IMPROVEMENT AND APPLICATION OF A HIGH-INTENSITY MAGNETIC-LENS TYPE OF MASS-SPECTROMETER

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ABSTRACT

A mass-spectrometer in which was used a new type of magnetic lens 'o focus ions from an extended source was already in existence when the research was begun, but no adequate source of positive ions had beer devised. The research concerns itself with the investigation of three types of intense, directed-beam ion sources and with the application of the completed mass-spectrometer to the separation of isotopes of potassium and lithium in quantity.

The ion-source finally used was adapted from a form designed by neglecting space charge and solving by a mechanical analogy the electrostatic problem of focussing the ions from a large surface into a plane parallel beam. Revised to allow for space charge, this source gives 0.3 m.a. of potassium ions in a flat beam, which is 12% of the emission from 30 cm².

Sources using a curved grid were set up and tested. A threeslit electrostatic lens was investigated mathematically and with the mechanical analogy. The effect of thermal velocities of ions at the hot surface of the source was calculated and found to account for almost the whole focal defect of the mass-spectrometer.

One-microgram samples of lithium isotopes were collected. Preliminary tests of radioactivity were hindered by contamination of the discs on which the samples were collected, and an apparatus has been constructed by which these tests are being made without possibility of contamination.

IMPROVEMENT AND APPLICATION OF A HIGH-INTENSITY

MAGNETIC-LENS TYPE OF MASS-SPECTROMETER

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I. INTRODUCTION

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Since the discovery in 1919 of isotopes of nonradioactive elements, many attempts have been made to obtain pure specimens of individual isotopes in amounts large enough for experimental purposes, with no success except in the application of differential electrolysis to hydrogen and of the Hertz diffusion method¹ to certain other gases. The method of Hertz is suitable only for the separation of the lightest and heaviest isotopes of a group which can be obtained in gaseous form, and to isolate any member of a group it is necessary to use some form of mass-spectrometer. Since collimating a beam of ions by slits makes the quantities inordinately small, there was necessary a mass-spectrometer which would use all ions emitted from a large area.

Such an instrument was developed by Dr. W. R. Smythe and further by Dr. L. H. Rumbaugh, who constructed in 1931 a well-corrected magnetic lens to focus the ions from an extended source onto a slit.² I shall devote the following pages to a description of certain types of intense ion-sources which I investigated after Dr. Rumbaugh's departure in 1932, of methods of collecting, and of the investigation of the radioactivity of potassium with the completed mass-spectrometer.

II. THE MAGNETIC LENS

The design, focal errors, and correction of the magnetic lens have been discussed to some length in Dr. Rumbaugh's thesis, but it is well to review the general features briefly here.

The boundaries of the uniform magnetic field are shown in Fig. 1.



Figure 1. -- Cylindrical magnetic lens formed by a uniform magnetic field with circular boundaries having centers at C and C'. Paths of positive rays are shown converging to C from the right and diverging from a virtual focus C at the left. If K³⁹ is focussed at C, K⁴¹ comes to a focus at the point checked just above C. The corrected form of the pole-piece is dotted in.



Figure 2. -- Perspective drawing of the mass-spectrometer. The outline of the pole-pieces is completed by dotted lines, and the source assembly and collecting arrangement are also dotted in.



Figure 3a. --Front view of apparetus.



Figure 3b. -- Rear view of apparatus, showing collector head at 1 and Pirami gauge at 2.



Figure 3c. -- Side view of apparatus, showing controls for wire and her potentials at 3, voltmeter for accelerating potential at 4, and potentiometer slider at 5.

These are circles passing through the origin, one having its center at the focal point C and the other at C' a distance r to the right of C, where r is the radius of curvature of the trajectories in the magnetic field. Since actually the field does not have sharp boundaries, the field near the edges of the pole-pieces was computed and a correction made, which resulted in reducing the radius of the convex edge from 28.735 cm to 28.100 cm and increasing the radius of the concave edge from 20.320 cm to 20.955 cm. The gap between the pole-pieces was 0.635 cm and the radius of the path in the field 20.320 cm. The actual outline of the pole-pieces is shown by a dotted line in Fig. 1 and in perspective in Fig. 2.

The ions enter in a nearly plane sheet parallel to the plane of Fig. 1 and are brought to a focus at C.

III. ION SOURCES

1) METHOD FOR THE SOLUTION OF PROBLEMS IN ELECTROSTATIC FOCUSSING

The simplest source of positive ions of uniform velocity consists or a rectangular slit placed at a sufficiently great distance above an equipotential plane emitting surface so that, when the emitter is maintained at a positive potential with respect to the slit, the field between them will be uniform over nearly the whole distance. (Source A) Under such circumstances, however, a space charge of ions will build up until the potential gradient at the emitting surface becomes zero, and hence the ion current is limited and can be shown to have the maximum value $i = \left(\frac{1}{9} + \frac{2e}{m} + \frac{v^3}{y}\right)^2$, where y is the distance between two parallel planes whose potential difference is V. If a grid is used to reduce the effect of this space charge, the beam is scattered badly and a considerable portion of it goes to the grid wires themselves. Source A was the type in use when I began work.

Another method of reducing space charge consists of moving the slit closer to the emitting surface, to obtain a higher field with the same potentials. Since the width of the slit must remain the same, so that the beam will fill a large part of the pole-piece slot, the emitting surface can no longer be plane, but must have some other shape such that ions emitted from all parts of it will ultimately have parallel trajectories. The electrostatic problem here is really two-dimensional, most or the source being effectively at a large distance from the ends. Poisson's equation is practically impossible to solve in this case, but a solution for Laplace's equation, space charge being neglected, was obtained in the following manner.

The deflections V of a stretched elastic membrane from a given plane obey a differential equation $\mathbf{f}_{\mathbf{i}} + \mathbf{f}_{\mathbf{i}} = \mathbf{0}$ when the deflections are small, which is the same as Laplace's equation in two dimensions. Now two particles can move in identical paths in a plane if their accelerations are the same at any two corresponding points of the paths, and we can even have $\overline{a_1} = c\overline{a_2}$, where $\overline{a_1}$ and $\overline{a_2}$ are the accelerations and c is a constant, for this is equivalent merely to changing the time scale for one particle from the case where $\overline{a_1} = \overline{a_2}$, the same path being traversed in different times. For a ball rolling in a gravitational field and deviating little from a horizontal plane,

$$(\frac{2}{5}m_1r^2 + m_1r^2)\frac{\overline{a}_1}{\overline{r}} = r\overline{P}$$

$$\overline{a}_1 = \frac{2}{5}\overline{F}/m_1 = \frac{2}{5}\overline{G}m_1/m_1 = \frac{2}{5}\overline{G}$$

where m_1 is the mass of the ball, r the radius of the ball, F the force, a₁ the acceleration of the center of gravity, and \overline{G} the gravitational field.

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For an ion in a two-dimensional electrostatic field,

 $m_{z}\overline{a}_{z} = e\overline{E}$ $\overline{a}_{z} = (e/m_{z})\overline{E}$

Hence, if the electrostatic field has at each point the direction of the gravitational field at the corresponding point and a magnitude differing only by a constant factor c=G/E, the accelerations for corresponding points in the two planes will have the same directions and their absolute magnitudes will be in the ratio

$$\frac{a_1}{a_2} = \frac{5/7 \text{ G}}{e/m_2 \text{ E}} = \frac{5m_2}{7e} \text{ c} = \text{const.}$$

The paths will therefore be identical and independent of the mass or charge of the ion.

2) DESIGN OF AN ELECTROSTATICALLY FOCUSSED SOURCE

Such a mechanical analogy was constructed by stretching sheet rubber uniformly over a horizontal plane frame of size 3x5 feet. Portions of the membrane were elevated with configurations and elevations corresponding to the shape and electrostatic potentials of the source electrodes and neighboring conductors, the height above the frame being never more than about one inch. At the boundary corresponding to the emitting surface were released steel ball-bearings 1/8 inch in diameter, so that their paths were those which ions would follow in the corresponding electric field. Deviations of the balls due to rolling friction, etc., were small, for changing the mass of the ball by a factor of eight produced little change in the path. The design obtained from this analogy (Fig. 4) consisted of a portion of a circular cylinder completely covered with emitter, close above which was placed a beveled slit RR. The curvature of the emitting surface, the width and bevel of the alit, and the distance between slit and source were so adjusted



Figure 4. -- Section of source assembly showing the heated block U, with the mitter shown in black, and the water-jacket V surrounding it. The accelerating potential is applied between UV and the water-cooled slit R. Paths of particles in the absence of space charge are shown by arrows. Evacuation holes are dotted at the sides.

that the emission is (in the absence of space-charge) concentrated into the narrow parallel beam shown in the figure. Suitable dimensions are those given below. They are adjusted so that the beam will fill the 6 mm slot between the pole-pieces.

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Width	of	beam				•		=	0.25 inch
Width	of	slit						a '	0.667
Width	of	emitting	surfac	e on	cho	ord		=	1.133
Shorte	st	distance	from p	late	to	emitting	surface	-	0.290
Radius	: 01	curvatu	e of e	mitti	ine	surface		=	0.761

To eliminate the effect of slight errors in allignment, there is installed a pair of focuscing bars SS, whose potentials may be adjusted to converge or diverge the beam or swing it to right or left.

Because the intensity of the electric field at the emitting surface is no longer uniform in this design, space charge will build up unequally and thereby destroy the focussing. It was found that the beam emerging from the slit is divided into three major components, one very weak beam in the central plane and two of more than 100 times as great intensity at angles of about 15° to this plane on the two sides. The equipotentials can be reduced to approximately the shape that they would have in the case without space charge, and from 12% to 20% of the indicated total emission thrown into the central plane beam, by removing the emitting material from a space at the center of the cylindrical surface and placing in the central plane, between emitting surface and slit and therefore lower than the center of curvature of the cylinder, a wire T whose potential can be varied at will. Fig. 5 and 6 show the focussing action of the wire and of the bars. In addition, the usable amount of the total emission can be increased five times by removing the emitter from a strip at each edge of the curved surface.



Figure 5. -- Focussing effect on intensity of current to collector obtained by varying the potential on one focussing bar (S, Fig. 3) while keeping the other grounded.



Figure 6. -- Focussing effect on intensity of current to collector obtained by varying the potential of the wire T (Fig. 3).

For a source of the dimensions given above, the central dead strip is 13/32 inch wide and the dead strips at the edges are 1/8 inch wide. The total remaining area of emitting surface is then about 30 sq. cm. A one-mil tungsten wire was used. This design will be called Source B. It is the design finally adopted, so the details of construction will now be described.

The source block U (Fig. 4) was machined from an iron bar and bored with two holes for the heating elements. Thus the potential drop along the emitting surface is very small even when the bar is used to conduct the heating current. To dissipate the 2000 to 3000 watts radiated by this block, it is surrounded by a heavy water-jacketed box V of 1/4 inch copper. Helf-inch holes were bored in the sides of this box to permit evacuation, which, as will be shown below, is of great importance. The slit plate is water-cooled and is supported at the ends by four lavite pillars screwed to the corners of the main box. the plate being hung from metal brackets by screws which permit vertical adjustment. Two lavite cross-bars on the tops of these pillars support the focussing bars, which are of 3/8x7/8 inch brass. The hot source block rests on the ends of three screws, which pass vertically through brackets hung below the cover of the copper box, two screws being in the uninsulated front bracket and one in an insulating lavite block screwed to the rear bracket. One of the front screws rests in a hole in the bottom of the source block and one slides against the flat face, while the rear screw slides in a longitudinal flat-bottomed groove in this lower surface. Thus longitudinal expansion of the source-block is permitted, while it is held rigidly against lateral movement. The rectangular copper box, on which the whole of the assembly is supported, is clamped by four thumb-screws Q (Fig. 2) into a tripod frame, whose legs can be screwed in or out and fit into depressions in three lavite

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blocks P fixed in position on the floor of the main chamber H of the apparatus. By screwing the legs in or out the source assembly can be tilted as desired and by the thumb screws it can be moved laterally. The lavite of which all insulating supports are made consists of talc which has been baked three hours at 1150° C, after being machined.

The accelerating voltage is supplied by a 6000 volt, 20 k.w. generator, which is very steady, the heating current is drawn from the 110 volt mains through an insulating transformer, and the current for the magnet is obtained from storage batteries.

The accelerating voltage is controlled by placing a 65000 ohm potentiometer across the generator leads, and a further potentiometer of 550,000 ohms is connected between the end and sliding tap of the first. Included in this 550,000 ohms is the voltmeter by which the accelerating voltage is read, along it may be tapped off the botentials for the focussing wire and bars, and from its ends are taken the leads to the slit and emitter.

3) ALTERNATIVE METHODS OF FOCUSSING

Two other methods of producing a nearly plane beam of ions were investigated, and these will now be discussed.

a) Grid and single slit.

While the method of controlling space charge used in Source B was fairly satisfactory, it was felt that more efficiency might be obtained if the mechanical analogy were applied to a situation in which no space

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charge would be present even in the actual apparatus. If, for instance, a cylindrical grid were placed concentric with the emitting surface with an electric field between them, the field would be almost uniform over this surface, so that the equipotentials would also be concentric and the ions would emerge between the grid wires convergent toward a line focus. Some scattering would occur, but this is small when the spacing of the grid wires is small compared to the distance between grid and emitting surface. The ions emerging from the grid would then be concentrated into a plane parallel beam by means of another field existing between the grid and a rectangular slit placed above it.

The configuration of this slit-and-grid design was determined from the rubber membrane analogy by starting the steel balls normal to the boundary representing the grid with a constant velocity. This was accomplished by rolling the balls down a small grooved block, so that their horizontal velocity was always the same at the start and their direction could be controlled. The resulting design is similar to that shown in Fig. 7, except that grid and emitting surface are concentric. If V_1 represent the potential difference between emitter and grid and V_2 that between grid and slit, the design calls for $V_1 = 2V_2$.

Now this design assumes that there is no interpenetration of the rields through the grid. However, such penetration does take place with the result that the equipotentials below the grid are no longer concentric with the grid. The maximum efficiency was actually found at $V_{g}/V_{1} = 30$. Numerous modifications were tried, and in the final design the radius of curvature of the emitting surface was smaller than the radius of the grid, the dimensions being as follows. (Source G) A diagram of the source is shown in Fig. 7.

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Figure 7. -- Section of Source G.







Figure 9. -- Section of three-slit lens.

Radius of curvature of emitting surface	8	0.4 inch
Radius of curvature of grid	=	1.0
Distance, slit to edge of grid	8	0.250
Chord of grid	=	0.5
Chord of emitting surface	=	0.56
Distance, edge of grid to edge of em. surf.	-	0.125
Width of slit	-	0.375

This Source G gave an ion current into the pole-piece slot of 35 microamperes at 2500 volts when the source block was bright red, which amounts to 7% of the indicated total emission. The actual efficiency was probably somewhat higher than this, for the indicated positive current to the grid (65% of the total emission) is composed to a large extent of electrons going in the opposite direction. This is known from a series of measurements carried out by grounding the grid and making the slit (which also becomes coated with potassium) slightly positive. The slit current was reduced by 70% when the slit-potential was changed from 0 to +150 volts.

b) Electrostatic lenses of three slits.

In the last few years the technique and theory of focussing electron beams by electric and magnetic lenses have been rather thoroughly investigated.^{3 4} Electric lenses of three slits, the central plate at high potential and the outer pair grounded, offer another solution to the problem of focussing an ion beam, and I considered a scheme by which the ions would be converged toward a line focus with a grid concentric to the emitting surface and then focussed to a plane parallel beam by a three-slit lens.

Picht's theoretical treatment⁴ gives the following formula

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for the focal length of such a lens.

 $f = \frac{4\sqrt{E}}{\int \frac{\partial^2 4}{\partial x^2} \frac{dx}{\sqrt{E-A(x)}}}$

where E is the potential through which the incoming particle would have to have fallen to gain its velocity and $\beta(y)$ is the potential distribution along the axis of the lens. The potential distribution for a symmetrical three-slit lens (Fig. 9) was obtained by means of a Schwarz transformation from a distribution $W = \frac{2U_0}{T} \int_{C_1^2 - Z_1^2}^{C_1^2 - Z_1^2}$ (Fig. 8), and the above integral was evaluated for the case $a_1 \approx c_1$. The formula for the focal length f is the following.

 $\frac{1}{g} = \frac{(a_1^2 + \frac{2L_{a_1}}{T})(a_1 - c_1^2)}{gL^2 a_1^2} \left(\frac{U_0}{E}\right)^2 \left\{\frac{\pi}{a_1} - \frac{\pi}{\sqrt{a_1^2 + 2L_0}} + \frac{3U_0\sqrt{a_1^2 - c_1^2}}{\pi E} \frac{1}{a_1^2} + \frac{3U_0\sqrt{a_1^2 - c_1^2}}{\pi E}\right\}$ - 34 101-612 by 101+240 + 1240,) (0,+240)(260) ya2+260 - 5260,

Unfortunately the approximation on which the formula is based holds only for ions whose paths make angles of not more than one or two degrees with the central plane of the lens. The limiting angle of the converging ion beam must be at least 20° in the case of the present apparatus because of mechanical difficulties, and the focal length can be expected to agree with this formula only near the central plane. The three slit lens was set up on the rubber membrane analogy and did actually have something akin to spherical aberration, so that the observed focal length was very indefinite, although it agreed fairly well with the formula near the axis. This type of lens, therefore, is not suitable for the present arrangement of the apparatus, for, in order to have the emission from a large surface in the beam, it is necessary to have the beam converge over a large angle. If the convergence of the beam were somewhat less, the scheme might be applied.

4) FCCAL DEFECTS

As remarked above, focal defects of the magnetic lens itself were discussed to some length by Dr. Rumbauch, but I think it desirable to re-examine errors originating in the ion beam in the light of more recent information. Such errors arise from three causes, (1) action upon the ion beam by the magnetic field of the heating coils, (2) interaction of the ions with the other ions in the beam or with gas in the apparatus, and (3) thermal velocities at the emitting surface.

Consider first the magnetic field of the heaters. It is possible to calculate, as did Rumbaugh, an upper limit for the rield, for which the paths of the ions are displaced through a given angle bo. However, the real circuit differs so much in current path and in surrounding masses of iron from any simple calculable system that definite information must be obtained from the actual apparatus. Now resolution curves (collector current against mass number) taken with heating current on and off are the same in shape and peak width to the limit of observational accuracy. Furthermore, while the return circuit for the molybdenum wire heating coils now used was at first made through the hot block itself (with coils in parallel), it was found adequate to connect the coils in series with no current passing through the block. Although the current shells are not concentric in this last case, the longitudinal components of the currents are close enough together to have little resultant magnetic field.

Interaction of the ions with each other can be shown by simple calculation to be negligible with such a low current density as is used. Interaction with gas molecules, however, may become very important indeed. To be effective, encounters with gas molecules must take place

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within the accelerating field except in rare instances. If the ions and gas molecules are taken to be simple spheres attracting one another in passing, one may calculate that for the high gas pressure of 10^{-3} mm of mercury and with ions which have fallen through 2000 volts, less than 3% of the ions are shifted by more than 0.01 mass number at the collector. Gas within the accelerating field can widen the peak in the resolution curve by three times or more, making it impossible to resolve K⁴¹. It is for this reason that great care must be taken to secure good contacts on the heating elements.

Thermal velocities of ions at the hot surface of the source produce the largest aberration in this type of mass-spectrometer. From the kinetic theory of gases it can be shown that the component of velocity parallel to the accelerating field corresponds to an accelerating potential of no more than a few hundredths of a volt, and is therefore wholly negligible. Nevertheless, the component velocity normal to the field will cause the ions to emerge from the electric field at an angle $\theta = \tan^{-1} v_n / v_0$ to their assumed direction, v_n being the normal component of velocity and v_0 the velocity imparted by the electric field. Hence the ion with velocity $v_n=v$ must have a different radius of curvature in the magnetic field than the ion for which $v_n=0$, if it is to reach the collecting slit, and it will therefore be observed at an accelerating voltage corresponding to a different atomic weight. Thus the curve of ion current to the collector against atomic weight has such a spread as is seen in Fig. 10.

Let us consider the magnetic lens without fringing field and compute the path of an ion which starts at an angle θ with the vertical from a point $\mathbf{x}_0, \mathbf{y}_0$ in Fig. 1 and passes through the principal focus C of the lens. Designate the convex edge of the lens by the subscript **d**,

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the concave edge by β , the region from source to lens by 1, the region in the lens by 2, and the region from lens to collecting slit by 3. The equations of the edges of the lens are then

$$x_1^2 - 2rx_1 + y_1^2 - 2cy_1 = 0$$

 $x_1^2 + y_2^2 - 2cy_1 = 0$

and the three segments of the trajectory of the ion,

$$\theta(y_{1} + y_{0}) = (x_{1} - x_{0})$$
$$(x_{2} - p)^{2} + (y_{2} - q)^{2} = r_{2}^{2}$$
$$y_{3} = mx_{3} + c$$

The boundary conditions require the continuity of the path segments and their tangents at the boundaries \mathbf{d} and $\mathbf{\beta}$. These conditions give four equations from which can be obtained an equation for θ in terms of \mathbf{r}_{g} and \mathbf{x}_{0} . In deriving this, θ^{g} was neglected with respect to 1, which made the final equation of the second degree in θ_{\bullet}

The limiting values of x_0 are determined by the length and position of the source, and r_z is obtained from the electric potential V through which the ion has fallen and the strength of the magnetic field. Further, if M₀ represent the atomic weight corresponding to V=V₀ and θ =0 (radius of curvature of trajectory = r), then

$$\frac{\mathbf{r}_{2}}{\mathbf{r}} = \sqrt{\frac{\mathbf{V}(1+\theta^{2})}{\mathbf{V}_{0}}} = \sqrt{\frac{\mathbf{M}_{0}}{\mathbf{M}}} (1+\theta^{2}) = \sqrt{\frac{\mathbf{M}_{0}}{\mathbf{M}}} \text{ to } 0.1\%$$

Now by the Maxwell-Boltzman distribution law we have for the number dN of ions having a normal component of velocity between v_n and $v_n + dv_n$ $dN = a \exp(-\frac{mv^2}{2kT}) dv_n = a^*(M, x_0) \exp(-\frac{mv_0}{2kT}^2 \theta^2) dM dx_0 = a^* \exp(-\frac{Ve\theta^2}{kT}) dM dx_0$ a is a constant and one can compute $a^*(M, x_0)$ for each pair of values of M and x_0 to the necessary accuracy. The function $a^* \exp(-\frac{mv^2}{2kT}\theta^2)$ was integrated graphically over the range of x_0 and the resulting function of M (which represents the intensity of the ion beam at the collecting



Figure 11. -- Theoretical curve showing width of K³⁹ and K⁴¹ peaks to be expected if the lateral thermal velocity of the ions is the only cause of focal defect. Accelerating voltage and temperature are the same as for Fig. 10, whose experimental points are shown in circles.

slit) was plotted against M. (Fig. 11) The circled dots represent the experimental points taken at the same voltage and temperature and forming the curve shown in Fig. 10. One sees from Fig. 11 that the aberration due to the thermal velocities constitutes almost the entire focal defact of the mass-spectrometer. The curve is not a true Gaussian error curve, but is somewhat wider at the base.

The close approximation of experimental results to the computed curve furnishes proof that the edge correction of the magnetic lens is nearly perfect. The run shown in Fig. 10 was taken at a collector current of 0.02 m.a., a source temperature of 1100° K., a voltage of 2350 volts, and a pressure of $5x10^{-5}$ mm of mercury, measured on a Pirani gauge attatched at Y in Fig. 2.

5) EMITTING MATERIALS

The ion source described above requires a solid emitting material to whose surface ions are supplied continuously. This requirement is satisfied by the preparations known as Kunsman catalysts⁵ and to a lesser degree by certain natural alkali-aluminum silicates.

Our Kunsman catalyst for potassium was furnished by Dr. A. Keith Brewer of the Fixed Nitrogen Research Laboratory, where it was prepared in an electric furnace. It was found, bowever, that a Kunsman catalyst for lithium could be made by melting down ferric oxide with 15% of its mass of LiCO₃ in an almost closed vessel by means of an oxygen-gas flame. This process left in the melt the desired 3% of lithium. In either case the melt was ground fine and reduced for three hours at dull red heat in an atmosphere of hydrogen before being packed into the source block. Longer reduction had little effect on the copiousness of emission.

The potassium-aluminum silicate called leucite and the lithiumsluminum silicate called spodumene have the property of emitting the corresponding positive alkali ion when they are heated. Nevertheless, this emission was observed to be at the most somewhat lower than that obtainable with a Kunsman catalyst at the same temperature, and the emission diminished to a tenth of its original value when the field had been applied for an hour. One presumes that the ions do not diffuse as raidly to the surface in the natural silicates as in the Funsman catalysts.

The highest emission observed was 100. microamperes/cm² for potassium at 1000°C. and 7. microamperes/cm² for lithium at the same temperature, Kunsman catalyst being used in both cases.

6) HEATING ELEMENTS

That the heaters for the source block should be long-lived and rugged is necessary, but a still more important condition is that they must in no way render the apparatus difficult to outgas. The necessity of this condition is found in the fact, which was mentioned above, that gas produces its chief effect on the ion beam within the electric field, and in the mearness of the heaters to this field.

The simple Source A constructed by Rumbaugh used as a heater a Globar, which is a rod of carborundum held together with a binding substance, and the same kind of heater was used when Source B was built. Contact was made with the end of the Globar by means of an iron cup containing a small piece of aluminum, which was supposed to melt and perfect the contact between Globar and cup. It happens, however, that

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gas is evolved at these contacts, due apparently to the evaporation of the aluminum or the formation of its oxide, and the development of an arc. Other types of contacts were tried, but no decrease in the evolution of gas could be obtained except by the use of tin with the aluminum, and tin evaporates too readily and makes holes in the cups by alloying with the iron.

The trouble with gas ceased immediately when the Globars were replaced by coils of 35 mil molybdenum wire wound to a diameter of 8 mm. These are mounted **inside** cylinders of Stupakoff tubing, which in turn slide into the heater holes in the source block. Good contact is easily made with the ends of these coils, and they can be reised to 2000° C. before the vapor pressure of molybdenum becomes appreciable. The amount of material that absorbs gas when the apparatus is opened is no greater than when Globars are used, and the ruggedness of the assembly is much increased, for Globars are brittle and difficult to remove without breaking.

IV: METHODS OF COLLECTING

The ion beams of the separated isotopes converge at points in the focal plane of the spectrometer whose distances apart are approximately proportional to the separation in mass numbers. The distribution of intensity in the focal plane is then given by such a curve as Fig. 10, the distance between K^{39} and K^{41} being about 1 cm and that between Li⁶ and Li⁷ about 3 cm. Hence by placing a slit of suitable width in the focal plane one can select any portion of this i tensity curve. Behind this slit is placed a cup, usually circular, upon which the atoms are to deposit. For the atoms actually to deposit, three conditions must be satisfied, (1) specular reflection must not occur, (2) the incident atoms must not have sufficient energy to dislodge those already collected, and (3) the cup must remain cool enough so that the atoms will not sublime off. The f² st two conditions can be fairly well satisfied by placing a grid due to the slit and applying a retarding field between grid and cup so that the ions strike with a relocity equivalent to a fall through only a few hundred volts. Whether the third condition is satisfied with this arrangement and with the cup at room temperature is not clear. The energy carried to the cup is small and should be easily dissipated, but probably the cup should be cooled with liquid air in order to collect all of the incident atoms.

The receiving assembly is shown at the upper left of Fig. 2. To find the best focus the cylindrical box which carries the slits at its lower end slides in two rings and is moved in or out by the screw W, which is rendered vacuum-tight by running beeswax around its head. The brass rods carrying the collecting cups are supported by the two insulating plugs shown. Each plug is a double cone of redmanol with a metallic sheath between the cones to shunt leakage current around the measuring instrument.

The samples of Li⁶ and Li⁷ were collected on platinum foil placed at the bottom of a shallow cup. The foil was subsequently treated with HF fumes and warmed gently, whereupon the bluish discoloration of the deposit became white. The quantity of each isotope was about one microgram.

The quantity of material deposited was estimated from the ion current to the cup, measured with a high resistance and electrometer

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or with a microammeter. In actual collecting one slit was always kept marrow (0.5 mm) to check the resolution from time to time.

V. THE RADIOACTIVITY OF POTASSIUM

It was discovered by Campbell and Wood⁶ in 1906 that potassium possesses a A-radioactivity not traceable to impurities of members of known radioactive series, and Kohlhorster⁷ in 1928 found a Y-radiation of hardness slightly less than that of the hard T-rays of RaC. Confirmation and extension of such results⁸ have made it of great interest to workers in the field of nuclear physics to know which is tope of potassium, if either, is the radioactive one. Von Hevesy⁹ in 1927 by differential distillation obtained potassium with an atomic weight 0.005 ± 0.001 units greater than that of ordinary potassium, and from measurement of the radioactivity ascribed it to the isotope K⁴¹. No other work appears to have been done on this subject.

I collected several samples of K⁴¹ of about half a milligram each, and Mr. W. W. Harper attempted to determine the radioactivity of these by means of a Geiger-Muller ion counter. However, the brass discs, on which the potassium was deposited, easily became contaminated with other radioactive material, and we constructed the following apparatus to make the measurements without possibility of such contamination.

A new collecting head was built for the mass-spectrometer as shown in Fig. 12. In this we fixed the slits permanently in the position discovered with the adjustable head to be the best. The lower slit S_g is narrow to observe the resolution and the upper stit S_1 is wide so that the isotope desired can be deposited on a cup carried on





Figure 12. -- Collector head to test the radioactivity of potassium (much simplified). At the upper left is seen the slit system which faces the magnetic lens, at the upper right a sectional side view, and below the top view.

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the circumfirence of the wheel A. On this wheel are mounted a collecting oup and a cup containing KCl, the two being separated by aluminum shields. The wheel is rotated by a knob B to bring the desired cup in front of the wide slit or the Geiger counter C, which is separated from the highly evacuated main chamber by a window of one-mil aluminum. A glass window is provided to permit observation of the cups. The shaft for the wheel A and the support for the lower cup are insulated from the case by blocks D of redmanol. The tests on this apparatus are now in progress.

VI. CONCLUSION

The collection of lithium isotopes for nuclear disintegration work is to be undertaken immediately with a collector head containing a cup cooled with liquid eir, this head now being under construction. Also to be constructed is an apparatus by which a potassium sample can be introduced directly into the chamber of a Geiger counter. Other possible applications of the mass-spectrometer have been adequately discussed elsewhere.²

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REFERENCES

1. G. Hertz --- Zeit. fur Phys., 79, 108, 1932. Naturwiss., 21, 884, 1933. 2. L. H. Rumbaugh --- 'Development of a High-Intensity Magnetic-Lens Type of Mass-spectrometer', Ph.D. Thesis, California Institute of Technology, 1932. 3. Davisson and Calbrick --- Phys. Rev., 38, 585, 1931, and 42, 580, 1932. Busch --- Ann. d. Phys., 81, 974, 1926. A. f. Elekt., 18, 583, 1927. Knoll and Ruska --- Zeit. fur Tech. Phys., 12, 389, 1931. Ang. d. Phys., 12, 607-660, 1932. Zeit. f. Phys., 78, 318-340, 1932. Knoll, Houtermans, and Schulze --- Zeit. fur Phys, 78, 339-360, 1932. Bruche and Johannson --- Naturwiss., 20, 49 and 353, 1932. Ann. d. Phys., 15, 145-166, 1932. Phys. Zeit., 33, 898. 4. Picht --- Ann. d. Phys, 15, 926-964, 1932. 5. Kunsman, Harnwell, and Parton --- Phys. Rev., 27, 737, 1926. 6. Campbell and Wood --- Proc. Camb. Phil. Soc., 14, 15, 1906. 7. Kohlhorster --- Naturwiss., 16, 28, 1928. 8. Rutherford, Chadwick, and Ellis, 'Radiations from Radioactive Substances' See references attatched to section on radioactivity of potassium and rubidium. 9. Hevesy --- Nature, 120, 838, 1927.