A REPORT OF A SOLID SOURCE DEMPSTER TYPE DOUBLE FOCUSING MASS SPECTROMETER

A Thesis

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Scope and Contents:

A description of a Dempster type double focusing mass spectrograph, converted to a mass spectrometer is reported. The latter part of the thesis deals with an investigation of a Shaw type solid ion source. Ten microgram samples of $BaCl_2$ and $SrCl_2$ yielded measurable ion currents of 1.0×10^{-11} amperes. Results with UF_4 and PbI_2 are also reported.

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DESCRIPTION OF A DEMPSTER TYPE DOUBLE FOCUSING MASS SPECTROMETER

1. Introduction

Early mass spectographs provided for either direction[1] or velocity[2] focusing. In order to study the heavy elements it was necessary to select a narrow bundle of rays having a small spread of energies. This selection resulted in an undesirable decrease in intensity.

In 1935, Dempster[3] described an instrument in which the particles were first deflected 90 degrees by an electric field in a cylindrical chamber, and then through 180 degrees by a magnetic field. In 1937 he[4] showed mathematically how this arrangement of fields produced both direction and velocity focusing.

In 1934, Mattauch and Herzog[5] published the general theory for mass spectrometers for all combinations of a radial electric field and a homogeneous magnetic field. Two years later Mattauch[6] showed that Dempster's double focusing mass spectograph was a special case of this general theory and that it satisfied the conditions for double focusing.

In 1945, Duckworth and Roberts built a duplicate of Dempster's double focusing mass spectograph in which the ions were recorded and measured photometrically. At McMaster, interest lay in the relative abundance of the isotopes, rather than in accurate mass determinations. Because of the difficulties inherent in obtaining these abundances[7] from the relative blackening of a photographic plate, it was decided to convert the instrument to a mass spectrometer in which the isotopic abundances are measured by positive ion currents. This necessitated the designing and rebuilding of the source, collector assembly, and the accompanying electronic units. The condenser chamber and magnetic analyser were unchanged.

Resolving Power

The resolving power of a mass spectrometer is given by the relation

$$\frac{M}{\Delta M} = \frac{r}{S_1+S_2}$$

where r is the radius of the ion path in the magnetic field

S_1 is the exit slit width and

S_2 is the entrance slit width.

For this spectrometer

[a]$r = 9.5$ cms.

$S_1 = .3$ mms.

$S_2 = .05, .1,$ or $.2$ mms.

giving a resolving power of approximately 271,238 or 190 respectively.

Relation between accelerating and deflecting voltages.

The initial energy of an ion of mass m, and charge e is given by the relation

$\frac{1}{2} mv^2 = Va.e.$ where v is the velocity of the ion and Va the accelerating potential.

In order that the ion travel through the electric field in the central path of radius a, the following condition must hold:

$\frac{mv^2}{a} = \frac{Vd}{x}.e$ where Vd is the deflecting voltage and x the distance between the deflecting plates.

Hence, $2\,Va\,e = a\,\frac{Vd}{x}.e$ or $Vd = \frac{2x}{a}.Va$

For this spectrometer a = 8.484 cms, d = .508 cms. giving Va = 8.35 Vd.

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[a] R. Shields placed the exit slit at a point corresponding to the position on the photographic plate where the lines were most clearly defined.

Thus the accelerating voltage should be 8.35 times as large as the voltage applied to the deflecting plates whereas in operation the ratio is about 7.

2. The Mass Spectrometer[b]

The mass spectrometer will be described under the following general headings; the source end, the condenser chamber, the magnetic analyser, the collector assembly, and the vacuum system. The construction of the instrument is all metal except for the necessary "Mykroy" insulators, and the glass envelope surrounding the source end. Both silver solder and soft solder were used in the construction of the instrument. This is very unsatisfactory in that the instrument cannot be "baked" in order to eliminate residual gases. A different method of assembling the tube, which would allow "baking", would increase the analysing efficiency of the instrument. All metal surfaces are cadmium plated to prevent rusting. The condenser assembly, magnetic analyser, and sylphon bellows are painted on the outside with glyptol varnish to render the spectrometer vacuum tight.

2.(a) The Source End.

The envelope surrounding the source assembly (figures 1 and 2) is a glass tube of 45 mm. I.D., flanged at one end and closed at the other by a brass and sylphon bellows assembly attached to the glass with Apiezon wax (W). A 3/16 inch steel rod passes through a Wilson vacuum seal attached to the bellows assembly and ends inside the glass envelope. A 1¼ inch length of 15 mills diameter tungsten wire, to which is secured the crucible containing

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[b] A general description of parts of the instrument unchanged in the conversion from a spectograph to a spectrometer is taken directly from a report by D. T. Roberts. For more detailed information concerning the condenser plates, pole pieces, etc. reference should be made to this report.

the sample to be analysed, is held in the end of the rod by a drilled hole and set screw. The Wilson seal enables the rod, and hence the crucible, to be moved in and out, and the bellows with three spring loaded tilting screws, allows the rod to be tilted in any direction.

Inside the glass envelope, the steel rod passes through a circular "Mykroy" spacer which is bounded at each end by a ring, grooved to fit the spacer, and clamped together by three threaded studs. Three other studs, bolted to the upper ring, support a disc which holds the shield. A tungsten wire fastened in the end of the steel rod, passes through a small hole in the centre of the disc and then supports a crucible inside the shield. The crucible is a ¼ inch length of 63 mills diameter tantalum wire, one end of which is recessed to hold the sample. The tungsten wire fits into a hole drilled in the other end of the crucible which is squeezed tightly against the wire.

Two tungsten rods, which support a tungsten filament (thickness .001 inch, width .030 inch) surrounding the crucible, are brought through holes drilled in the disc and "Mykroy" spacer, and serve as conductors for the filament current. Two copper-tungsten wire press seals connect the filament current supply to the filament through the glass envelope.

One side of the filament is grounded to the assembly such that the filament and shield are at the same potential. The high voltage is connected directly to the filament which raises the entire source above ground potential and hence the necessity of an isolation transformer. A lead from the crucible current stablilizer, clipped on the end of the steel rod, raises the potential of the crucible 500 volts above that of the filament. The crucible is heated by electron impact and positive ions originating within the crucible are accelerated to the hole in the shield by the same potential that accelerates the electrons from the filament to the crucible. After emerging from the hole in the shield, the ions are further accelerated by the applied potential between the shield and the entrance slits, which are at ground potential.

A flange soldered to the source end of the condenser chamber is threaded on the outside and grooved on the surface facing the source to take a rubber gasket. A threaded (female) knurled brass ring, engaging with the threaded flange, presses the flanged end of the glass envelope against the gasket, thus making a vacuum tight joint between the source end and the condenser chamber.

The entrance slit fits into a counterbore in the holder that supports the deflection plate assembly such that the distance from the slit to the effective boundary of the electro-static field is ⅛ inch. The slit is aligned by means of a dowel beside the counterbore in the holder. There are three slits available of widths .05, .1, and .2 mms.

2. (b) The Condenser Chamber

The condenser chamber (figure 3)[c] made of brass, which is silver soldered together, contains the curved plates used to produce a 90 degree deviation of the ions. The plates (figure 4) made of Armco magnet iron for magnetic shielding, are insulated from each other, and from the grounded brass chamber, and have leads which are brought out through the chamber

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[c] Note: Upper flange joint shown in figure 3 is not used. See figure 1.

wall by means of small kovar-glass seals. The deflecting plate assembly is an independent unit and can readily be removed from the chamber for cleaning purposes. The end of the chamber remote from the source carries one-half of a flange joint which is bolted, with a rubber gasket in between, to a similar flange soldered to the magnetic analyser.

2. (c) The Magnetic Analyser

The magnetic analyser (figure 5) fits between the poles of the electro-magnet and is built up of two Armco iron pole-pieces separated by a semi-circular brass spacer. A totally enclosed semi-cylindrical chamber is formed by a backing plate across the diameter which carries at one end a half flange joint fitting to the one on the condenser chamber. At the other end is a large hole, 2 inches in diameter, bounded by four threaded rods which support the collector assembly. The gap over which the magnetic field is effective is 1/8 inch and by automatically varying the magnet current, isotopes of different masses are focused on the exit slit.

The magnet (figure 6) is made of Armco magnetic iron and the surfaces of the poles are precision ground to a low tolerance for the pole gap which receives the magnetic analyser chamber. To enable the current to be stabilized electronically, the coils were randomly wound with a large number of turns of small diameter wire.

2. (d) The Collector Assembly

The collector assembly (figure 7) is machined from a brass cylinder $2\frac{3}{8}$ inches O.D., and turned down to 2 inches O.D. at both ends to form two shoulders. Two tungsten leads are brought out of the closed end of the cylinder by two kovar seals. Inside the cylinder one end of a small brass rod is soldered to one lead. The other end of the rod is recessed to form a deep Faraday cup to collect the positive ions. The other kovar seal supports a suppressor plate kept at a negative potential of 45 volts with respect to the Faraday cup. This negative potential repels any secondary electrons emitted from the collector cup when it is bombarded by the positive ions. Four counter-sunk screws in the wall of the chamber hold the exit slit plate over the open end of the collector assembly with the .3 mm. slit directly in front of the collector cup.

The shoulder near the open end of the collector assembly sits upon a thick rubber gasket surrounding the 2 inch I.D. hole in the backing plate. A ring, 2 inches I.D., with four attached sleeves which fit over the four threaded rods in the backing plate, presses against the upper shoulder of the assembly, thus making a vacuum-tight joint between the collector assembly and the backing plate. The four sleeves allow the collector assembly to be tightened against the backing plate until the exit slit just clears the pole pieces.

2. (e) The Vacuum System

The spectrometer is evacuated through a horizontal pumping lead soft-soldered to the curved surface of the condenser chamber on the side remote from the source end. A 1½ inch kovar-glass seal joins this lead to a large stop-cock (approximately 15 mm. bore) which changes the direction to a vertical one. Glass tubing, 25 mms. in diameter, continues down from the stop-cock into a liquid air trap. The side arm of the trap is sealed to the top of a large capacity mercury condensation pump. This pump is of a design favoured by the Clarendon Laboratory and is of the single jet inverted umbrella type. The backing side of this pump is connected by rubber tubing to a Duo-Seal fore pump. A 1949 R.C.A. ionization tube is sealed in between the stop-cock and trap while a McLeod gauge is connected between the trap and the top of the condensation pump. The lowest pressure obtained is 10^{-6} mm. Hg. which rises quickly when the filament is turned on to about 10^{-4} mm. Hg. After a few moments an operating pressure of approximately 10^{-5} mm. Hg. is obtained.

3. Electronic Units

3.(a) Magnet Current Stabilizer

Since the mass which is focused on the exit slit varies as the square of the magnetic field it is essential that the magnet current be maintained constant within narrow limits. The magnet is wound with approximately 40,000 turns of No. 26 B&S. Formvar magnet wire and requires a current of about 150 ma. for saturation of the yoke. This current is obtained from a standard 2000 volt supply which has a variac (R_1 , figure 11) in the primary circuit for adjustment of the output voltage. A complete description of the stabilizer is given by R. Shields.[8] Because the control tube is a triode there is an optimum plate voltage depending on the plate current and the recommended plate voltages for various values of magnet current are listed below.

Magnet Current (ma)	Plate Voltage (volts)
10	190
20	220
30	245
40	270
50	295
60	315
70	330
80	355
90	370
100	390

A small A.C. motor driving a 10 K "Helipot" provides automatic magnetic scanning. The rate of scanning is varied by a 100 K potentiometer in parallel with the "Helipot".

3. (b) Deflecting Voltage Supply

The deflecting voltage source, also described by R. Shields,[8] supplies one of the electrostatic deflecting plates with a positive voltage and the other with an equal negative voltage with respect to ground. Because of the critical relationship between the accelerating voltage and deflecting voltage a 100 K "Helipot" was substituted for the potentiometer R_{20}. (See figure 7, R. Shields Thesis).

3. (c) Accelerating Potential Supply

The accelerating potential is supplied from a special high voltage transformer giving a maximum output of 40 Kv. at 10 ma. D.C. The primary of the transformer is fed from two variac controls (R_5, R_6, figure 11) in parallel to provide a coarse and fine adjustment and regulated as shown in figure 8. Across the output is a 0-1 ma. meter in series with a chain of corona shielded precision resistors giving a total resistance of 20 megohms and a full-scale deflection of the meter for an output voltage of 20 Kv.

3. (d) The Crucible Current Stabilizer.

The crucible current stabilizer is fed through a variac control (R_2, figure 11) followed by an isolation transformer. The circuit (figure 9) was originally designed to supply a crucible current of about 10 ma. with a potential difference of 500 volts between the crucible and filament. To obtain a larger current, the secondary of a 2.5 volt filament transformer was connected in series with the filament. The crucible current controls

(R_3, R_4, figure 11) are mounted on a panel separate from the rest of the unit.

The temperature of the crucible depends largely on the electron power supplied and can be approximately calculated from the Stefan-Boltzmann[9] radiation equation.

3. (e) The D.C. Amplifier and Recorder[10]

The D.C. amplifier is a duplicate of the type used on the other mass spectrometers in this laboratory. The 954 chamber is mounted on the framework holding the magnet and is connected to the collector lead by a 7 inch length of polystyrene shielded wire. The manual shunt selector has five ranges (figure 10), the relation between any two consecutive ranges being approximately 3:1. The 7000 ohm resistor, in series with the precision shunt resistors, is connected directly to the cathode of the 12 J 5 vacuum tube. The signal is fed to a Brown "Electronik" Recorder which has a maximum input impedance of about 500 ohms for satisfactory operation. Two Brown Recorders are available. One, with a four second response, has a sensitivity of 5 millivolts full-scale deflection and is used when running microgram samples. The other recorder, which is used with milligram samples, has a two second response but is only half as sensitive.

4. The General Assembly

The dolly supporting the mass spectrometer consists of a framework of 1½ inches steel tubes. Rubber tired casters are attached to the four corner uprights to facilitate moving. The whole of the base of the dolly is covered with a ¼ inch steel sheet and a similar piece covers the top leaving an opening on the left for the mounting of the diffusion pump. The top sheet supports the magnet and mass spectrometer. The front of the dolly is covered by three panels on which are mounted various controls. With reference to figure 11, S_1 is a switch and indicator light for the fore pump, S_2 for the diffusion pump, S_3 for the high voltage supply, S_4 for the crucible current supply, S_5 for the magnet current supply, and S_6 for the ionization gauge.

Two uprights on the left side, extending about 4 feet above the level of the top of the dolly, support a wooden platform fitted with bakelite insulators which carry the high tension leads to the spectrometer. The top of the rear upright is bored to fit a $1\frac{1}{8}$ inch flexible aluminium conduit which carries the leads from the electrical units. The leads pass down the inside of the tubular uprights, emerge under the top of the dolly, and are then distributed to the various controls.

On top of the wooden platform is the crucible current supply and isolation transformer. The crucible current controls are mounted on a bakelite panel secured to the wooden platform. The ionization gauge circuit is mounted on the right hand side of the dolly underneath the top plate. (not shown in figure 11)

With the exception of the D.C. amplifier and Brown Recorder which are on the right of the dolly, the rest of the electrical units are housed in two cabinets on the left. An emergency switch (S_7, figure 11) is mounted between the two cabinets and so arranged that the magnet current, accelerating voltage, and crucible current can be open circuited immediately.

INVESTIGATION OF A SHAW TYPE SOLID ION SOURCE EMPLOYING A DOUBLE FOCUSING MASS SPECTROMETER

1. Introduction

In mass spectrometry there are several methods[a] of obtaining ion currents from solid samples. In one method the sample is reduced to a molecular beam by vapourization in a furnace or crucible after which ionization occurs by electron impact. Another procedure involves the thermionic emission from a filament, impregnated with the sample. The source discussed here is one originated by A. E. Shaw[11] in which a tantalum crucible, containing the solid sample and heated by electron impact, produces beams of positive ions.

It is evident that ionization in this type of source depends to a large extent on the sample being studied, that is, whether or not the sample is a good ion emitter. According to Jones,[12] the necessary condition for positive thermionic emission is that the electron work function of the crucible must exceed the ionization potential of the sample. Blewett and Jones[13] suggest that the sample should have an ionic lattice structure and be thermally stable so that sufficient energy can be supplied to the lattice to effect a partial breakdown of the structure and permit singly-charged ions to escape with the aid of a drawing out potential. The alkali metal salts, the rare earths, and the alkali-alumino-silicates are satisfactory thermionic ion emitters.

2. Operation

To introduce a sample the knurled ring holding the flanged end of the glass envelope against the condenser chamber is unscrewed and the glass envelope containing the source assembly is removed. The filament

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[a] A Survey of the various types of sources has been compiled by Mr. R. H. Mills, M.Sc. Thesis, McMaster University, 1949.

leads are disconnected from the press seals and the source assembly is removed from the glass envelope by sliding the steel rod forward. The wire, holding the crucible filled with the solid sample, is fastened in the end of the steel rod and lined up such that the open end of the crucible lies directly in line with the centre of the hole in the shield. The source assembly is then put back in the glass envelope, the flanged end of which is coated with cello-seal grease before connecting to the condenser chamber again. By adjusting the sylphon bellows the centre of the hole in the shield is lined up with the entrance slits.

The crucible is heated slowly at the beginning to prevent the sample from spattering. Because the ions have approximately the same initial energy, the ratio of the accelerating potential to the deflecting plates voltage is very critical. (See page 3) In adjusting to obtain this relation the instrument is lined up on the potassium ion currents which are always large, even at very low temperatures. Potassium is present as an impurity in the tantalum metal. Sitting on top of the $K_{39}{}^+$ peak the accelerating potential and source are adjusted for maximum ion current. Then while scanning over the mass range under study the temperature of the crucible is increased until the desired peaks appear. The accelerating potential and source assembly are then readjusted for maximum ion current before beginning a run.

3. General Discussion

Results obtained indicate that ions are formed by electron impact as well as by thermal ionization. With a sample of $BaCl_2$, the following ions are obtained, Ba^+, Ba^{++}, $BaCl^+$, $BaCl^{++}$, $BaCl_2^+$. As no one has reported finding doubly-charged barium ions formed by thermionic emission, the presence of these ions suggests that there is some ionization by electron impact. With a sample of PbI_2, which is very volatile and therefore not likely to be thermally ionized, Pb^{+} and Pb^{++} ions are found. Both have maximum ion currents when the crucible is as far away from the entrance slits as possible. The same is true for the $Hg.^{+}$ and $Hg.^{++}$ peaks which are always present. On the other hand the Ba^{+} peaks are a maximum when the crucible is about 1.2 cms. from the entrance slits. Similarly there is an optimum position at about the same point for K^{+} ions which are emitted directly from the hot crucible.

Referring to figure 12 it can qualitatively be seen that there is an optimum position of the crucible for the maximum number of ions to enter the slits if all the ions originate inside the hole in the crucible, that is, they are the result of thermionic emission (case b). When the crucible is moved further back than this optimum position as in case c, molecules ionized by the electron beam in the region between the filament and the crucible lie within the permissible cone and can therefore get through the slits. As the crucible is moved back further, more ions created by electron impact will be detected until a limiting position is reached, determined by the size of the hole in the shield.

This explanation, although it agrees with the experimental results, may not be entirely correct as no account has been taken of the field distribution between the crucible and the shield, and between the shield and entrance slits. Both of these fields would influence the actual path of the ions. The possibility of scattered electrons passing in front of the crucible should also be considered.

If the filament is placed between the open end of the crucible and the shield as shown by the dotted lines in figure 12 B, instead of around the centre of the crucible, larger ion currents are obtained. In this case, the electrons which are attracted to the open end of the crucible ionize any molecules which are vapourized from the sample. The ions produced in this way, added to those formed by thermal emission, increase the detected ion current. As an example the potassium ion currents were increased about threefold.

However, when the filament is in this position small peaks due to the residual gases in the spectrometer appear at practically every mass unit up to mass 140. This is also the case, but to a much lesser extent, when the crucible is back beyond the optimum position as in case c. By keeping the distance between the end of the shield and the crucible as small as possible, about 1 mm., most of the background peaks are eliminated.

The actual position of the crucible and filament then will depend largely on the sample. If the isotopic mass of the sample is under 140, then the filament should be about the centre of the crucible and the crucible as close to the shield as possible. The shield should be set about 1.1 cms. from the entrance slits and then varied while operating the instrument to obtain the optimum position. On the other hand, for volatile substances such as PbI_2 and UF_4 which have isotopic masses above 200, the filament should be placed between the shield and the crucible and the crucible set as far back from the slits as possible. As there are no residual peaks in this mass range, full advantage can be taken of ionization by electron bombardment.

4. Sources of Error

In considering the corrections which must be applied to the observed abundances, possible preferential emission of the lighter mass ions due to their higher thermal velocities should be considered.[14] If any such mass discrimination exists the last determination in a run would have larger ratios of heavier masses than did the first determinations. No such effect was noted with any of the samples.

The possible dependence of ion emission on the mass of the isotope should be considered. However, as mentioned before, since in this type of source the efficiency of ionization depends on the ionization potential of the sample and the work function of the crucible and not on the mass, it is concluded that this effect is negligible.

The condition for no discrimination between light and heavy ions in their passage through a mass spectrometer is that the paths for different ions be the same.[15,16] This condition is satisfied by bringing different masses to focus at a fixed point, the exit slit, by varying the magnetic field and keeping the accelerating potential constant.

Inaccuracy could be caused by non-linearity of the 10^{11} ohm grid leak resistor. However, the grid leak resistor is of the same type as the ones used on the other mass spectrometers in this research laboratory and they have been checked and found to be constant within one percent. Therefore, measurements are assumed to be in error, due to the grid leak resistor by no more than this amount.

Since ion currents are measured over different ranges, abundance measurements are dependent on the shunt factors. The shunt factors were checked twice in three months and found to be constant within a third of one percent.

In view of the above discussion of errors it may be concluded that the ion currents are proportional to the isotopic abundances to an accuracy of less than one and one-half percent.

The greatest difficulty with a solid source mass spectrometer is encountered not in the instrumentation but in the small impurities which may be present either in the instrument itself or in the sample. These impurities may produce small ion currents in the range being observed and therefore incorrect abundance measurements are obtained. Impurities in the instrument itself can be detected by running an empty crucible. Measurements are then made in a region free from residual ion currents but if this is not possible the abundances must be corrected accordingly. Impurities detected in the $BaCl_2$, and $SrCl_2$ samples emphasize the need for better methods of obtaining chemically pure substances. This becomes increasingly difficult in the preparation of microgram samples of radioactive material.

5. Results

Lead Iodide

An analysis of PbI_2[b] revealed the presence of Pb^+ and Pb^{++} ions under the following operating conditions:

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[b] This sample was prepared by Mr. J. MacNamara from a sample of pitchblende ore which originated from Great Bear Lake.

Accelerating Voltage	3040 volts
Deflecting Voltage	448 volts
Magnet Current	52 ma.
Entrance Slit	.1 mm.

A tungsten crucible was employed with a small cap which fitted over the open end of the crucible. A very fine hole in the centre of the cap allowed a molecular beam to escape from the crucible. The filament was placed between the shield and the filament to take full advantage of ionization by electron impact. A sample set of the Pb^+ peaks is shown in figure 13. The average results of ten sets of peaks is tabled below and compared to results obtained by Aston.

Table 1

	Percentage Relative Abundance of Mass		
Experimental Results	206	207	208
This Work	90.6±.9	7.6±.1	1.8±.1
Aston, F. W.[17]	89.8	7.9	2.3

Uranium Tetrafluoride

Several samples of UF_4 were tried with the filament around the centre of a tantalum crucible, but the ion currents decayed too quickly to obtain any measurement of the U^{238}/U^{235} ratio. With the filament between the crucible and the shield steady ion currents were obtained corresponding to U^+, U^{++}, UF^+, UF^{++}, UF_2^+, UF_2^{++}, UF_3^+, UF_3^{++}, UF_4^+, UF_4^{++}. The results tabled below were obtained from the U ion currents. The U^{235} peak is not completely resolved as shown in figure 13, but by drawing in the base with the aid of a french curve good precision was obtained.

Table 2

Experimental Results	Ratio U^{238}/U^{235}
This Work	141.2 ± 1%
Nier, A. O.[18]	139.0 ± 1%

Rubidium

Rubidium was detected in two radioactive samples of strontium carbonate. They appeared at a comparatively low temperature and disappeared quickly when the temperature was raised. Because the ratio of the rubidium isotopes is in close agreement with that obtained with natural rubidium, and that if radioactive rubidium were present it could be there only in very small quantities, it is concluded that this is natural rubidium, probably introduced as an impurity in the chemical reagents used in the preparation of the sample. The first result tabled below is the average of 9 sets of peaks while the second is the average of 5 sets.

Table 3

Experimental Results	Ratio Rb^{85}/Rb^{87}
This Work, 1st result	2.56±.01
This Work, 2nd result	2.60±.06
This Work, average	2.58
Brewer, A. K.[19]	2.59±.04

Barium Chloride

Measurable ion currents of 1.0×10^{-11} amperes have been obtained with 10 microgram samples of $BaCl_2$. As barium has seven known isotopes, it takes several minutes to scan one complete set of peaks. The sample decays quite rapidly at first, with the result that only 3 or 4 complete sets of peaks can be obtained. The sample does not entirely disappear for ion currents can still be detected after several hours; however, they are so small that it is impossible to detect Ba^{130} and Ba^{132}. Figure 4 is a sample of the barium peaks obtained. The results tabled below are the averages of three complete sets.

The operating conditions for both $BaCl_2$ and $SrCl_2$ (reported below) are:

Accelerating Potential	6780 volts
Deflecting Voltage	900 volts
Magnet Current for Ba	60-70 ma.
Magnet Current for Sr.	44-46 ma.
Entrance Slits	.2 mm.

Table 4

	Percentage Relative Abundance of Mass						
Experimental Results	130	132	134	135	136	137	138
This Work	.106 ± .002	.099 ±.001	2.33 ±.03	6.47 ±.04	7.70 ± .03	11.03 ± .03	72.13 ± .16
Nier, A. O.[20]	.101	.097	2.42	6.59	7.81	11.32	71.66

Strontium Chloride

Five and ten microgram samples of $SrCl_2$ mixed with various amounts of $BaCl_2$, up to one milligram, yielded ion currents of the order of 1.0×10^{-11} amperes. At a crucible current of approximately 4 ma. the first peaks which were detected in the strontium range were due to $BaCl^{++}$ ions. As the current was increased to 10 ma. these peaks disappeared[c] and singly-charged strontium ions were detected. The strontium ion currents lasted long enough with each sample to obtain at least 6 complete sets of peaks. SrCl and Sr peaks obtained with a ten microgram sample of $SrCl_2$ are shown in figure 15. Table 5 contains the average results of 5 different microgram samples of $SrCl_2$.

Table 5

	Ratio of	
Experimental Results	Sr^{88}/Sr^{87}	Sr^{88}/Sr^{86}
This Work	11.75 ±.09	8.54 ±.05
White and Cameron[21]	11.89	8.49

Thorium and Uranium Oxide

Several samples of thorium and uranium oxide were introduced into the mass spectrometer but in each case only small lead peaks were detected. In the thorium sample the Pb^{208} peak was the largest and with the uranium oxide the Pb^{206} peak was the largest. This is as expected, because the lead detected is the end product in the natural decay of the thorium and uranium samples.

Conclusion

The results obtained with the microgram samples of $SrCl_2$ suggest the possibility of measuring the relative abundances of radioactive samples

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[c] Probably at a low temperature the thermal energy supplied is not sufficient to dissociate the $BaCl_2$ molecule completely and $BaCl^+$ ions are emitted which become doubly-charged by electron bombardment. At the higher temperature the thermal energy supplied may be sufficient to separate the $BaCl_2$ molecule into Ba^{++} and $2Cl^-$.

such as strontium and caesium obtained from fission products. Work along this line is at present being carried out.

Several changes with regard to the mass spectrometer are contemplated. With the present crucible current supply the maximum temperature to which the sample can be heated is not hot enough to obtain thermionic emission from the oxides of the heavy elements. A circuit capable of delivering up to 100 ma. with different crucible potentials up to 500 volts would supply sufficient energy to the crucible and at the same time permit experiments for the investigation of the effect of various accelerating potentials for the electron beam. As mentioned previously the instrument is operated at a pressure of 10^{-5} mm. Hg. An O3B oil diffusion pump has been obtained and will be installed as soon as possible in order to reduce the operating pressure to 10^{-6} mm. Hg. or better.

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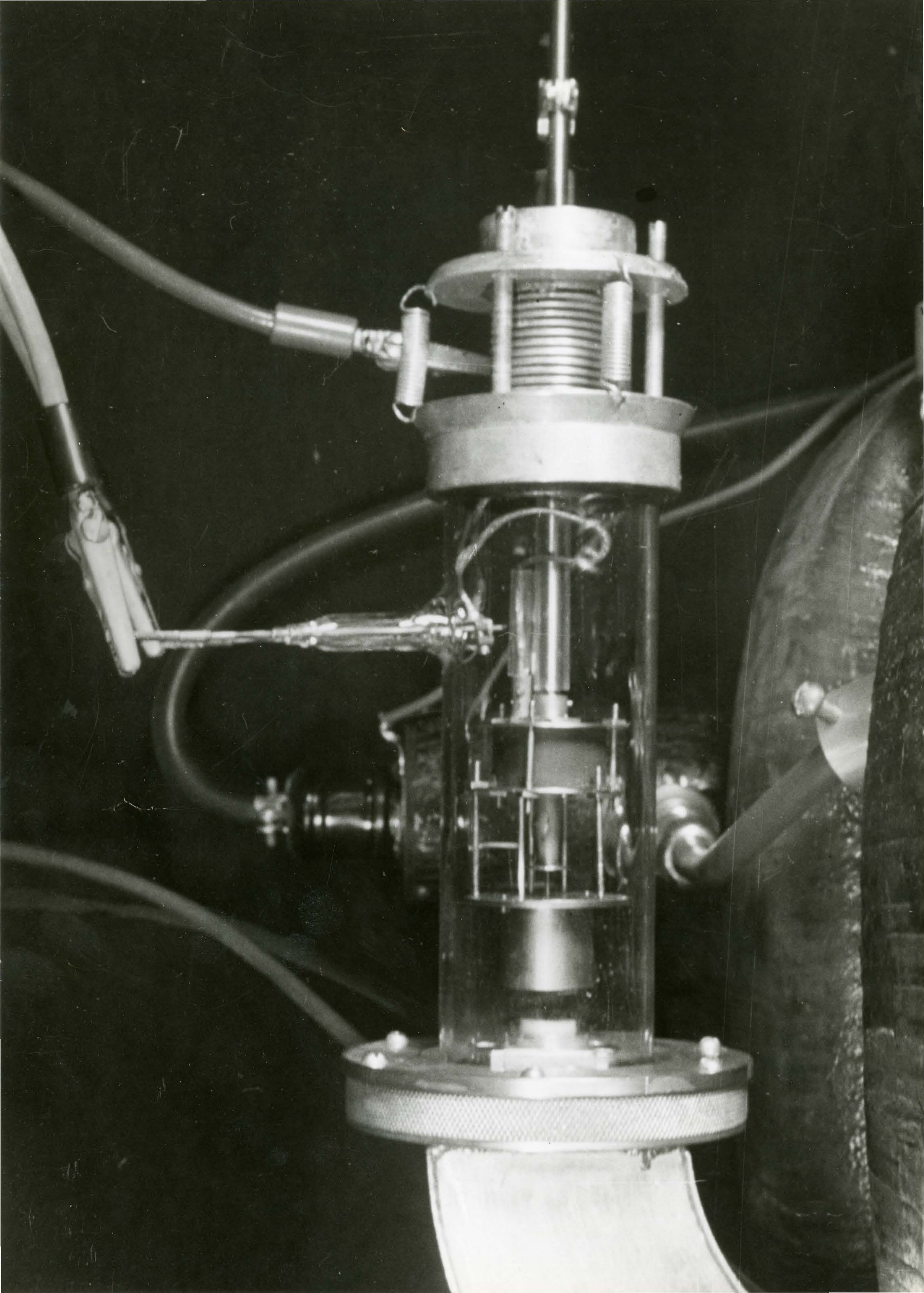
List of Illustrations



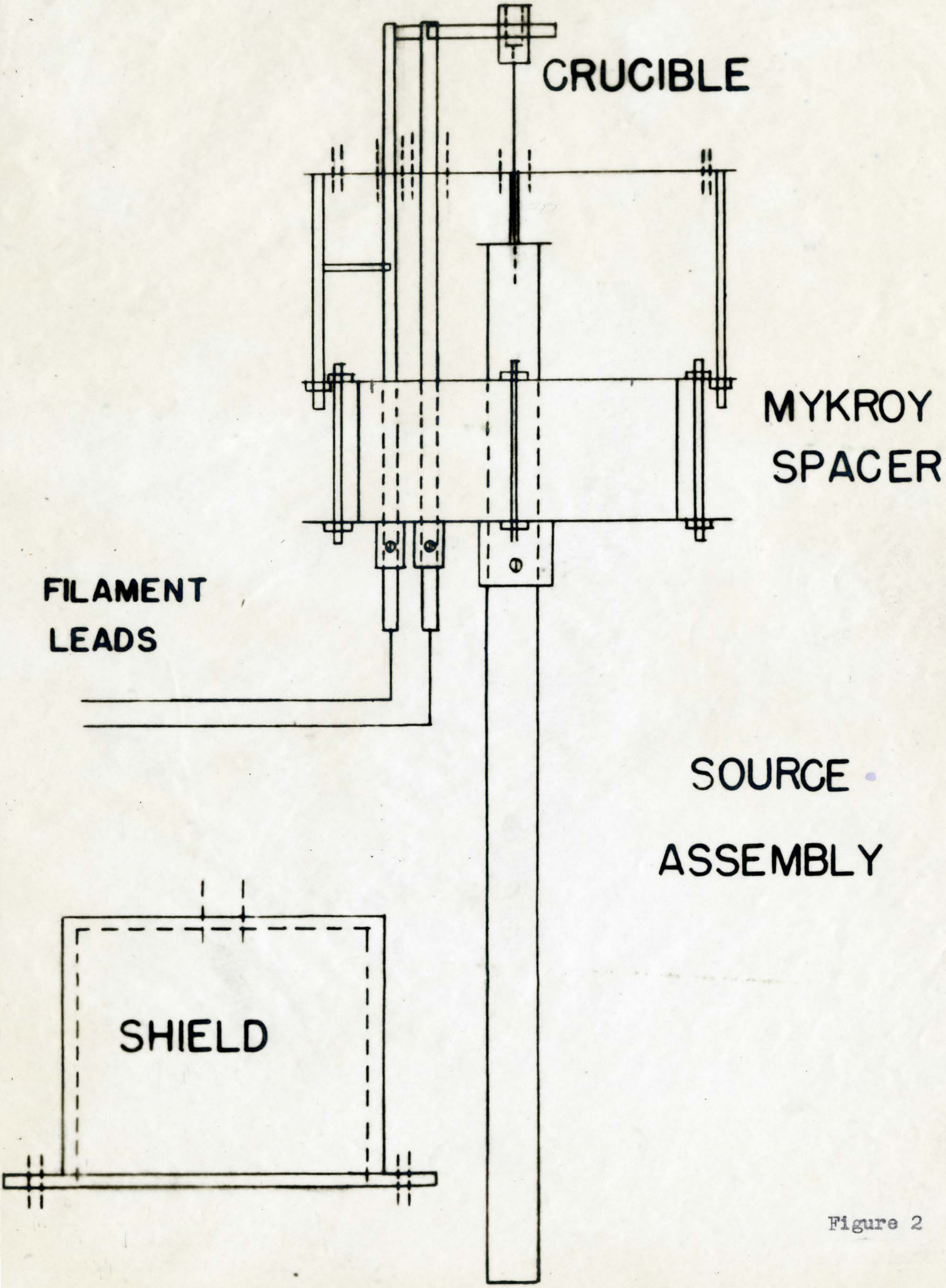

CRUCIBLE
MYKROY SPACER
FILAMENT LEADS
SOURCE ASSEMBLY
SHIELD

Figure 2

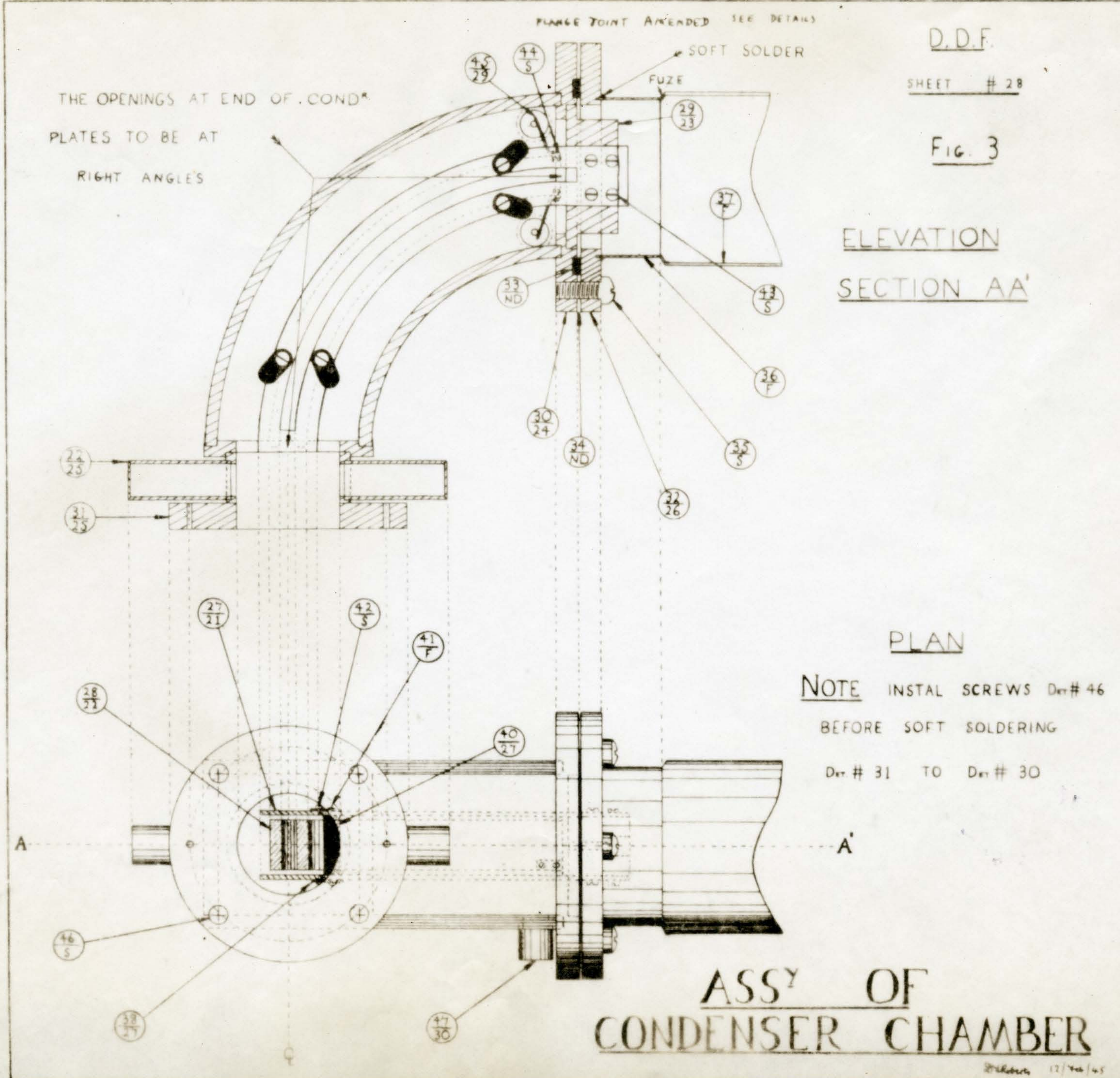
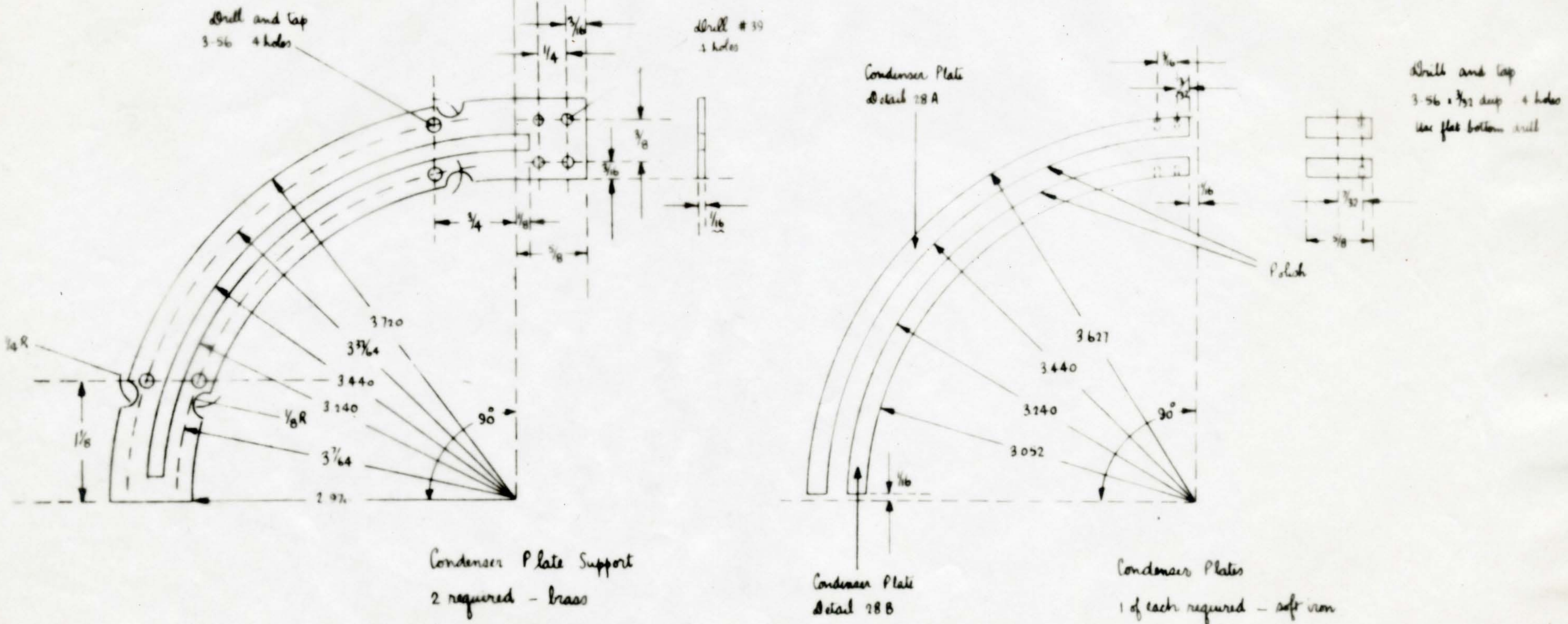

FLANGE JOINT AMENDED SEE DETAILS
SOFT SOLDER
FUZE
THE OPENINGS AT END OF COND^R PLATES TO BE AT RIGHT ANGLES
D.D.F.
SHEET # 28
FIG. 3
ELEVATION
SECTION AA'
45/29
44/S
29/23
27/F
43/S
36/F
35/S
32/26
34/ND
30/24
33/ND
22/25
31/25
PLAN
NOTE INSTAL SCREWS DET# 46 BEFORE SOFT SOLDERING DET# 31 TO DET# 30
27/21
42/S
41/F
28/21
40/27
46/S
38/27
47/30
A
A'
ASS^Y OF CONDENSER CHAMBER
12/Feb/45

Figure 3



Condenser Plate Support
2 required - brass
Drawing DDF - 27
Sheet 21

H.E. Duckworth
31/1/45

Condenser Plates
1 of each required - soft iron
Drawing DDF - 28
Sheet 22

H.E. Duckworth
31/1/45

FIG. 4

Figure 4

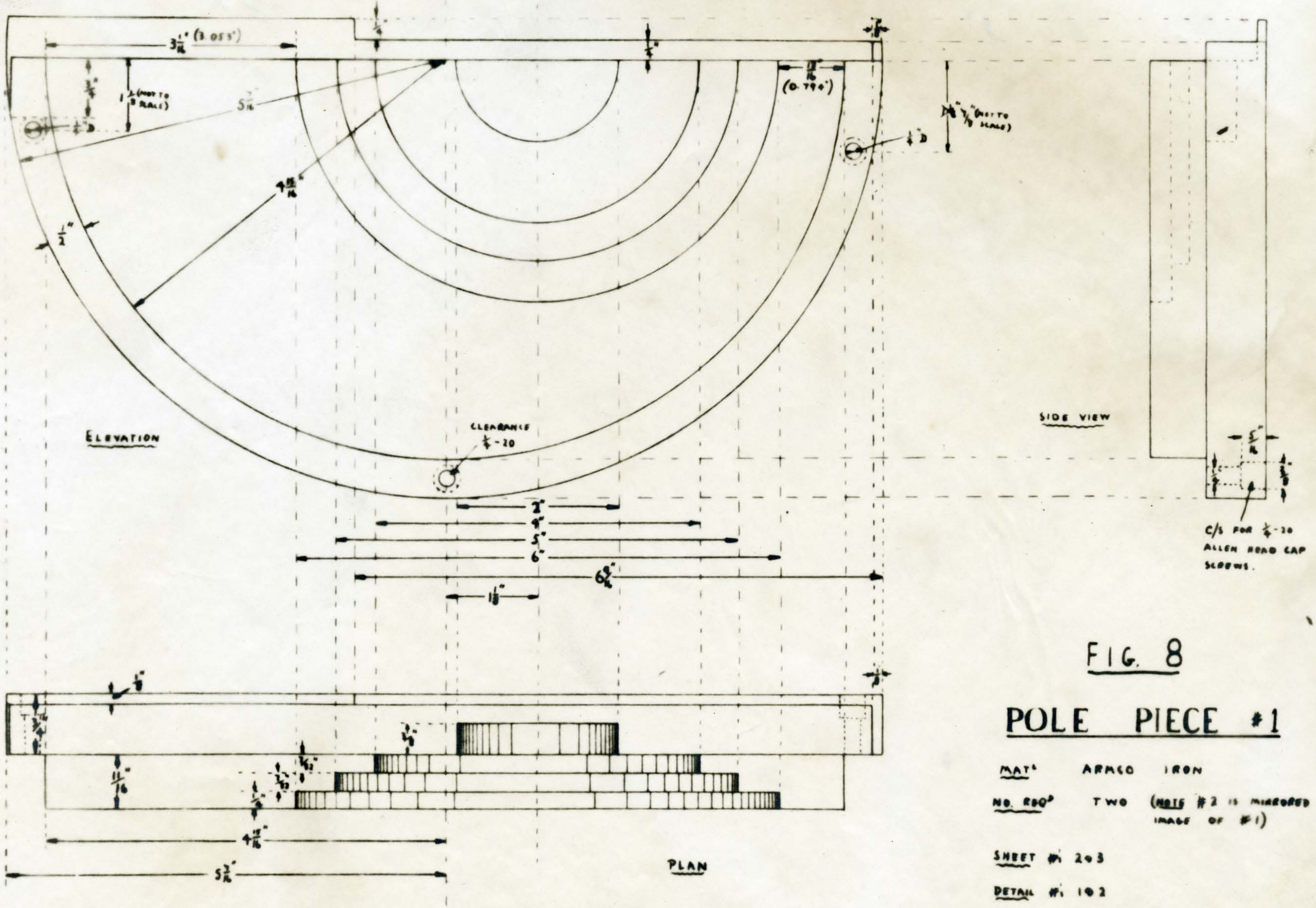


FIG. 8

POLE PIECE #1

MAT'L ARMCO IRON

NO. REQ'D TWO (NOTE #2 IS MIRRORED IMAGE OF #1)

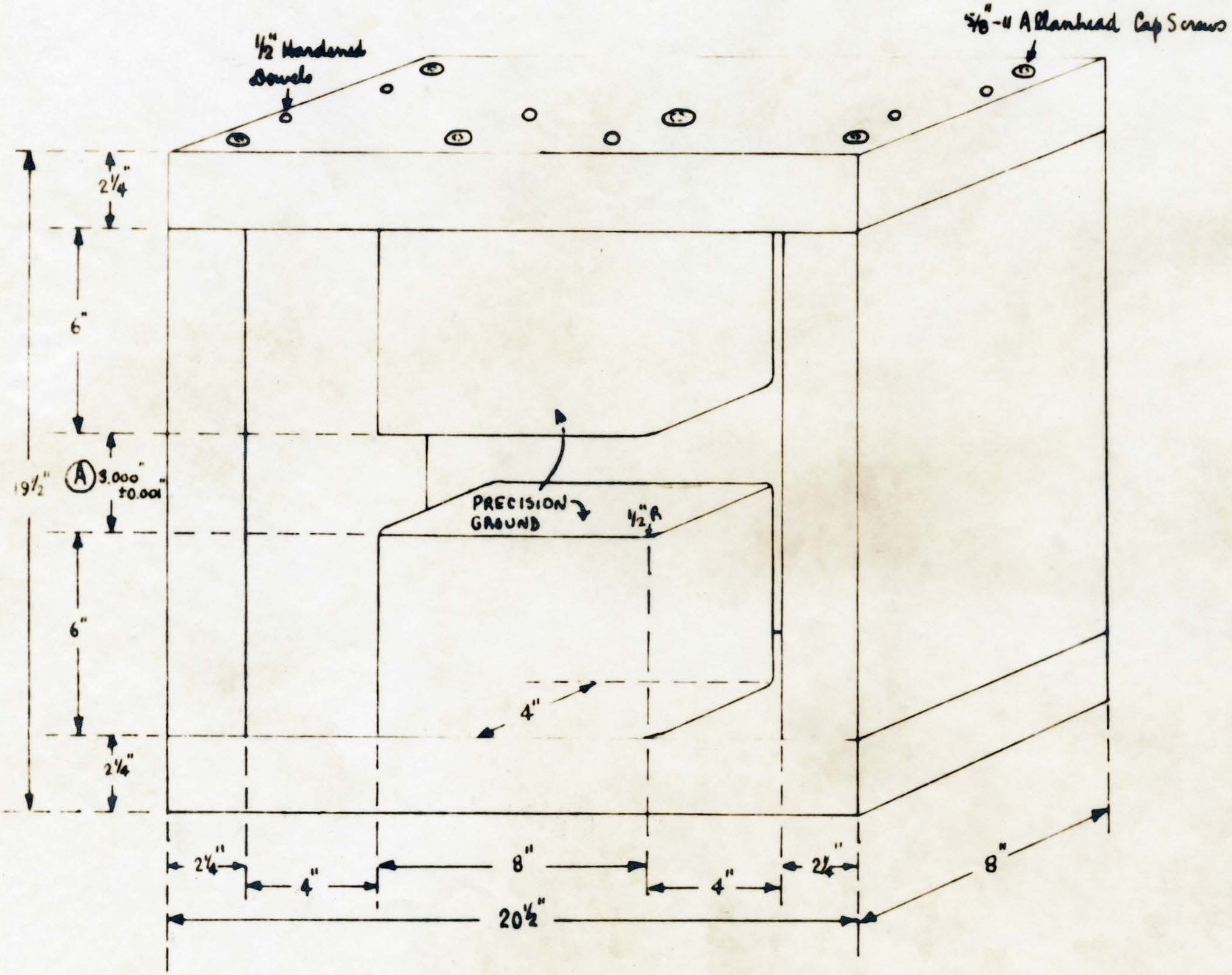
SHEET #: 2 of 3

DETAIL #: 1 of 2

NOTE FIGS IN BRACKETS ARE DIMENSIONS MEASURED AFTER MACHINING (6" CUTTER WAS OVERSIZE)

14 Aug 1945

Figure 5



Supplied:

Armco Magnetic Ingot Iron Billets as follows

2 - 2¼" x 8" x 21" Top + Bottom

2 - 2¼" x 8" x 16" Sides

2 - 4" x 8" x 6½" Poles

FIG. 11

Magnet Assembly

Material - Armco Iron

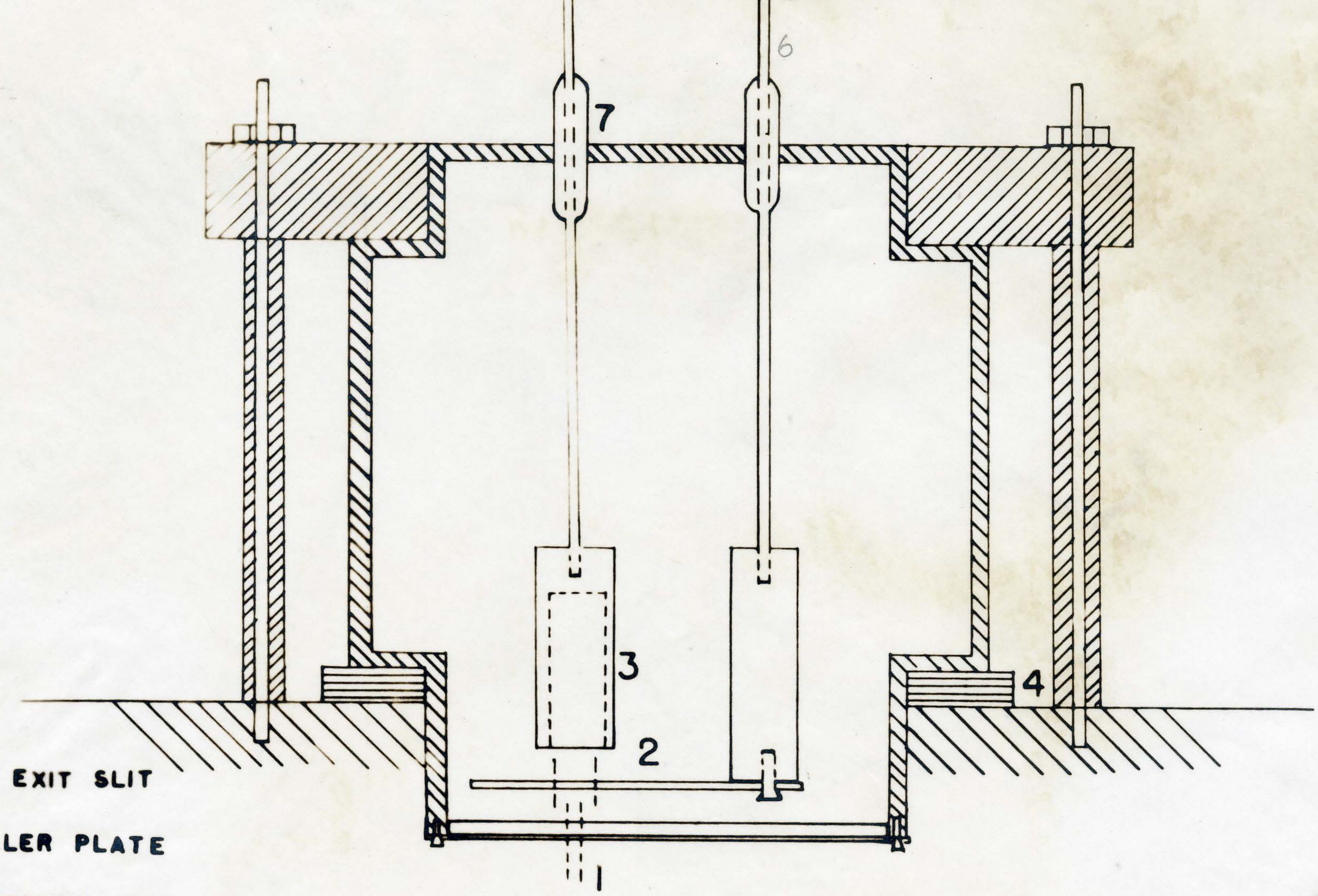
No. required - 2

Tolerance - when assembled A = 3.000 ±0.001"

Drawing DDF-0

W.E. Duckworth 20/2/45

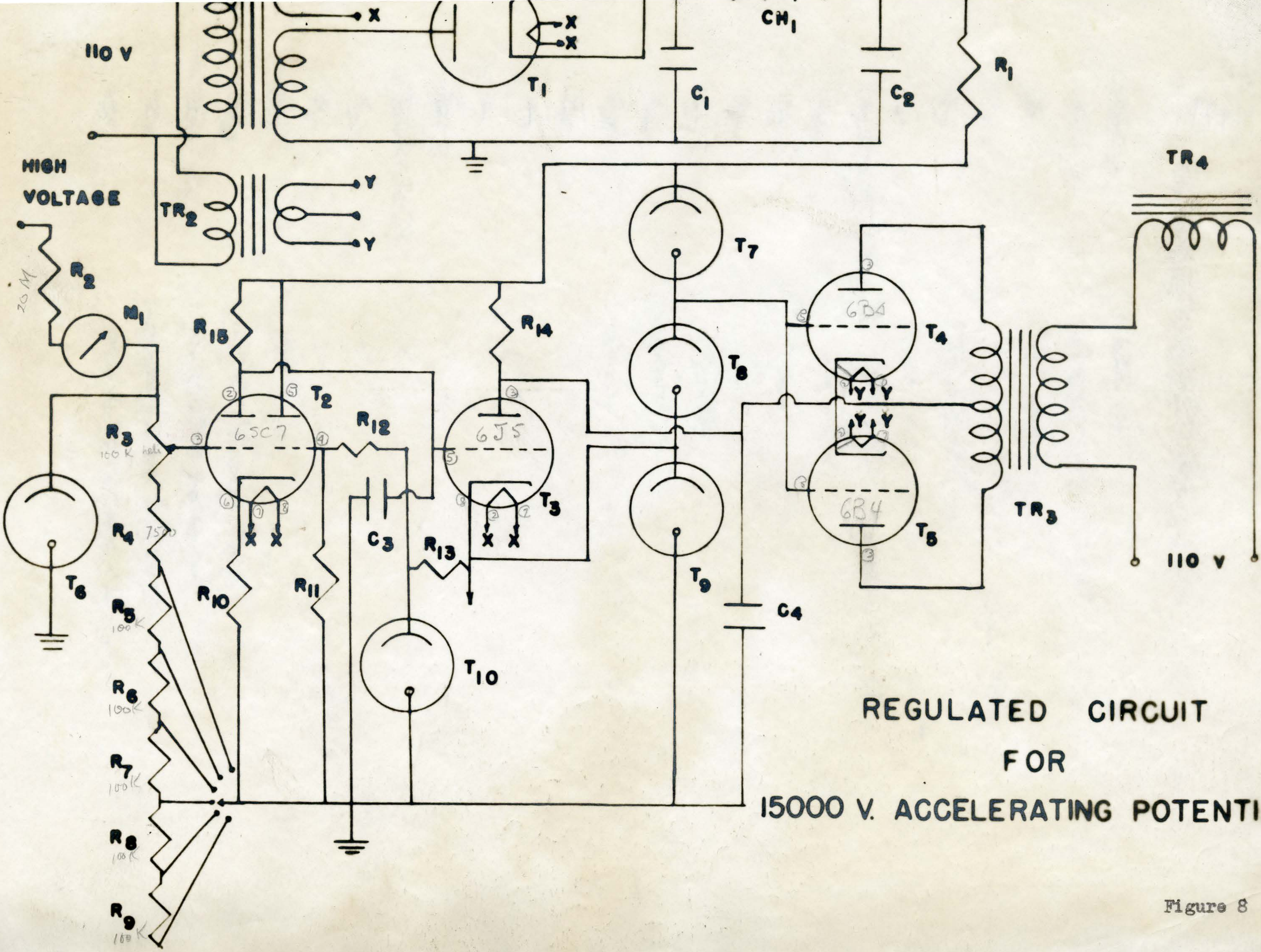
Figure 6



1 ·3MMS. EXIT SLIT
2 REPELLER PLATE
3 COLLECTOR CUP
4 RUBBER GASKET
5 CONNECTION TO REPELLER VOLTAGE
6 CONNECTION TO 954
7 KOVAR SEAL

COLLECTOR ASSEMBLY

Figure 7

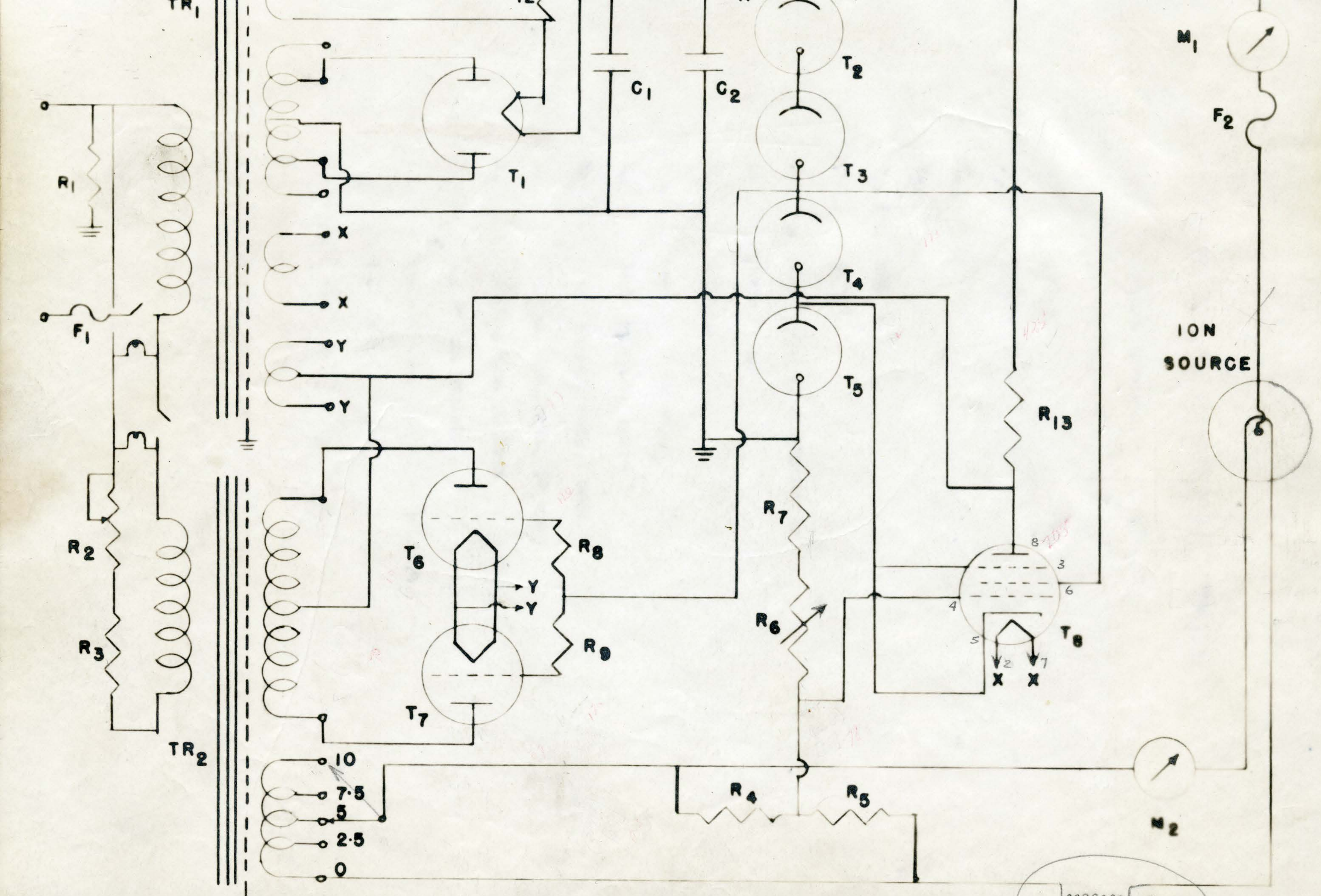


REGULATED CIRCUIT
FOR
15000 V. ACCELERATING POTENTIAL

Figure 8

PARTS LIST FOR ACCELERATING POTENTIAL CIRCUIT

C_1, C_2,	16mfd. 600 v.
C_3,	.002 mfd. 600 v.
R_1,	3,000 ohms, 20 watt
R_2,	4, 5 megohm shielded precision resistors
R_3,	100,000 ohms, helipot
R_4,	7,500 ohms, 8 watts
R_5, R_6, R_7, R_8, R_9,	100,000 ohms, precision resistors, 20 watt
R_{10},	100,000 ohms, 1 watt
R_{11},	50,000 ohms, 20 watt
R_{12},	20,000 ohms, 20 watt
R_{13},	5,000 ohms, 20 watt
R_{14},	100,000 ohms, 1 watt
R_{15},	1 megohm precision resistor, 1 watt
T_1,	1 V
T_2,	6SC7
T_3,	6J5
T_4, T_5,	6R4
T_6, T_7, T_8, T_9,	VR-105
T_{10},	VR-75
M_1,	0-1 MA. meter
CH_1,	30-65 choke
TR_1,	Hammond Transformer #269
TR_2,	Hammond Transformer #167
TR_3,	Hammond Transformer #18032
TR_4,	Primary High Voltage Transformer



CRUCIBLE SOURCE STABILIZER

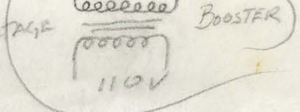
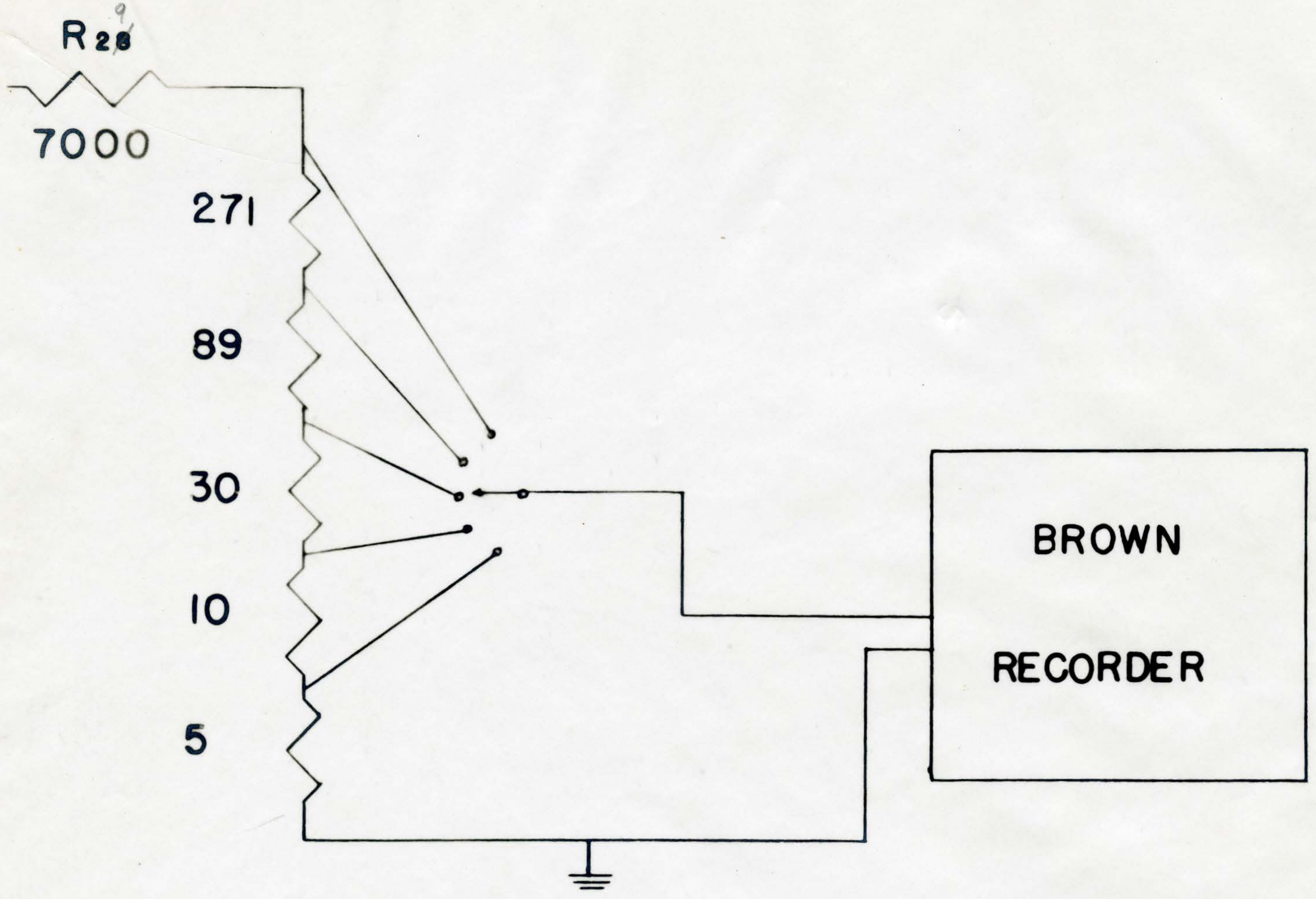


Figure 9

PARTS LIST FOR CRUCIBLE SOURCE STABILIZER

TR_1,	Hammond Transformer #24207
TR_2,	Hammond Transformer #20273 (331114) (See 5-10-204)
T_1,	5U4-G
T_2, T_3,	VR-150
T_4,	VR-~~150~~ 105
T_5,	VR-75
T_6, T_7,	6B4
T_8,	6SJ7 (6SH7)
C_1, C_2,	8 mfd. 600 V. electrolytic
F_1,	5 amp. fuse
F_2,	1/32 amp. fuse
M_1,	0-25 MA. D.C. meter
M_2,	0-10 amp. A.C. meter
R_1,	1 megohm resistor, 1 watt
R_2,	150 ohms w.w. pot., 20 watt
R_3,	150 ohms w.w. 20 watt
R_4, R_5,	one 50 ohm resistor, C.T., 20 watt
R_6,	100,000 ohms, precision helipot.
R_7,	3,000 ohms, 10 watt (150 Ω)
R_8, R_9,	5,000 ohms, 1 watt
R_{10}, R_{11},	1500 ohms 20 watt
R_{12},	50 ohms, C.T. 20 watt
R_{13},	200,000 ohms, precision resistor, 1 watt



SHUNT ARRANGEMENT

Figure 10

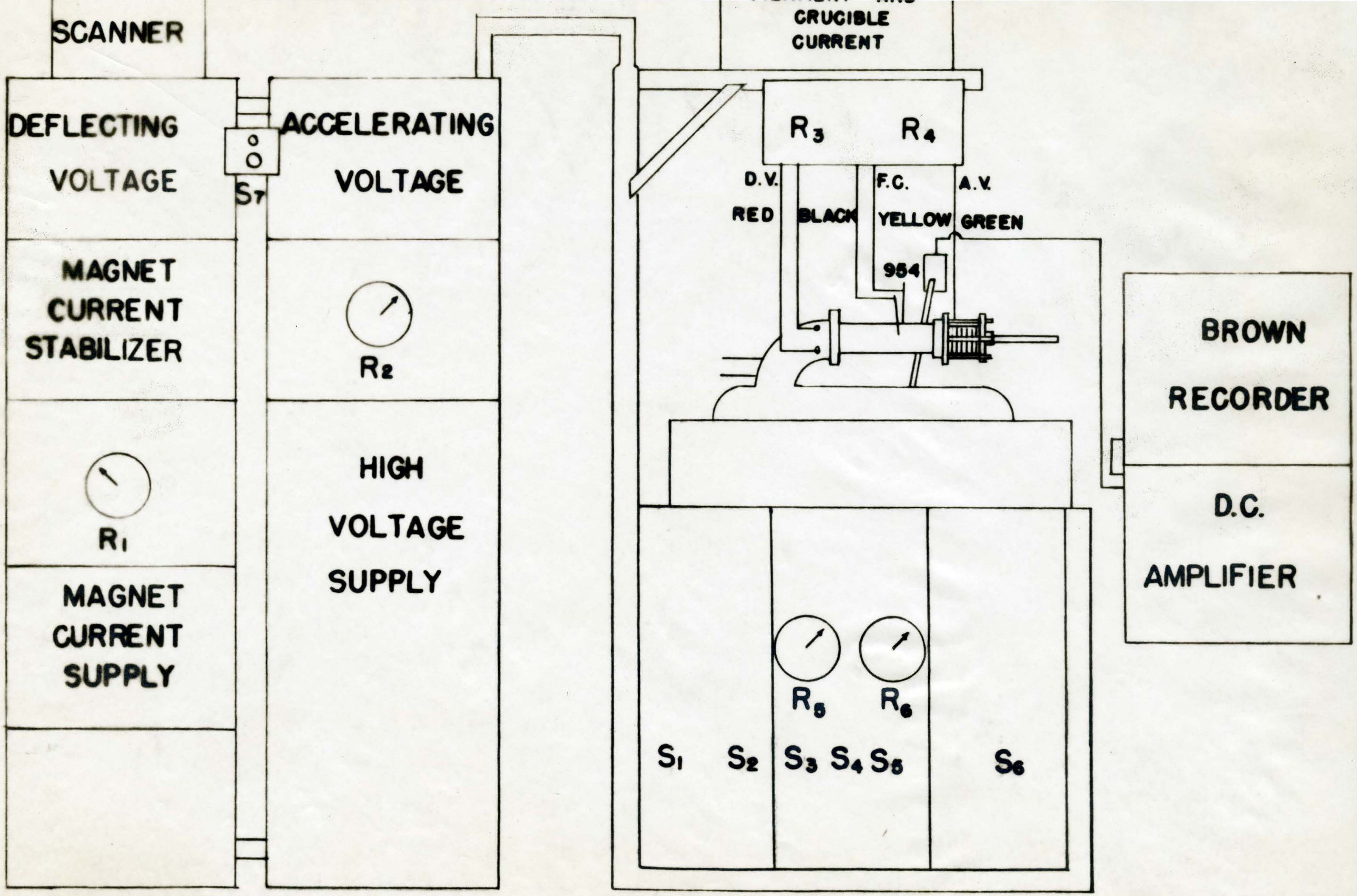
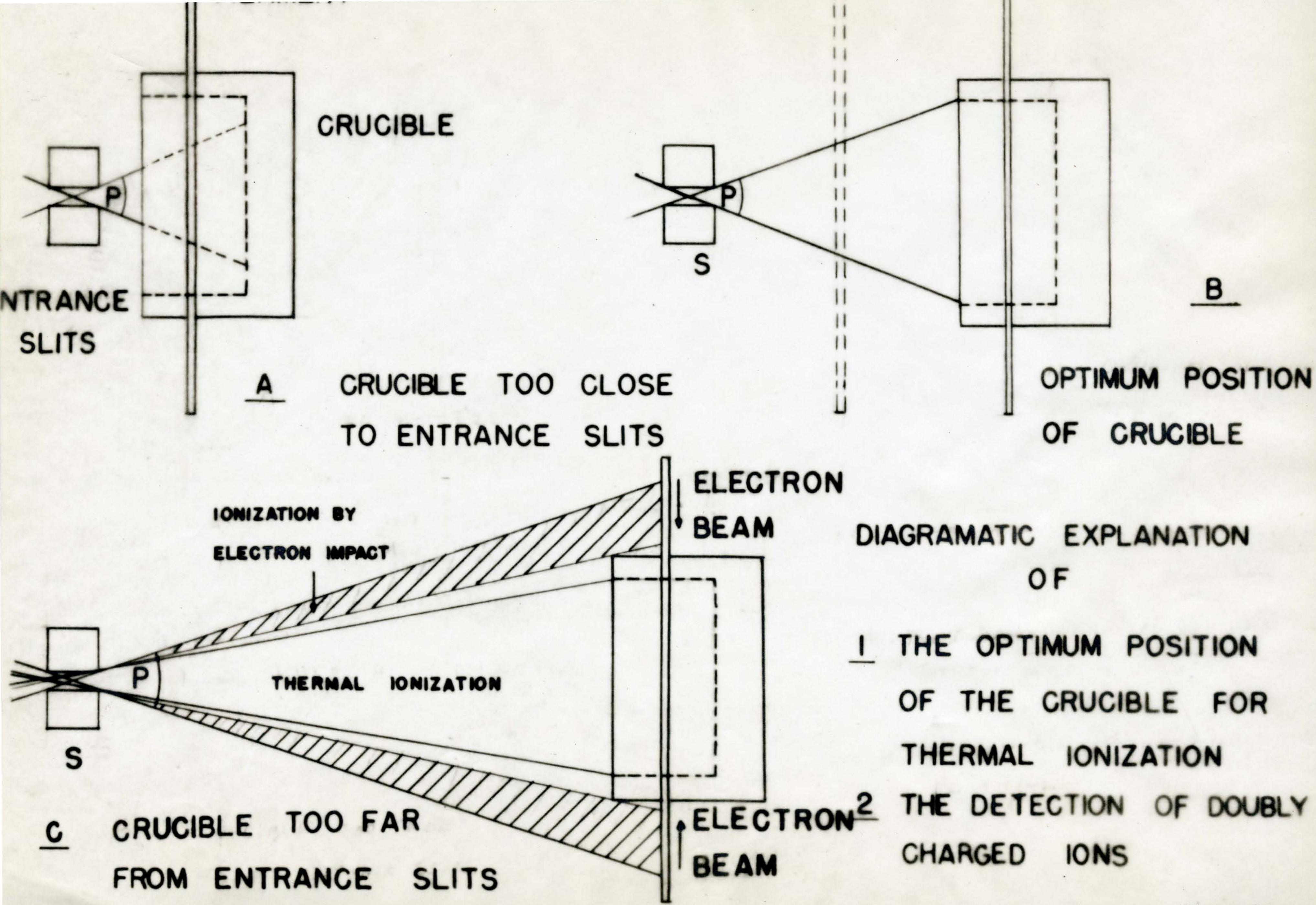

SCANNER
CRUCIBLE
CURRENT
DEFLECTING
VOLTAGE
S_7
ACCELERATING
VOLTAGE
R_3
R_4
D.V.
F.C.
A.V.
RED
BLACK
YELLOW
GREEN
954
MAGNET
CURRENT
STABILIZER
R_2
BROWN
RECORDER
R_1
HIGH
VOLTAGE
SUPPLY
D.C.
AMPLIFIER
MAGNET
CURRENT
SUPPLY
R_5
R_6
S_1
S_2
S_3
S_4
S_5
S_6

Figure 11



A CRUCIBLE TOO CLOSE TO ENTRANCE SLITS

B OPTIMUM POSITION OF CRUCIBLE

C CRUCIBLE TOO FAR FROM ENTRANCE SLITS

DIAGRAMATIC EXPLANATION
OF
1 THE OPTIMUM POSITION OF THE CRUCIBLE FOR THERMAL IONIZATION
2 THE DETECTION OF DOUBLY CHARGED IONS

Figure 12

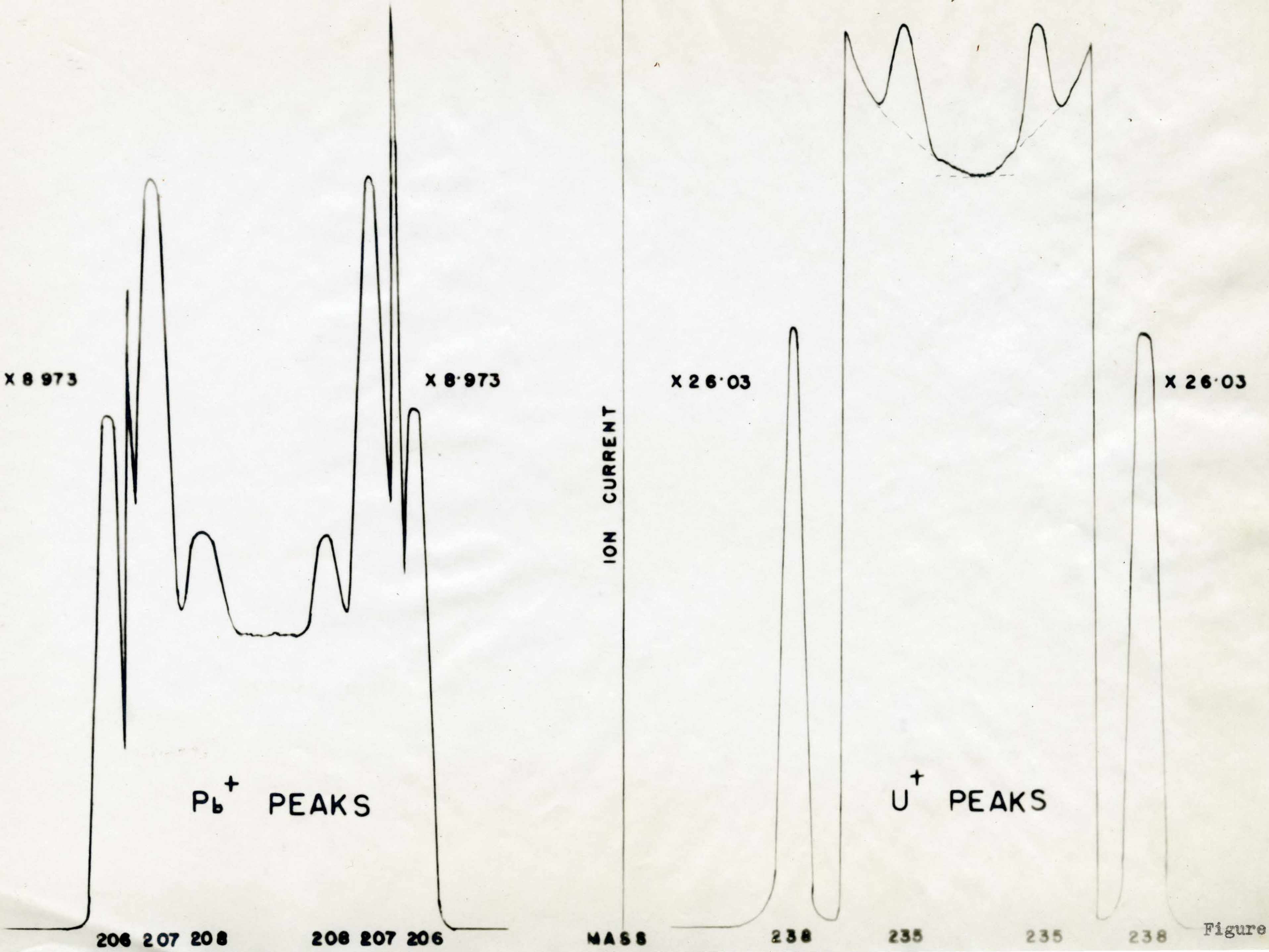

X 8 973
X 8·973
X 26·03
X 26·03
ION CURRENT
Pb$^+$ PEAKS
U$^+$ PEAKS
206 207 208
208 207 206
MASS
238
235
235
238

Figure 13

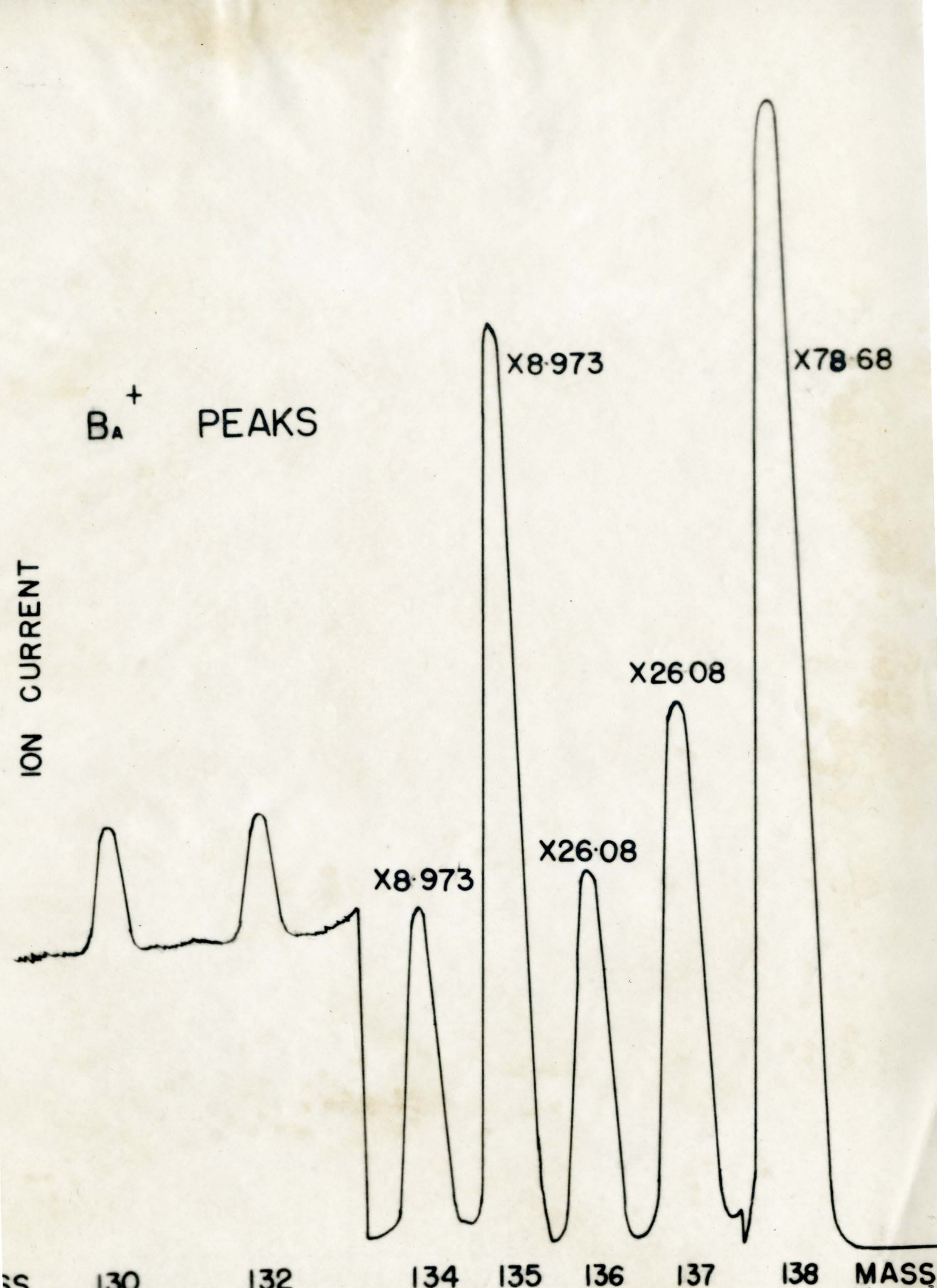
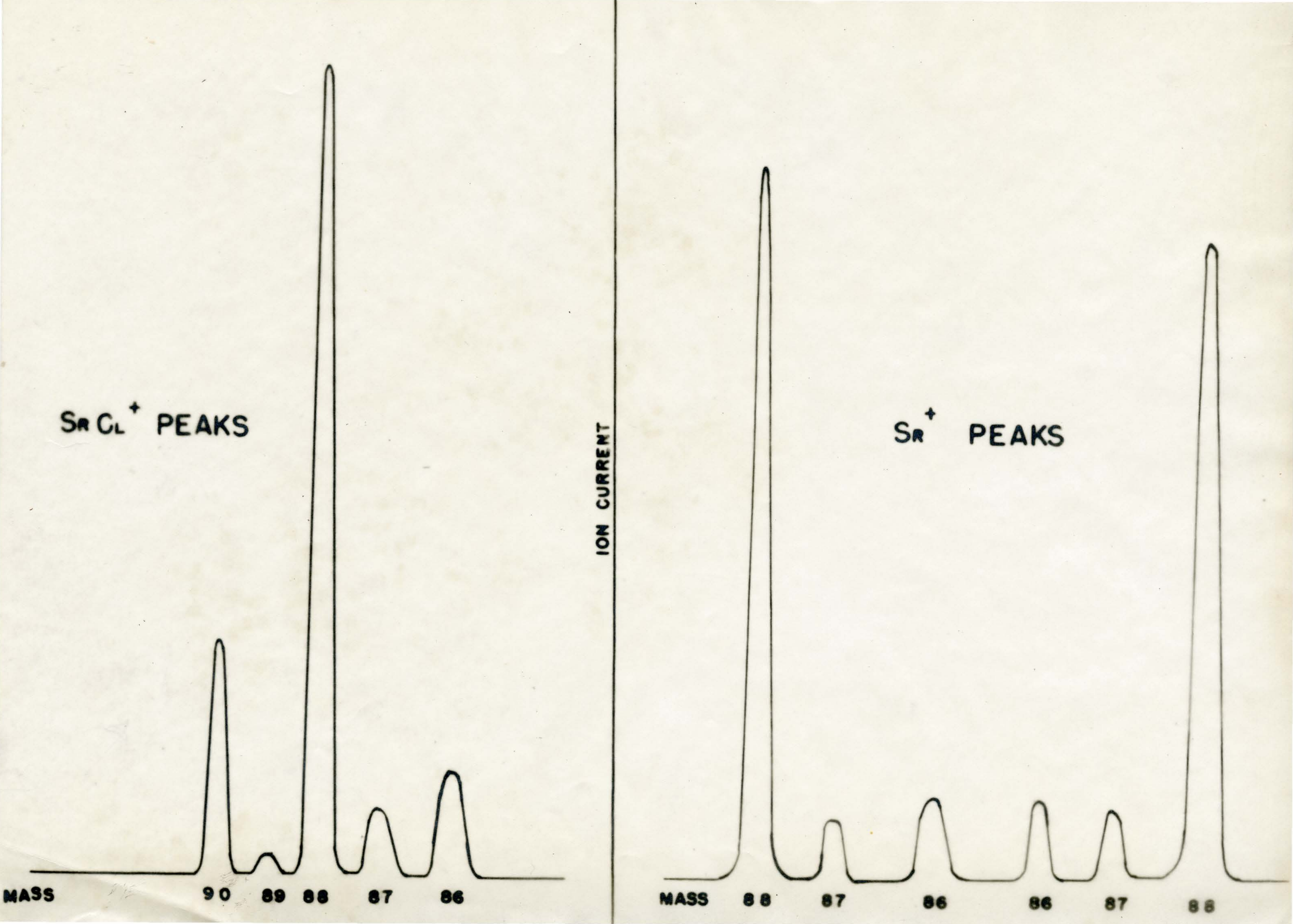

BA+ PEAKS
ION CURRENT
X8·973
X8·973
X26·08
X26·08
X78·68
SS
130
132
134
135
136
137
138
MASS

Figure 1


SR CL+ PEAKS
MASS
90
89
88
87
86
ION CURRENT
SR+ PEAKS
MASS
88
87
86
86
87
88