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CALIBRATION OF FIVE GAMMA-EMITTING NUCLIDES FOR EMISSION RATE



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CALIBRATION OF FIVE GAMMA-EMITTING NUCLIDES FOR EMISSION RATE

J. M. R. Hutchinson

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ABSTRACT

Mercury-203 and niobium-95 were calibrated by a $4\pi\beta-\gamma$ coincidence method for γ -emission rate, zinc-65 by comparison with the 1.12 Mev peak of scandium-46, sodium-22 by a γ -annihilation-quanta coincidence method and by a triple coincidence method, and strontium-85 by x- γ coincidence counting. The accuracy of the calibration in all cases was ± 2 %. The half-life of the isomeric state of rubidium-85 was measured and found to be .98 microseconds.

Calibration of Five γ -Emitting Nuclides for Emission Rate

J. M. R. Hutchinson

Introduction

In order to supply a need for point source γ -ray standards for calibration purposes in nuclear physics, five γ -emitting nuclides have been calibrated by the coincidence method for γ -emission rate. The nuclides are listed below with some of their properties.

Table I

Nuclide	Emitted rays	γ Energy(mev)	Half-life	
Mercury-203	γ,.β	0.245	47a	
Strontium-85	γ , x (k capture)	0.513	64a	
Niobium-95	γ, β	0.765	35a	
Zinc-65	γ, β^+, x	1.119	245a	
Sodium-22	γ, β ⁺	1.28	2.8y	

The nuclides will be provided in solid, point source (diameter of deposit < 1 cm) standard samples of γ -emission rate. Ideally one desires nuclides emitting a single γ -ray, whose energies are well known and evenly spaced with respect to energy from 0.1 Mev to 2.0 Mev and whose half lives are long enough to make them useful as accurate standards for a period of years. In the kit initially produced the range of

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energies used is indicated in Table I. The activity of each standard is approximately $5 \times 10^4 \gamma$'s/sec. The estimated accuracy of the standardization for γ -emission rate is $\pm 2\%$.

This article will be divided into three parts devoted to (i) methods of source preparation, (ii) a description of the calibration of the standards, (iii) various checks on the γ -emission rate of the standards.

SOURCE PREPARATION

Mounting

It is desirable that in a small solid-angle, at least, the γ radiation emitted by the source should be unscattered. Also the mounting has to be firm in order to allow a fixed geometry intercomparison with other sources of the same nuclide. Toughness and resistance to temperature changes are also desirable qualities. All these requirements were satisfied by covering an annulus of aluminum 2" o.d. 1 1/2" i.d. with thin adhesive polyester tape under tension. The source was deposited on the adhesive side of the tape, approximately in the center and covered with another layer of similar polyester tape.

Source material was received in highly concentrated form and diluted to a strength of approximately $5 \ge 10^4 \gamma$'s/sec per 50 mg. of water solution. 5 ml. ampoules containing accurately weighed amounts of the diluted solution were prepared and sealed. The ampoules were

then measured in the N.B.S. ionization chamber for ion current per gram of solution relative to a fixed radium source (See Emission Rate Checks). The ampoules were then broken open and a number of sources of approximately 50 mg were weighed onto the source mounts using a picnometer and a balance accurate to 0.1 mg. The latter operation was performed quickly to prevent much evaporation during the weighing. The accuracy of these weighings in the worst case as checked by relative counting rates in a fixed geometry was found to be \pm 0.6%. Since each source was checked for counting rate relative to a fixed standard, the error in intercomparing standard samples is less than \pm 0.4%. The sources were allowed to dry, after which tape was placed over them securing them in a sandwich.

CALIBRATION

Mercury-203 ($4\pi\beta$ - γ coincidence counting)

The energy of the conversion electron near the high energy limit of the continuous β spectrum caused difficulty in monitoring β 's in the continuous spectrum without counting the conversion electrons. Moreover the total conversion is too large (22.6%) to be corrected for accurately in any easy manner in low efficiency counters. The obvious solution is to use a $4\pi\beta$ counter which has high enough efficiency so that most of the time a beta is counted in, say a coincidence apparatus, a conversion electron is also counted, the two being recorded as one pulse in the β channel. For 100%

 β efficiency the counts in the β channel equal the β emission rate alone without any contribution from the accompanying conversion electrons.

In view of these considerations a $4\pi\beta-\gamma$ coincidence measurement of Hg²⁰³ was made. Since the coincidence analyser could not accept counting rates of 10⁴/sec an accurate further dilution of the source material was made and deposited by picnometer onto thin collodion source holders. Several sources were counted for β 's, γ 's and coincidences. Approximate disintegration rates and efficiencies were determined after correcting for background in all channels. The observed β efficiency was approximately 94%. A final disintegration rate was determined after correcting for (i) accidentals (ii) dead time losses (iii) internal^{1/2} conversion. The accidental rate was given by N_{β} N_{γ} C(2- ϵ_{β} - ϵ_{γ}). The dead time loss rate for both channels was small.

A multiplicative factor correcting for internal conversion is

$$1 + \frac{\alpha(1-\epsilon_{\beta})\epsilon_{ce}}{\epsilon_{\beta}(1+\alpha)}$$

Thus the final disintegration rate is given by: $N_{\beta} = \beta$ channel rate $N_{\gamma} = \gamma$ channel rate $\epsilon_{\beta} = \beta$ efficiency

$$N_{o} \frac{(N_{\beta}-B_{\beta})(N_{\gamma}-B_{\gamma})}{(N_{c}-B_{c}-A)} \frac{1}{1+\frac{\alpha(1-\epsilon_{\beta})\epsilon_{c}}{\epsilon_{\beta}(1+\alpha)}} \epsilon_{\gamma} = \gamma \text{ efficiency}}$$

$$\alpha = \text{total convers:}$$

 ϵ_{ce} = conversion electron efficiency τ = resolving time (1.45 x 10⁻⁶ sec)

lon coefficient

and inserting some typical numbers
(all in per second)

$$N_{o} = \frac{(356.87-0.90)(11.879-4.109)}{(7.3227-0.1160-0.00639)} \frac{1}{1.0113}$$

$$B_{\beta} = \beta \text{ background}$$

$$B_{\gamma} = \gamma \text{ background}$$

$$B_{\gamma} = \gamma \text{ background}$$

$$B_{c} = \text{coincidence background}$$

$$A = \text{accidental rate}$$

A method similar to the above was used to calibrate Nb⁹⁵ and to check the disintegration rate of Na²² described in later sections of this article. A total conversion coefficient of 22.57% based on the numbers of Nijgh and Wapstra^{2/} was used.

Strontium-85 $(x-\gamma \text{ coincidence count})$

The long half-life (10^{-6} sec) of the isomeric state of Rb^{85} required special consideration when making an x- γ coincidence count on the decay of Sr^{85} . In order that all the coincidences be recorded, a resolving time long compared to the half life of the isomeric state was necessary. It was also important to make the resolving time small enough to minimize the error due to variation in accidental rate. The half-life was determined and coincidence counts were made at two resolving times.

Half-life determination of the 0.513 Mev state of Rb⁸⁵

The schematic experimental arrangement used in the half-life determination is shown in Figure IV. A 1-mm thick NaI crystal with an electron-multiplier phototube was used to detect the x-rays and a 2" x 2" NaI crystal with phototube was used to detect the 0.513-Mev γ radiation. The γ ray was received by the γ channel some time after the x-ray had been detected in the x-ray channel. To measure the half-life of the isomeric state a plot on semi-log paper was made of the number of real coincidences divided by the product of the two singles rates versus delay in the x channel for a resolving time short compared to the half life. A straight line resulted and the half-life was read from the graph. The resolving time was 0.2 µs. The accidentals were found by increasing the delay to 20 µs. The variable delay was calibrated with a crystal oscillator. Six values of the half life of the isomeric state of Rb⁸⁵ were obtained - 0.96, 0.96, 0.97, 1.00, 0.98, 1.00 giving an average corrected value of

 $\tau_{1/2} = 0.98 \pm 3\%$

The major source of error in this measurement is due to inaccurate calibration of the time delay.

Calibration of disintegration rate of Sr⁸⁵

The disintegration rate of a Sr^{85} standard sample was measured by coincidence counting with the equipment of Figure IV using resolving times of 2.57 µs and 8.5 µs in the coincidence analyser. Knowing the half life of the isomeric state of Rb⁸⁵ and the resolving time of the coincidence circuit, the fraction of the total real coincidences which were lost due to the delay of the γ in decaying from the isomeric state

were calculated and a correction in the disintegration rate was made for them. To obtain the 8.5 µs resolving time a gate 7.3 µs long was placed in the γ channel and a pulse of 1.2 μ s shaped by a shorted delay line was introduced into the x channel. The time relationship between the two channels was found by placing an annihilating positron source between the detectors and adjusting the delay in the x channel to where coincidences just disappeared, i.e., where the total delay in the x channel was 7.3 us longer than the delay in the γ channel. The channels were then adjusted to overlap by approximately 0.6 µs (i.e., the delay in the x channel was reduced by 0.6 μ s) to remove any loss of coincidences due to differing rise times of the pulses in various parts of the circuit. Under these circumstances γ rays which were emitted within 7.3 + 1.2 - 0.6 = 7.9 μ s after the associated x-rays were emitted could produce a coincidence. The coincidence equation for this experimental arrangement is then: $N_{o} = \left(1 - \frac{1}{\frac{7 \cdot 9}{0 \cdot 98}}\right) \frac{N_{x}N_{\gamma}}{N_{c}}$

where N_x = singles counts in the x channel, N_γ = singles counts in the γ channel, N_c = coincidences.

A coincidence count of Sr^{85} was also made with resolving times shaped by a shorted delay line of $\frac{2.57}{2}$ µs in both channels. The pulses were made to overlap by 1.034 µs. The disintegration rate for this setup was then calculated with the following formula

$$N_{o} = \left(1 - \frac{1}{2 \cdot 11}\right) \frac{N_{x}N_{\gamma}}{N_{c}} \text{ where } 4.11 = 2.57 - 1.034 + 2.57.$$

The disintegration rate of the Sr^{85} master solution obtained using these two resolving times agreed with each other within 1% and both fell within 1% of the predicted N₀/gm of the master solution using the ionization chamber curve.

Zinc-65 (comparison with the 1.12 mev scandium-46 peak)

Zinc was calibrated for γ emission rate by comparing the 1.112 Mev peak of Zn⁶⁵ as seen in a 2" x 2" crystal with the upper 1.119 Mev peak of a calibrated Sc⁴⁶ source. The disintegration scheme of Sc⁴⁶ is similar to that of Co⁶⁰.

A Sc⁴⁶ source was calibrated by $\gamma - \gamma$ and $\beta - \gamma$ coincidence counting. The $\gamma - \gamma$ coincidence method used in calibrating Sc⁴⁶ followed that of Hayward, Hoppes and Mann in their calibration of Co⁶⁰.⁵ Briefly, their method was to set their crystals at different angles with respect to the source and monitor both channel's singles rates and the coincidence rate. Corrections for the angular correlations of the cascade γ 's and the finite size of the crystals can be computed using theoretical values given by Rose.⁶ The fact that the crystals cannot be made to monitor the γ 's separately can be taken into account by adjusting the discriminator settings on the amplifier outputs to give equal efficiencies and by introducing a factor of 1/2 into the coincidence equations.⁷ For two, two incl. crystals each at a distance of four inches from the source, the theoretical ratio $\frac{N_1N_2}{N_c}$ at π and $\frac{\pi}{2}$ for a 4-2-0 transition is 1.155. The experimental ratio was 1.155 ± 0.010.

A β - γ measurement on the same source was also made and the value for the disintegration rate fell within 0.5% of that obtained by γ - γ coincidence counting. The estimated accuracy of the measurement of the Sc⁴⁶ source was ± 0.5%.

A comparison of the Sc⁴⁶ and Zn⁶⁵ sources was made by comparing the counting rate in the 1.12 Sc⁴⁶ peak as "seen" through a 5v window with the counting rate of the Zn⁶⁵ γ peak as seen through the same window. Measurements were made at different settings to check the effect on the ratio of γ emission rates of the slightly differing energies of the two γ 's. Comparison in this manner of a Sc⁴⁶ source and a Zn⁶⁵ source provided the same ratio of Sc⁴⁶ to Zn⁶⁵ for differential discriminator settings varying over a range of 0.03 Mev - i.e., a range known to be greater than the difference in energy between the upper Sc⁴⁶ peak and the Zn⁶⁵ peak.

Sodium-22 (triple coincidence and γ -annihilation quanta coincidence)

Sodium-22 decays 90% by positron emission and 10% by electron capture to the 1.28 Mev excited state of Ne²². This state then decays promptly to the ground state. The coincidence experiment was as follows.

A source was encased in a lead sheath to produce complete annihilation of the positrons within a small volume. The annihilation quanta were received in coincidence in a_1 and a_2 which were 180° apart with respect to the source (see Figure VIII) and fed through amplifiers into coincidence analyser C_a . The coincidence pulses out of C_a represented a sampling of positrons. These pulses were fed into coincidence

analyser $C_{\beta\gamma}$. Pulses into the γ channel were amplified (A_{γ}) and discriminated against annihilation quanta (D) and also fed into $C_{\beta\gamma}$. Thus at $C_{\beta\gamma}$ one had the arrangement for an oridnary β - γ coincidence experiment. β , γ , and coincidence rates were taken. Accidental and background rates were also taken. Turning to the β channel again, part of the β counts resulted from coincidences between the 1.28 Mev γ and annihilation photons. This so-called γ background in the " β " channel and other effects such as accidentals in the " β " channel etc. were corrected for by subtracting the counts in S_{β} when a_1 was rotated away from the 180° position with respect to the source from the " β " singles rate. The advantage of this method lies in the fact that the " γ background in the β channel" can be subtracted directly without making an independent estimate of the 1.28 Mev γ efficiency in the 0.511 Mev region.

γ - Annihilation γ Coincidence Count

A coincidence count was then taken between the 1.28 Mev γ and the annihilation γ 's. The experimental arrangement consisted of an ordinary two channel coincidence analyser with two, 2" x 2" crystals facing each other but which had the freedom to rotate with respect to the line connecting the source and other crystal. One channel was fed through a window which contained the annihilation peak. The positrons were again completely annihilated close to the source by a lead sandwich. The coincidence equations are in general:

$$\begin{split} N_{\gamma} &= N_{o} \stackrel{\epsilon}{\gamma} + n_{\beta\gamma}^{\gamma} \\ N_{\beta} &= N_{o} \stackrel{\epsilon}{\beta} + n_{\gamma}^{\prime} + n_{\beta\gamma}^{\beta} \\ N_{\beta} &= N_{o} \stackrel{\epsilon}{\beta} + n_{\gamma}^{\prime} + n_{\beta\gamma}^{\beta} \\ N_{c} &= N_{o} \stackrel{\epsilon}{\beta} \stackrel{\epsilon}{\gamma} + n_{\beta\gamma}^{\gamma} \stackrel{\epsilon}{\epsilon} \\ \stackrel{\prime}{\beta} \end{split} \qquad \begin{aligned} N_{c} &= N_{o} \stackrel{\epsilon}{\beta} \stackrel{\epsilon}{\beta} \\ N_{c} &= N_{o} \stackrel{\epsilon}{\beta} \stackrel{\epsilon}{\gamma} + n_{\beta\gamma}^{\gamma} \stackrel{\epsilon}{\epsilon} \\ \stackrel{\prime}{\beta} \end{aligned} \qquad \begin{aligned} N_{c} &= n_{o} \stackrel{\epsilon}{\beta} \stackrel{\epsilon}{\gamma} + n_{\beta\gamma}^{\gamma} \stackrel{\epsilon}{\epsilon} \\ \stackrel{\prime}{\beta} \end{aligned} \qquad \begin{aligned} N_{c} &= probability \text{ that a } \beta \text{ will be detected through } \beta \text{ window if the } \beta \text{ strikes the crystal} \end{aligned}$$

Hence if the two crystals oppose each other with the source in the center we get:

$$\frac{\frac{N_{\gamma}N_{\beta}}{N_{c}}}{N_{c}} = \frac{\frac{(N \cdot \epsilon_{\gamma} + n_{\gamma}' + n_{\beta\gamma}')(N \epsilon_{\beta} + n_{\beta\gamma}')}{N_{c} \epsilon_{\beta} \epsilon_{\gamma} + n_{\beta\gamma}' \epsilon_{\beta}'}$$

If the crystals are not aligned with the source $n_{\beta\gamma}^{\gamma} \epsilon_{\beta}^{*} = 0$ since the annihilation quanta are emitted in opposite directions. From a detailed analysis of the Na²² spectrum ϵ_{β}^{*} was estimated at 50%. $n_{\gamma}^{'}$ was measured by counting the Compton recoils from a calibrated Co⁶⁰ source through the β window and by assuming that the two Co⁶⁰ γ 's with an average energy of 1.25 Mev would produce twice as many Compton recoils per disintegration in the region of 0.511 Mev as one Na²² γ with an energy of 1.28 Mev.

 $n_{\beta\gamma}^{\gamma} \in {}_{\beta}^{\prime} (0.5 n_{\beta\gamma}^{\gamma})$ was estimated from the difference between $\frac{N_{\beta}N_{\gamma}}{N_{c}}$ when the crystals and the source were and were not aligned. Some typical numbers will help illustrate the calculation. $\frac{N_{\beta}N_{\gamma}}{N}$

at 180° was found to be 20% smaller than at 90°. Thus $N_{\beta\gamma} \epsilon_{\beta}^{\gamma} \epsilon_{\beta}^{\gamma}$ was approximately 20% of the true coincidence rate. N_c was about 1 count/sec, hence $n_{\beta\gamma}^{\gamma} \epsilon_{\beta}^{\prime}$ (0.5 $n_{\beta\gamma}^{\prime}$) = (20%) 1 count/sec = 0.2 counts/sec. Hence $n_{\beta\gamma}^{\gamma}$ = 0.4 counts/sec. Now N_{γ} was about 70 counts/sec hence the summing correction in the γ singles channel was approximately $\frac{0.2}{(70)(0.5)} = 0.6\%$. With regard to the " β " channel, it was evident that the summing should be less than in the γ channel because the β " channel was "seen" through a window and the γ channel was not. Analysis of the spectrum showed this estimate to be correct--in fact the summing in the β channel was calculated to be approximately 0.1% of the annihilation γ counting rate.

 N_{O} was counted at an angle of 180°. After the above corrections were made and the background and accidental coincidences were sub-tracted, a disintegration rate of the Na²² agreeing within 1% of the triple coincidence measurement was counted.

Two Other Checks on the Calibration of Na²²

Calibrated sources of Sr^{85} and Co^{60} were used in the right proportions to reproduce the spectrum of Na²² in the region of the annihilation peak, the Co^{60} reproducing the Compton-recoil background. Counting through a 6v window (this just took in the annihilation peak) an intercheck on the calibration of three sources was made since the Co^{60} gave degraded quanta which were equivalent to the 1.28 Mev Na²² quanta in that energy region and the Sr^{85} produced γ 's which were of the same energy (within 0.5%) as the annihilation quanta. The following formula was used to intercompare the sources:

$$\frac{N_{s}}{N_{os}} = 2(0.898) \frac{N_{r}}{N_{or}} + \frac{N_{c}}{N_{oc}}$$

where N_s , N_r , N_c are the counting rates of Na^{22} , Sr^{85} and Co^{60} sources respectively and N_{os} , N_{or} , N_{oc} are the corresponding disintegration rates.

In this equation the fact that two annihilation quanta appear for every positron in a Na²² disintegration and the fact that for each decay of Co⁶⁰ there are two γ 's emitted were taken into account. $0.898^{8/}$ is the positron branching fraction from a decaying Na²² nucleus. Using an average value of disintegration rate of both double and triple coincidence counting for Na²², the left hand side of the equation agreed with the right hand side to within 1.5%.

A second intercomparison was provided by the fact that the peak efficiencies of the upper peak of Co^{60} , the 1.28 Mev peak of Na²² and the peak of Zn⁶⁵ fell on a straight line on a log-log-graph in agreement with the curves of Lazar, Davis and Bell.2/

EMISSION RATE CHECKS

In addition to the source calibration two checks on the accuracy of calibration of the disintegration rate of the standard samples were made, these were namely (i) the final weighed sources were intercompare for counting rate thus checking the weighed amount of the master solution, and (ii) a comparison with a group of nuclides calibrated by

 $4\pi\beta$ - γ coincidence counting which included among others Na²², Nb⁹⁵, Hg²⁰³ but not Sr⁸⁵, Zn⁶⁵ was made using the ion chamber as a means of comparison. A smooth curve was drawn through the points given by these $4\pi\beta$ - γ calibrated nuclides on a graph of $\frac{\text{ion current}}{\text{gm }\gamma/\text{sec}}$ versus energy. Interpolated values of $\frac{\text{ion current}}{\text{gm }\gamma/\text{sec}}$ were obtained for Sr⁸⁵ and Zn⁶⁵, and a comparison of these nuclides was made. It should be emphasized that in the cases of Sr⁸⁵ and Zn⁶⁵ the quoted values for the γ emission rates of the samples were those obtained directly by other methods, the ion chamber measurements served only to check the limits to within 2 or 3%. As it turned out, calibration of these two nuclides using interpolated values on the $\frac{\text{ion current}}{\text{gm }\gamma/\text{sec}}$ versus energy curve. The disintegration rate of the Na²² master solution obtained in other ways agreed with that obtained by $4\pi\beta$ - γ coincidence measurement.

In conclusion the author gratefully acknowledges continued interest in the work by R.W. Hayward, numerous helpful discussions with S. Garfinkel and D. D. Hoppes, and aid with the dilutions by R. Medlock.

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Figure II. Niobium-95 decay scheme: (11)





Figure IV. Diagram of the experimental arrangement for the half-life measurement of the isomeric state of Rubidium-85.



Figure V. Decay curve of the isomeric state of Rubidium-85.



Figure VI. Zinc-65 decay scheme. (13)



Figure VII. Sodium-22 decay scheme. (14)



Figure VIII. Schematic diagram of the triple coincidence apparatus for the determination of the absolute disintegration rate of Sodium-22 sources.



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