CHAPTER IV

EXPERIMENTAL RESULTS AND DISCUSSIONS

4.1 The Study of the Mass Spectrometer.

4.1a Using KCl as the Salt to be Investigated.

The arrangements inside the body of the instrument were as follows :-

the radius of the deflecting path of positive ions = 4.0 cm.
the width of the slit S_1 = 0.02 cm.
the width of the slit S_2 = 1.38 cm.
the width of the slit S_3 = 0.12 cm.

When the pressure of the vacuum system was below 10^{-5} m.m. of mercury, the magnetic field strength 3100 gauss was applied by using the magnet current of 9.6 amp. d.c. Due to the heating current of 2 amp. a.c. and the appropriate accelerating voltage, potassium ions emitting from the heated KCl on the tungsten filament deflected in a semicircle and then reached the ion collector. The ion currents were measured by the Avometer in conjunction with the measuring amplifier. The accelerating voltage varied and the ion currents measured were recorded. The results were tabulated in Table 4-1 and their relation was shown in Fig. 11. From Fig.11, the results were obtained as follows :-

1. The two adjacent peaks were observed corresponding to the accelerating voltage 179 volts and 189 volts. By calculating from Eq. (2.7), it was found that the accelerating voltage 179 volts and 189 volts corresponded to the atomic mass 41.4 a.m.u and 39.2 a.m.u respectively. Therefore the separation of the isotpes K^{39}, K^{41}

Table 4 - 1 the potassium ion currents and the accelerating voltages.

Accelerating Voltages Volts	Ion Currents X 10^{-9} Amps.
150	0 ± 0.03
170	0 ± 0.03
176	0.09 ± 0.03
180	0.21 ± 0.03
182	0.03 ± 0.03
186	0.9 ± 0.03
190	1.89 ± 0.03
195	0.3 ± 0.03
200	0.03 ± 0.03
210	0 ± 0.03
230	0 ± 0.03

The width of the slit S_1 = 0.02 cm.

The width of the slit S_2 = 1.38 cm.

The width of the slit S_3 = 0.12 cm.

Magnet current = 9.6 amp. d.c.

Heating current = 2.0 amp. a.c.

The radius of the circular orbit = 4.0 cm.

Magnetic field strength = 3100 gauss

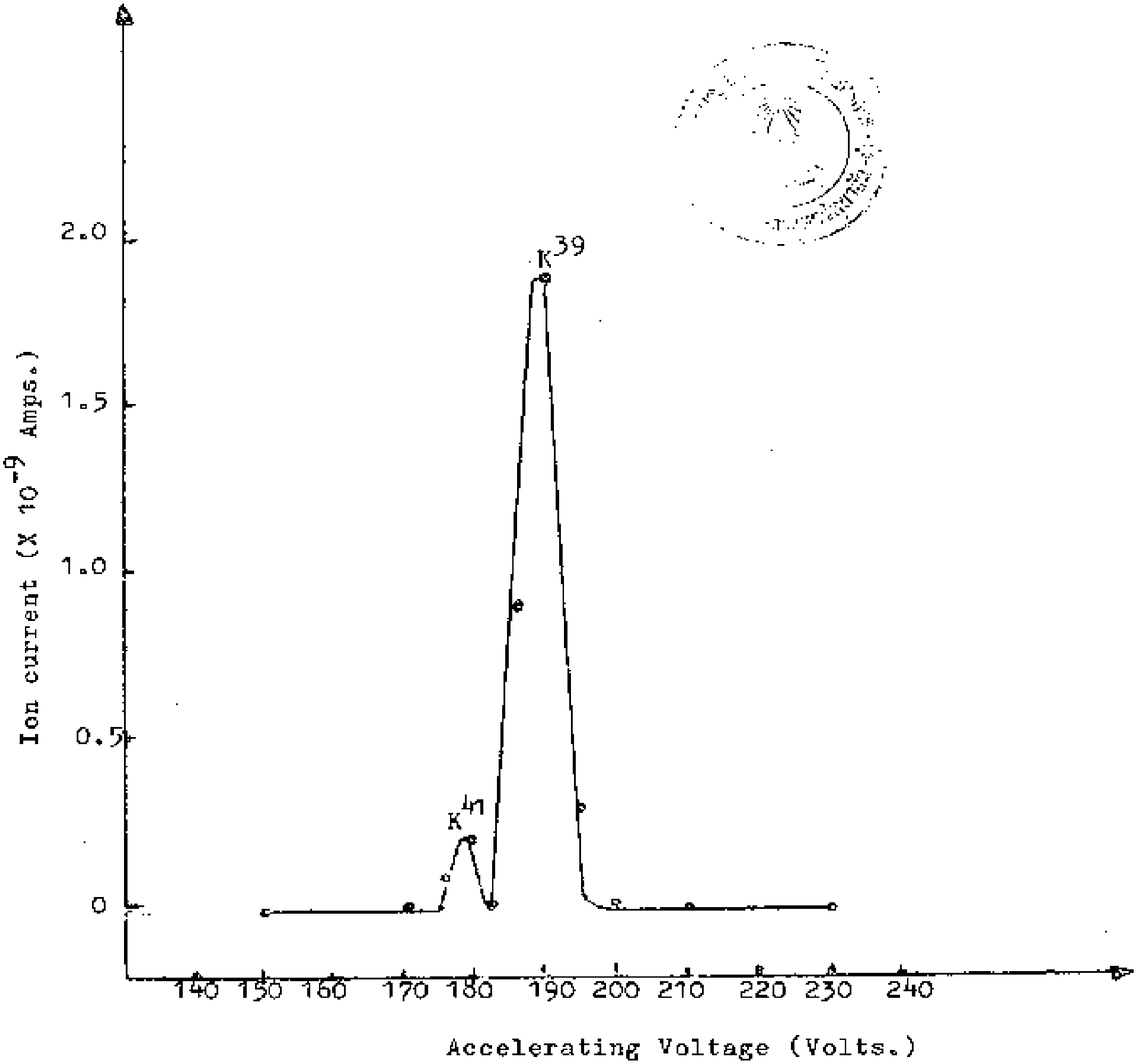


Fig.11 The Curve of Potassium Ion Currents Versus Accelerating Voltages

was indicated.

2. From the areas below the K^{39} and K^{41} peaks, the relative isotopic abundance of K^{39} and K^{41} was found to be 94.3 % and 5.5 % respectively.

3. The total image width was 0.14 cm. The resolving power calculated by Eq. (2.13) was 29. Nevertheless, the half width at the base of the K^{39} peak was 6 volts and by Eq. (2.14), the resolving power was 31. This showed that the proportional error of the result may be up to 3.2 %.

The probable error of the atomic mass of the potassium ion may be calculated from Eq. (2.15) and Eq. (2.17). From Eq. (2.17), the value of $\left(\frac{\Delta B}{B}\right)^2$ was 0.000859, by substituting

M = 50 millihenry ; ΔM = 0
I = 10 milliamp. ; ΔI = 0.1 milli amp.
α = 4.6 cm. ; $\Delta\alpha$ = 0.01 cm.
β = 0.4 cm. ; $\Delta\beta$ = 0.01 cm.
N = 30 turns ; ΔN = 0
r = 1.4 cm. ; Δr = 0.008 cm.

Then from Eq. (2.15), the ratio $\frac{\Delta m}{m}$ was 0.058 by substituting

$\left(\frac{\Delta B}{B}\right)^2$ = 0.000859
R = 4.0 cm. ΔR = 0.005 cm.
V = 179 volts, ΔV = 1 volt.

Hence the error of the result, resulting from the dimensions of the instruments may be up to 5.8 %.

4.1b Using Li_2SO_4 as the Salt to be Investigated.

In the case of Li_2SO_4, the magnetic field strength of 1500 gauss was applied by using the magnet current of 4 amp. d.c.. The heating current was 2.8 amp. a.c. The ion currents and the accelerating volages were recorded in Table 4 - 2 and their relation was shown in Fig.12. From Fig.12, the results were obtained as follows :-

1. Two peaks were observed corresponding to the accelerating voltage 248 volts and 289 volts. From Eq. (2.7), it was obvious that the accelerating voltage 248 volts and 289 volts corresponded to the atomic mass 6.9 a.m.u and 6.0 a.m.u. respectively. Thus the separation of isotopes Li^7, Li^6 was indicated.

2. From the areas below the Li^7 and Li^6 peaks, the relative isotopic abundance of Li^7 and Li^6 was 90.9 % and 9.1 % respectively.

3. The half width at the base of the Li^7 peak was 8 volts and by Eq. (2.14), the resolving power was 31. This showed that the proportional error of the result may be up to 3.2 %.

The probable error of the atomic mass of lithium ions may be calculated from Eq. (2.15) and Eq. (2.17). From Eq. (2.17), the value of $\left(\frac{\Delta B}{B}\right)^2$ was 0.000875, by substituting $\Delta M = 0$; $\Delta N = 0$

$I = 10$ milliamp. ; $\Delta I = 0.1$ milliamp.

$\alpha = 2.2$ cm. ; $\Delta\alpha = 0.01$ cm.

$\beta = 0.4$ cm. ; $\Delta\beta = 0.01$ cm.

$r = 1.4$ cm. ; $\Delta r = 0.008$ cm.

Then from Eq. (2.15), the ratio $\frac{\Delta m}{m}$ was 0.059 by substituting $\left(\frac{\Delta B}{B}\right)^2 = 0.000875$

$R = 4.0$ cm. ; $\Delta R = 0.005$ cm.

$V = 248$ volts ; $\Delta V = 1$ volt.

Table 4-2 The lithium ion currents and the accelerating voltages.

Accelerating Voltages Volts.	Ion currents X 10^{-12} Amps.
220	0 ± 0.03
230	0 ± 0.03
240	0.3 ± 0.03
248	1.2 ± 0.03
250	0.96 ± 0.03
255	0.09 ± 0.03
260	0.03 ± 0.03
270	0 ± 0.03
280	0 ± 0.03
285	0.06 ± 0.03
289	0.24 ± 0.03
295	0 ± 0.03
310	0 ± 0.03

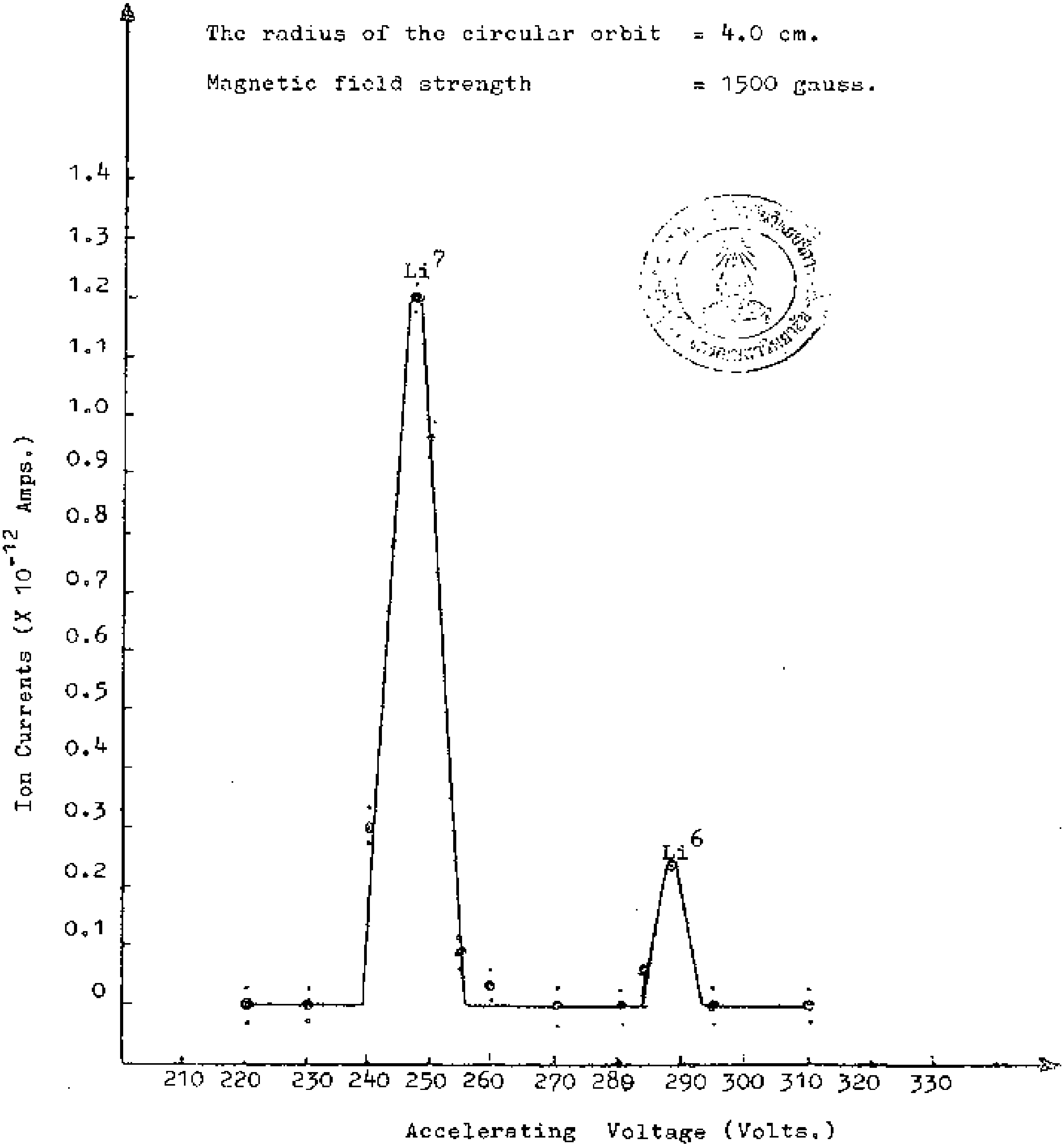
The width of the slit S_1 = 0.02 cm.

The width of the slit S_2 = 1.38 cm.

The width of the slit S_3 = 0.12 cm.

Magnet current = 4.0 amp. d.c.

Heating current = 2.8 amp. a.c.



The radius of the circular orbit = 4.0 cm.

Magnetic field strength = 1500 gauss.

Fig.12 The Curve of Lithium Ion Currents versus Accelerating Voltages.

As a result, the error of the result due to the dimensions of the instruments may be up to 5.9 %.

4.1c Using Na_2SO_4 as the Salt to be Investigated.

In the case of Na_2SO_4, the magnetic field strength 2700 gauss was applied by using the magnet current of 8.2 amp. d.c. The heating current was 2.5 amp. a.c. The ion currents and the accelerating voltages were recorded in Table 4-3 and their relation was shown in Fig. 13.

From Fig.13, the peak was observed corresponding to the accelerating voltage 243 volts. By calculating from Eq. (2.7), the accelerating voltage 243 volts, corresponded to the atomic mass 23.1 a.m.u. Therefore, the atomic mass of Na^{23} was determined. Besides this, the half width at the base of the Na^{23} peak was 8 volts. Then by Eq. (2.14), the resolving power was about 31. This showed that the proportional error of the result may be up to 3.2 %.

The probable error of the atomic mass of sodium ion may be calculated from Eq. (2.15) and Eq. (2.17). From Eq. (2.17), the value of $\left(\frac{\Delta B}{B}\right)^2$ was 0.000861 by substituting $\Delta M = 0$, $\Delta N = 0$

$I = 10$ milliamp. ; $\Delta I = 0.1$ milliamp.

$\alpha = 4.0$ cm. ; $\Delta\alpha = 0.01$ cm.

$\beta = 0.4$ cm. ; $\Delta\beta = 0.01$ cm.

$r = 1.4$ cm. ; $\Delta r = 0.008$ cm.

Then from Eq. (2.15), the ratio $\frac{\Delta m}{m}$ was 0.058 by substituting

$\left(\frac{\Delta B}{B}\right)^2 = 0.000861$

$R = 4.0$ cm. ; $\Delta R = 0.005$ cm.

$V = 243$ volts ; $\Delta V = 1$ volt.

Table 4 - 3 The sodium ion currents and the accelerating voltages

Accelerating Voltages Volts.	Ion currents X 10^{-12} Amps.
210	0 ± 0.03
230	0 ± 0.03
235	0.3 ± 0.03
240	1.5 ± 0.03
244	2.4 ± 0.03
248	1.2 ± 0.03
255	0 ± 0.03
270	0 ± 0.03

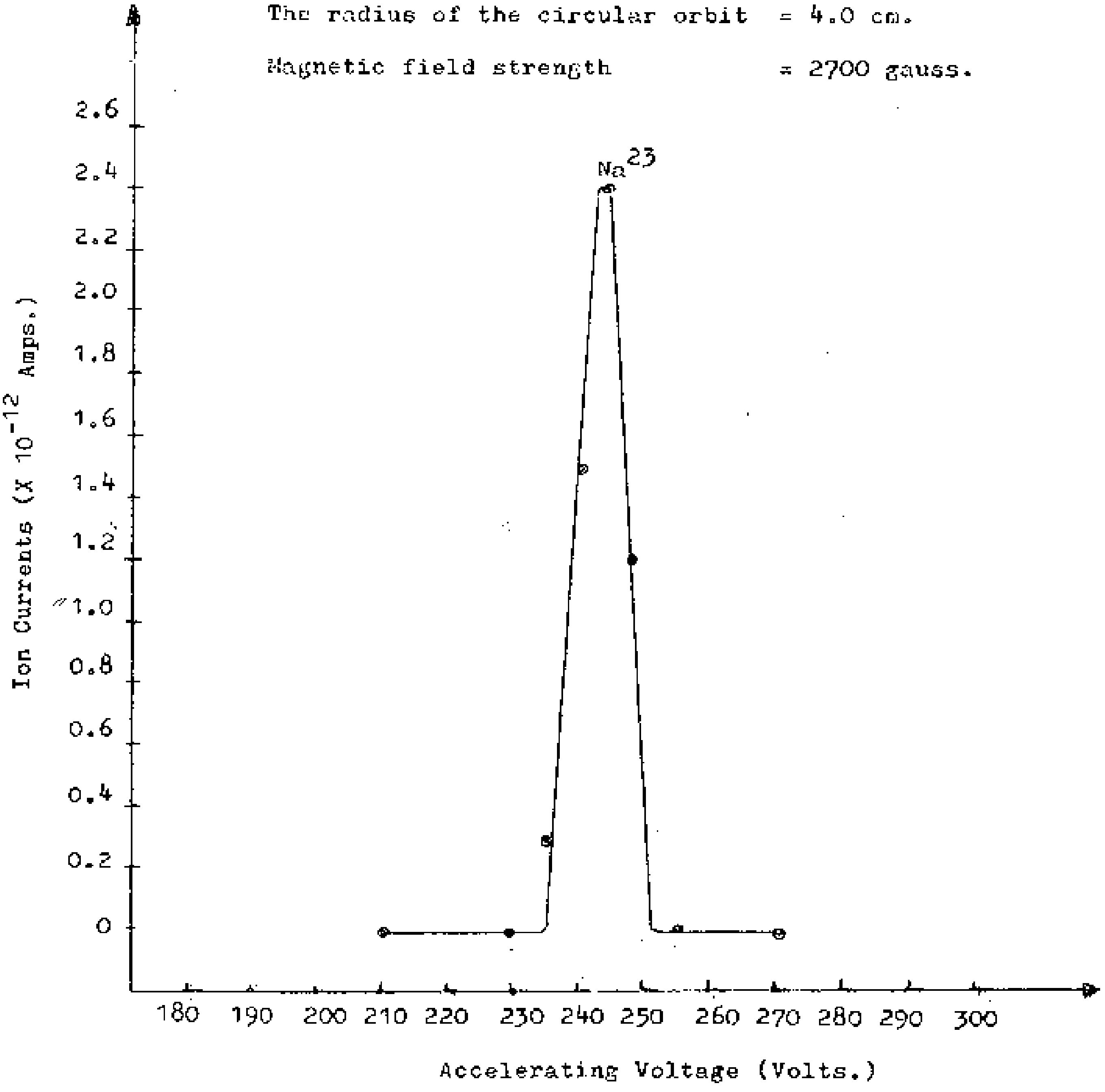
The width of the slit S_1 = 0.02 cm.

The width of the slit S_2 = 1.38 cm.

The width of the slit S_3 = 0.12 cm.

Magnet current = 8.2 amp. d.c.

Heating current = 2.5 amp. a.c.



The radius of the circular orbit = 4.0 cm.

Magnetic field strength = 2700 gauss.

Fig.13 The Curve of Sodium Ion Currents versus Accelerating Voltages.

Thus, the error of the result owing to the dimensions of the instruments may be up to 5.8 %.

4.1d Using $BaCl_2$ as the Salt to be Investigated.

In the case of $BaCl_2$, the magnetic field strength 2500 gauss was applied by using the magnet current of 7.5 amp. d.c. The heating current was 2.8 amp. a.c. The ion currents and the accelerating voltages were recorded in Table 4 - 4 and their relation was shown in Fig.14.

From Fig.14 there was only one flat topped peak. Thus it was difficult to point out the exact accelerating voltage. Nevertheless, the accelerating voltage of 35 volts was corresponding to the point at the half width of the flat topped peak. From Eq. (2.7), the voltage 35 volts corresponded to the atomic mass 137.7 a.m.u. The half width at the base of the peak was 1.1 volts. Then, by Eq. (2.14) the resolving power was about 31.

The probable error of the atomic mass of barium may be calculated from Eq. (2.15) and Eq. (2.17). From Eq. (2.17), the value of $\left(\frac{\Delta B}{B}\right)^2$ was 0.000862 by substituting $\Delta M = 0$; $\Delta N = 0$

$I = 10$ milliamp. ; $\Delta I = 0.1$ milliamp.

$\alpha = 3.7$ cm. ; $\Delta\alpha = 0.01$ cm.

$\beta = 0.4$ cm. ; $\Delta\beta = 0.01$ cm.

$r = 1.4$ cm. ; $\Delta r = 0.008$ cm.

Then from Eq. (2.15), the ratio $\left(\frac{\Delta m}{m}\right)$ was 0.058 by substituting $\left(\frac{\Delta B}{B}\right)^2 = 0.000862$

Table 4-4 The barium ion currents and the accelerating voltages.

Accelerating Voltages Volts.	Ion Currents $\times 10^{-12}$ Amps.
31	0 ± 0.03
33	0 ± 0.03
34	0.09 ± 0.03
34.1	0.3 ± 0.03
34.7	0.6 ± 0.03
35.0	0.6 ± 0.03
35.5	0.6 ± 0.03
37.7	0.36 ± 0.03
36	0.03 ± 0.03
37	0 ± 0.03
39	0 ± 0.03

The width of the slit S_1 = 0.02 cm.

The width of the slit S_2 = 1.38 cm.

The width of the slit S_3 = 0.12 cm.

Magnet current = 7.5 amp. d.c.

Heating current = 2.8 amp. a.c.

The radius of the circular orbit = 4.0 cm.

Magnetic field strength = 2500 gauss.

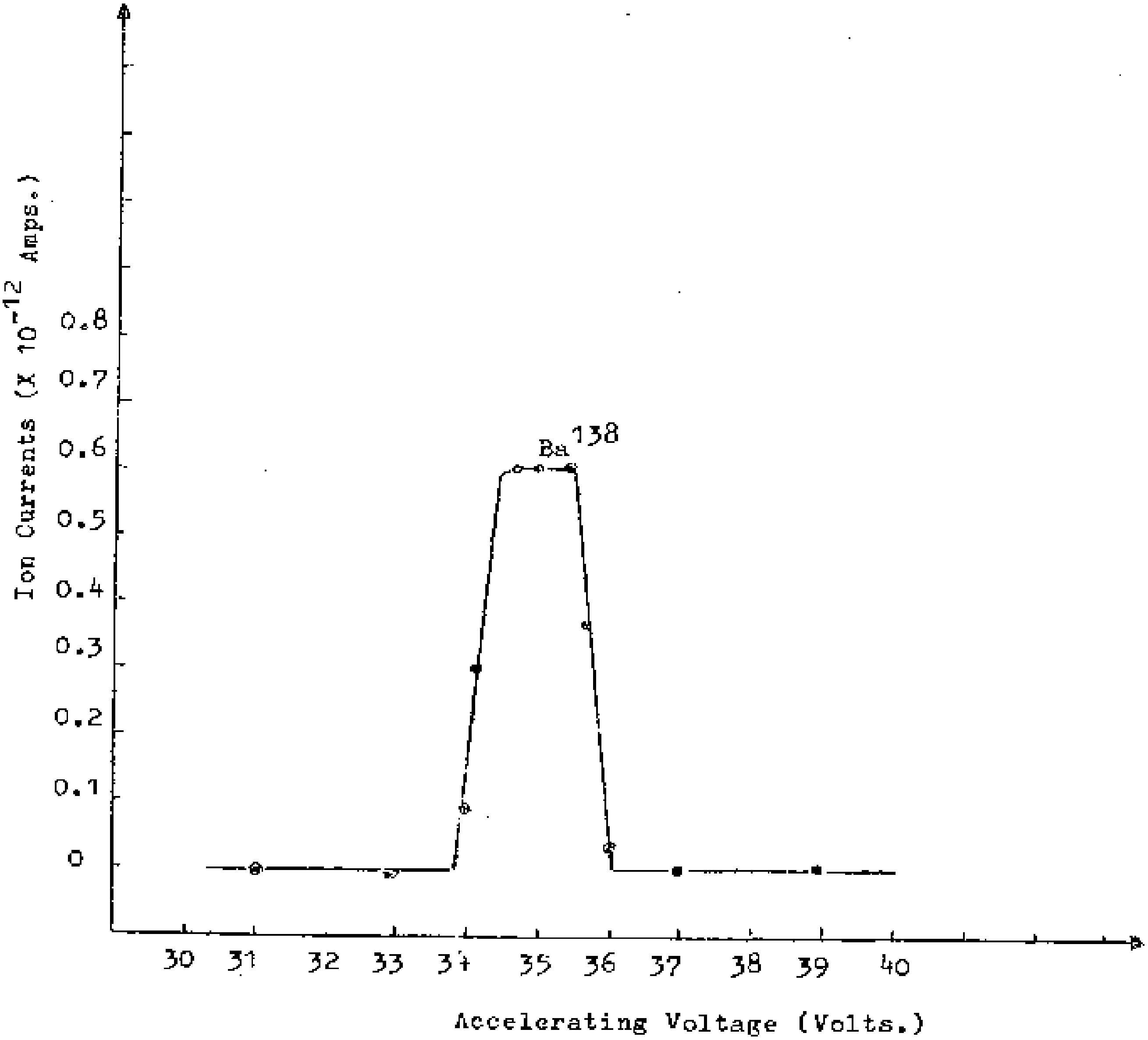


Fig.14 The Curve of Barium Ion Currents versus Accelerating Voltages.

$R = 4.0$ cm. ; $\Delta R = 0.005$ cm.

$V = 35$ volts ; $\Delta V = 0.1$ volt.

Hence, the error of the result owing to the dimensions of the instruments may be up to 5.8 %.

4.1e Using $CaCO_3$ as the Salt to be Investigated.

The magnetic field 2500 gauss was applied by using the magnet current 7.5 amp. d.c. The heating current was 3.0 amp. a.c. The ion currents and the accelerating voltages were recorded in Table 4 - 5 and their relation was shown in Fig.15.

From Fig.15, the results were obtained as follows :-

1. Two peaks were observed corresponding to the accelerating voltage 108 volts and 118 volts. From Eq.(2.7), it was clear that the accelerating voltage 108 volts and 118 volts corresponded to the atomic mass 44.6 a.m.u. and 40.8 a.m.u. respectively. Hence the separation of isotopes Ca^{44} and Ca^{40} was carried out.

2. From the areas below the Ca^{44} and Ca^{40} peaks, the relative isotopic abundance of Ca^{44} and Ca^{40} was 1.9 % and 98.0 % respectively.

3. The half width at the base of the Ca^{40} peak was 4 volts. Then from Eq. (2.14), the resolving power was about 30.

The probable error of the atomic mass of positive calcium ions may be calculated from Eq. (2.15) and Eq. (2.17). The value of $\left(\frac{\Delta B}{B}\right)^2$ was 0.000862. It was the same value as that in the use of $BaCl_2$ because of using the same value of the magnet current then from Eq. (2.15), the ratio $\frac{\Delta m}{m}$ was 0.058 by substituting

$$\left(\frac{\Delta B}{B}\right)^2 = 0.000862$$

Table 4-5 The calcium ion currents and the accelerating voltages.

Accelerating Voltages Volts	Ion currents X 10^{-12} Amps.
95	0 ± 0.03
100	0 ± 0.03
105	0.03 ± 0.03
108	0.06 ± 0.03
111	0 ± 0.03
115	1.5 ± 0.03
119	3.0 ± 0.03
122	0.9 ± 0.03
125	0.03 ± 0.03
135	0 ± 0.03

The width of the slit S_1 = 0.02 cm.

The width of the slit S_2 = 1.38 cm.

The width of the slit S_3 = 0.12 cm.

Magnet current = 7.5 amp. d.c.

Heating current = 3.0 amp. a.c.

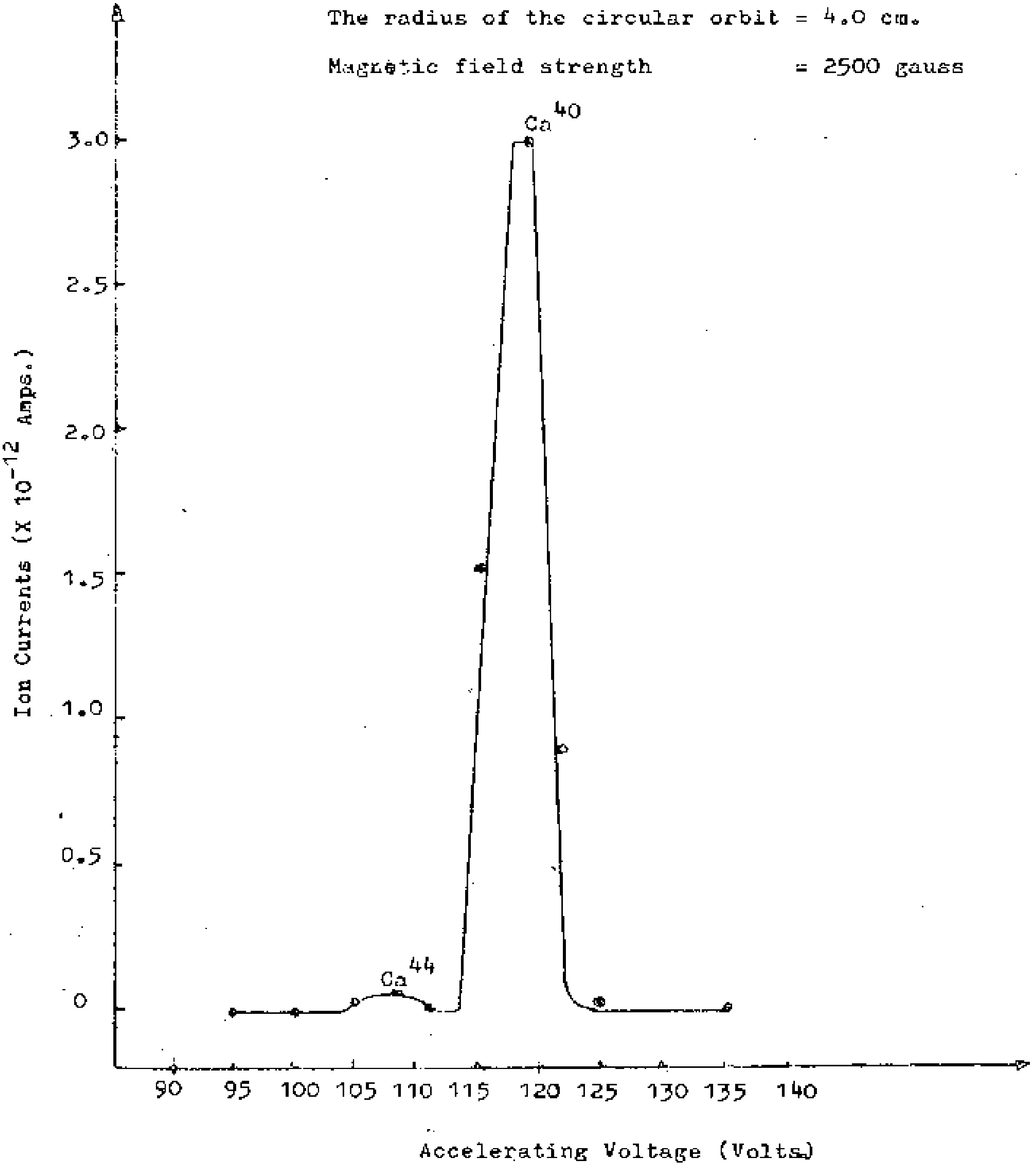


Fig.15 The Curve of Calcium Ion Currents versus Accelerating Voltages.

$R = 4.0$ cm., $\Delta R = 0.005$ cm.

$V = 118$ volts. $\Delta V = 0.2$ volt.

Hence, the error of the result owing to the dimension of the instruments may be up to 5.8 %.

4.2 Discussions

In order to obtain a good vacumm system there were several factors to be considered. In the first place, the body of the instrument must be kept clean free from dust and grease. Secondly, the alkali salt coated on the tungsten filament should be absolutely dry. Thirdly, the joints were vacuum-tight.

If the pressure inside the instrument did not fall below 10^{-5} m.m. of mercury, positive ions from the heated salt would collide with the atoms of the residual gases and scatter. For a given element, the yield of positive ions from a coated filament depends strongly upon the chemical compound used. Nevertheless the salts used for the investigation except $CaCO_3$ were the suitable ones as mentioned by Robert G. Macley (11). The heating temperature of the filament resulting from the heating current should be high enough, otherwise no positive ions could be released from the salt. The slightly red glow at the end of the electrodes of the heating unit was the indicating point for appropriate temperature. Three slits must be carefully adjusted, otherwise positive ions could not be detected owing to missing the slits. The mass spectrometer placed perpendicularly between the pole faces of the electromagnet, was to be arranged properly so that the circular path of positive ions was wholly maintained under the influence of the magnetic field.

Eventhough, the probable error which may be caused by the dimensions of the instrument was about 6 %, the experimental values of the atomic masses of the isotopes (K^{39}, K^{41}, Li^{6}, Li^{7}, Na^{23} and Ca^{40}, Ca^{44}) were in good agreement with their mass numbers and were good enough in comparison with those of E. Segre (20) with the percentage of error about 1 - 2 %.

The relative isotopic abundance of the element was sometimes varied owing to the salt non-uniformly coated on the filament. However the experimental values of the relative isotopic abundances of the potassium isotopes (K^{39}, K^{41}) , the lithium isotopes (Li^{6},Li^{7}) and the calcium isotopes (Ca^{40}, Ca^{44}) were satisfactory in comparison with those of E. Segre (20). In the case of the sodium isotope, there was only the Na^{23} peak. Hence the abundance ratio of sodium isotope was 100 %.

The experimental value of the resolving power of the instrument was about 31 and it was nearly the same value as that of the one calculated.

The maximum ion currents of positive potassium ions were about 10^{-9} amps. and those of positive lithium, sodium, barium and calcium ions were about 10^{-12} amps. These showed that the tungsten filament having work function greater than the ionization potential of potassium atom would emit large quantities of potassium ions. On the contrary, the lithium, sodium, barium and calcium atoms having ionization potentials greater than the work function of tungsten would emit small quantities of the appropriate ions. The heating temperature of the filament was the important factor of the emitting of positive ions from the heated salt. It was found that if the heating temperature was raised higher, the heated salt would emit larger amount of positive ions.

In the determination of the atomic mass of barium ions, one of the alkali earth metal elements by using $BaCl_2$ as the sample, the flat topped peak was obtained. If the width of the flat topped peak was bisected, the three points, one point at the middle and the other two points at both rims were corresponding to the accelerating voltages of 34.5 volts, 35.0 volts and 35.5 volts. By calculating from Eq. (2.7) those three voltages corresponded to the atomic mass 139.7 a.m.u., 137.7 a.m.u. and 135.7 a.m.u. respectively. It was possible that the instrument was not good enough to separate the isotopes of barium 138, 137, 136, 135 containing in a large portions, and of course the isotopes of barium 134, 132, 130 containing as very small compositions in the barium salt were not detected. By this reason, the atomic masses of barium isotopes were in the range 139.7 a.m.u. to 137.7 a.m.u. However, the point at the middle of the width of the flat topped peak was reckoned at the accelerating voltage 35.0 volts corresponding to the atomic mass of barium ion 137.7 a.m.u. This showed that the atomic mass 137.7 a.m.u. belonged to the atomic mass of barium 138, having the most relative isotopic abundances in the barium salt.

In the determination of calcium isotopes, the two isotopes of Ca^{40} and Ca^{44} were detected, whereas the isotopes of Ca^{42}, Ca^{43}, Ca^{46}, Ca^{48} could not be detected because of their very small abundance ratios in the salt. During the operation it was noticeable that the pressure in the vacuum system was not as low as that when other salts were used. It might be due to the small amount of CO_2 that would come out from heated $CaCO_3$. Nevertheless, the calcium ions were detected.