AN ION SOURCE

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PREFACE

In Chapter I a brief summary of the work done to date on the mass spectrometer under construction at Oklahoma A. and M. College is given. The electron gun used for producing ions is described in Chapter II and some values for the ion currents obtained are given. In Chapter III the means used for admitting gas samples to the ionization chamber is described, and in Chapter IV some of the difficulties encountered in attempting to make the unit function when attached to the mass spectrometer are described. Some suggestions as to the cause for the failure of the unit to operate when attached to the spectrometer and possible remedies are given in Chapter V.

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CHAPTER I

INTRODUCTION

In 1948 Truman Franklin started the construction of a Nier type mass spectrometer. Since that time work has been done on the vacuum system, coated filaments, the detection and measurement of small currents, suitable filaments for use in an electron gun, and operating procedure. The next step in the development of the spectrometer was the replacement of the coated filament as a source of ions by an electron gun and gas inlet system. The carrying out of this step was the author's task.

CHAPTER II

THE ELECTRON GUN

At first the electron gun was mounted in a glass envelope containing two metal sheets spaced 9 millimeters apart with slits and a metal plate located 13 millimeters below the last sheet containing a slit. The first slit corresponded to the defining slit in the spectrometer head, the second one to the slit at the entrance to the spectrometer tube, and the plate to the slit at the collecting end of the spectrometer tube. Figure 1 shows the electrical arrangement of the apparatus. The microammeter D measures the electrom current flowing between the filament and the plate; this current is called the emission current. The wire leading from the slits and plate C to the amplifier was shielded. This was also true for the side of the ionization chamber toward the operator.

The preliminary tests of this arrangement were run with the slits A and B, and plate C connected together and with no gas in the system other than the air left by the pumps. With this arrangement it was possible to obtain an ion current. When the heater of the diffusion pump was disconnected, the variation in pressure was so rapid that it was impossible to assign a given ion current to a given pressure. Hence it was not possible at that time to find the variation of ion current with pressure.

After readings had been obtained with all three elements connected to the amplifier, slit A was connected to ground and the remaining elements left connected to the amplifier. Then slits A and B were connected to

ground with only plate C connected to the amplifier. The data listed in Table I show that the ion current was sufficiently large to be detected by the use of the amplifier and a galvanometer at hand. The sensitivity setting of the amplifier was 2. The full sensitivity of the amplifier, approximately 4×10^{-12} amperes per millimeter, is obtained when the sensitivity is set at 18.

As it was possible to obtain readings with plate C alone connected to the amplifier, the wiring arrangement was modified somewhat in order that the ions might be accelerated by a potential independent of the emission current and the operating conditions of the mass spectrometer more closely approximated. The ground was removed from the positive terminal of the ammeter measuring the filament current and only plate C connected to the amplifier. Slit B was grounded and connected to the negative terminal of a 90-volt battery whose positive terminal was connected to slit A. A 3-volt battery was placed between slit A and the plate (negative terminal to slit A). Table II shows some of the values obtained. The readings given in column A were taken with the amplifier sensitivity set at the same value as that for the readings in Table I; however, it must be remembered that the pressure in the ionization chamber was not necessarily the same for all data. The data shown in parts B, C, D, and E of Table II were taken with the same setting of the amplifier sensitivity (6). Again the data show that the ion current is sufficiently large to be easily detected by the use of the amplifier and galvanometer.

Table 1. Ion Currents in Residual Gases

Part A
(A, B, and C connected to amplifier)

Galvanometer Deflection in Centimeters	Emission Current in Microamperes
5.6	0.9
12.0	1.0
17.8	1.2

Part B
(A connected to ground, and B and C to amplifier)

Galvanometer Deflection in Centimeters	Emission Current in Microamperes
0.4	1.2
1.2	3.2
2.3	5.6
3.1	7.9
4.0	10.5
4.9	13.7
5.5	15.5

Part C
(A and B connected to ground, and C to amplifier)

Galvanometer Deflection in Centimeters	Emission Current in Microamperes
0.3	1.0
0.6	2.3
1.4	4.9
1.9	6.8
2.6	9.3
3.6	13.5
3.8	14.0
4.2	15.5

Table 2. Ion Currents in Residual Gases with Increased Accelerating Potentials

Galvanometer Deflection in Centimeters	Emission in Microamperes	Galvanometer Deflection in Centimeters	Emission in Microamperes
A		B	
0.2	1.3	0.7	1.0
0.7	3.8	2.1	4.0
1.1	6.0	3.6	8.5
1.3	8.4	4.1	10.0
1.8	12.3	4.8	11.3
2.0	12.5	5.0	12.0
2.3	15.0	4.9	11.5
2.4	15.4	7.8	15.2
2.5	15.9	6.5	12.1
2.6	16.0	0.3	1.9
C		D	
1.8	1.1	1.5	1.6
1.9	3.0	2.6	3.0
1.9	2.8	3.3	4.0
2.3	4.0	3.9	5.0
2.6	5.1	4.6	6.0
3.0	6.1	6.2	7.2
3.4	7.1	7.1	8.0
4.6	9.1	8.2	9.0
		9.0	10.0
		7.9	8.0
E			
1.9	1.9		
3.3	3.1		
4.1	4.1		
5.0	5.1		
5.6	6.0		
6.6	7.2		
7.0	7.9		
8.2	9.2		
8.8	10.0		
9.0	10.0		
9.0	9.7		

CHAPTER III

THE GAS INLET SYSTEM

The development of the gas inlet system may easily be divided into two phases. One deals with the construction of the leak itself and the other with the construction of a containing vessel so made that only the gas which is to be analyzed may enter the leak. It will be seen later that both of these phases are closely related in that they both affect the rate at which the gas enters the ionization chamber.

The Leak

There are various kinds of gas leaks that may be used. In general, these fall into two classes: (1) those whose size of opening is fixed, and (2) those whose size of opening is variable. The Hopfield leak[1] and a leak made of copper tubing wrapped around a movable drum[2] are examples of the latter type. The capillary leak and the gold foil leak are examples of the fixed leak. With a gold foil leak there is the danger that the foil may rupture and let the pressure rise abruptly to a rather high value.

Because of its simplicity the capillary leak was chosen. A hole was drilled in the brass top plate of the electron gun and a tapered glass

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[1] "Glass Variable Microleaks for Gases," Review of Scientific Instruments, XXI (1950), 671.

[2] A. O. Nier, E. P. Ney, and M. G. Ingham, "Adjustable Gas Leak," Review of Scientific Instruments, XVIII (1947), 191.

tube led through the brass top plate and down to and through a hole in the U-shaped part of the plate. The upper end of the glass tube had a stopcock attached to it. Capillaries were drawn from several sizes of tubing and capillary tubing. Since it was not known what size capillary would work and also since there were no means readily available for measuring the size of the capillaries, it was necessary to seal the capillaries (with the small end closed), one at a time, onto the glass tube leading into the ionization chamber and break off the closed end while watching the pressure gauges.

When the end of the first capillary was broken off, the pressure rose rapidly to well over 100 microns. When the second capillary was broken off, the pressure started rising at the rate of about one micron every one or two seconds. This rate of leak was too rapid and hence the capillary was removed and a third one sealed on. With the third capillary the pressure rose from approximately 1.4×10^{-4} to 1.7×10^{-4} millimeters of mercury. Small pieces were broken off this capillary until less than one-half of the original remained, at which time the pressure was 3.4×10^{-4} millimeters. The capillary was subjected to atmospheric pressure when this value was obtained.

In an article by Nier in the 1940 volume of the Review of Scientific Instruments (page 212) the value 10^{-4} millimeters is given as the estimated value of the pressure in the ionization chamber; hence, it was decided that the capillary was of suitable size---particularly since the capillary could be enclosed in a vessel and the pressure in the vessel made either greater or less than atmospheric pressure. Thus the pressure in the ionization chamber could be varied about the value 3.4×10^{-4} millimeters.

The Reservoir

In order to provide a means for letting a given gas into the ionization chamber and to provide a means of controlling the rate of flow of the gas, and hence, the pressure in the chamber, a glass system was built around a capillary (not the one previously discussed). The system is shown in Figure 2. It was necessary to open the system several times and shorten the capillary before the opening was such that the pressure in the ionization chamber could be controlled over a considerable range.

With a 3-volt battery between the plate and slit A and a 90-volt one between slits A and B and the other connections as shown in Figure 1 (except for the modifications stated on page 3), it was found that the ion current increased as the pressure increased for a constant emission current of 10 microamperes. Since the ion current was sufficiently large to be measured and means had been developed for admitting gas samples to the ionization chamber, the unit was then mounted on the mass spectrometer.

CHAPTER IV

SOURCE MOUNTED ON SPECTROMETER

The electron gun was slipped into the mass spectrometer head, and some modifications were made in the lengths of tubing of the gas inlet system. It is to be noted that although the pumps are approximately the same distance from the ionization chamber as before, the pressure gauges are now connected to the side wall of the chamber.

The electrical connections were made as shown in Figure 3. The heat evolved by the filament caused sufficient expansion that the plate came into contact with slit S_1, Figure 4. In order to correct this the electron gun was removed from the spectrometer head and pieces of mica glued to the plate with shellac. After two days of outgassing (filament used as heater) an attempt to find a peak was made. The accelerating potential and emission current were held constant, and the magnetic field and pressure were varied. The pressure in the ionization chamber was varied by admitting argon to the gas reservoir. No peak was found. The sensitivity of the amplifier was increased by changing the grid resistor from 9,700 megohms to 110,000 megohms, but still no abrupt change occurred in the galvanometer reading.

In order to determine whether or not ions were being produced, a galvanometer (0.7 mm./microvolt, CDRX 330 ohms, and resistance 49 ohms) and a source of potential (A, Figure 3) were placed between the plate and slit S_1. The change in magnitude of the galvanometer deflection as the

potential was varied and the polarity of the applied potential were such as to suggest that the current flowing through the galvanometer was due to electrons or negative ions. If this current were due to electrons, it might be an indication that slit S_1 was not sufficiently distant from the electron beam. In order to increase this distance the assembly was again removed from the spectrometer and the rods supporting the elements of the electron gun were shortened about 6 millimeters. Measurements showed that before the rods were shortened the distance between the electron beam and slit S_1 (Figure 3) was much closer than that between the electron beam and slit A (Figure 1). While the gun was dismounted, the mica was removed from the plate and the filament replaced.

After the unit had been placed in the spectrometer head again and outgassed, no peak was found although the emission current was set at 35 to 40 microamperes and the pressure varied from 3×10^{-5} to 2×10^{-4} millimeters of mercury.

The system was outgassed for several hours at a higher temperature. A galvanometer (0.7 mm./microvolt) was placed between the plate and the A battery (Figure 3). The emission current was approximately 35 microamperes and the pressure 3×10^{-5} millimeters. A deflection of 3 millimeters in such a direction as to indicate positive ions was obtained. Slits S_1 and S_2 were connected together, and the entire accelerating potential (138 volts) and the galvanometer placed in series between the plate and the slits. The galvanometer deflection was 5 millimeters and remained constant over a pressure range of 3×10^{-5} to 2×10^{-4} millimeters of mercury. When the emission current was run up to 35 microamperes or from there down to zero the galvanometer deflected over 10 centimeters, but then returned to either the reading or the rest position. This indicates a capacitive effect, the cause for which has not been determined.

CHAPTER V

SUMMARY AND CONCLUSION

The electron gun and gas inlet system are capable of delivering a measurable ion current when the distance between the second slit and the collecting plate is a few millimeters and when the system is pumped through a large tube connected to the ionization chamber. When the assembly was mounted on the mass spectrometer, no peak was detected.

Time did not permit any further modifications to be made. However, there are several things that might be done. During the preliminary part of this work the electron gun was mounted in a glass envelope. When the unit is mounted in the metal spectrometer head the electric field in the chamber is somewhat modified. Also, a glass envelope is easily outgassed whereas a metal one continues to give off gases for a considerable length of time.

If the pressure in the spectrometer tube is sufficiently high the ions will collide with each other frequently and never reach the collecting chamber. To remedy this a hole might be drilled in the spectrometer head and a tube (larger than the spectrometer tube) run from the head to the diffusion pump. Not only would this provide for pumping from the head end, but the tube would also by-pass the narrow slits in the spectrometer tube through which the system must now be pumped.

It might also be noted that most spectrometers have an insulated metal strip mounted on the plate. This strip is called a pusher, and a potential is applied to it in such a manner that the positive ions formed are pushed

toward the slits. This, however, did not seem to be necessary in the glass enclosure.

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APPENDICES

Figure 1. Ion Source
(after a drawing by Wei)

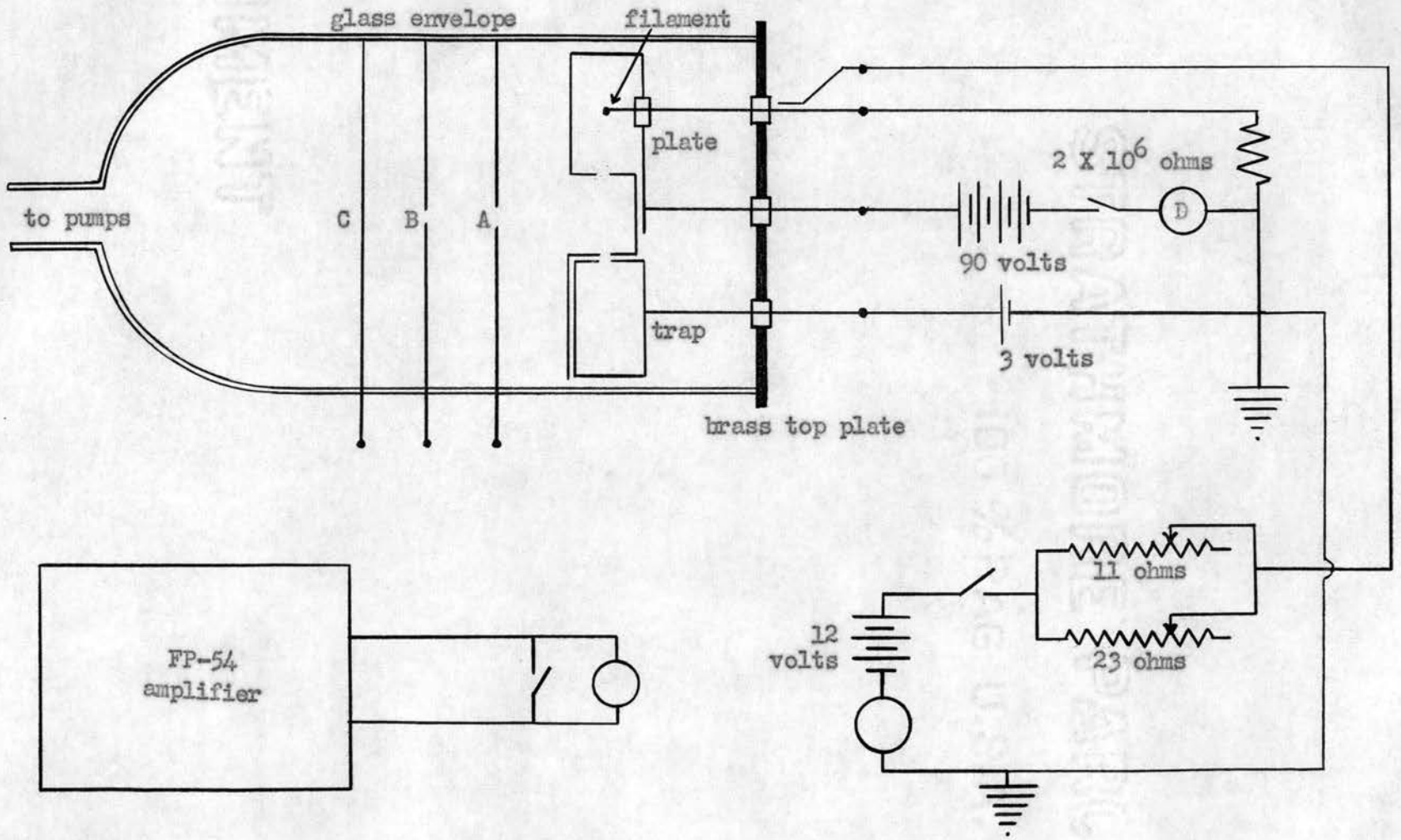


Figure 2. Gas Inlet System

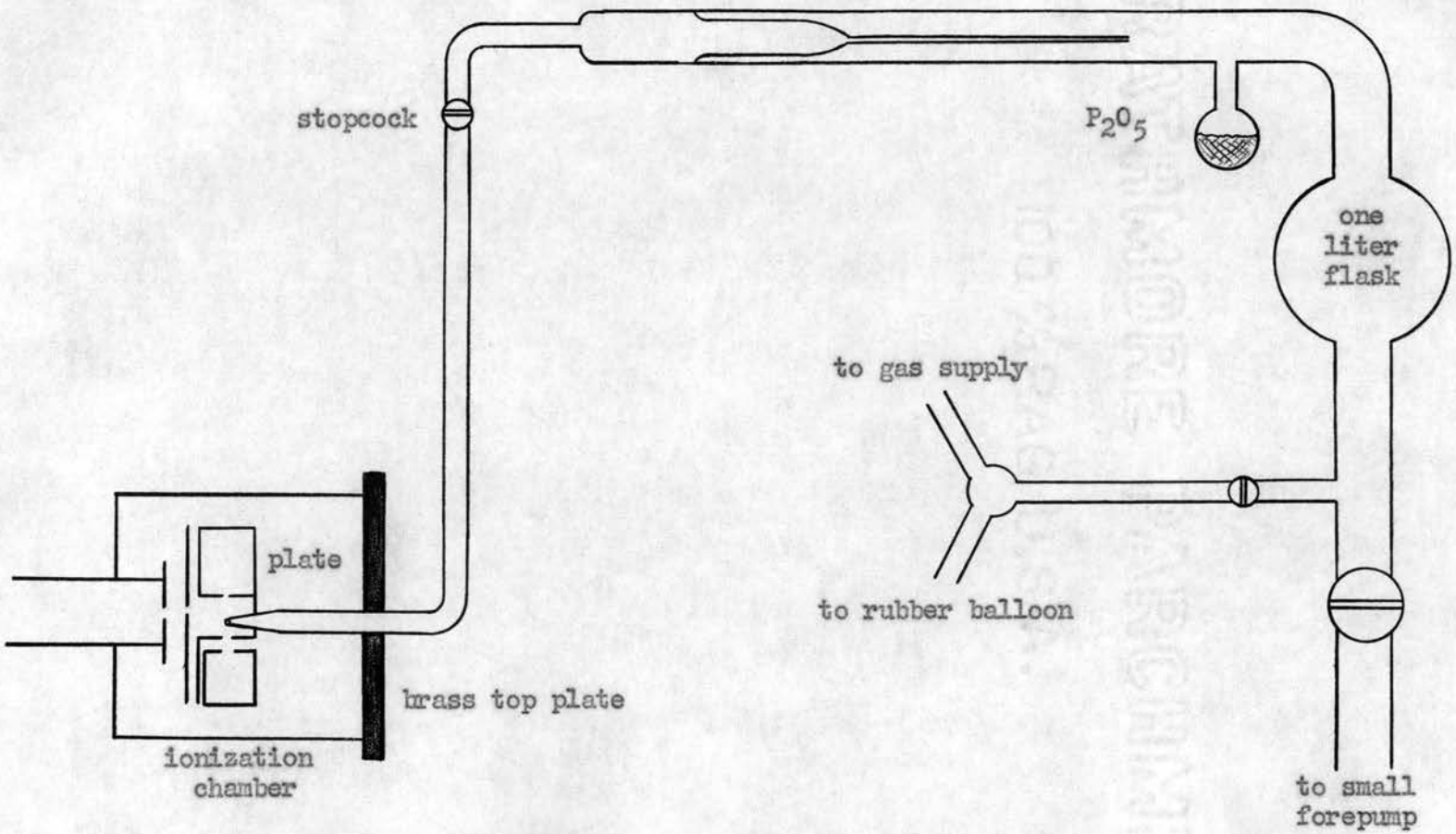


Figure 3. Electrical Connections for Mass Spectrometer
(after a drawing by Lull)

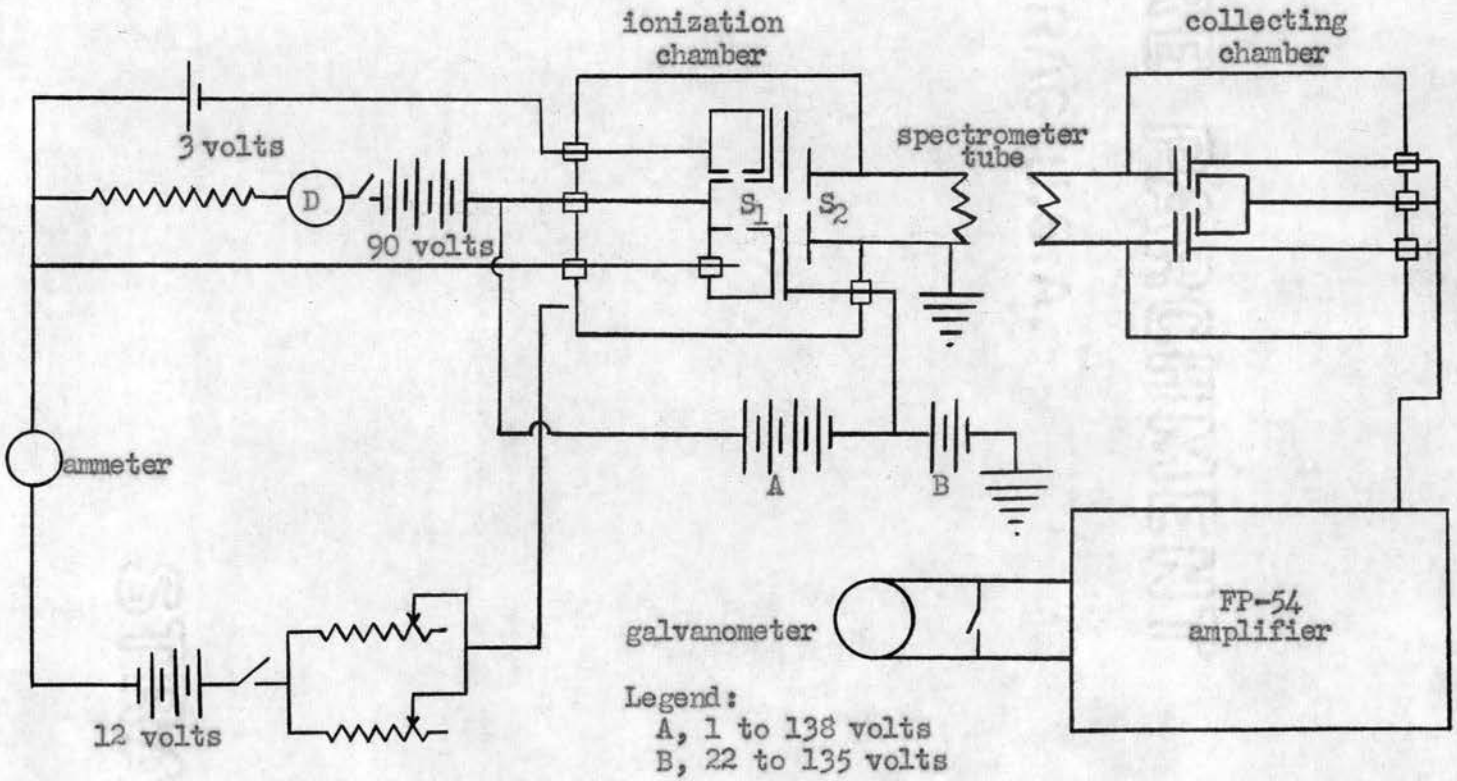
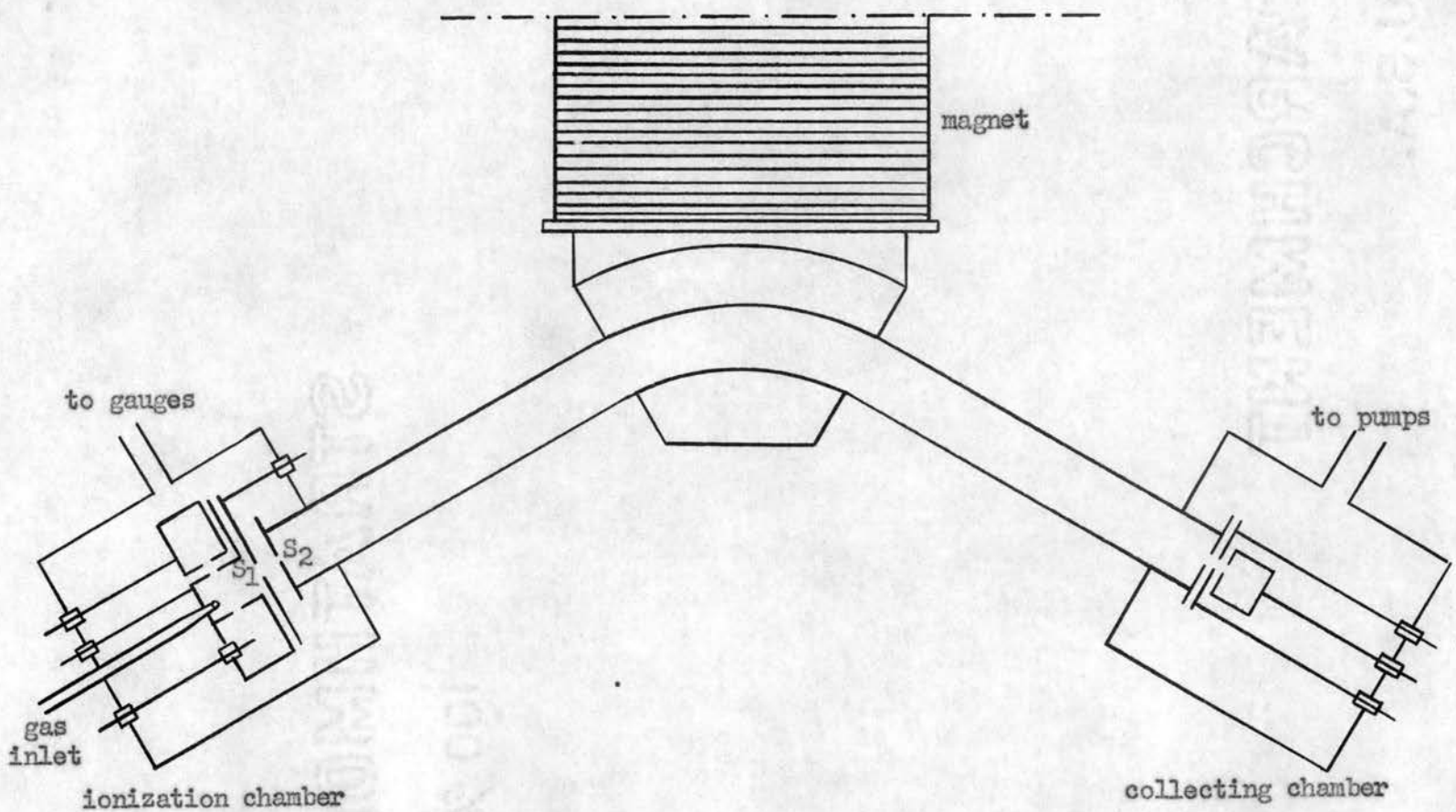


Figure 4. Nier Type Mass Spectrometer
(after a drawing by Lull)



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