

Radiations from Cd^{115} , $^*\text{In}^{115}$, and $\text{Hf}^{181}\dagger$ C. E. MANDEVILLE, M. V. SCHERB,^{††} AND W. B. KEIGHTON^{†††}
Bartol Research Foundation of the Franklin Institute, Swarthmore, Pennsylvania

(Received October 8, 1948)

The characteristic radiations of Cd^{115} , $^*\text{In}^{115}$, and Hf^{181} have been measured by absorption and coincidence methods. The beta-gamma-coincidence rate of Cd^{115} indicates the presence of an inner beta-ray group at 0.46 Mev. Coincidence measurements show that the 4-hour isomer of indium (115) decays with the emission of a single partially converted gamma-ray. Hf^{181} was found to emit 0.52 Mev beta-rays (aluminum absorption) and 0.17- and 0.47-Mev gamma-rays (lead absorption). Beta-gamma-coincidence data indicate that the beta-ray spectrum of Hf^{181} could be complex. Beta-beta- and gamma-gamma-coincidence data were also obtained.

INTRODUCTION

THE disintegration schemes of Cd^{115} , $^*\text{In}^{115}$, and Hf^{181} have been investigated by absorption and coincidence methods which have been previously described.¹

The 2.5-day period of Cd^{115} and its 4-hour daughter activity, $^*\text{In}^{115}$, were induced in metallic cadmium irradiated by slow neutrons in the Oak Ridge pile. The time of exposure was only one hour, so that little of the 43-day cadmium was present. Chemical separations were carried out for the removal of iron, calcium, and phosphorous as possible impurities. All measurements on the cadmium fraction were completed within four days after removal of the irradiated cadmium from the pile.

The 46-day period was induced in Hf_2O_3 by pile neutrons. Chemical separations should have removed any Cu, Fe, Ag, Ni, Pb, Sn, and Zn. A spectrographic analysis indicated that zirconium was present with an abundance of two-tenths of one percent by weight. Since the chemistry of zirconium is identical with that of hafnium, the elements could not be separated. However, the characteristic radiations and coincidence rates of Zr^{95} have been previously investigated in this laboratory.² Those data show that the present measurements on Hf^{181} are uninfluenced by the presence of any zirconium.

[†] Assisted by the Joint Program of the Office of Naval Research and the Atomic Energy Commission.

^{††} Now at Princeton University.

^{†††} Also of the Department of Chemistry, Swarthmore College.

¹ C. E. Mandeville and M. V. Scherb, Phys. Rev. **73**, 143 (1948).

² C. E. Mandeville and M. V. Scherb, Phys. Rev. **73**, 1434 (1948).

Cd^{115}

The energies of the beta-rays and gamma-rays of Cd^{115} and its daughter element, the 4-hour isomer of indium (115), have been previously studied in considerable detail.³⁻⁶ The beta-rays have been reported to have an energy as high as 1.25 Mev.⁶ Lawson and Cork⁴ have found a maximum beta-ray energy of 1.13 ± 0.03 Mev for the beta-rays of Cd^{115} . Their spectrogram also gave evidence of an inner spectrum having a maximum energy of about 0.6 Mev. Those au-

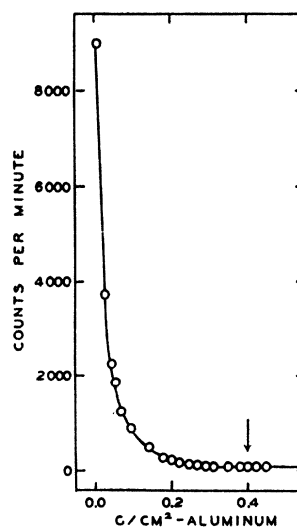


FIG. 1. Absorption in aluminum of the beta-rays of Cd^{115} .

³ J. M. Cork and J. L. Lawson, Phys. Rev. **56**, 241 (1939).

⁴ J. L. Lawson and J. M. Cork, Phys. Rev. **57**, 982 (1940). References 3 and 4 contain a complete list of all papers published in 1940 and prior to that year relating to Cd^{115} and In^{115} .

⁵ C. E. Mandeville and H. W. Fulbright, Phys. Rev. **64**, 265 (1943).

⁶ R. P. Metcalf, MDDC-614GG.

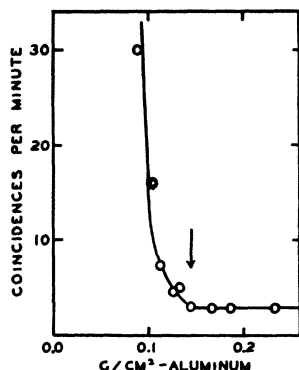


FIG. 2. Coincidence absorption of the Compton electrons of the gamma-rays of Cd^{115} .

thors also reported a gamma-ray energy of 0.54 Mev, measured by lead absorption. Mandeville and Fulbright⁵ have reported 0.65 Mev as the energy of this gamma-ray.

The beta-ray and gamma-ray energies of Cd^{115} have been remeasured in the course of the present experiments. An absorption curve in aluminum of the beta-rays of the 2.5-day cadmium is shown in Fig. 1. The end point shown at 0.40 g/cm² in aluminum corresponds to a maximum energy of 1.03 Mev.

A coincidence absorption experiment gave the curve of Fig. 2. The end point of the Compton recoils corresponds to a quantum energy of the order of 0.6 Mev. This estimate is not particularly accurate, since the calibration of the coincidence counting set used in the coincidence ab-

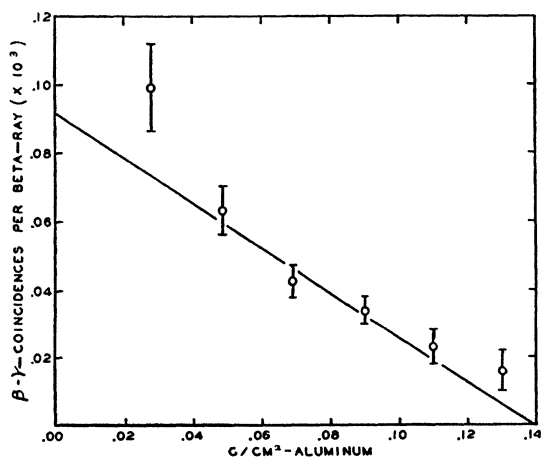


FIG. 3. The beta-gamma-coincidence rate of Cd^{115} as a function of the surface density of aluminum placed before the beta-ray counter.

sorption measurements has not been extended below 0.75 Mev.²

The curves of Figs. 1 and 2 do indicate that after chemical separation the cadmium was free of impurities.

A source of Cd^{115} , freshly separated from the 4-hour indium daughter activity, was placed between two counters in coincidence (resolving time 0.10 microsecond), and beta-gamma-coincidences were observed as a function of the surface density of the aluminum placed before the beta-ray counter. The data thus obtained are plotted in Fig. 3, where the beta-gamma-coincidence rate is seen to decrease from an extrapolated value of 0.093×10^{-3} coincidence per beta-ray at zero absorber thickness to zero at 0.14 g/cm², corresponding to a beta-ray energy of 0.46 Mev.⁷ The level diagram given by Lawson and Cork⁴ is thus confirmed.†

A small gamma-gamma-coincidence rate, 0.08×10^{-3} coincidence per gamma-ray recorded in the gamma-ray counter, was measured, suggesting cascade emission of gamma-rays in the cadmium fraction. No gamma-rays, other than the 0.54-Mev gamma-ray, have been reported. A search was made for beta-beta-coincidences, but none were found in the cadmium fraction.

* In^{115}

Absorption and coincidence measurements were carried out on the conversion electrons of

⁷ L. E. Glendenin, *Nucleonics* 2, No. 1, 12 (1948).

† In a note added in proof in their 1940 paper, Lawson and Cork remark that the beta-gamma-coincidences shown in Fig. 3 of this paper were not observed by Mitchell and Langer of Indiana University.

Although no data are given in the note concerning the value of the coincidence resolving time of their circuit, the published literature of that period indicates that a resolving time of about 3.2×10^{-6} second was employed by the Indiana group. It seems probable that for almost any reasonable counting rates in either counter, the relatively small beta-gamma-coincidence rate of Cd^{115} would be partially obscured by accidentals. A considerable advantage was held by the writers in that the coincidence resolving time of their circuit was only 0.10×10^{-6} second.

Calibration of the gamma-ray counter gave a beta-gamma-coincidence rate of 2.61×10^{-3} coincidence per beta-ray for Sc^{46} . Were each beta-ray of Cd^{115} followed by the 0.54 Mev gamma-ray, the coincidence rate would have been 0.7×10^{-3} coincidence per beta-ray. Thus, 0.093×10^{-3} coincidence per beta-ray at zero absorber thickness suggests that the soft spectrum has an intensity about one eighth as great as that of the 1.03 Mev beta-rays. This is a lower limit of the intensity, since no correction has been made for the fact that the soft spectrum is more heavily absorbed and scattered in the thin window of the beta-ray counter.

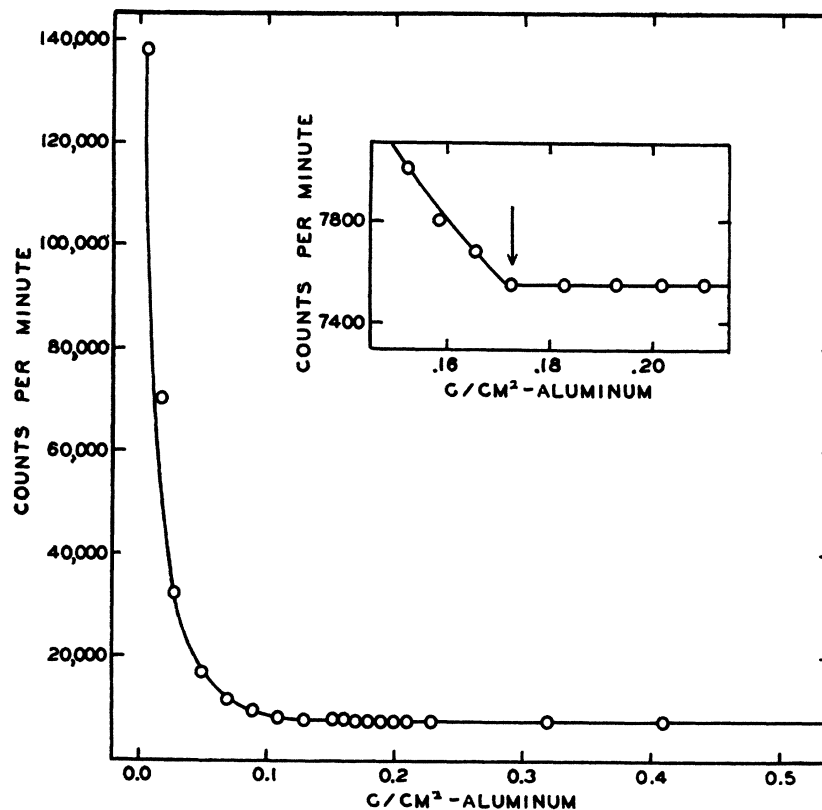


FIG. 4. Absorption in aluminum of the beta-rays of Hf¹⁸¹.

the 4-hour *In¹¹⁵, separated from its cadmium parent. Conversion electron-gamma- and gamma-gamma-coincidences were observed, but all coincidence rates could be accounted for by the

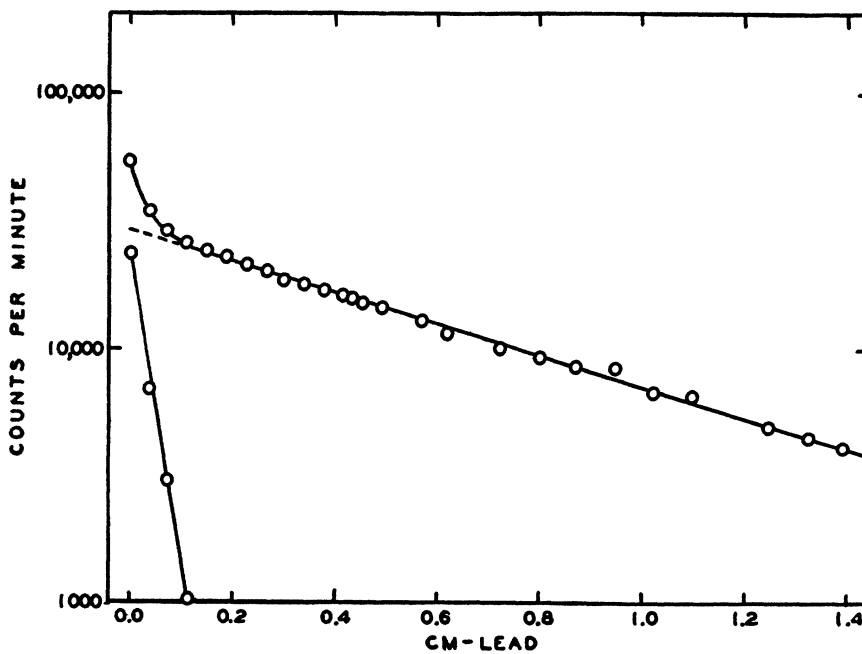


FIG. 5. Absorption in lead of the gamma-rays of Hf¹⁸¹.

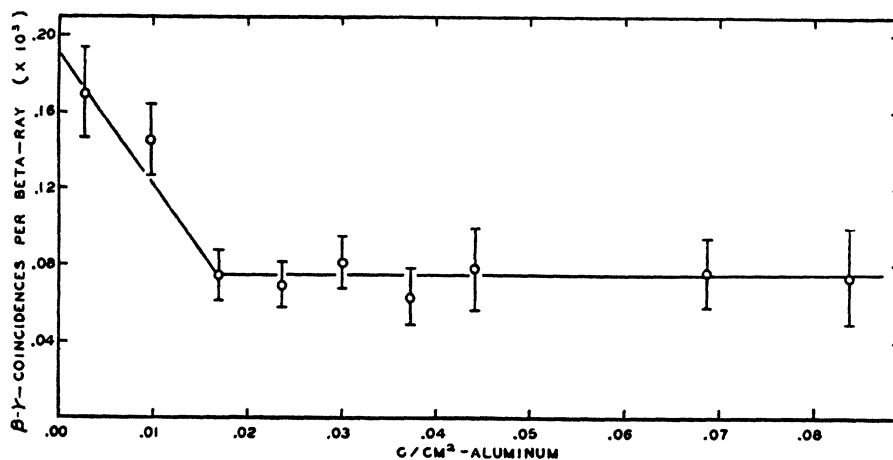


FIG. 6. The beta-gamma-coincidence rate of Hf^{181} as a function of the surface density of aluminum placed before the beta-ray counter.

presence of conversion electron-(x-ray) coincidences and (x-ray)-(x-ray) coincidences. It was concluded that the 4-hour level decays with the emission of a single partially converted gamma-ray and its associated x-rays. Lawson and Cork⁴ have given a spectrometric energy value of 0.338 Mev for this gamma-ray.

Hf^{181}

The radiations of the 46-day hafnium (181) have been previously investigated by DeBenedetti and McGowan,⁸ Wiedenbeck and Chu,⁹

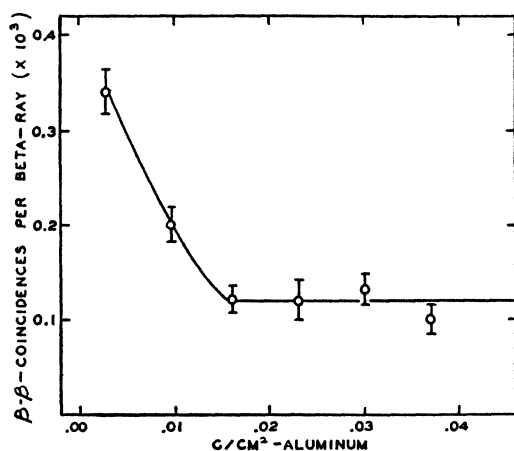


FIG. 7. Beta-beta coincidence rate of Hf^{181} . The aluminum absorbers were placed before both beta-ray counters.

⁸ S. DeBenedetti and F. K. McGowan, *Phys. Rev.* **70**, 569 (1946).

⁹ M. L. Wiedenbeck and K. Y. Chu, *Phys. Rev.* **72**, 1164 (1947).

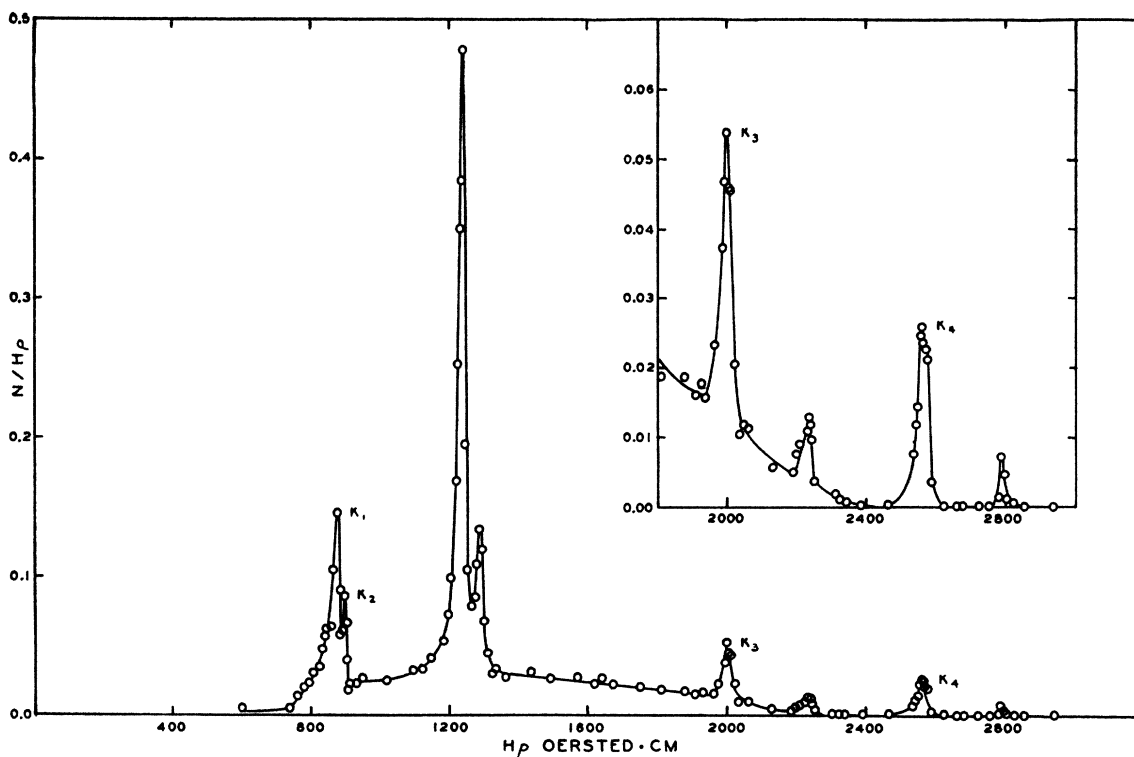
Voigt and Thamer,¹⁰ and by Beněš, Ghosh, Hedgran, and Hole.¹¹

The energy of the beta-rays of Hf^{181} , absorbed in aluminum as shown in Fig. 4, was found to be 0.52 Mev. Absorption in lead of the gamma-rays gave two components having energies of 0.17 and 0.47 Mev. These data are given in Fig. 5. Spectrometric measurements¹⁰⁻¹² have shown gamma-rays present having energies of 0.128, 0.342, and 0.472 Mev. The beta-gamma-coincidence rate as a function of aluminum absorber thickness inserted before the beta-ray counter is shown in Fig. 6. From the curve, it is clear that the beta-gamma-coincidence rate decreases from an extrapolated value of 0.192×10^{-3} coincidence per beta-ray at zero absorber thickness to 0.075×10^{-3} coincidence per beta-ray at 0.017 g/cm² or 0.11 Mev⁵ and remains constant thereafter. The curve of Fig. 6 was taken with a coincidence resolving time of 0.10 microsecond. On increasing the resolving time to one microsecond, it was noted that the curve remained unchanged in shape and absolute magnitude. It was very difficult to attain any high statistical accuracy beyond 0.05 g/cm², because the relatively strong gamma-gamma-coincidence rate begins to interfere with the beta-gamma-meas-

¹⁰ A. F. Voigt and B. J. Thamer, *Phys. Rev.* **74**, 1264 (1948).

¹¹ J. Beněš, A. Ghosh, A. Hedgran, and N. Hole, *Nature* **162**, 262 (1948).

¹² J. M. Cork, R. G. Shreffler, and C. M. Fowler, *Phys. Rev.* **72**, 1209 (1947).

FIG. 8. Beta-ray spectrum of Hf^{181} .

urements. The curve of Fig. 6 shows that the beta-ray spectrum of Hf^{181} may be complex.

The beta-beta-coincidence rate measured in two "bubble" counters (bubble thickness 2.5 mg/cm²) is plotted in Fig. 7. The coincidence rate did not decrease to zero but remained roughly constant beyond 0.016 g/cm². This was interpreted as showing that some of the higher energy gamma-rays are to some extent converted. As in the case of the beta-gamma-coincidence rate of Fig. 6, the beta-beta-coincidence rate of Fig. 7 remained unchanged in shape and absolute magnitude when the resolving time was changed from 0.10 microsecond to 1.0 microsecond.

At this point, it should be remarked that Wiedenbeck and Chu⁹ have published a beta-gamma-coincidence curve for Hf^{181} which rises from a relatively low value at zero absorber thickness to a plateau in the vicinity of 0.05 g/cm². Since their data differed so markedly from those of Fig. 6, the writers communicated to them their results and learned that they had independently resumed their investigation of Hf^{181} . They have now obtained a beta-gamma-

curve substantially in agreement with the measurements of Fig. 6. Their newly obtained data on Hf^{181} are given in the paper following this one. They have also kindly observed the beta-ray spectrum and associated conversion lines of Hf^{181} in their spectrometer, using a portion of the material purified and used by the writers. The spectrogram is shown in Fig. 8. Since it is identical with that of their sample, the purity of the materials used by both groups of investigators is confirmed.

APPENDIX I

Chemical Procedure for the Purification of Cadmium

One gram of irradiated cadmium metal with about 50 mg of Ag, 50 mg of Pb, and 50 mg of Cu, added as carriers for possible impurities in the cadmium, were dissolved in nitric acid and the solution was diluted with water. The silver was precipitated and filtered out by adding NH_4Cl . The sulfides of lead, copper, and cadmium were precipitated with H_2S from a solution 0.3N in H. The filtrate, which would contain any In, Fe, Ca, or P, was rejected.

Cadmium sulfide was dissolved from the precipitate by dilute (1:2) HCl, and the solution was evaporated with

H₂SO₄, diluted with water, and allowed to stand to separate out lead as PbSO₄. After filtering out the PbSO₄, about 50 mg of indium, dissolved in H₂SO₄, was added as a carrier, and the acidity was adjusted to 1*N* in H₂SO₄. Cadmium sulfide was precipitated from this solution by H₂S, washed with water, and dried at 105°C.

APPENDIX II

Separation of Indium from the Purified Cadmium

After completion of the measurements on CdS, this same precipitate was dissolved in HCl; 30 mg of indium was added, and the acid concentration was adjusted to 0.3*N*. Cadmium sulfide was precipitated with H₂S, removed by filtration, and the acidity of the filtrate was adjusted to 0.05*N*. Indium sulfide was then precipitated with H₂S, washed with water, and dissolved in HNO₃. From this solution, In(OH)₃ was precipitated with an excess of ammonia. The precipitate was washed with a 2 percent NH₄NO₃ solution, dried, and ignited to In₂O₃.

APPENDIX III

Chemical Procedure for the Purification of Hafnium

Three hundred milligrams of Hf₂O₃ were fused with borax, and the melt was dissolved in 2*N*-HCl. The hy-

droxide was precipitated with ammonia in slight excess and washed with water. After dissolving the precipitate in *N*-H₂SO₄, about 20 mg of copper (as a carrier for Ag, Sn, Pb, and Cu) and about 20 mg of iron (as a carrier for Fe, Ni, and Zn) were added in solution. The solution was diluted to 0.3*N*, and H₂S was passed into the cold solution until the copper was completely precipitated.

Tartaric acid was added to prevent precipitation of hafnium and, without filtering, the solution was made alkaline, with ammonia and saturated with H₂S, until the iron was precipitated. This precipitate was filtered out and discarded. After neutralizing the filtrate with H₂SO₄, 10 percent by volume was added in excess, and the H₂S was boiled off. About 20 mg of erbium were added, in H₂SO₄ solution, as a carrier for rare earths. Hafnium was precipitated from the cold solution by cupferron reagent; the precipitate was washed with *N*-HCl and ignited to the oxide. This was dissolved in hydrofluoric acid, evaporated to a small volume, and diluted with ten volumes of water. The hafnium, in solution, was separated by decantation and filtration from carbon and fluorides of the rare earths.

To the filtrate containing the hafnium was added a few milliliters of H₂SO₄, after which the solution was evaporated to expel HF. Water and H₂SO₄ were added to the solution to bring the volume to 300 ml and the acid concentration to 10 percent by volume. Hafnium was again precipitated with cupferron. After washing, the precipitate was ignited to HfO₂.

The Radiations from Hf¹⁸¹

K. Y. CHU* AND M. L. WIEDENBECK

Department of Physics, University of Michigan, Ann Arbor, Michigan

(Received October 8, 1948)

The beta-spectrum and photoelectron spectrum of Hf¹⁸¹ have been studied with a semicircular focusing beta-ray spectrograph of 2 percent resolving power. In addition to the continuous beta-spectrum, four partially converted gamma-rays were found. An estimate of the relative intensities of the two higher energy gamma-rays can be made from the Compton and photoelectron intensities. Data have also been obtained from β - γ - and β - e^- -coincidence measurements. An energy level diagram is discussed.

INTRODUCTION

THE beta- and conversion electron spectrum of Hf¹⁸¹ has previously been studied by Cork, Shreffler, and Fowler,¹ Voigt and Thamer,² and by Beněš, Ghosh, Hedgran, and Hole.³ Coin-

cidence work on the radiations of Hf¹⁸¹ is also discussed in the preceding paper by Mandeville, Scherb, and Keighton.

BETA-SPECTRUM OF Hf¹⁸¹

We have studied the beta-spectrum of Hf¹⁸¹, using a semicircular focusing spectrograph of 2 percent resolving power. The spectrograph was calibrated from the 0.400-Mev line⁴ of Au¹⁹⁸. The

* Rackham Predoctoral Fellow, 1948-49.

¹ J. M. Cork, R. G. Shreffler, and C. M. Fowler, *Phys. Rev.* **72**, 1209 (1947).

² A. F. Voigt and B. J. Thamer, *Phys. Rev.* **74**, 1254 (1948).

³ J. Beněš, A. Ghosh, A. Hedgran, and N. Hole, *Nature* **162**, 261 (1948).

⁴ K. Siegbahn, *Proc. Roy. Soc.* **187**, 527 (1947).