A Study of the Radiations from Iridium (194), Iridium (192), Lanthanum (140), Antimony (124), and Zirconium (95)*

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The beta- and gamma-radiations of radioactive isotopes of iridium, lanthanum, antimony, and zirconium, prepared by slow neutron bombardment in the Clinton pile, have been investigated by absorption and coincidence methods. The results are given in summary below.

(1) Iridium (194): The beta-gamma coincidence rate decreases from 0.06×10^{-3} coincidence per beta-ray at zero absorber thickness to zero at 0.150 g/cm², corresponding to 0.48 Mev, indicating a soft beta-ray spectrum of low intensity, and that the hard beta-rays of energy 2.2 Mev are non-coincident with gamma-radiation. The gamma-gamma coincidence rate is $(0.54\pm0.05)\times10^{-3}$ coincidence per gamma-ray, indicative of cascade emission of gamma-rays in the nineteen-hour period. Coincidence absorption experiments revealed the presence of a gamma-ray of quantum energy 1.43 Mev. The momentum distribution of secondary electrons ejected from a thick aluminum radiator in a magnetic spectrograph gives an energy of 1.35 ± 0.03 Mev. A disintegration scheme is given for Ir^{194}.

(2) Iridium (192): The beta-rays have a maximum energy of 0.56 Mev as measured by absorption in aluminum and Feather analysis. The beta-gamma coincidence rate is 0.41×10^{-3} coincidence per beta-ray, independent of the beta-ray energy, suggesting a simple spectrum. The gamma-gamma coincidence rate is $(0.23\pm0.01)\times10^{-3}$ coincidence per gamma-ray recorded in the gamma-ray counter. The coincidence experiments show that on the average, each beta-ray is followed by 0.6 Mev of gamma-radiation.

(3) Lanthanum (140): The beta-gamma coincidence rate is 1.63×10^{-3} coincidence per beta-ray and seems to be independent of the beta-ray energy. A strong gamma-gamma coincidence rate, $(0.85\pm0.03)\times10^{-3}$ coincidence per gamma-ray, was also observed. The coincidence experiments show that gamma-rays are emitted in cascade and that 2.3 Mev of gamma-radiation follow each beta-ray.

(4) Antimony (124): Coincidence experiments show a decrease in the beta-gamma coincidence rate from 0.92 $\times10^{-3}$ coincidence per beta-ray at zero absorber thickness to 0.38×10^{-3} coincidence per beta-ray at 0.110 g/cm². Beyond that point, the coincidence rate remains constant. A gamma-gamma coincidence rate of $(0.59\pm0.03)\times10^{-3}$ coincidence per gamma-ray was also observed. From the coincidence rates it may be concluded that gamma-rays are in cascade and that the high energy beta-rays are followed by 0.55 Mev of gamma-radiation.

(5) Zirconium (95): The 63-day activity was found to emit 0.40-Mev beta-rays. The maximum energy of the gamma-rays was 0.91 Mev as measured by coincidence absorption. The beta-gamma coincidence rate was 0.21 $\times10^{-3}$ coincidence per beta-ray, independent of the beta-ray energy. The gamma-gamma coincidence rate, greater than the beta-gamma coincidence rate, was (0.29 ± 0.02) $\times10^{-3}$ coincidence per gamma-ray. The coincidence experiments suggest that the 0.91-Mev gamma-ray is non-coincident with the beta-rays and may be related to some other decay process.

Chemical purification was carried out in all cases, except that of antimony (124). The calibration curve for the coincidence absorption counting set is given as well as a short discussion of some previously measured beta-ray spectra.

1. INTRODUCTION

THE characteristic radiations of a number of activities prepared at the Clinton pile have been investigated by absorption and coincidence methods. Beta-ray energies were measured by inserting aluminum foils before a single G-M counter, while the gamma-ray measurements were carried out with the aid of a calibrated coincidence counting set, using the method of coincidence absorption.

In all cases beta-gamma and gamma-gamma coincidence measurements were made. A general description of the counters and the procedure of the experiments has been given previously.[1]

2. IRIDIUM (194)

The nineteen-hour activity has long been known to emit hard beta-rays and gamma-rays.[2–6] In addition to the high energy beta-rays

* Assisted by the Office of Naval Research.

[1] C. E. Mandeville and M. V. Scherb, Phys. Rev. **73**, 141 (1948).
[2] A. I. Alichanian, A. I. Alichanow, and B. S. Dzelepow, Physik. Zeits. Sowjetunion **10**, 78 (1936).
[3] E. McMillan, M. Kamen, and S. Ruben, Phys. Rev. **52**, 375 (1937).
[4] C. M. Witcher, Phys. Rev. **60**, 32 (1941).
[5] C. E. Mandeville and H. W. Fulbright, Phys. Rev. **64**, 265 (1943).
[6] L. J. Goodman and M. L. Pool, Phys. Rev. **71**, 288 (1947).

of energy 2.2 Mev,[2,4] indications of a spectrum of lower energy have been obtained also. Witcher,[4] using a thick magnetic lens spectrometer, has concluded that the end point of the low energy beta-ray group lies between $3000H\rho$ and $3500H\rho$. The gamma rays of Ir^{194} were reported to have an energy of 1.35 Mev by Mandeville and Fulbright[5] who took this value from the end point of a momentum distribution of secondary electrons ejected from a thick aluminum radiator in a semicircular, focusing magnetic spectrograph. In a recent paper by Goodman and Pool,[6] Ir^{194} was shown to emit gamma-rays having energies of 0.38 and 1.65 Mev, as determined by absorption in lead. The 0.38-Mev gamma-ray would not have been detected in the apparatus of Mandeville and Fulbright because the spectrograph was unsatisfactory for the measurement of gamma-ray energies less than 0.5 Mev.

IrO_2 was irradiated by slow neutrons for 30 minutes in the Clinton pile. The short exposure time made the 19-hour activity large as compared to the 68-day period. Measurements were commenced about twenty hours after removal of the isotope from the pile.

A coincidence absorption experiment, carried out with a calibrated double coincidence counter

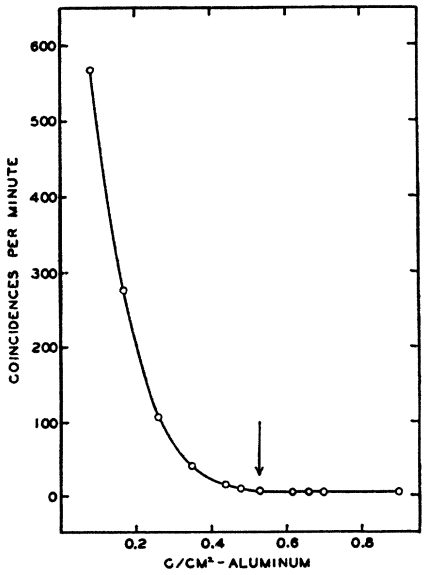

FIG. 1. Coincidence absorption of the secondary electrons of the gamma-rays from Ir^{194}. The end point corresponds to a quantum energy of 1.43 ± 0.05 Mev.

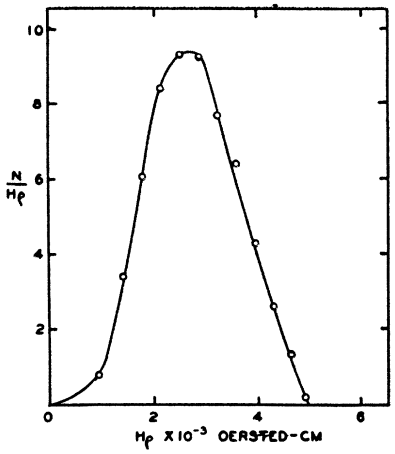

FIG. 2. Momentum distribution of the secondary electrons ejected from a thick aluminum radiator by the gamma-rays of the nineteen-hour Ir^{194}.

set, revealed the presence of a high energy gamma-ray having an energy of 1.43 ± 0.05 Mev. The plot of these data is given in Fig. 1. This value is to be compared with 1.35 ± 0.03 Mev, as calculated from the end point of the curve[7] of Fig. 2. This is a momentum distribution of secondary electrons knocked out of a thick aluminum radiator. Both of the values quoted above would indicate that the 1.65-Mev estimate of the Ohio State University group is somewhat high.

A thin source of chemically purified IrO_2 was placed between two G-M counters in coincidence, and the beta-gamma and gamma-gamma coincidence rates were measured. The beta-gamma coincidence rate is shown in Fig. 3 as a function of the surface density of aluminum absorber placed before the beta-ray counter. The curve of Fig. 3 was taken about 24 hours after the radioactive material was removed from the pile. The beta-gamma coincidence rate is seen to decrease from 0.09×10^{-3} coincidence per beta-ray at zero absorber thickness to zero at 0.150 g/cm². These data were later corrected for the presence of the 68-day Ir^{192} and are plotted in Fig. 4. From these measurements it may be concluded that in addition to the spectrum of maximum energy

[7] This distribution, previously unpublished, was observed in the semi-circular focusing spectrograph at the Rice Institute in April, 1943. As mentioned in the text, the quantum energy taken from the end point is calculated to be 1.35 ± 0.03 Mev and was so quoted a number of years ago (see reference 5).

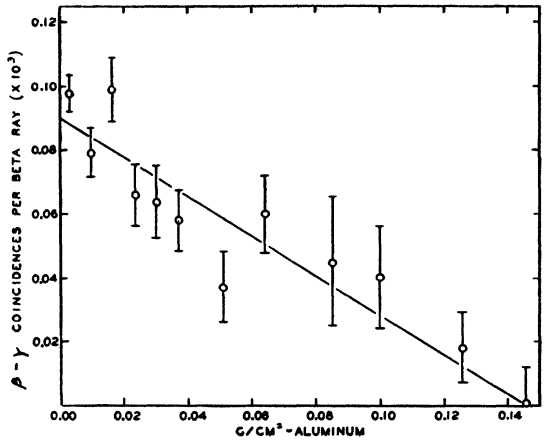

FIG. 3. Beta-gamma coincidence rate of Ir^{194} as a function of the surface density of aluminum inserted before the beta-ray counter.

2.2 Mev, a second spectrum, coupled with gamma-radiation, is present, having an end point at 0.150 g/cm². This corresponds to an energy of 0.48 Mev.[8] It is also clear that no gamma-rays are coupled with the spectrum of maximum energy 2.2 Mev. The high energy spectrum then leads to the ground state of the Pt^{194} residual nucleus. Gamma-gamma coincidences indicating cascade emission of gamma-rays were found in the nineteen-hour period and when corrected for the presence of Ir^{192}, give a gamma-gamma coincidence rate of $(0.54\pm0.05)\times10^{-3}$ coincidence per gamma-ray.

The measurements of this paper, coupled with those of reference 4 and reference 6, make it possible to construct the level diagram of Fig. 5.

3. IRIDIUM (192)

The beta-rays of the 68-day activity were found to have a visual absorption limit at 0.160 g/cm², or 0.50 Mev. This is shown in Fig. 6. Feather analysis, given in Fig. 7, results in an end point at 0.56 Mev. The values reported previously were 0.67 Mev[9] and 0.617 Mev[10].

The beta-gamma coincidence rate, shown in Fig. 8, is clearly independent of the beta-ray energy, in agreement with the findings of Wiedenbeck and Chu.[11] This suggests a simple beta-ray spectrum. According to the calibrated gamma-ray counter of the beta-gamma coincidence counting set, the beta-gamma coincidence rate of 0.41×10^{-3} coincidence per beta-ray indicates

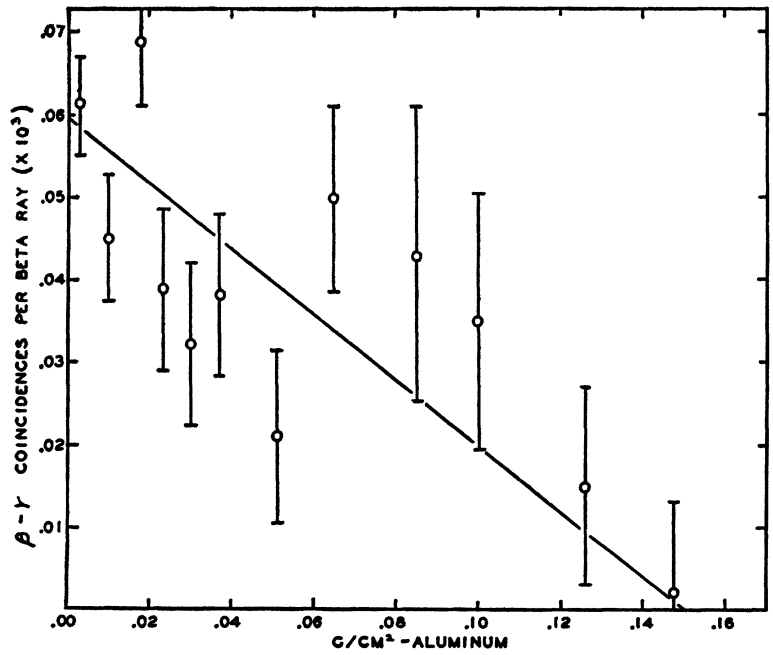

FIG. 4. Beta-gamma coincidence rate of Ir^{194} corrected for the presence of Ir^{192}.

[8] L. E. Glendenin, Nucleonics 2, No. 1, 12 (1948).
[9] P. W. Levy, Phys. Rev. 72, 352 (1947).
[10] Swami Jnanananda, Phys. Rev. 72, 1124 (1947).
[11] M. L. Wiedenbeck and K. Y. Chu, Phys. Rev. 72, 1164 (1947).

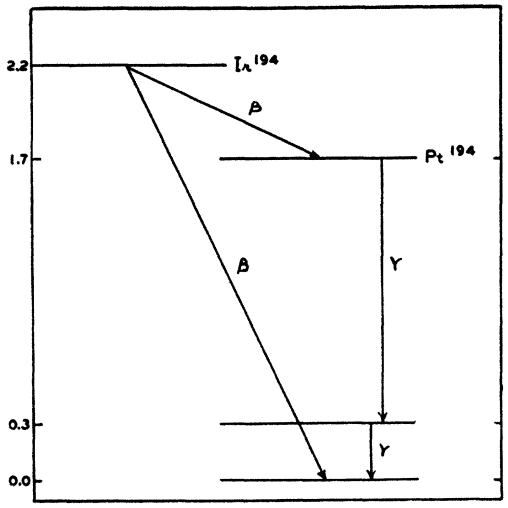

FIG. 5. Proposed disintegration scheme for Ir^{194}.

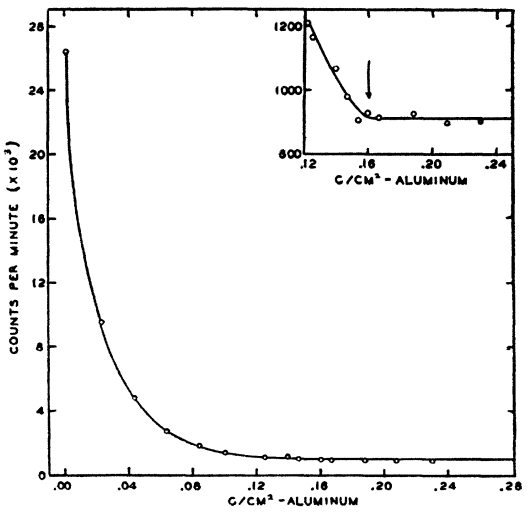

FIG. 6. Absorption in aluminum of the beta-rays of Ir^{192}.

that, on the average, each beta-ray is followed by 0.6 Mev of gamma-radiation. This seems reasonable since the most energetic gamma-ray emitted by Ir^{192} has been shown to have an energy of 0.63 Mev.[5,12] A gamma-gamma coincidence rate of $(0.23\pm0.01)\times10^{-3}$ coincidence per gamma-ray recorded in the gamma-ray counter was also detected.

4. LANTHANUM (140)

The 40-hour activity was induced in La_2O_3, irradiated by slow neutrons in the Clinton pile. In order to measure the gamma-ray energies, a coincidence absorption experiment was performed. It was possible to resolve the curve into two components, giving quantum energies of 1.60 Mev and 2.16 Mev, in agreement with the several previous estimates.[13–15]

A thin source of the radioactive material was placed in the standard position in the beta-gamma coincidence counting arrangement. The beta-gamma coincidence rate, as a function of the surface density of the aluminum absorber thickness placed before the beta-ray counter, is plotted in Fig. 9, where it is seen to be 1.63×10^{-3} coincidence per beta-ray recorded in the beta-ray counter, independent of the beta-ray energy. This result is strikingly similar to that obtained by the Indiana University group.[14] Since observation of the beta-gamma coincidence rate as a function of the surface density of the aluminum placed before the beta-ray counter constitutes an integral method, it does not have the sensitivity of the spectrometric measurements of Osborne and Peacock.[13] Their data have indicated three spectra of energies 0.90, 1.40, and 2.12 Mev rather than a simple spectrum.**

However, a few interesting conclusions can be drawn from the measurements of Fig. 9:

(1) All of the beta-ray spectra of La^{140} lead to excited states of the Ce^{140} residual nucleus, since genuine coincidences were observed as far out as 0.64 g/cm², 1.48 Mev as calculated by Feather's equation.[16]

(2) Using the coincidence rates of Sc^{46} as a calibration point, the beta-gamma coincidence rate of Fig. 9, 1.63×10^{-3} coincidence per beta-ray indicates that each beta-ray is followed, on the average, by 2.3 Mev of gamma-radiation.

[12] J. M. Cork, Phys. Rev. **72**, 581 (1947).
[13] R. K. Osborne and W. C. Peacock, Phys. Rev. **69**, 679 (1946).
[14] A. C. G. Mitchell, L. M. Langer, and L. J. Brown, Phys. Rev. **71**, 140 (1947).
[15] C. E. Mandeville, Phys. Rev. **64**, 147 (1943).

** *Note added in proof:* These beta-gamma coincidence data on La^{140} demonstrate that the method cannot be depended upon to reveal the presence of spectra having a low intensity relative to the principal component. This has bearing upon the many interesting experiments which have been conducted to ascertain whether the beta-ray spectrum of Au^{198} is simple or complex. Experiments of this type would give evidence only of a simple spectrum, though a second component might be present with a relative intensity of several percent.

The differential analog of this method, coincidences on a spectrometer, would obviously constitute a more sensitive approach.

[16] N. Feather, Proc. Camb. Phil. Soc. **34**, 599 (1938).

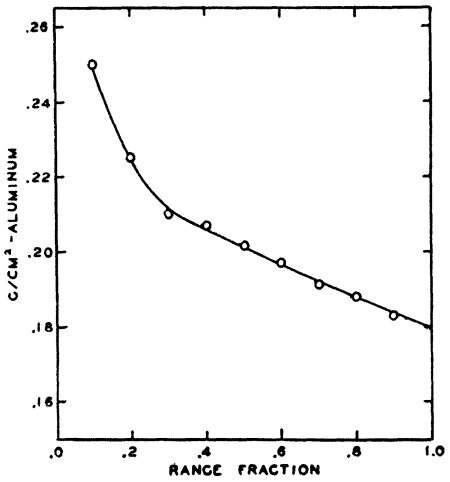

FIG. 7. Feather analysis of the data of Fig. 6.

(3) On considering the statistical probable errors, the beta-gamma coincidence rate can only be represented as a straight line, having a value independent of the beta-ray energy. This would indicate that the 0.90-Mev beta-ray spectrum reported by Osborne and Peacock must be of very low intensity. This is consistent with their report.

A gamma-gamma coincidence rate of $(0.85 \pm 0.03) \times 10^{-3}$ coincidence per gamma-ray recorded in the gamma-ray counter was observed in the disintegration of La^{140}. If the quantum efficiency of the gamma-ray counters is assumed to be constant with energy rather than linear with energy as is actually the case, it is found that two gamma-rays follow each beta-ray.[17] This result also agrees with the Indiana University report.[14]

5. ANTIMONY (124)

The first coincidence experiments in connection with the 60-day activity were carried out by Mitchell, Langer, and McDaniel.[18] Their early data seemed to indicate a simple beta-ray spectrum. Since that time, Meyerhoff and Scharff-Goldhaber[19] have reported a rise in the coincidence rate at low beta-ray energies. This result has been verified by several groups of investigators.[11, 20, 21]

For the sake of completeness, the beta-gamma coincidence rate as a function of the surface density of aluminum before the beta-ray counter is given in Fig. 10. The coincidence rate is seen to decrease from an extrapolated value of 0.92×10^{-3} coincidence per beta-ray at zero absorber thickness to 0.38×10^{-3} coincidence per beta-ray beyond 0.110 g/cm² of aluminum, corresponding to an energy of 0.39 Mev.[8] Beyond this absorber thickness, the calibration point of Sc^{46} indicates that on the average, each beta-ray is followed by 0.55 Mev of gamma-radiation. The gamma-gamma coincidence rate was measured and was found to be $(0.59 \pm 0.03) \times 10^{-3}$ coincidence per gamma-ray.

The radioactive Sb^{124} was prepared when metallic antimony was irradiated for two hours in the pile. The material was aged for three months before the measurements on Sb^{124} were made. No chemical separation was performed.

6. ZIRCONIUM (95)

The long-period zirconium activity has been reported to have a half-life of 63 days.[22] Absorption measurements have indicated beta-rays of energy of 0.29 Mev and gamma-rays of energy 0.94 Mev.[23] Subsequent spectrometric

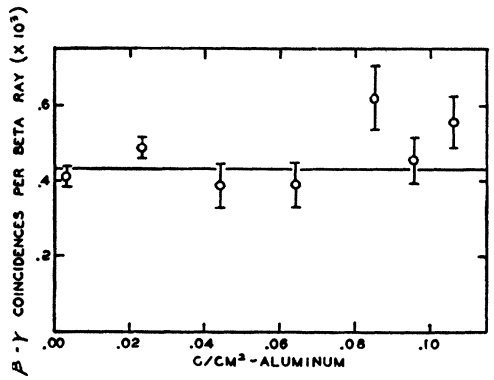

FIG. 8. Beta-gamma coincidence rate for Ir^{192}. This should indicate a simple spectrum.

[17] This result is derived from the following equation:

$$K/(K-1) = \alpha/\beta.$$

α = beta-gamma coincidence rate. β = gamma-gamma coincidence rate. κ = number of quanta per disintegration. For La^{140}, $K = 2.09$.

[18] A. C. G. Mitchell, L. M. Langer, and P. W. McDaniel, Phys. Rev. **57**, 1107 (1940).

[19] W. E. Meyerhof and G. Scharff-Goldhaber, Phys. Rev. **72**, 273 (1947).

[20] E. T. Jurney and A. C. G. Mitchell, Bull. Am. Phys. Soc. **23**, No. 2, 51 (1948).

[21] M. V. Scherb and C. E. Mandeville, Bull. Am. Phys. Soc. **23**, No. 2, 40 (1948).

[22] R. Sagane, S. Kojima, G. Miyamoto, and M. Ikawa, Phys. Rev. **57**, 1180 (1940).

[23] W. N. Moquin and M. L. Pool, Phys. Rev. **65**, 30 (1944).

measurements have given a beta-ray energy of 0.394 Mev and gamma-ray energies of 0.73 Mev and 0.92 Mev.[24] In the course of these latter measurements, a beta-ray spectrum of maximum energy 1.0 Mev and of relative intensity 2 percent was also reported.[24]

Absorption in aluminum of the beta-rays of Zr^{95}, shown in Fig. 11, gives a visual absorption limit of 0.093 g/cm^2, corresponding to an energy of 0.34 Mev.[8] Analysis of these data by the method of Feather,[16] illustrated in Fig. 12, gives a maximum energy of 0.40 Mev. The coincidence absorption curve of Fig. 13 gives a maximum gamma-ray energy of 0.91 Mev, in good agreement with the measurements already cited. This value for the quantum energy was obtained with the aid of the calibrated coincidence counting arrangement.

The beta-gamma coincidence rate for Zr^{95} is given in Fig. 14, where it is seen to be 0.21×10^{-3} coincidence per beta-ray, independent of the beta-ray energy. The coincidence counting set was calibrated with Sc^{46} for which the beta-gamma coincidence rate was 1.4×10^{-3} coincidence per beta-ray. Miller and Deutsch[25] have shown that 2 Mev of gamma-ray energy follows each beta-ray emitted by Sc^{46}. Since the gamma-ray counter used in the coincidence experiments of this paper had a glass cathode coated with Aquadag and was shielded by a thick aluminum block, the gamma-ray efficiency curve may be assumed to be linear with energy, a good assumption for counters of "low Z" materials. The beta-gamma coincidence rate for Zr^{95} would suggest that each beta-ray is followed by a total gamma-ray energy of about 0.3 Mev. It would seem that the hard gamma-rays are associated with some other decay process such as positron emission or K-electron capture. This view is strengthened by the fact that the gamma-gamma coincidence rate was found to be $(0.29\pm0.02)\times10^{-3}$ coincidence per gamma-ray recorded in the gamma-ray counter, larger than the beta-gamma coincidence rate. K-electron capture or positron emission would lead to the Y^{95} residual nucleus. However, the view has been expressed[24] that Y^{93} and Y^{95} are precedents of isotopes of zirconium. This would suggest a possible confusion of isotopic assignments in the zirconium region. The possibility could also exist that the 0.91-Mev gamma-ray is associated with another zirconium activity of about the same half-period.

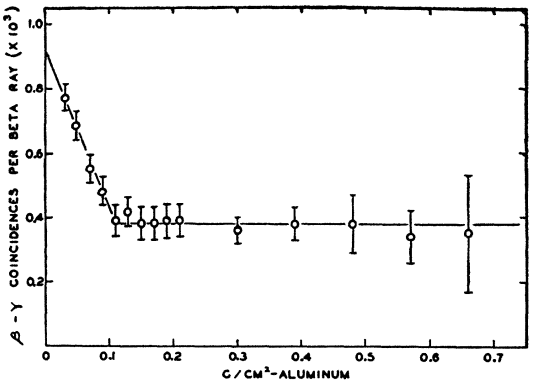

FIG. 10. Beta-gamma coincidence rate for Sb^{124}.

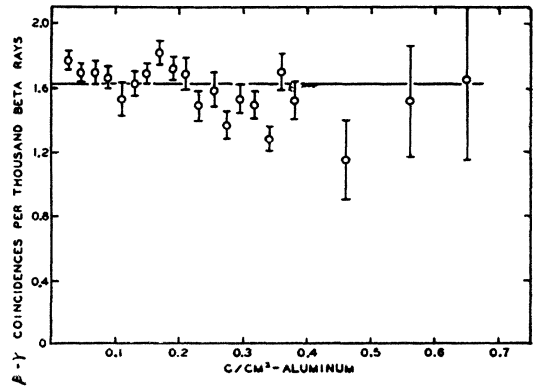

FIG. 9. Beta-gamma coincidence rate for La^{140}.

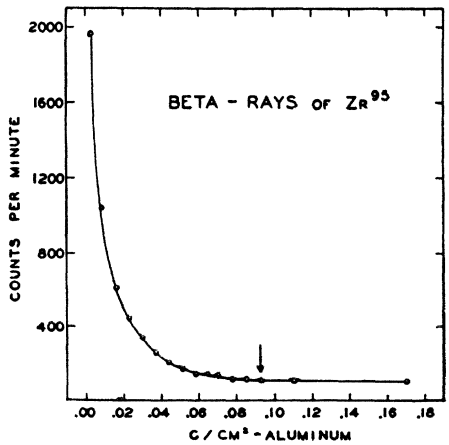

FIG. 11. Absorption in aluminum of the beta-rays of Zr^{95}.

[24] Plutonium Project Report, "Nuclei formed in fission," Rev. Mod. Phys. **18**, 513 (1946).

[25] A. E. Miller and M. Deutsch, Phys. Rev. **72**, 527 (1947).

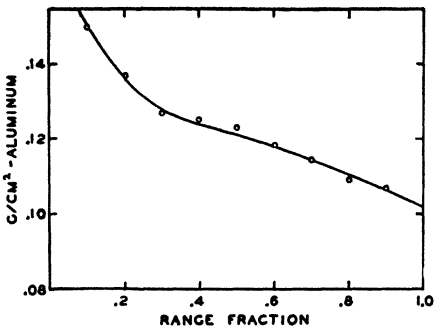

FIG. 12. Feather analysis of the data of Fig. 11.

In observing beta-gamma coincidences, the coincidence resolving time of the circuit was varied from 0.10 microsecond to 12 microseconds. The genuine coincidence rate was found to remain unchanged with the resolving time, so that the low beta-gamma coincidence rate cannot be attributed to coincidence losses arising from a delay in the emission of the gamma-rays which follow the beta-rays. *The role of the 0.91-Mev gamma-ray, and for that matter, of all other gamma-rays emitted by Zr^{95} of energy greater than 0.3 Mev, is unclear.*** It should be added that the coincidence rates were measured immediately after separation of the zirconium activity from its daughter activities of columbium.

7. THE CALIBRATION CURVE FOR COINCIDENCE ABSORPTION MEASUREMENTS OF THE GAMMA-RAY ENERGIES

When coincidence absorption measurements were first undertaken, the coincidence counting arrangement was uncalibrated. Quantum energies were determined by estimating the recoil electron energies from the range-energy relation for homogeneous beta-rays and adding an appropriate amount of energy for collisions in the forward direction. However, as work progressed, many isotopes were investigated upon which spectrometric measurements had already been made. Taking the spectrometric determinations to be correct, the absorption thicknesses (including wall thicknesses of the G-M counters) are plotted against the quantum energies. These data are given in Fig. 15.

The maximum energy of the gamma-rays of As^{76} was recently reported to have a value of 1.98 Mev.[26] The calibration curve of this paper is a revised one, based upon Hayne's value of 2.51 Mev for Ga^{72}. The new value for the maximum energy of the arsenic gamma-rays is then 2.12 Mev.

8. OTHER BETA-RAY SPECTRA

No particular attempt has been made to obtain really high accuracy in the absorption measurements on beta-ray spectra. They were usually made so as to establish the identity of the isotope and to check the validity of the chemical separation. Feather analyses[16] have been made of some data from which end points were previously determined only by visual inspection. The newly determined end points are:

Element	Energy
Sc^{46}	0.32 Mev
Ti^{51}	0.45 Mev
Ta^{182}	0.45 Mev

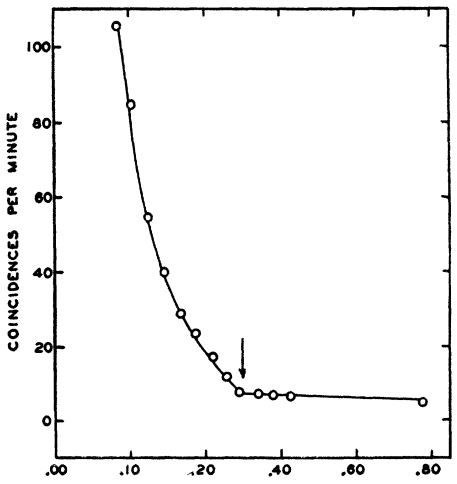

FIG. 13. Coincidence absorption of the secondary electrons of the gamma-rays of Zr^{95}.

*** *Note added in proof May 11, 1948:* The difficulties relating to the presence of the 0.91-Mev gamma-ray are now resolved. A source of the thirty-five day Cb^{95} was separated from the fission fragments of the uranium pile at Oak Ridge. After chemical purification, coincidence and absorption measurements were carried out in the usual manner. A coincidence absorption experiment showed that the 0.91-Mev gamma-ray is emitted by the columbium daughter element, and beta-gamma coincidence measurements indicated that the gamma-ray follows the soft beta-rays emitted by Cb^{95}. Assignment of this gamma-ray to Zr^{95} in the text of this paper apparently resulted from an incomplete chemical separation and from the fact that the 0.91-Mev gamma-ray is very intense.

Gamma-gamma coincidences were found to be 0.28×10^{-3} coincidence per gamma-ray for the fission fragment of Cb^{95}. The gamma-gamma coincidence rate reported in the paper itself for Zr^{95} is therefore probably incorrect.

[26] M. V. Scherb and C. E. Mandeville, Phys. Rev. **73**, 418 (1948).

ACKNOWLEDGMENTS

The writers wish to thank Professor W. B. Keighton of Swarthmore College for having made the chemical separations. They wish also to acknowledge the continued interest of Dr. W. F. G. Swann, Director of the Bartol Research Foundation.

APPENDIX I: CHEMICAL PROCEDURE FOR IRIDIUM

IrO_2, irradiated by neutrons in the pile, was dissolved in HBr. Aqua regia was added, and the solution was evaporated to dryness. Two cc of H_2O were then added, and to this new solution NH_4Cl was added until saturation occurred. After standing for thirty minutes, a precipitate, $(NH_4)_2IrCl_6$, was formed. This was filtered out and washed with a cold saturated solution of NH_4Cl. This procedure should have removed palladium, the outstanding impurity which might have been present.

APPENDIX II: CHEMICAL PROCEDURE FOR LANTHANUM

To 38 mg of irradiated La_2O_3 were added 350 mg of inactive La_2O_3, 10 mg of CaO, 10 mg of BaO, and Na_3PO_4 as carriers for calcium, barium, and phosphorous. This mixture was dissolved in 200 ml of water and a minimum quantity of HCl. Lanthanum hydroxide was precipitated from the boiling solution with ammonia. After washing the precipitate with water containing a small amount of NH_3, the precipitate was dissolved in 0.5N HCl, and lanthanum oxalate was precipitated from the boiling solution with oxalic acid. After washing, the precipitate was ignited to La_2O_3.

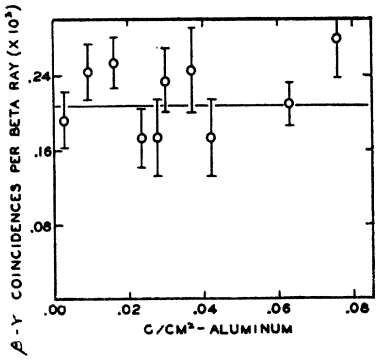

FIG. 14. Beta-gamma coincidence rate for Zr^{95}.

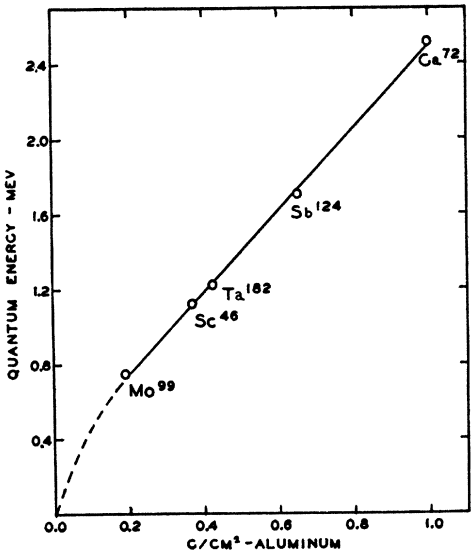

FIG. 15. Calibration curve for the double coincidence counting set used in the coincidence absorption experiments. References:

[1] Ga^{72}—S. K. Haynes, Phys. Rev. 73, 187 (1948).
[2] Sb^{124}—A. Wattenberg, Phys. Rev. 71, 497 (1947).
[3] Ta^{182}—W. Rall and R. G. Wilkinson, Phys. Rev. 71, 321 (1947).
[4] Sc^{46}—A. E. Miller and M. Deutsch, Phys. Rev. 72, 527 (1947).
[5] Mo^{99}—L. C. Miller and L. F. Curtiss, Phys. Rev. 70, 982 (1946).

APPENDIX III: CHEMICAL PROCEDURE FOR ZIRCONIUM

To 0.5g of $Zr(OH)_4$ were added 10 mg of CaO, of Fe_2O_3, of KH_2PO_4, and of Ta_2O_5 as carriers for calcium, iron, phosphorous, and columbian, respectively. The mixture was fused with 5g of K_2CO_3 and 100 mg of KNO_3. After cooling, the melt was leached with 10 ml of cold water and filtered. The residue containing ZrO_2 was thoroughly washed with cold H_2O to remove tantalum, phosphorous, and columbium. The residue was fused with $KHSO_4$, and the melt was dissolved in dilute H_2SO_4. Zirconium was precipitated from the acid solutions as $ZrH_2(PO_4)_2$ by an excess of $(NH_4)_2HPO_4$. After washing, the precipitate was ignited to ZrP_2O_7. Immediately before making observations on the zirconium activity, the phosphate precipitate with Ta_2O_5 carrier was again fused with K_2CO_3 and KNO_3 and was leached with cold water to separate out any accumulated columbium.