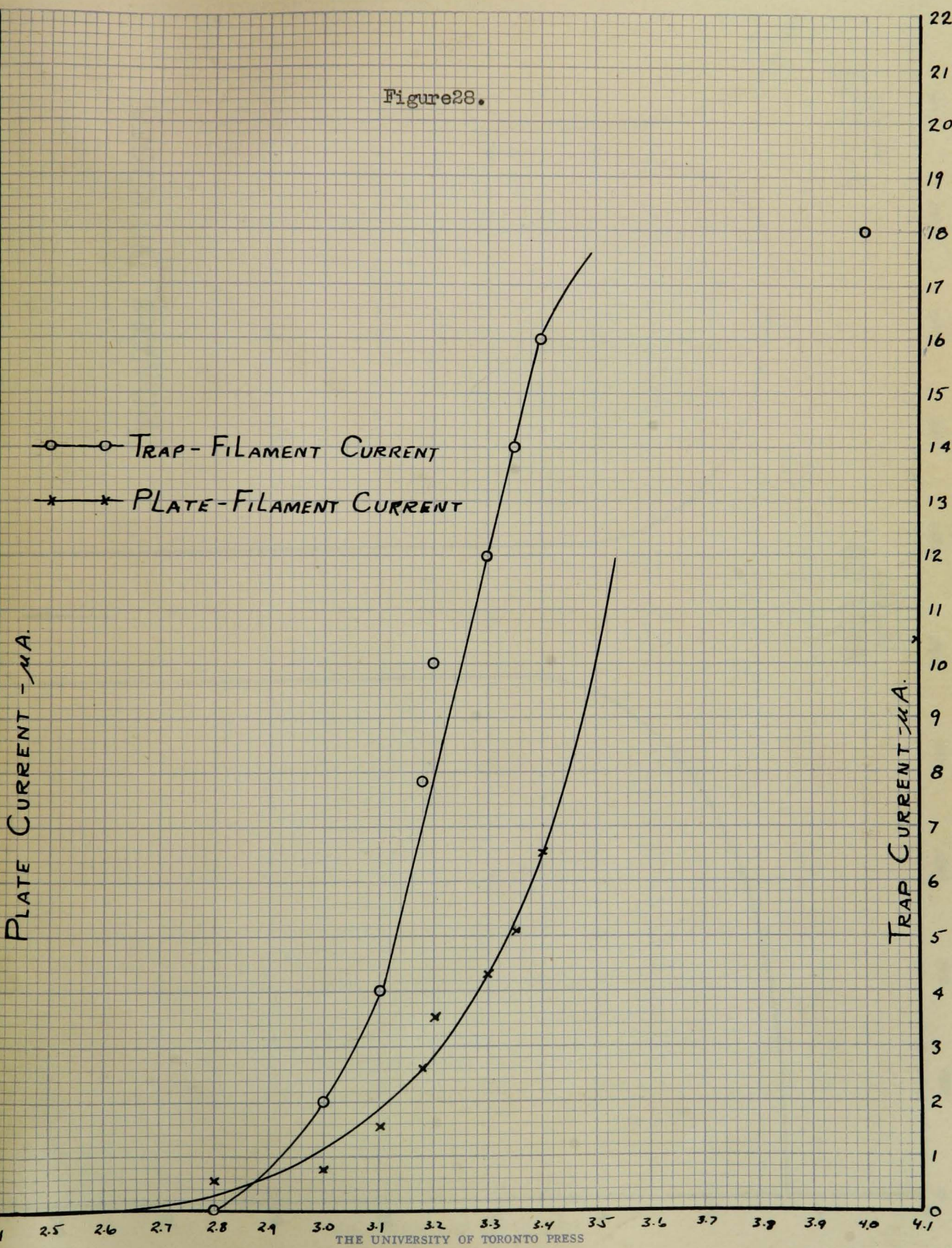
$$\text{therefore } r = \frac{10 - 11}{2} = 10.5 \text{ mils.}$$

since r_0 is equal to 5 inches or 5000 mils,

$$\frac{m}{m} = \frac{5000}{2 \times 10.5} = 238$$

The instrument should therefore distinguish between mass 237 and mass 238.

Figure 28.



○ — ○ TRAP-FILAMENT CURRENT
* — * PLATE-FILAMENT CURRENT

PLATE CURRENT - μA.

TRAP CURRENT - μA.

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Filament Current - Amperes.

Figure 29.

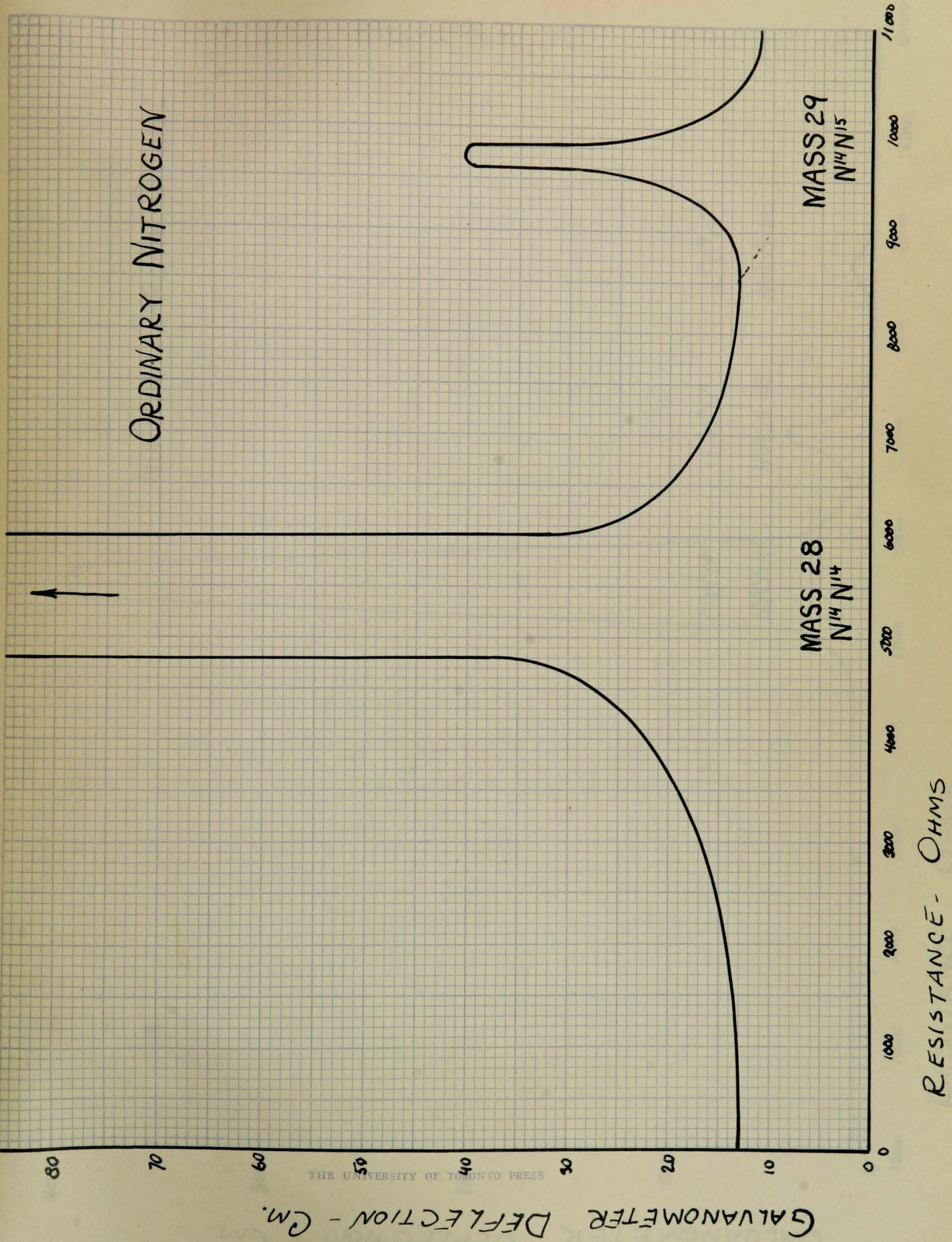
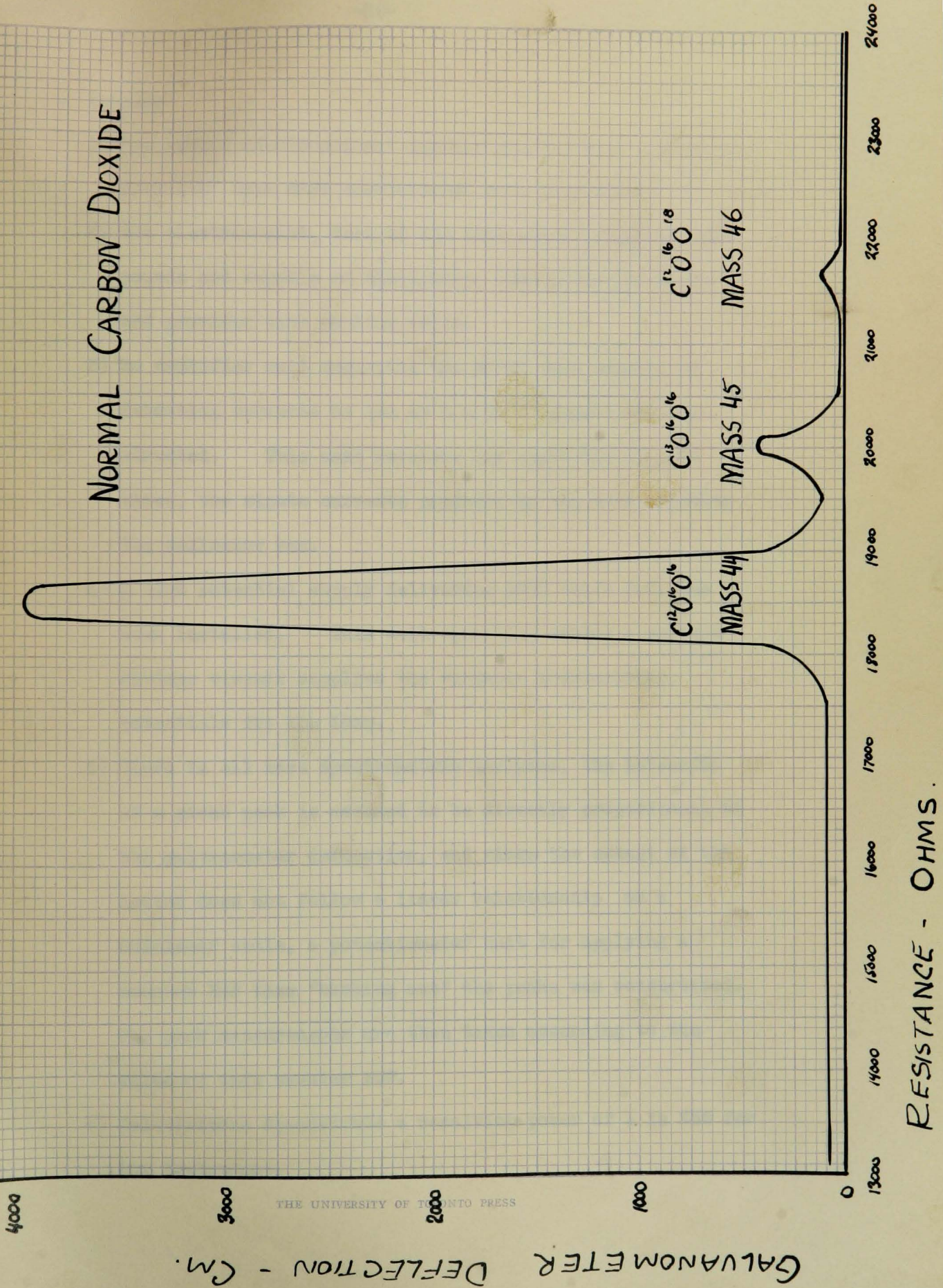


Figure 30.

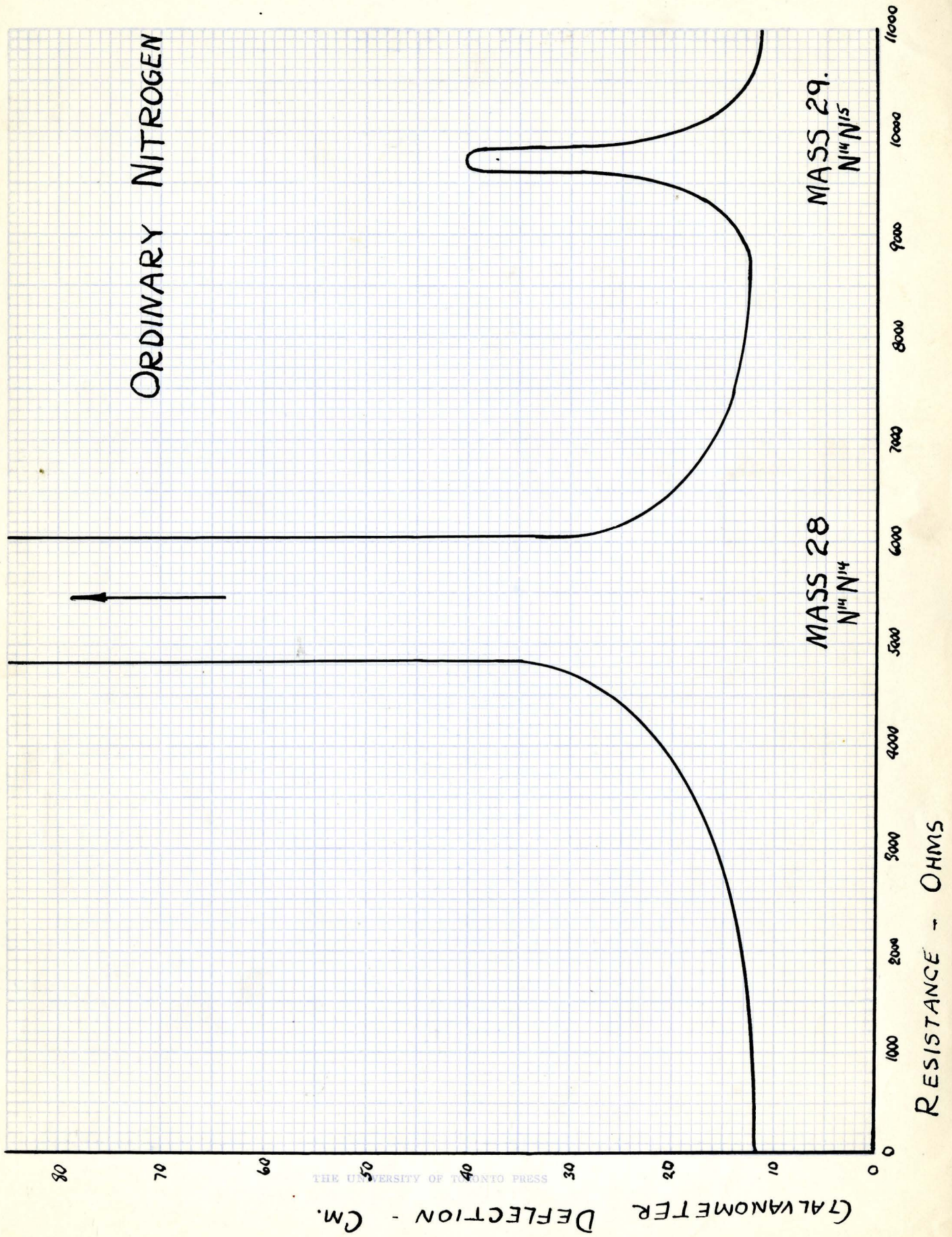


Summary

- 1) The entire constructional details for building a modified Nier 180° type mass spectrometer have been given.
- 2) The electromagnet, cast of SAE 1010 steel, weighing 3893 pounds and capable of dissipating 2360 watts was built and provided with cooling coils.
- 3) An amplifier unit employing the 6X4 tube capable of detecting currents as low as 5×10^{-18} ampere was constructed. This unit receives and amplifies 250,000 times, the minute currents produced by the ions striking the collector box.
- 4) A high potential supply delivering 1300 volts D.C. has been installed. This unit operating across a voltage divider circuit supplies the variable accelerating potentials for the ions.
- 5) Since in all mass spectrometric analyses, the intensity of a given peak is assumed to be directly proportional to the galvanometer deflection, and since the extent of the latter does not follow a linear relationship on a uniplanar scale, a potentiometer unit for applying a counter EMF thus "backing out" the peaks was constructed. The peak measurements are then taken according to the value of this counter EMF.
- 6) Calculations demonstrate a resolving power of 1 in 238 for our instrument.

Acknowledgements

The writer wishes to express his profound appreciation to Dr. H.G. Thode for his untiring efforts and most helpful suggestions, without whose help this research could not have progressed. Our joint gratitude is expressed to the National Research Council of Canada for a grant permitting construction of this instrument. Sincere thanks are due Dr. Hans Lundberg of Hans Lundberg, Limited, for the donation of the electromagnet used in our spectrometer, and to Mr. D. Drysdale for his valued help in constructing the electronic circuits. We are greatly indebted to Dr. N.B. Keevil, University of Toronto, for the loan of drawings, equipment, and for his help during the course of this research. Finally, thanks are expressed to all members of the Science Staff who gave freely of their time and advice in overcoming difficulties as they arose.



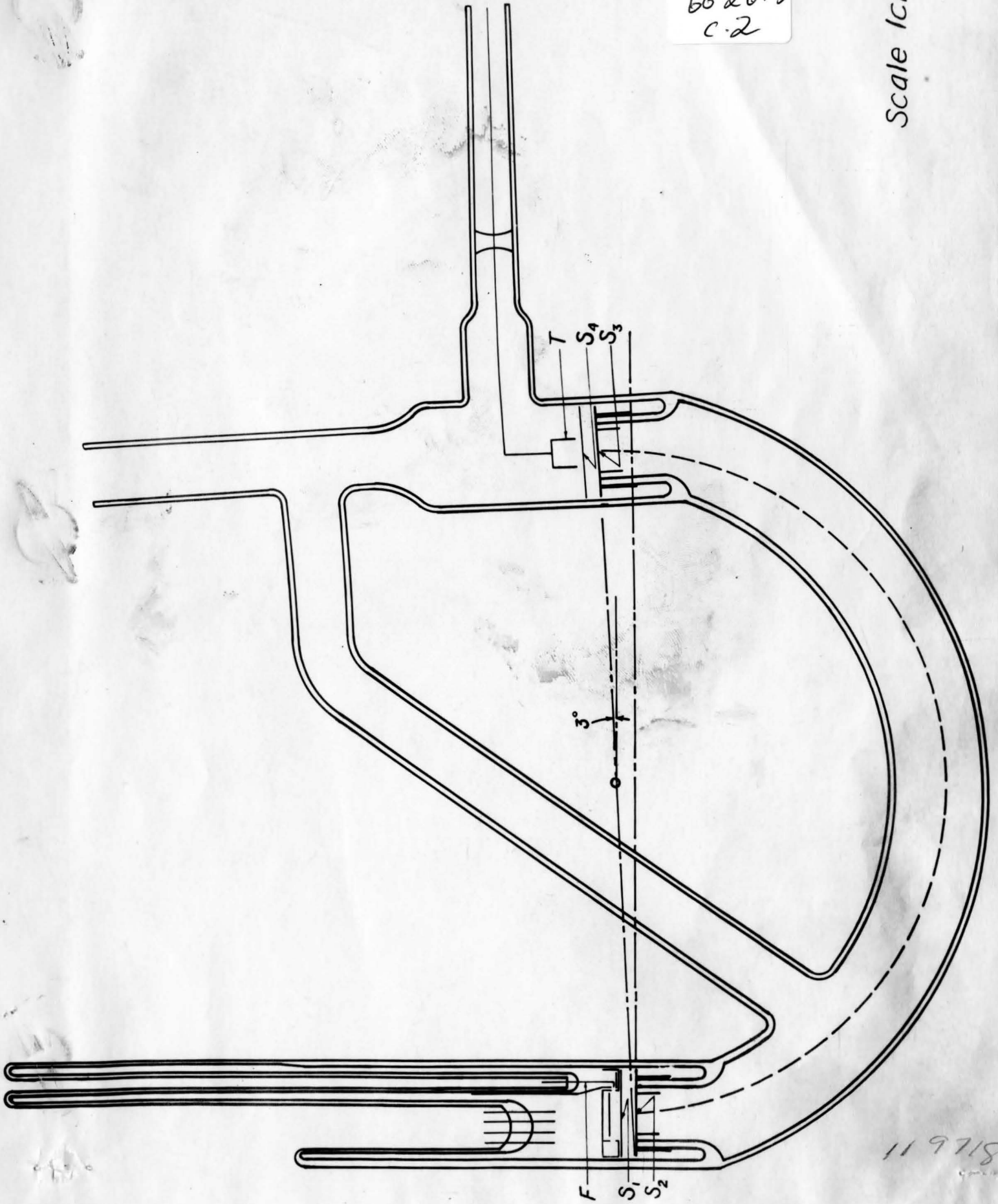
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Thode
Thesis
QD
662615
C-2

Scale 1cm = 1cm.



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