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COUNTING INDIVIDUAL ALPHA
ACTIVITIES IN MIXTURES

by

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ABSTRACT

A reproducible method for directly determining individual alpha activities in mixtures in which there are sufficient differences in alpha energies to give separation of the spectrum peaks was developed. A gridded ionization chamber, connected with a 10-channel analyzer, at first, and later to a 100-channel analyzer, was used together with suitably thin source.⁽¹⁾ The chamber gas was a mixture of 90% argon and 10% methane.

INTRODUCTION

The alpha-particle spectra of thin sources consist of a main peak, a tail which extends from the main peak to zero energy, and smaller peaks caused by decay to excited states. The smaller peaks usually merge with the main peak; if they do not and are of sufficient magnitude, they can be handled as separate alpha energies.

The problem of counting individual alpha activities can be considered as being twofold: (1) a method to produce thin sources must be developed in order to concentrate as many counts as possible in the main peak, and to make the main peak as narrow as possible; and (2) in the case of the overlapping of spectra, a procedure to determine with sufficient accuracy the total number of counts belonging to each spectrum must be developed.

PREPARATION OF SOURCES

The common method of source preparation involves the deposition of a solution containing radioactive material on a solid backing and evaporating the solvent until the residue is deposited. The area covered by the solution at the time that saturation is reached during the evaporation process determines largely the area that the residue will cover, since deposition usually occurs first at the edges of the solution. It can be readily seen that if the solution is spread over a larger area at the time of saturation, a thinner deposit will usually be obtained.

To some extent the spreading of the solution is governed by the following equation:

$$\cos \theta = \frac{\gamma_S - \gamma_{LS}}{\gamma_L},$$

where θ is the angle of contact that the edge of the solution makes with the solid backing, γ_S is the surface tension or free surface energy of the solid backing surface, γ_{LS} is the interfacial tension between the solution and solid backing, and γ_L is the surface tension of the solution. As the value of the fraction increases, the contact angle becomes smaller and the solution will cover a larger area. At a value of the fraction equal to one, the entire surface will be wetted. Thus it is seen that the lower is the surface tension of the solution, γ_L , the larger will be the spreading.

Thin sources have been prepared by various investigators from solutions of radioactive materials dissolved in organic solvents which, of course, usually have lower surface tensions than those of aqueous solutions. In general, the surface tension of the solid surfaces of materials used for source mounts would be sufficiently large to cause considerable spreading of the solution were it not that such surfaces are contaminated rather quickly after preparation; this results in a reduction of the free surface energy and thereby hinders spreading. Thus the cleanliness of surface mounts is an important factor in the preparation of thin sources.

Various materials were tried as mounts for the determination of Am^{241} activity (see Table 1). In some cases new surfaces were used to take advantage of the high free surface energy. The highest contrast between uncleaned and clean surfaces was found in the case of platinum. Two sources of Am^{241} activity were prepared on new platinum discs by identical preparation methods, except that one disc was heated with an induction heater to a red heat and allowed to cool prior to depositing the solution. For a 10-channel region which included the spectrum peak and covered a 170-kev region, the unflamed disc source gave 54% of the total 2π count, while the flamed disc source gave 83%; a 20-channel region for the flamed disc gave 92%. Figure 1 shows the total 50-channel plot. It can be readily seen that if the first 20-channel count of the spectrum could be obtained directly, a 10% error in the estimation of the remainder would result in only about 1% error in the total count.

In this report the channels of a spectrum or spectra are numbered in order of decreasing energy, the one of highest energy being channel 1, the second highest channel 2, and so forth. The channel numbers on the abscissa of the figures, being different, are from the instrument used and are arranged in a manner which was most convenient at the time.

New, flamed platinum discs were found to be the best backing materials tried. These could be used with ordinary procedures to provide a suitable standard method of preparation of thin sources.

Table 1

THE EFFECTS OF VARIOUS MOUNTING MATERIALS
IN DETERMINING Am^{241} ACTIVITIES

Backing Material	Percent of 2π count obtained with 10-channel analyzer, for 170-kev region including spec- trum peak	Percent of 2π count for 50-channel ana- lyzer, for 850-kev region with peak
Stainless steel disc, cleaned with conc. nitric acid and carbon tetrachloride	47.1	70.8
Stainless steel disc, sanded to remove old surface	74.2	87.8
New platinum disc; no cleaning	53.6	79.9
New platinum disc; cleaned by heating with induction heater	83.1	92.2
Aluminized mylar; no cleaning	74.8	91.5
Freshly split mica	76.9	87.9

Fig. 1
Spectrum of Am^{241} activity. Runs at
five settings of the 10 channel band in
adjacent parts of the spectrum were
required to get the 50 channel combi-
nation. Channel width is 17 kev.

SINGLE THIN ALPHA SOURCES

The procedure of actually making a count while keeping the gain settings constant on the pulse analyzer consisted of determining the complete spectrum to sufficient accuracy, adding up a chosen number of channels

which included the peak, and multiplying this total by a previously determined factor to get the total 2π count. A total of 50 channels was chiefly used, and the spread of 17 kev per channel was used throughout for thin sources. The general method for estimating the contribution from the tail or low-energy part of the spectrum, where overlapping occurred, into another spectrum was based on the assumption that for thin sources all spectra are sufficiently similar to permit application of the same factors or ratios within the accuracy required here.

A comparison of the distributions of counts in various portions of the spectrum was made for individual sources of Am^{241} (5.48 Mev), Pu^{239} (5.15 Mev), and Th^{230} (4.68 Mev). The results are summarized in Table 2 in which some of the data have been normalized to the counting rate obtained in a standard 2π alpha counter. A consistent value of 0.95 was obtained for the fraction of total counting rate in the 50 channels (850 kev) extending downward from the high-energy portion of the peak. The fraction (0.92) obtained for 26 channels was also consistent enough to permit use where counting time was limited with the 10-channel analyzer used.

Table 2

DISTRIBUTION OF COUNTS IN SPECTRA OBTAINED
FROM SOURCES OF Am^{241} , Pu^{239} , and Th^{230}

Nuclide	F_{50}	F_{26}	K_{27}	K_{50}	Final Slope after 50 Channels (% Decrease per 10 Channels)
Am^{241}	0.949	0.934	728	2184	13.6
Pu^{239}	0.949	0.910	295	899	46.0
Th^{230}	0.944	0.911	359	960	19.7
Average	0.947	0.918	461	1348	26.4

F_{50} = cpm in 50 channels/cpm in 2π counter

F_{26} = cpm in 26 channels/cpm in 2π counter

K_{50} = Ratio of the sum of counts in channels to the right of channel containing 0.1 of the number of counts in the peak channel (cf. Fig. 1) to the number of counts in channel 50.

K_{27} is defined for channel 27 as K_{50} was defined for channel 50.

The values of "K" and the final slopes in Table 2 are measures of the fraction of total activity to be found in the tails of the spectra. As

expected from the smaller difference between F_{50} and F_{26} for Am^{241} than for the other two nuclides, a considerably smaller fraction of the counts is found in channel 27 or channel 50 for Am^{241} than for Pu^{239} or Th^{230} . On the other hand, the number of counts per channel beyond channel 50 decreased more slowly for Am^{241} , as is to be expected from the constant values of F_{50} . Thus, if one extrapolates a spectrum beyond 26 channels on the basis of average values of K_{27} , K_{50} , and slope (Table 2), errors in the region from channels 27 to 50 tend to be cancelled by errors in the region beyond channel 50. Figure 2 shows an extrapolated slope for the Am^{241} spectrum given in the first figure.

Fig. 2

Am^{241} data of Fig. 1 plotted on an expanded vertical scale. Crosses at channels 27 and 50 designate the positions of estimated channel counts that were used as guides for work with multiple spectra. Channel width is 17 kev.

THIN MULTIPLE ALPHA-PARTICLE SOURCES

The procedure adopted for analysis of multiple spectra was as follows:

- (1) The total counting rate of the nuclide with the highest energy alpha particle was determined by applying standard factors (e.g., F_{26}) to the observed counts in the region of the peak.
- (2) The spectral tail of the nuclide was extrapolated, using values of K_{27} , K_{50} , and the slope.
- (3) The contribution of the first spectrum to the next peak was subtracted to provide the net value of the activity of the nuclide with the next most energetic alpha particles.

(4) The total counting rate and extrapolated tail of the second group were determined, and the process continued until the spectra were fully resolved.

The accuracy of determining components in a mixture of alpha activities is affected by the spread in alpha-particle energies, the relative intensities of the components, and the thickness of the source. Where one or more components have a low count rate relative to the others, a modification in the method must be used; this is dealt with later. Furthermore, thicker sources also require modification and they too are covered later.

As a means of determining how well the average factors obtained in Table 2 might be applied to multiple sources, sources consisting of mixtures of Th^{230} (4.68 Mev), Pu^{239} (5.15 Mev), and Am^{241} (5.48 Mev) of known activity were prepared in which the ratios of the activities of the components was varied 20 or 30 percent. These were analyzed for individual activities, and a typical plot is shown in Fig. 3. The agreement between the determined count and the known count was within 3 percent.

Fig. 3

Typical spectra obtained from thin source containing Th^{230} , Pu^{239} , and Am^{241} activities which was used to check reliability of method for determining individual activities. Channel width is 17 kev.

Certain cases require individual variation of the above general method. The spectrum of Ra^{226} , for example, is obtained complete for 50 channels, but superimposed on a small background from daughters of higher energies. Estimating the counting rate for Ra^{226} in channel 50 by

means of K_{50} and subtracting it from the observed value at that channel gives the count from other activities at this point. By drawing a line from this point to that where the Ra^{226} should provide zero count to the right of channel 1 enables one to estimate the contribution from other sources to the counting rate in the 50-channel region. The activity of a Ra^{226} solution which had been calibrated by radon measurement was checked, and the determined count was within 2% of the radon measurement.

THIN MULTIPLE ALPHA-PARTICLE SOURCES WITH COMPONENTS OF LARGE VARIATION IN ACTIVITY

By a modification of the above method, counting of components of low activity, present with those of high activity and higher energy, can be made. Complete spectra of the lower activities are determined first, starting with that of highest energy and going downward in energy. After subtraction of the spectra of low activities from the combined spectra, the nuclides of higher activities can be counted by the method already given.

The isolation of a low-activity spectrum for counting in cases where there was considerable energy separation of the nuclides, 300 kev or greater, was accomplished by drawing a trial curve to channel 27, as is shown in Fig. 4, this curve (the broken line) representing the zero level of the lower activity. For the spectrum thus isolated, the channel 27 count was calculated as described previously and compared with the value on the plot. The curve was redrawn (not shown) and the channel 27 count recalculated until agreement was reached between the calculated and observed channel 27 values. The spectrum could readily be extrapolated to completion if necessary. With a knowledge of the 26 channels of the spectrum it is possible to calculate the 2π count as before.

Am^{241} - Pu^{239} MIXTURES

A thin source containing Am^{241} and Pu^{239} activities of known count and of approximate count ratio 10 to 1, respectively, was used to check the method. The 100-channel analyzer set at 17 kev per channel was used here. Fifteen determinations of the Pu^{239} activity, of approximately 10,000 counts each, were made. The difference between the determined and known counts was less than 4% in all cases, except one where it was 6%. Figure 4 shows a plot used for an isolation of the Pu^{239} spectrum. Determinations of the Am^{241} activity agreed within 3% of the known value.

UNIFORM THICKER SOURCES

It is difficult to put a boundary between thin and thicker sources. Here, thin sources were those, in general, whose contribution to the full width at half height of the peak was 25 kev or less. Thicker sources whose

Fig. 4

Plot used to isolate Pu^{239} spectrum from combined Pu^{239} , Am^{241} spectra. Count of channel 27 of the Pu^{239} spectrum, indicated by a cross, was used in determining the final isolating curve. Channel width is 17 kev.

contribution to the peak width was as high as 120 kev were considered usable for estimation of individual activities. Source contribution to width at one-half peak height was checked by comparison with a peak obtained with a pulse generator.

The general variation in pulse distribution with thickness of source is shown in Fig. 5. The differences in thicknesses of the three Am^{241} sources on platinum were caused by variation in preparation. The distribution with the lowest peak is from a source on a new platinum disc with no cleaning. The distribution with intermediate peak height is the same source after flaming, and the one with the highest peak is from a new platinum disc cleaned by flaming before deposition of the Am^{241} solution and flaming again after preparation. The 2π count rate of all three sources was normalized to the same value.

Fig. 5

Three spectra from Am^{241} activity on new platinum backings normalized to the same count rate. Channel width is 17 kev. $\circ$ Platinum disc not cleaned before deposition of Am^{241} solution, not flamed afterward. $\square$ Above source after flaming. $\triangle$ Platinum disc, flamed before deposition of solution, flamed after preparation.

Some totals from the data used for Fig. 5 are shown in Table 3. As can be seen, the three totals under T_1 and T_2 do not show very large variation. This behavior can be used in the process of estimating counts of thicker, uniform sources.

For thicker sources it was more advantageous to work with 35 kev per channel. The quantities K_{21} and K_{30} were determined with individual thin sources of Am^{241} , Pu^{239} , and Th^{230} by the same procedures as K_{26} and K_{50} were determined previously (Table 2), except here the fraction of the peak channel count used was 0.05 instead of 0.1, since the energy per channel was approximately doubled. Spectra of individual thicker sources of the same nuclides were also used and the number of channels of the thicker sources equivalent to 21 and 30 channels for thin sources were determined in the same manner as channel equivalents for 11 and 20 channels were

Table 3

SMALL VARIATION IN TOTALS OF EQUIVALENT NUMBERS
OF CHANNEL COUNTS FROM Am^{241} SPECTRA

Characteristic feature of Am^{241} source preparation using new platinum discs	<u>T₁</u>	<u>N₁</u>	<u>T₂</u>	<u>N₂</u>
Platinum disc cleaned by flaming, source flamed after preparation	48,000	11 channels	52,000	20 channels
Platinum disc not cleaned, flamed after preparation	45,000	25 channels	49,000	38 channels
Platinum disc not cleaned, source not flamed after preparation	44,000	30 channels	48,000	45 channels

T_1 = total of channel counts to right of channel containing 0.1 of the number of counts in the peak channel.

N_1 = number of channel (counts) included to get T_1 .

T_2 = total for 20 channels or total of channel counts equivalent to 20 channels. The equivalent number (N_2) was obtained by dividing the channel 20 count of the top distribution into its T_1 value to get K_{20} . K_{20} was divided into the other T_1 values to get the corresponding channel count whose location on the corresponding spectrum was used to get the corresponding value of N_2 (number of channels to right of identified channel).

N_2 = number of channel counts included to get T_2 .

determined in Table 3. From the data obtained, Fig. 6 was plotted; this shows the number of channels equivalent to 21 and 30 channels versus full width at one-half peak height of the spectra of the sources of varying thickness. For all individual thicker sources used, an average factor, call it F_{21} , was determined, which represented the ratio of the total of the 21 channel equivalent counts to the 2π count, and was similar to F_{26} and F_{50} of Table 2.

As a visual aid in extrapolating individual spectra of mixtures, single spectra of thicker sources were combined graphically; one such combination is shown in Fig. 7, which is a combination of Am^{241} , Pu^{239} , and Th^{230} spectra.

Fig. 6

Number of channels for thicker sources that are equivalent to 21 and 30 channels of thin sources. The total of the channel counts of equivalent channel numbers are approximately equal. Plots used for work with multiple spectra from thicker sources.

The procedure for the analysis of multiple spectra of thicker sources was almost the same as for thin sources, the chief exception being, of course, that the positions of the 2 channel counts that were determined for guides in extrapolation of the tail varied with source thickness. These positions were determined from Fig. 6 for a first trial. If the extrapolation in question were too different from a graphic combination (Fig. 7), by comparison, then the extrapolation was drawn from past experience, as an estimation.

The slope after channel 30 or channel equivalent to channel 30 was found to be close to zero.

The spectrum finally arrived at was treated in the customary manner; 0.05 of the peak channel count gave the count which isolated the channels for the first total; this total divided by the appropriate factor (K_{21}) gave the count which isolated the channels equivalent to 21 channels; the total of these channel counts was divided by the appropriate geometry factor (F_{21}) to get the 2π count.

Figure 8 shows a situation where the locations of the positions of the channel counts to be used as guides in extrapolating the tail for Pu^{239} , determined from the plot of Fig. 6, were considered to be inaccurate, so, in addition, an extrapolation was made as an estimation from past

Fig. 7

Single spectra of thicker sources of Th^{230} , Pu^{239} , and Am^{241} combined graphically, used as a visual aid for extrapolation of single spectra of multiple sources. Channel width is 35 kev.

experience. The final 2π count for Pu^{239} , using the values from Fig. 6 in the extrapolation, gave an error of 10%, while the case of the extrapolation made visually gave an error of 5%.

Two thicker source mixtures of known count were used for a check of the method. After some experience with thicker sources, the error of the determined count compared with the known count of the individual activity was 6% or less for these two sources.

Fig. 8

Spectra obtained from thicker source containing Th^{230} , Pu^{239} , and Am^{241} activities. Crosses on extrapolations of Am^{241} and Pu^{239} indicate 21 and 30 equivalent channel positions obtained from Fig. 6. In the case of Pu^{239} (dotted line) the extrapolation was too different from Fig. 7 and therefore not used; the solid line drawn as an approximation was used instead. Channel width is 35 kev.

APPLICATIONS

Standard Th^{228} Solutions

The ratio of activities of Th^{228} to Th^{232} was checked for two solutions used as standards for Th^{228} . One which was prepared from thorium purified sometime between 1906 and 1917 and having a calculated ratio of

$0.99^{+0.005}_{-0.01}$ was analyzed, and the ratio found to be 0.99 ± 0.05 . The purification of the thorium of the second solution dated back to 1946. Its calculated ratio was 0.63 ± 0.015 . Two runs were taken on it and both gave 0.67 ± 0.03 . The possible errors associated with results, here and elsewhere in the report, are 0.9 errors.

The same thorium solutions were found to contain Th^{230} as an impurity. The ratio of activities of Th^{230} to Th^{232} in the older solution was found to be 0.21 ± 0.01 , while for the newer solution it was 0.12 ± 0.01 .

Tooth Sample

The ratio of Th^{232} to Th^{228} was also determined for thorium isolated from a tooth obtained from a patient who had taken thorium salts internally. The thorium was extracted into a mixture of carbon tetrachloride and dibutyl phosphate, and the organic phase ashed on a platinum disc. The activities found were: 2.24 ± 0.10 dpm of Th^{228} , 0.58 ± 0.05 dpm of Th^{232} , and a very small amount of Th^{230} . A plot of the activities is shown in Fig. 9. Similar results were obtained on the same sample by comparison of Th^{228} measured by the dibutyl phosphate procedure⁽²⁾ combined with the total alpha counting rate of the thorium in the ashed DBP as measured with a 2π counter: values of 2.6 ± 0.2 dpm and 0.8 ± 0.4 dpm were obtained for Th^{228} and Th^{232} , respectively.

Fig. 9

Thorium isotopes present in tooth sample. For each channel, instrument background was subtracted from the total channel count. Some channels have a negative count because of statistical fluctuation, (see curve).

U²³⁵ In Natural Uranium

A determination of U²³⁵ in natural uranium was made as a rigorous check of the method as applied to those cases in which the magnitudes of the individual activities are different. In order to obtain a sufficiently high count rate for U²³⁵ from a platinum mount, $\frac{15}{16}$ inches in diameter, some thickness in the uranium source had to be tolerated. Figure 10 shows the spectra of U²³⁸, U²³⁵, and U²³⁴ as recorded by the 100-channel analyzer at a count rate of approximately 5 counts per minute of U²³⁵. A check showed the U²³⁴ count to be equal to the U²³⁸ count within 2%. From the data for U²³⁴, a factor for calculating the channel 12 count was determined, as well as the ratios of the highest count channel to the counts of the other channels of the peak. In isolating the U²³⁵ spectrum, the procedure was almost the same as before for the case of the mixture of Pu²³⁹ and Am²⁴¹, shown in Fig. 4. One exception was that the highest count channel was first estimated for U²³⁵ and, using the ratios from the U²³⁴ data, the other channels of the peak were calculated. These were used as guides for drawing the trial curve isolating the U²³⁵ spectrum. For the isolated spectrum, channel 12 was calculated and compared with the observed count in the isolated

Fig. 10
Spectra of U²³⁸, U²³⁵, and
U²³⁴ activities in natural
uranium. Channel width is
17 kev.

spectrum. The process was repeated until the calculated and observed counts were in agreement. Figure 11 shows the isolated U^{235} spectrum. The ratio of the sums of the counts for U^{235} and U^{238} in the first 12 channels was used to calculate the percentage of U^{235} present. Two runs were taken of approximately 4,000 and 6,000 counts of U^{235} , and values of 0.65% and 0.69% were obtained, respectively, for the percentage of U^{235} present as compared with an accepted value of 0.71%.

Fig. 11

Isolated spectrum of U^{235} . Crosses are zero level points that were calculated and used as guides in drawing the isolating curve. Channel width is 27 kev.

Percent of Ra^{226} Decay to the Excited State

The percent of Ra^{226} atoms that decay to the excited state was determined. The source used was Ra^{226} adsorbed to fused quartz, and two mil Lektromesh was used for collimation to avoid trouble from conversion electrons.⁽³⁾ The resolution of the excited state peak is seen in Fig. 12. The excited state count and total count were obtained. Results of four runs are given in Table 4, and the average of the four runs, each weighted according to the total number of counts taken, is 5.7% which is the same value as that obtained by Asaro and Perlman.⁽⁴⁾

Table 4

RUNS WITH Ra^{226} FOR DETERMINATION OF EXTENT
OF DECAY TO THE EXCITED STATE

Run	Time for Run	Excited State Count	Total Count	Percent Decay to Excited State
1	1,486 min.	590	10,200	5.8
2	1,487 min.	590	10,400	5.7
3	1,486 min.	620	10,500	5.9
4	6,832 min.	2,700	47,400	5.7
weighted average				5.7

Fig. 12
Alpha particle spectrum of Ra^{226} . Source used was Ra^{226} adsorbed to fused quartz with 2 mil Lektromesh for collimation.

VARIATIONS IN SOURCE PREPARATION

In order to have the final evaporation from an organic solvent in some cases, a few drops of ethylene glycol were added to the active solution on the platinum disc. The initial evaporation removed most of the water and the final deposition was from an ethylene glycol solution with possibly a small amount of water. It was checked beforehand, of course, that there would be no chemical reaction of the radioactive solution with the ethylene glycol.

For some solutions containing considerable residue, an inert gas was blown on the deposited solution to keep it spread out during evaporation. Furthermore, solutions containing barium salts appeared to contaminate clean platinum surfaces rather quickly which prevented spreading of the solution. This procedure was useful for this type of situation.

These variations of source preparation are useful for measurement of ratios of activities, but carry some risk in preparing sources for absolute measurement since some of the activity can be lost by their use. The individual technique using these variations should be checked for loss of activity if application is considered to absolute work.

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