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ION-RETARDING LENS TO IMPROVE THE
ABUNDANCE SENSITIVITY OF
TANDEM MASS SPECTROMETERS

by

Kurt A. Kaiser and
Charles M. Stevens

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Printed in the United States of America

Available from

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Price: Printed Copy \$3.00; Microfiche \$0.65

ANL-7393
Engineering and Equipment
(TID-4500)
AEC Research and
Development Report

ARGONNE NATIONAL LABORATORY
9700 South Cass Avenue
Argonne, Illinois 60439

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Chemistry Division

November 1967

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ION-RETARDING LENS TO IMPROVE THE ABUNDANCE SENSITIVITY OF TANDEM MASS SPECTROMETERS*

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ABSTRACT

This report describes the design, construction, and performance of a retarding lens used to increase the abundance sensitivity of tandem magnetic-analyzer mass spectrometers. Under typical operating conditions in a 30-cm-radius, two-stage system, the abundance sensitivity improved by a factor of about 10 on the high-mass side and about 10^3 on the low-mass side.

I. INTRODUCTION

A persistent problem in mass spectroscopy is the measurement of isotopes of very low abundance in mass positions adjacent to an isotope of high abundance. When the mass spectrum is scanned, "tails" are detected on either side of the mass position of the high-abundance isotope, and these tails are frequently larger than the low-abundance isotopes we may wish to measure. In curves A and B in Fig. 1, the "tails" result from collisions between the ions of the high-abundance isotope beam and residual gas molecules in the instrument. Generally in these collisions the ion is deflected and loses energy, and in some instances may transfer its charge

Fig. 1

The Fractional Number of Ions of Mass M Detected in the Focal Plane, Plotted against the Distance (in mass units) Away from the Mass M Position. Curves A and B are for a single magnetic analyzer, C and D are for two magnetic analyzers in tandem, and E and F are for two magnetic analyzers in tandem plus the retarding lens. The curves are typical results obtained with 30-cm-radius analyzers for elements with masses in the range 170-250. The pressure in the instruments was about 2×10^{-7} Torr. The source slit was 0.2 mm, and the defining slit was 0.45 mm.

* After this manuscript was prepared, a paper by N. J. Freeman, N. R. Daly, and R. E. Powell, describing the use of a similar lens on a single-stage mass spectrometer, appeared in *Rev. Sci. Instr.* **38**, 945 (1967). See also *J. Sci. Instr.* **44**, 956 (1967).

to the neutral gas molecule. Calculations¹⁻³ and observed spectra show that the "tails" fall off logarithmically and extend over several mass units.

One method that has been used to reduce these "tails" employs a mass spectrometer containing two magnetic analyzers in tandem^{4,5} as in Fig. 2. Reanalyzing the beam from a single stage produces a substantial decrease in the "tail" on the high-mass side (curve D in Fig. 1). However, on the low-mass side, some scattered ions, which have lost energy, still pass through the defining slit as shown in Fig. 2. The resulting "tail" (curve C in Fig. 1) is unfortunately only slightly improved over that for the single stage (curve A in Fig. 1). The most common way to further decrease these "tails" or increase the abundance sensitivity* has been to add a complex and costly electrostatic analyzer as a third stage.⁶

Fig. 2

Mass Spectrometer with Two Magnetic Analyzers in Tandem. At the focal plane, the scattered beams are spread out from their nominal mass towards lower masses. Thus scattered ions of isotopes with mass greater than that of the one collected will enter the defining slit.

This report describes a simple and relatively inexpensive retarding lens, which greatly increases the abundance sensitivity for those isotopes that lie farther from the high-abundance isotope than the energy cutoff of the lens. The lens is a high-pass energy filter with a cutoff of about 40 volts below 15,000 volts which is equivalent to about two-thirds of a mass unit at mass 250. Although retarding plates have been used in mass spectrometers before,^{7,8} it has not been appreciated that a lens as described in this report would work so effectively in increasing abundance sensitivity. For the measurement of low-abundance isotopes (as close as $\pm$ one mass unit at mass 250), the lens performs as well as systems employing an electrostatic analyzer, although the lens does not have the analyzer's energy-focusing property, which may be useful in some circumstances.

* Abundance sensitivity is defined as the reciprocal of the fractional number of ions of mass M which are detected in the focal plane $\pm$ one mass unit away from M (i.e., at positions $M \pm 1$).

II. THE LENS

The lens described in this report is of an einzel or unipotential type with thick center electrodes. The lens-focusing properties were derived from a two-dimensional potential field with an axis of symmetry, by numerically integrating the paraxial trajectory equation,

$$r'' + \frac{[1 + (r')^2][r'V_0' + rV_0'']}{2V_0} = 0,$$

where

r is the radial coordinate,

$' = d/dx$,

x is the axial coordinate,

and

V_0 is the axial potential $= V(r, x)|_{r=0}$.

The axial potential, V_0 , was measured from a conducting-paper analog of the lens. V_0' and V_0'' were calculated numerically from V_0 . The lens electrodes are very wide in the z direction (i.e., perpendicular to the r, x plane); thus the z component of the ions motion is unaffected by the lens.

The lens is placed between the spectrometer defining slit and the detector, the defining slit becoming part of the first lens electrode (Fig. 3). In this position, the lens does not affect the instrument optics. A schematic drawing of the lens is shown in Fig. 4, and a section drawing in Fig. 5. For an ion-accelerating voltage of 15 kV and about 40 volts below 15 kV, or 14,960 volts on the center electrodes, the lens focuses on the detector, which is 8 cm away. This lens, which operates at 15 kV, is only 6 cm long and consequently was readily installed in the existing collector system.

Fig. 3. Tandem Mass Spectrometer with the Retarding Lens in Position

Fig. 4. Schematic Diagram of Retarding Lens. Dimensions are in centimeters.

Fig. 5. Section Drawing of Retarding Lens. All parts are of Pyrex or Type 304 stainless steel.

The normal construction procedures used for any high-voltage apparatus were followed. Of most importance were: (1) rounding all corners and edges that "see" the high voltage, (2) electropolishing all surfaces that "see" the high voltage, and (3) thoroughly ion-cleaning* the lens before use. Some arcing occurred when the high voltage was applied to the lens for the first time, but after high-vacuum conditioning (about 3 weeks at 10^{-7} Torr with occasional bakeouts), the frequency of arcing was reduced to about one per day.

III. RESULTS

The performance of this lens was determined by (1) applying the focus voltage to the center electrodes to turn the lens on, or (2) grounding the lens center electrodes, thereby effectively turning the lens off. These steps were performed alternately while holding the other instrument parameters constant to give the comparisons that follow. In Fig. 1, curves C and D show typical scattering "tails" for a two-stage machine with the lens off, and curves E and F show the "tails" with the lens on. The high-mass "tail" is lowered (curve F); the low-mass "tail" (curve E) follows curve C until the lens cutoff voltage is reached and then drops suddenly to very low values.

Figures 6 and 7 show tracings of actual recorder charts taken during mass scans of rhenium and uranium. Figure 6 shows the improvement in the definition of a 1.5-ppm W^{186} impurity peak in the valley between the normal rhenium peaks at masses 185 and 187. Figure 7 shows a spectrum of what was believed to be normal uranium until we turned the lens on and found the U^{236} impurity at an abundance of about 5×10^{-8} .

*The procedure given in Ref. 9 was followed.

Fig. 6. Mass Spectrum of Normal Rhenium Containing a Small W^{186} Impurity
(a) Without Lens (b) With Lens

Fig. 7. Mass Spectrum of Normal Uranium Containing a Small U^{236} Impurity (a) Without Lens (b) With Lens

The aberrations of the lens for ion trajectories far from the optic axis become large, thereby destroying the focusing properties. This can happen if the collector defining slit is greater than about 1.3 mm. Since narrowing the defining slit improves the abundance sensitivity, we generally use a relatively narrow defining slit so that these aberrations are not a problem.

REFERENCES

1. G. M. Roe, Appendix to Knolls Atomic Power Laboratory Report, KAPL-873 (Rev) (1952).
2. K. A. Kaiser, internal report, University of Minnesota (1961).
3. M. Menat, Can. J. Phys. 42, 164 (1964).
4. M. G. Inghram and D. C. Hess, reported in M. G. Inghram and R. J. Hayden, Handbook on Mass Spectroscopy, National Research Council, Nuclear Science Report No. 14, 24 (1954).
5. F. A. White and T. L. Collins, Appl. Spectry. 8, 169 (1954).
6. F. A. White, F. M. Rourke, and J. C. Sheffield, Appl. Spectry. 12, 46 (1958).
7. J. A. Hipple, R. E. Fox, and E. U. Condon, Phys. Rev. 69, 347 (1946).
8. A. O. Nier, C. M. Stevens, et al., J. Appl. Phys. 18, 30 (1947).
9. N. R. Daly, Rev. Sci. Instr. 34, 1116 (1963).