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THE MEASUREMENT OF DETECTOR CURRENTS
OF A MASS SPECTROMETER

by

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Bachelor of Science

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MASTER OF SCIENCE

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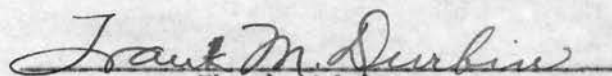
THE MEASUREMENT OF DETECTOR CURRENTS OF A MASS SPECTROMETER

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THESIS AND ABSTRACT APPROVED:


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The Measurement of Detector Currents of a Mass Spectrometer

I. Introduction

The purpose of this paper is to describe two vacuum-tube electrometers used with the Nier-type mass spectrometer currently undergoing adjustment in the Physics Department at Oklahoma A. and M. College. This spectrometer was constructed by Mr. Truman Franklin, and readings first taken by Mr. Richard Barron and the author in the Spring of 1950. The author worked with the particular project of measuring the ion currents present after acceleration by the source potential and deflection in the magnetic field of the instrument.

Already available for this task in the department was a vacuum-tube electrometer, or current amplifier, incorporating a FP - 54 type tube, built by Mr. Julian B. Grafa. This instrument served very successfully as a recording device. The author built a second circuit around a new miniature tube, the number 954. The characteristics and relative virtues of these two units will be discussed.

II. General Characteristics of the Mass Spectrometer

Figure I presents a simplified schematic diagram of the mass spectrometer used in this research. Ions emitted by the coated filament, F, are accelerated by a known voltage in the source chamber and an ion beam is directed down the deflection tube by the defining slits, S₁ and S₂. The ratio of charge to mass of these ions determines their velocity, as given by the equation

$$v^2 = \left(\frac{e}{m} \right) (2 \times 10^6) V$$

where v is ion velocity in $\frac{\text{cm}}{\text{sec}}$, V is the accelerating potential in volts, and $\left(\frac{e}{m} \right)$ is the ratio of charge, in e.m.u., to mass in grams.¹ As this beam passes through the magnetic field of the deflecting magnet its path has a curvature of radius r , given by the equation

$$r = v \div \left(\frac{e}{m} \right) H$$

where H is the strength of the magnetic field in Oersteds.² Only ions of one particular $\left(\frac{e}{m} \right)$ will be deflected in the proper direction to strike the collector plate in the detector chamber. Thus separation of the many ions of the source into individual groups is accomplished.

The author was concerned with the task of measuring the ion current which passed through the instrument. In well adjusted instruments this current is of the order of 10^{-10} amperes.³ It was expected that in our particular case this value would not be reached, since there had been no previous adjustment for optimum operating conditions; this was the initial

¹ Hoag and Korff, Electron and Nuclear Physics, p. 236.

² Ibid.

³ Graham et al., "A. C. Operated Mass Spectrometer", Journal of Scientific Instruments, 24 (1947), p. 119 ff.

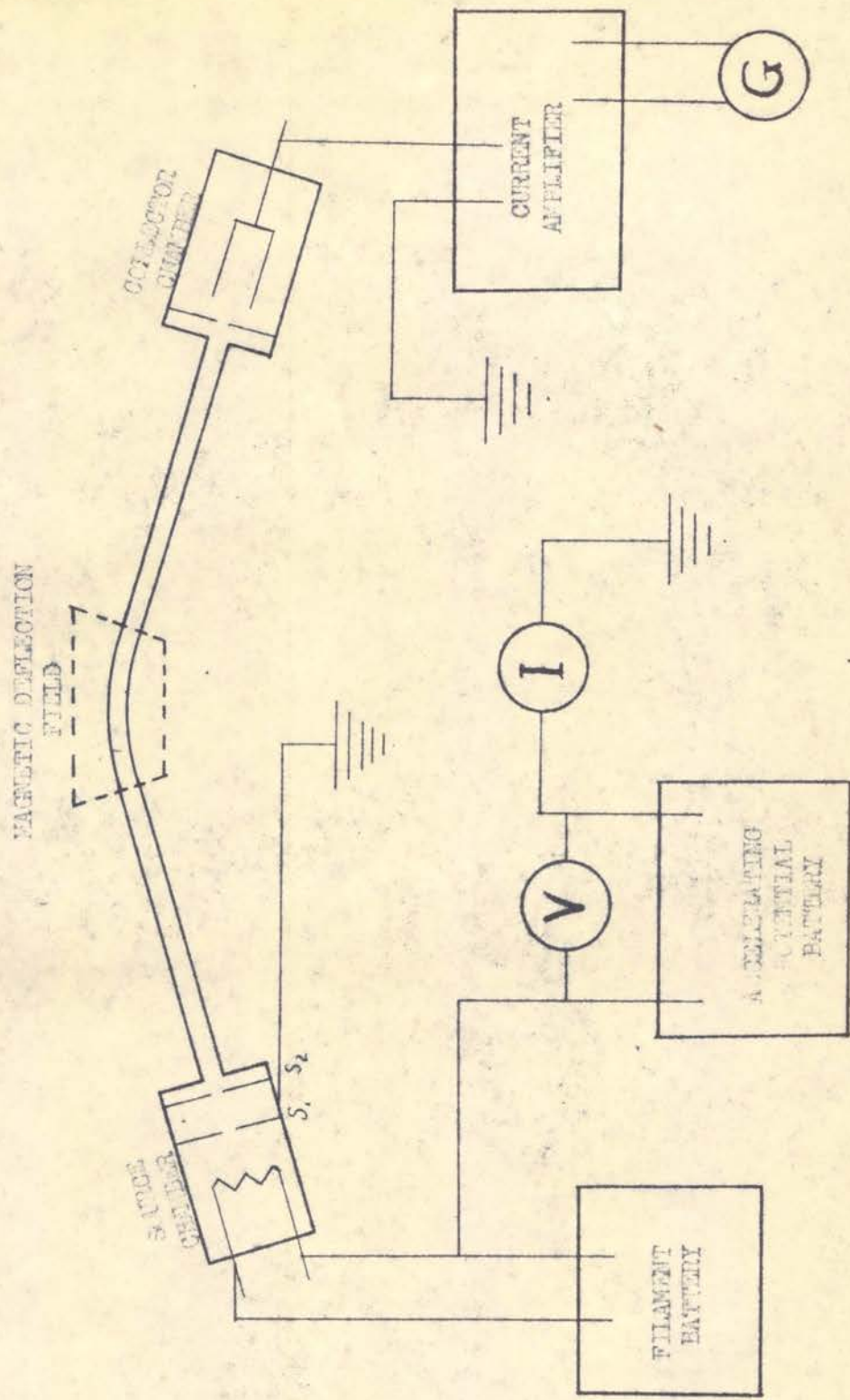


Figure 1 - NIER TYPE MASS SPECTROMETER

trial of the equipment. Actually, the largest current read was 3.9×10^{-12} amperes, which illustrates that better performance may be expected with careful alignment of the device. Ion currents were first read through the use of an FP-54 current amplifier, since it was available in the department, and because its performance was known to be highly reliable. A current amplifier built around a 954 tube was later devised as a trial at more compact and versatile instrumentation.

To review briefly the other operating characteristics of the mass spectrometer, it may be noted that the total emission from the coated filament was maintained at a value of approximately 3.5×10^{-7} amperes, with accelerating potentials around 100 volts. Readings have been taken with higher accelerating potentials, but the total ion emission from the filament could not be read accurately, apparently due to secondary emission of an electron current from the material forming the source chamber. This effect was not noted at lower accelerating potentials.

The measuring of this emission current is no simple feature in itself. A large storage battery capable of furnishing four amperes of filament current, as well as associated wiring and two Stupekoff insulators leading into the vacuum chamber must be isolated from ground, so that leakage currents due to the high accelerating potential will not mask the minute emission currents to be measured. To accomplish this the resistance to ground should be greater than 10^{10} ohms. Maintaining such a high value requires that all meters, the batteries, and lead wires be supported by glass or similar insulators.

The magnetic deflecting field was measured with a search coil and ballistic galvanometer. An accompanying graph, Figure 2, shows values of

5
Magnetization of Deflection Magnet
of Hier - type Mass Spectrometer

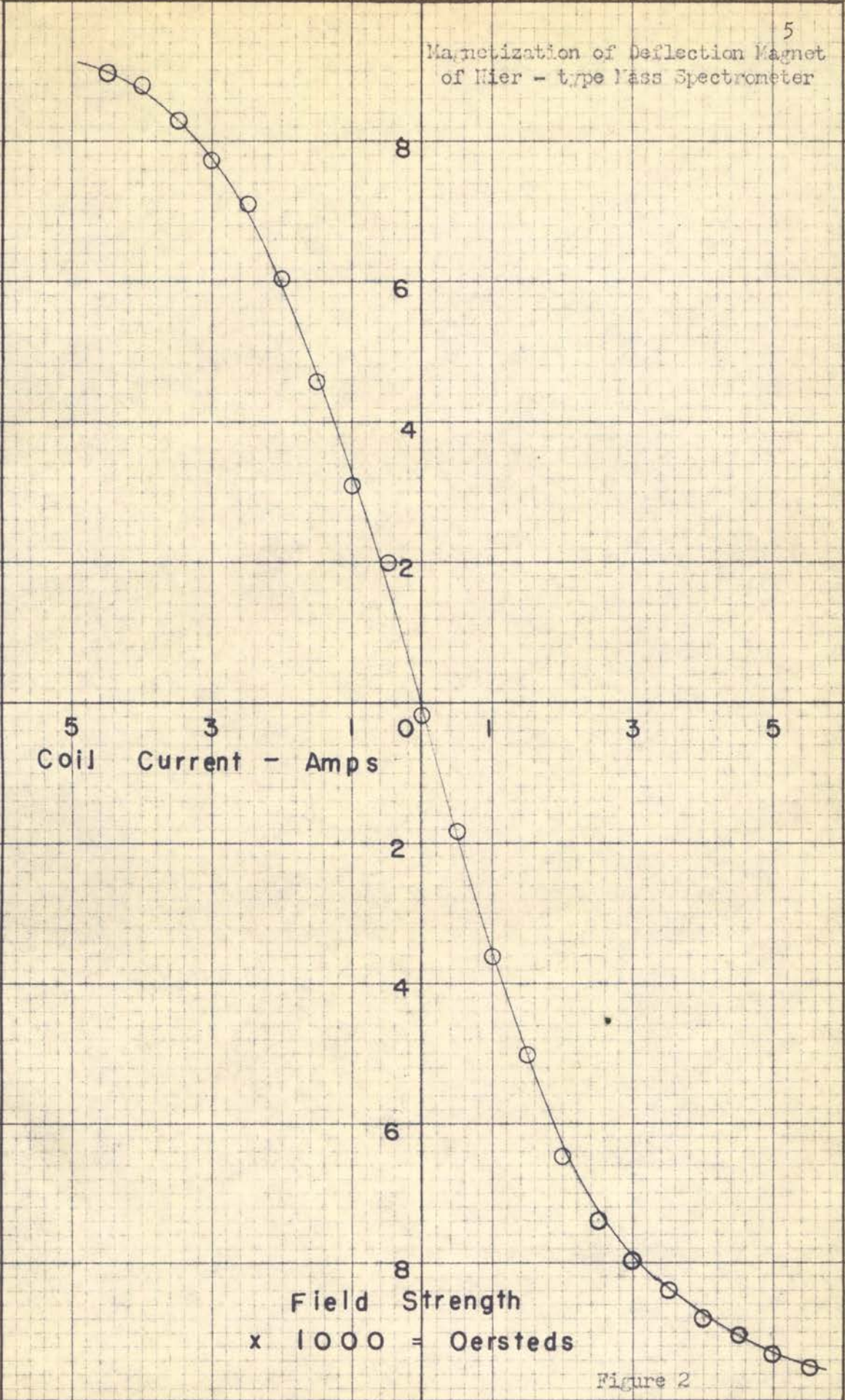


Figure 2

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the magnetic field of the large magnet used with the instrument. With accelerating potentials below 800 volts there will be little occasion to use excitation current values for the magnet greater than two amperes, since masses up to atomic weight of 400 will, if singly charged, be focused by such a value. It will be noted that under two amperes the magnetization of the field is approximately proportional to the field current.

The entire chamber of the mass spectrometer is maintained at a high vacuum by the action of a mechanical forepump and an oil diffusion pump. As read with an ionization gauge mounted at the source chamber, the minimum pressure attained was 10^{-6} millimeters of mercury. Due to emission of gases from the heated filament, operating pressures were usually around 10^{-5} millimeters of mercury.

III. The FP-54 Current Amplifier

The measuring of the ion current through the mass spectrometer is not a simple matter of connecting a recording meter to the plates in the detector chamber. No mechanical meters are available which indicate such small currents. The only direct method of detecting such currents is through the discharge of an electroscope connected to the collector plate,¹ but such readings would be tedious and slow. Some method is desired whereby ion current may be read instantaneously as, say, the magnetic field is varied, yielding a mass "spectrum". As in so many other applications, it is possible to apply vacuum tube amplifier circuits to this problem. Figure 3 represents such a circuit, utilizing a tube especially designed for such service, the FP-54. This is the circuit used by the author in his early investigations.

Several factors must be considered in the design of these circuits and tubes. Since the currents to be measured are so small, extreme care must be taken to eliminate current due to the control grid of the tube. The sensitivity is limited by the size of the control grid current. Grid current is caused by a number of factors. There may be an electron current flow to the grid from the cathode, or due to secondary emission from other tube elements. There may be a positive ion current caused by electron bombardment of residual gas molecules within the tube, or perhaps caused by ionizing radiations, such as cosmic rays, or soft X-rays generated by electrons striking the plate. Ions sometimes are emitted from the filament, along with the desired electron flow. Leakage currents over the physical dimensions of the tube will also contribute to

¹ Richtmyer and Kennard, Introduction to Modern Physics, p. 547.

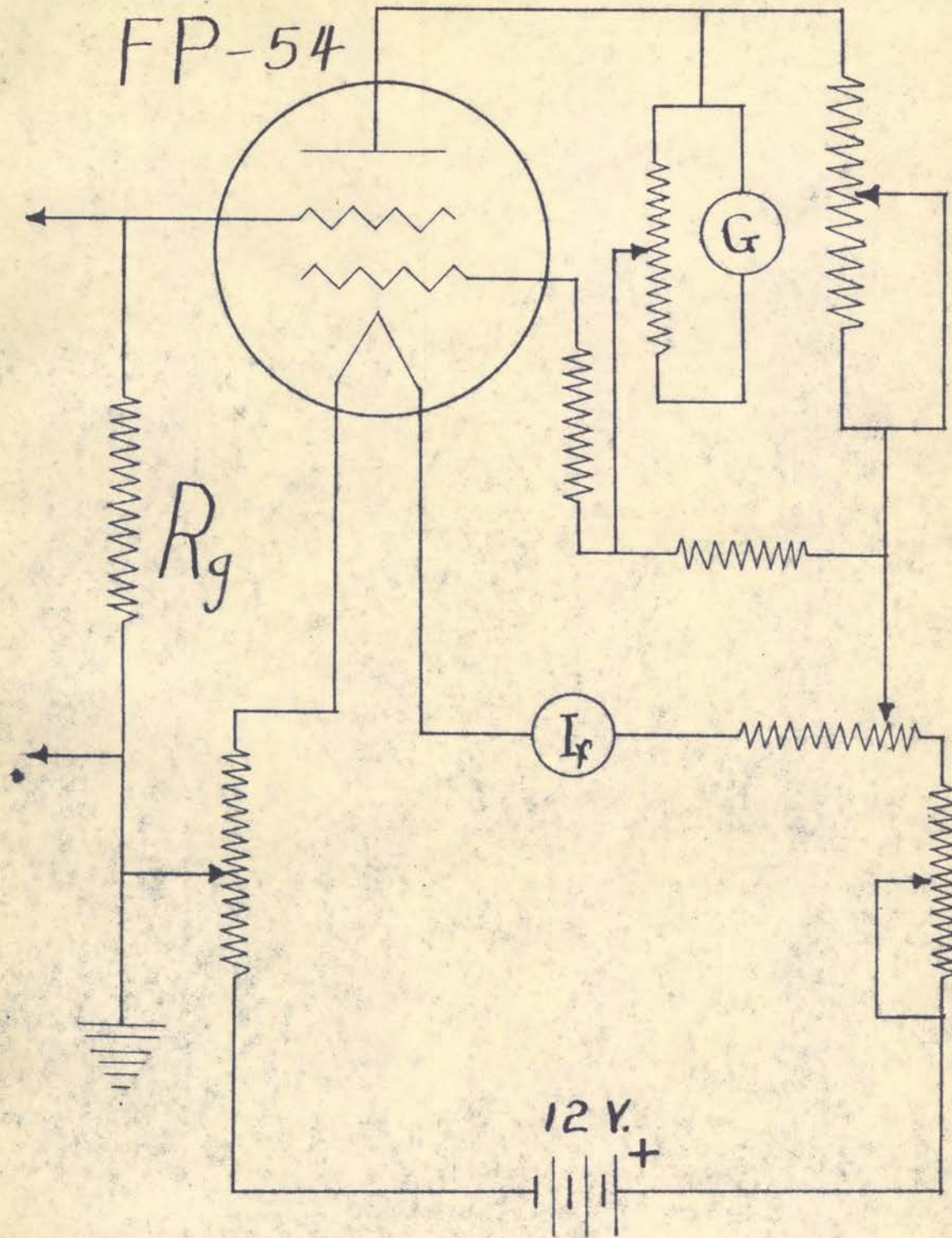


Figure 3 - CIRCUIT DIAGRAM OF FP-54 CURRENT AMPLIFIER

the total grid current. In order that the control grid current may be kept as low as possible, several unique methods are employed. The control grid is maintained at a negative potential, so that electrons will be repelled from it. All electrodes are maintained at a very low potential, so that secondary emission and other bombardment effects will be lessened. Note that in the figured circuit the maximum voltage available is twelve volts. The filament is operated at temperatures greatly reduced from those found in normal applications, so that erratic electronic emission, and ion currents, will be minimized. Since the grid resistor may have a value as high as 10^{12} ohms, the tube must be clean and dry so that leakage paths across impurities on the surface of the glass will not exist. With the FP-54 tube grid current is often as small as 10^{-17} amperes.

Fundamentally, the operation of the vacuum tube electrometer may be explained as follows: The current to be measured flows through the grid resistor, R_g in Figure 3, causing a voltage drop equal to the product IR . This potential is applied to the grid of the vacuum tube, causing a change in current flow to the plate, and varying the current through the indicating meter, G . Figure 4 is the voltage calibration curve obtained with the FP-54 instrument when connected to an indicating galvanometer with sensitivity of 2.86×10^{-7} amps per centimeter. Note the excellent linear response of the amplifier. The average voltage sensitivity for the amplifier as obtained from this graph is 8.55×10^{-3} volts per centimeter. Using this number we may obtain the current sensitivity of the circuit by simply dividing by the value of the grid resistor installed in the current amplifier, thus:

$$\text{current sensitivity} = \frac{8.55}{R_g} \times 10^{-3} \text{ amps per centimeter.}$$

Voltage Sensitivity for 5 $\frac{1}{2}$ Current Amplifier

23
21
19
17
15
13
11
9
7
5
3
1
0
1
3
5
7
9
11
13
15
17
19
21
23

Scale
Readings,
cm.

BK

RD

Grid Volts

.05 .10 .15 .20 .25 .30 .35 .40

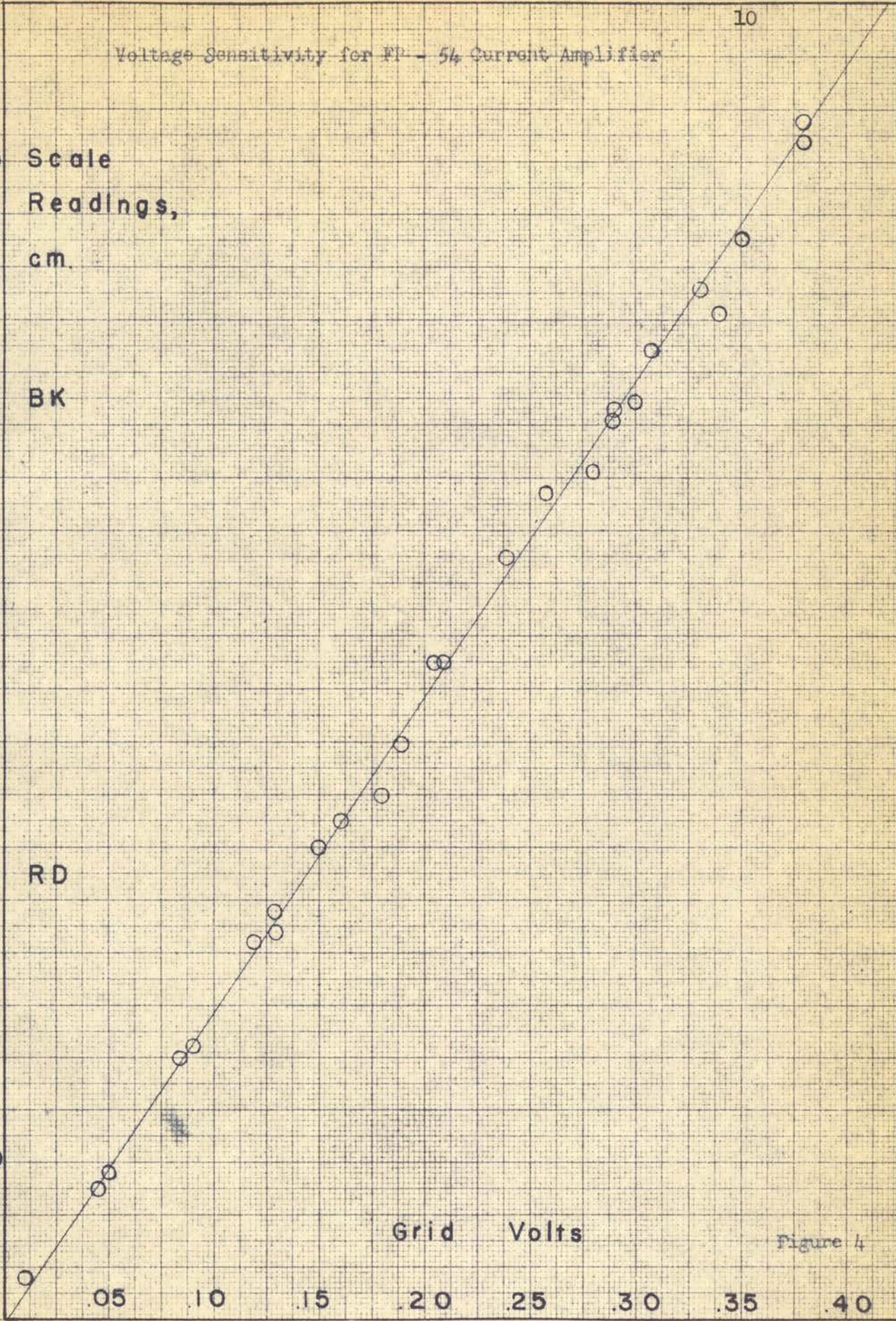


Figure 4

Since the grid resistor usually connected in the circuit had a value of 1.1×10^{11} ohms, the current sensitivity was 7.78×10^{-14} amps per centimeter. To change this sensitivity, either the grid resistor may be changed, or the sensitivity of the indicating meter may be altered.

The circuit built around the FP-54 tube is very useful and stable. After a warm-up period of only one-half hour the zero reading of the galvanometer shows very little tendency to drift. Further shifts are not over five millimeters per hour, a quite tolerable limit in itself, and after several hours of operation the zero shift is negligible over a laboratory period of three hours. Such excellent stability was not attained with any other unit. However, certain disadvantages were encountered in the use of this design. The physical dimensions of the FP-54 tube are large. As illustrated in plates 1 and 2, the size of the tube and associated controls necessitates a cabinet of quite large size. For the sake of convenience in positioning equipment and taking readings, the cabinet was placed several feet away from the connection points on the mass spectrometer. In order that readings could be taken, the wires carrying the current to be measured had to be completely shielded from external electrical fields. Without a shield, the static electricity generated as people in the room walked, or moved caused the indicating meter to fluctuate violently. However, shielding the lead-in wires posed the problem of how to protect without shunting the necessary 10^{11} ohm resistor with unwanted leakage resistances. To accomplish this the input wire was enclosed in a quartz tube, a braided shielding wire around this tube was connected to ground, and the connections at the spectrometer were enclosed in a metal can. The entire cabinet holding

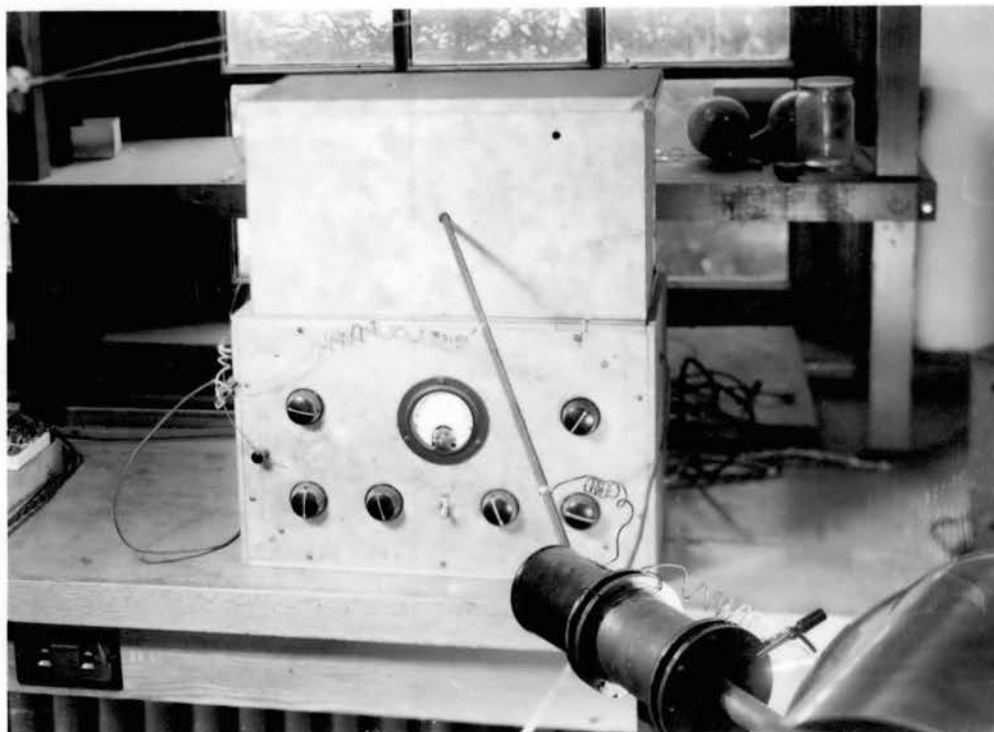
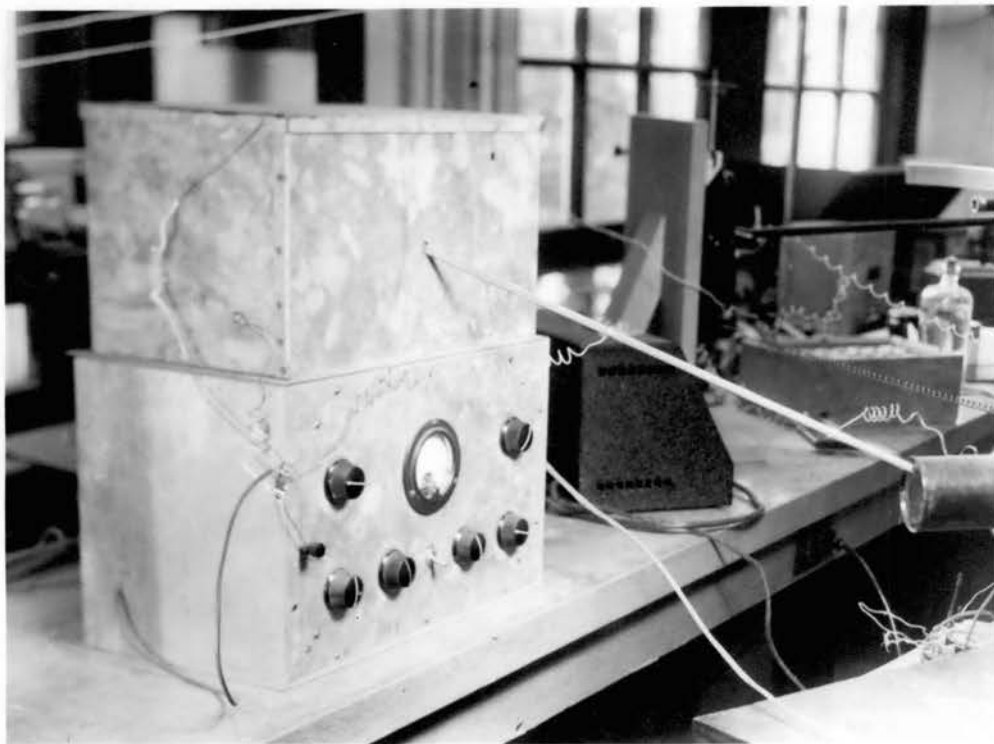


Plate 1 - The FP-54 Current Amplifier Cabinet and Connections to the Spectrometer.

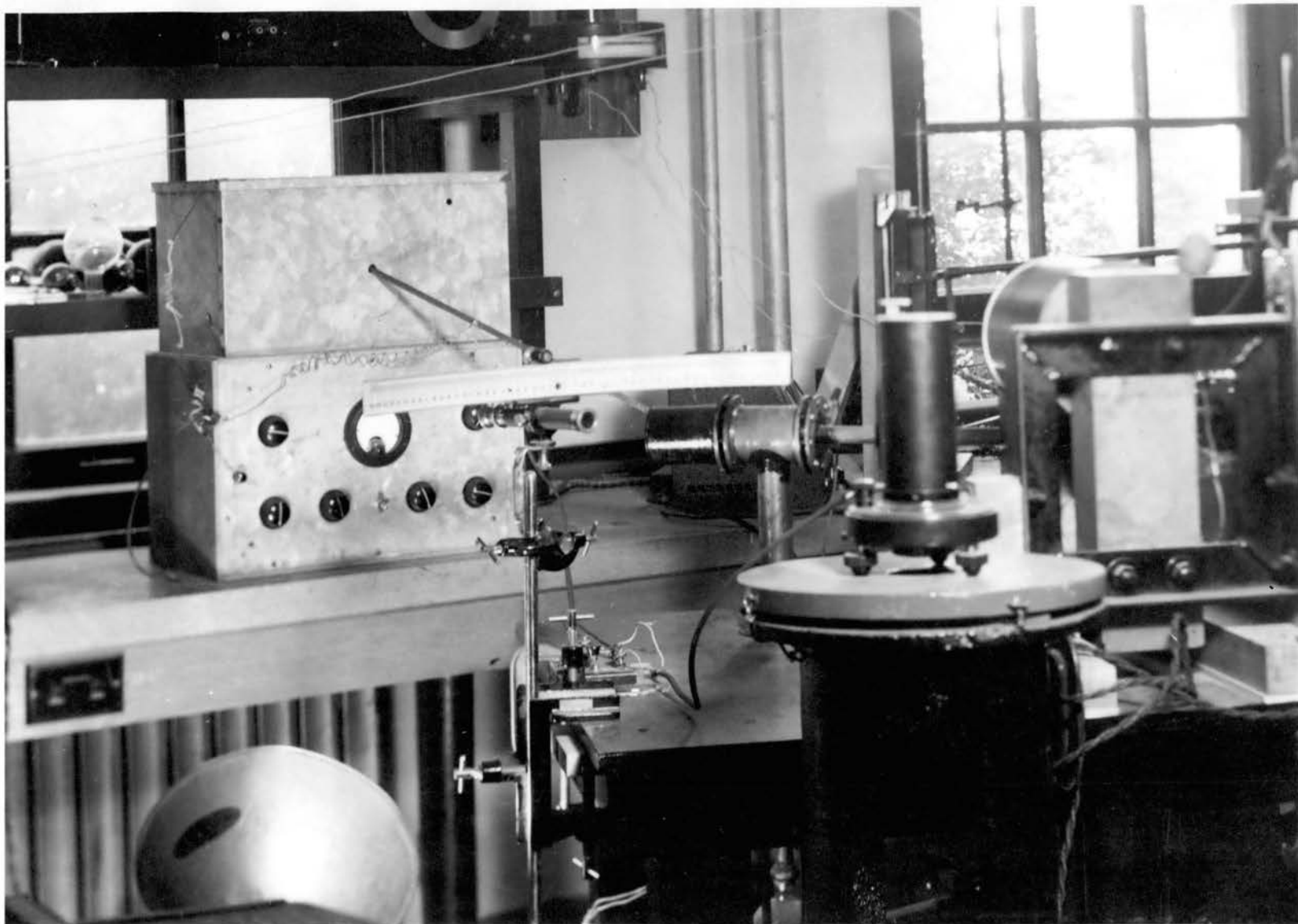


Plate 2 - General View of the Mass Spectrometer and Detector
Instruments, Using the FP-54 Current Amplifier.

the tube and controls was enclosed in a metal shield when first constructed. With this arrangement no fluctuation due to external causes was noted, and the resistance to ground of the input circuit was found to be lowered from 1.1×10^{11} ohms to 4.6×10^{10} ohms, not a detrimental loss. One difficulty was encountered which could not be eliminated. This was the added capacity to ground caused by the long shielded sections. At first thought there may be some surprise that capacity effects should be considered when dealing with a "direct current" amplifier. Capacity is usually a matter of consequence only when dealing with alternating currents. But with such a high resistance to ground as encountered here the time for a charge on the grid to leak off may become undesirable if there is too much capacity present in the circuit. While the actual capacity of the grid circuit was never measured, it was noted that a time of almost twenty-five seconds was required after removal of the applied signal for the indicator to drop from a reading of fifty centimeters (full scale) to a reading of one centimeter. Since the peaks to be measured are quite sharp, such a time lag made exact reading of the maximum current difficult. The only way to avoid this effect was to mount the tube itself closer to the output of the spectrometer. Rather than attempt to rebuild the mounting of the FP-54 tube, it was decided to use another type tube in a circuit slightly different from that associated with the FP-54.

IV. The 954 Current Amplifier

A new circuit was modified from a plan shown in Electronics by Elmore and Sands.¹ No data on the adaptability of this circuit for the proposed application was given in Elmore and Sands, but other sources^{2,3} mentioned the use of the same tube without giving any circuits. The circuit adopted used a miniature tube, the number 954, a regular commercial tube, not specifically designed for use as an electrometer tube, as was the FP-54. This small tube was mounted in a metal can directly attached to the spectrometer chamber. A ten foot cable, containing only noncritical wiring for the filament and plate, leads to the cabinet housing the various batteries, resistances and controls. Plate 3 is an illustration of this instrument. The black can contains the 954 tube and the grid resistance, R_g , while the cabinet contains all controls and batteries. The only other equipment needed is an indicating meter, G , which as a basis for comparison, is the same galvanometer used with the FP-54 unit. Figure 5 is the circuit diagram for this amplifier. Notice that it is quite different from the FP-54 circuit in that several batteries are employed, rather than the "balanced" circuit utilizing only one battery.

There are several advantages to be gained from this new arrangement. The size of the tube would allow installation even inside of the spectrometer vacuum chamber, if desired. By mounting the tube directly outside the spectrometer, the long shielded wiring in the grid circuit necessary

¹ Elmore and Sands, Electronics, p. 186

² Graham et al, Loc Cit.

³ Gabus and Pool, "Characteristics of Electrometer Tubes", Review of Scientific Instruments, 8 (1937) p. 196

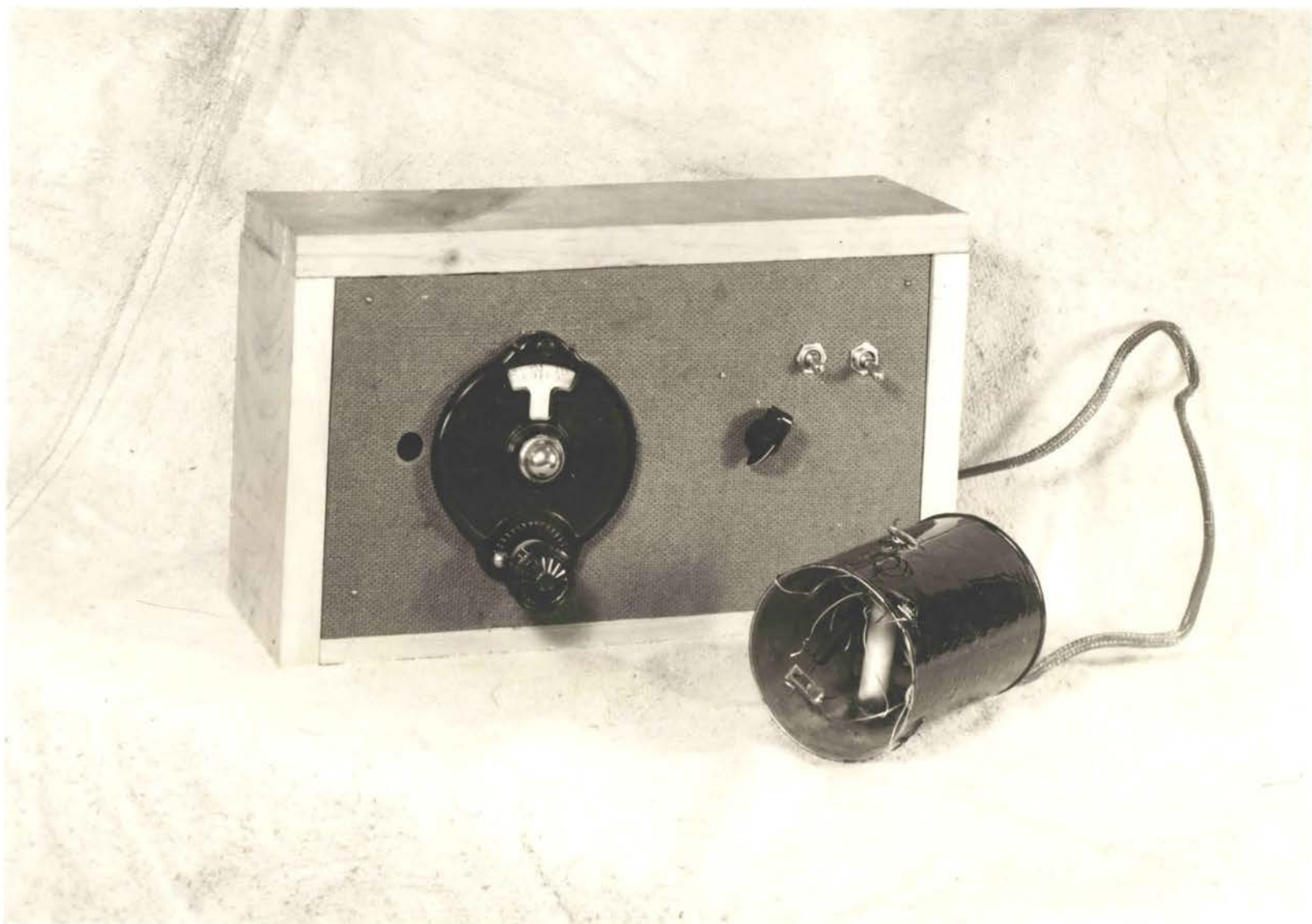


Plate 3 - The 954 Current Amplifier Cabinet and Tube Shield.

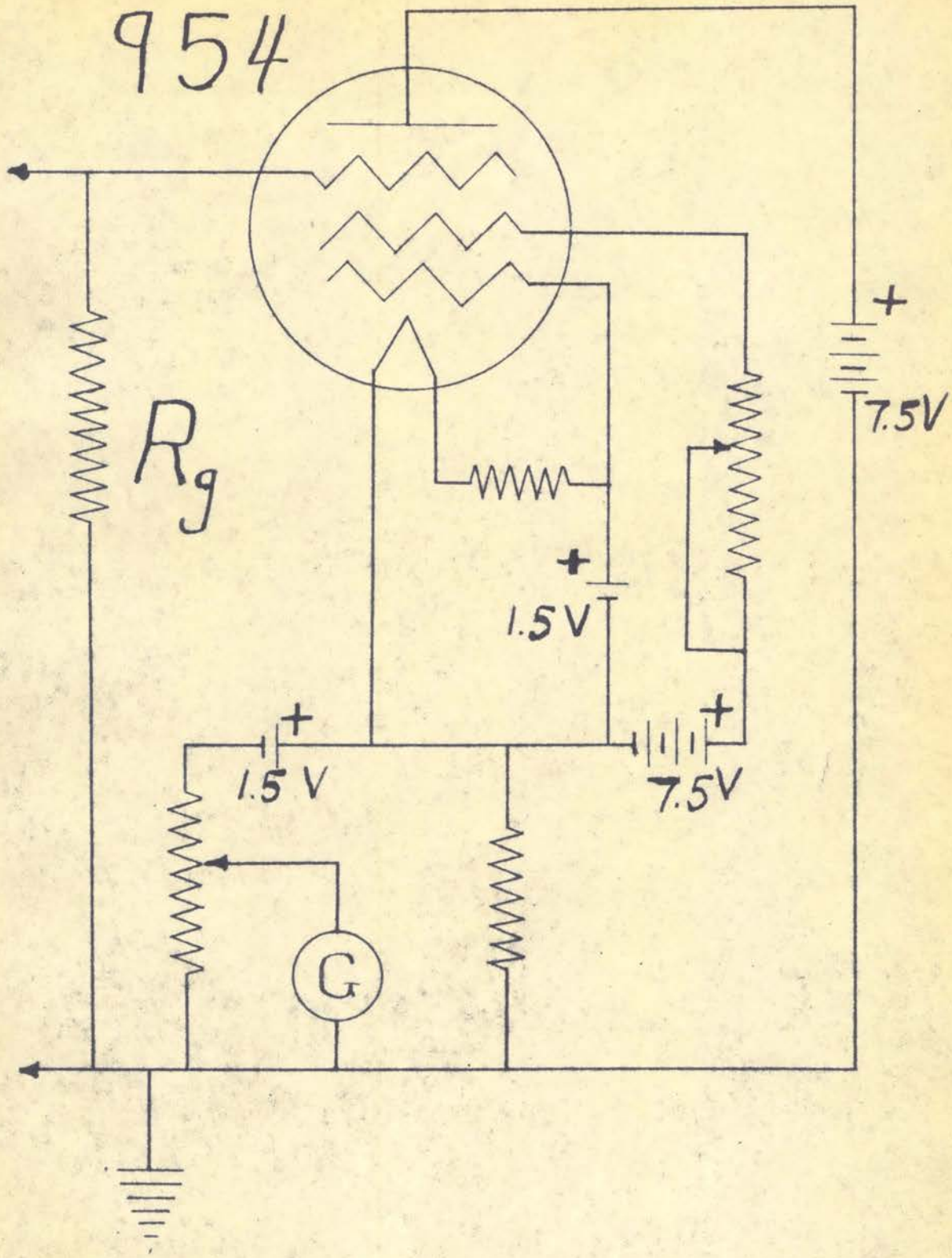


Figure 5 - CIRCUIT DIAGRAM OF 954 CURRENT AMPLIFIER

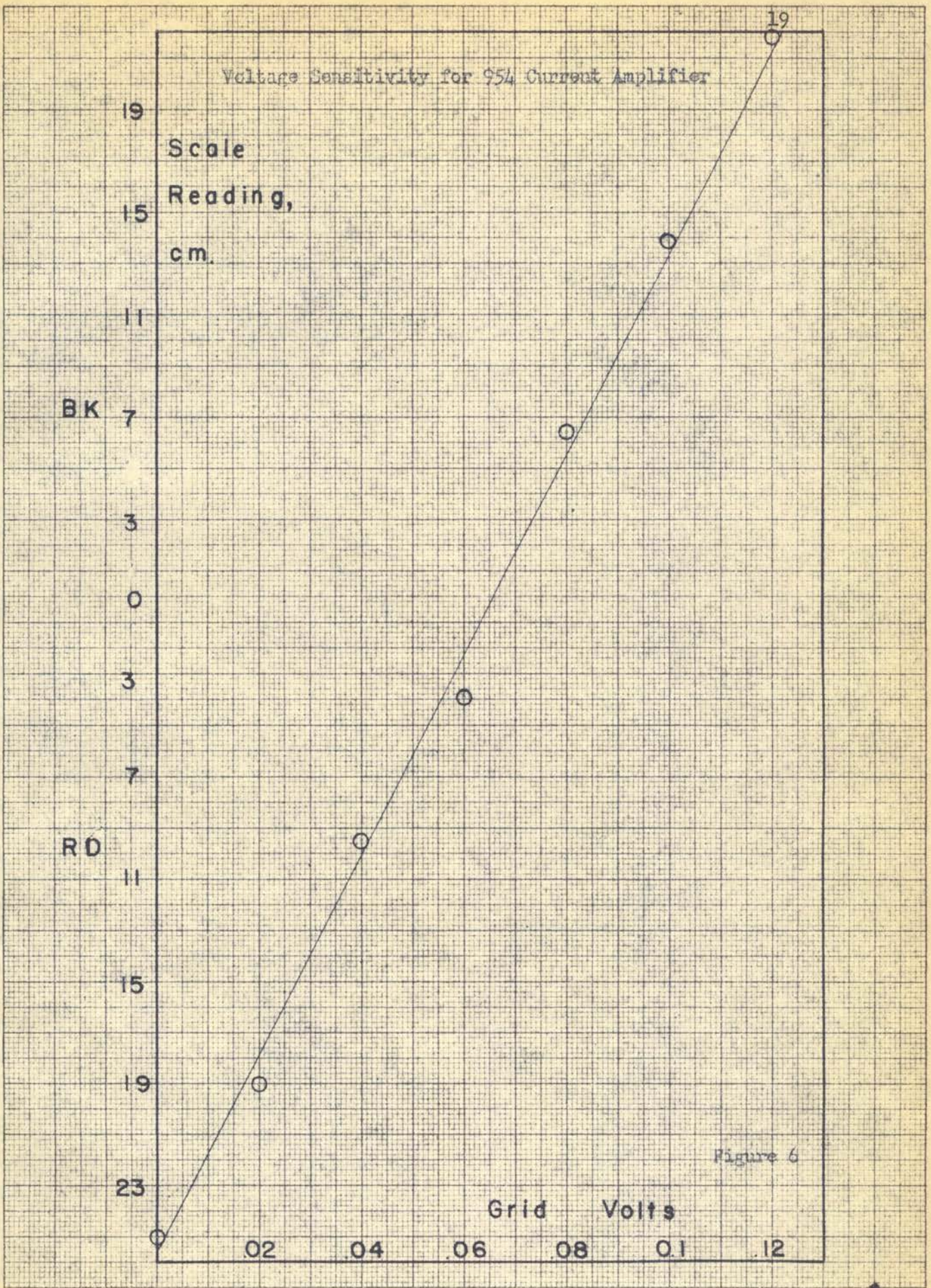
with the previous amplifier is eliminated. Leakage currents and external disturbances are reduced to a minimum. Also, this new tube is much cheaper in cost, five 954's may be bought for the price of one FP-54.

Reference to Figure 6 will show the voltage sensitivity curve for the 954 electrometer. The average value obtained from this curve is 2.57×10^{-3} volts per centimeter. For the 954 vacuum tube electrometer the sensitivity when connected to a meter with sensitivity of 2.86×10^{-7} amps per centimeter is:

$$\text{current sensitivity} = \frac{2.57}{R_g} \times 10^{-3} \text{ amps per centimeter.}$$

By comparison with the value given for the FP-54 (page 9) we see that the 954 has more than three times as much sensitivity. With a 1.1×10^{11} ohm grid resistor the sensitivity is 2.34×10^{-14} amps per centimeter. It is interesting to note that another investigator was able to measure currents of 3×10^{-15} amperes with the 954⁴, a figure very closely approached here. The tube does not possess the excellent stability exhibited by the FP-54, however. This is understandable, since the latter is undoubtedly manufactured under more careful control, and its design more specifically directed toward stability. By testing several 954 tubes under actual operating conditions, one will usually be found which is entirely satisfactory. The author worked with two tubes, one of which was not nearly so desirable as the other. During the first hours of operation of the 954 electrometer, very violent and unstable fluctuations were noted. The original diagram from which the circuit was taken suggested the use of a 0-50 micro-ammeter as the indicating movement, and initially such a meter was

⁴ Graham et al, Loc Cit.



connected, to aid in adjustment and avoid damage which might result from the use of a more sensitive meter. During the first few hours small movements were noted even on this meter. The value of these variations must have been of the order of 10^{-7} amperes, far too large a value to allow use of the instrument to measure the small currents desired. But careful mounting of all parts, use of good precision resistors and a week of ageing resulted in greatly improved stability.

There are still some fluctuations which cannot be eliminated. Figure 7a shows the shift in the zero reading of the circuit after being allowed to warm up for a period of one hour. Note that there is a rapid and fairly constant shift in one direction. After the amplifier has been left turned on for twenty-four hours this shift is eliminated and there is only a random fluctuation of one millimeter about the zero point. When readings were to be taken over a period of several days, the filament current of the tube was left on continuously, so that maximum stability would be obtained. This was possible since the current drain is very low, being only 50 milliamperes. One external effect was found which caused a change in the readings of the 954 current amplifier. This was a zero shift with changing values of magnetization of the deflection field. Figure 7b illustrates the magnitude of this change. There is a violent shift at the zero field value, due to the disturbances associated with reversing the current in the field coil; the inductance of the windings resists changes in current flow very strongly. No doubt this zero shift is due to the close proximity of the vacuum tube to the poles of the magnet and it could probably be lessened by the use of a shield designed to reduce magnetic as well as electric fields.

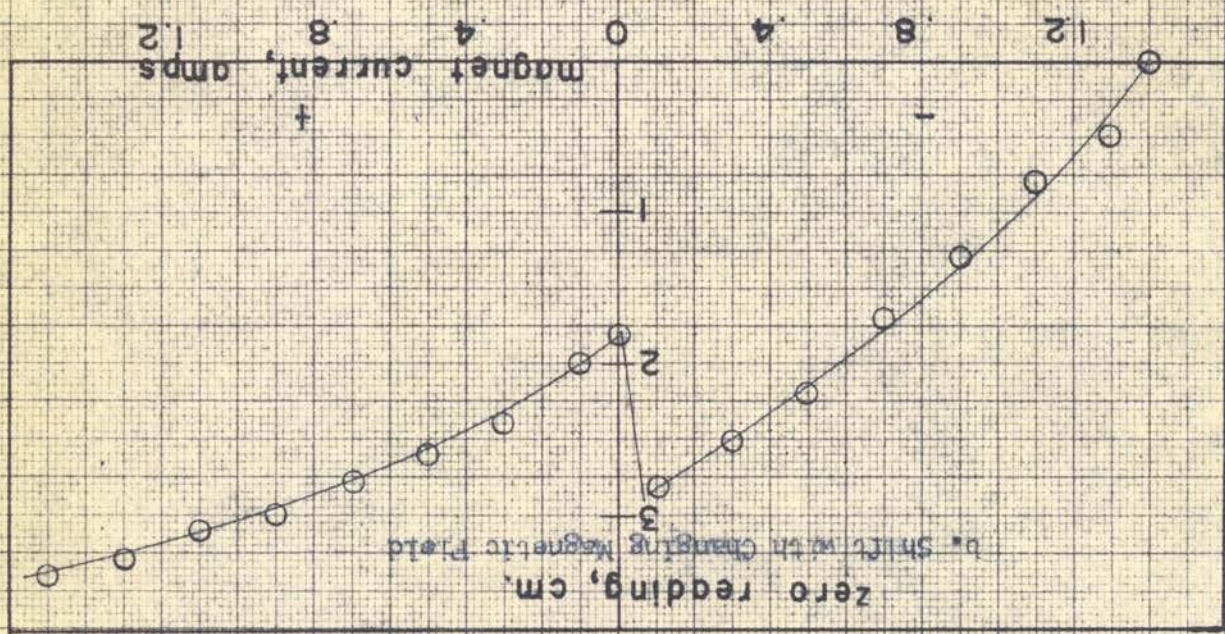
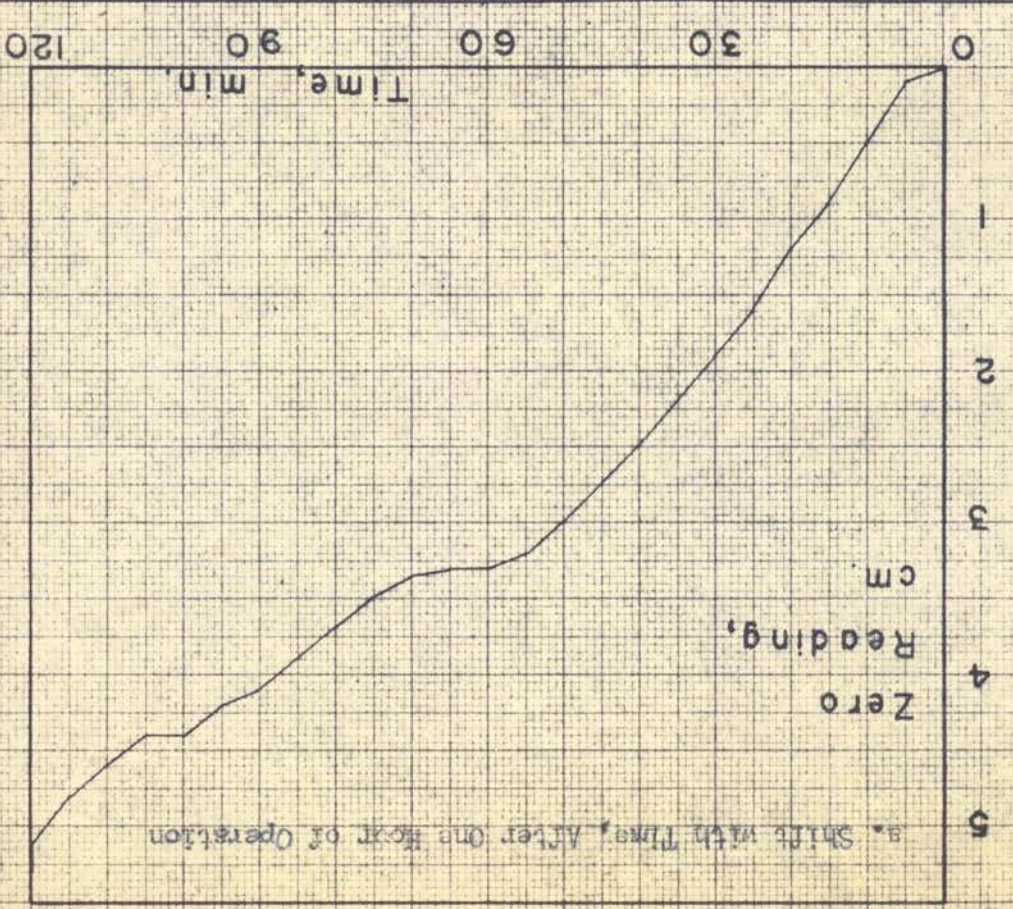


Figure 7 - Zero shift of 95% current amplifier



The operation of the 954 circuit is extremely simple, only two variable controls are included in its design. No loss of versatility has been discovered because of this minimum number of adjustments. The FP-54 amplifier has six adjustments, but not all of them seem to be vitally necessary. All of the batteries used with the 954 are small dry cells. (These batteries are: two Burgess type W5BP, 7 1/2 volt cells, a Burgess 2F2H, 3 volt cell, and a Burgess 4FH, 1 1/2 volt cell.) All are housed inside of the control cabinet. The unit makes a very compact installation due to this fact. Only the meter requires critical attention. The galvanometer employed an optical lever indicating system, so that considerable space was necessary, and the mounting of the meter could not be subject to vibration. In this particular installation the meter was mounted on a heavy cast iron pedestal which rested on rubber feet. This elaborate arrangement was necessary since the mechanical forepump in the vacuum system caused considerable vibration which could not be eliminated in any other way.

The 954 type electrometer was by no means found to be perfect. Its instability was at times annoying, perhaps a "balanced" circuit similar to that used with the FP-54⁵ would improve stability by decreasing the number of separate batteries involved. Better shielding should be studied so that the magnetic field would not cause variations. Also, one of the primary objectives, the reduction of the time constant observed in the R-C circuits, was not too successful. Apparently the grid-cathode capacity of the 954 is much higher than that of the FP-54.

⁵ Turner and Siegelin, "An Improved Balanced Circuit for Use with Electrometer Tubes", Review of Scientific Instruments, 4 (1933), p. 429.

The time constant observed with the former, while the long shields were eliminated, still appeared to be about the same magnitude as that of the early circuit using the 6P-54 tube. The only explanation for this would be increased capacity effects within the tube itself.

V. Other Problems

As an illustration of the use to which the current amplifier may be put Figure 8 is included. Figure 8 gives a small portion of a "mass spectrum" curve, with an attempt to show the present resolving power of the instrument. (Remember that the curve is taken at low accelerating potentials, which result in poorer resolution than higher voltages.) The high peak is probably due to cesium, while the broad plateau from 90 to 94 volts is possibly due to barium isotopes present as an impurity. To obtain this plot the emission current and magnetic deflection field were maintained at a constant value and the accelerating voltage was varied over one volt intervals. With the present controls it is a very difficult task to measure exactly the peak current caused by any particular ion, because they are very sharp.

Table 1 is an attempt to show the actual performance of the current amplifier compared to the more familiar operation of a current galvanometer. The readings of the vacuum tube current amplifier are compared with the emission from the filament as read on a mechanical meter. The relation obtained is not linear. However the current read by the vacuum tube device is that due to one particular ion, probably cesium, emitted from the filament, while the current read by the mechanical meter is that due to total emission. There is a distinct possibility that larger emission currents do not necessarily mean that one element has its emission proportionately larger. At higher emission other elements may be given off in higher proportion. That is, as the emission is doubled it may be that the increased current is not due to added emission of cesium, but rather by a much larger emission of some other element, say sodium or aluminum.

PORTION OF MASS SPECTRUM

magnet current = 0.3 amp
emission current = 3.5×10^{-7} amp.
filament coating: Pollucite

Detector
Current,
 $\times 10^{-13} =$ amps

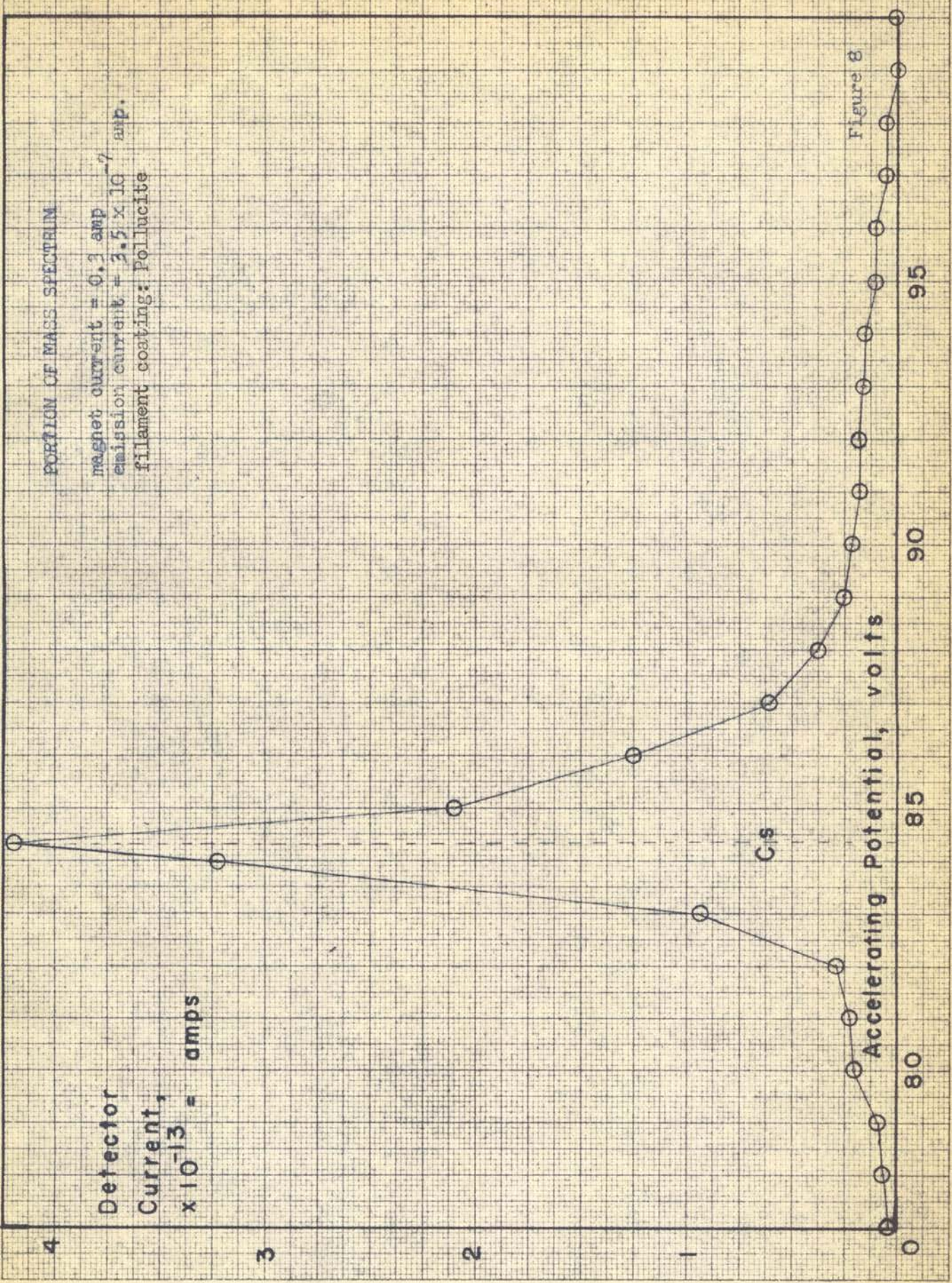


Figure 8

Table 1

Variation of detector reading with changing emission

Detector	Emission	Ratio: $\frac{\text{Emission}}{\text{Detector}}$
0.1 cm	0 cm	—
2.9	4.2	1.45
6.1	7.8	1.28
10.9	14.4	1.32
14.3	19.2	1.34
18.1	24.1	1.33
22.4	29.3	1.31
22.8	34.5	1.51
25.2	41.2	1.63
27.7	46.8	1.69

Several problems still remain to be worked out on the mass spectrometer. The resolution of the instrument could be improved. Better alignment of the unit would be a great help. By proper alignment of the magnetic field and slits the number of ions reaching the detector would be increased, reducing the problems met with very small currents. Arrangements are under way which will result in the ability to admit gases to the vacuum chamber where they will be ionized and analyzed. This will make the instrument of practical value to other organizations on the campus, while again posing problems in analysis and interpretation. Before accurate readings may be taken better control of the variable quantities associated with the spectrometer must be worked out. The magnetic field should be readily measurable as well as more easily controlled. This magnet also affords an excellent opportunity to study hysteresis effects. The effective radius of the curve followed by the ion beam should be known for qualitative studies of the device, as well as what effect the fringe of the magnetic field causes. Many other fields of investigation still remain connected with the mass spectrometer. Work with these problems will no doubt prove as stimulating and perplexing as has the author's work with the current amplifier.

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THESIS ADVISER: FRANK M. DURBIN

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