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A METHOD OF ASSAY OF ^{235}U , ^{238}U , AND ^{237}Np FISSION FOILS

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A combination of 2π and low geometry α counting was used to assay a set of vacuum-evaporated ^{235}U , ^{238}U , and ^{237}Np foils to be used in the measurement of their relative high-energy neutron fission cross sections. The masses assigned to the uranium foils rest on experimentally determined specific α activities; the neptunium masses depend on low geometry counting and the published half-life.

INTRODUCTION

The assay of a set of ^{235}U , ^{238}U , and ^{237}Np fission foils was undertaken as part of an effort at this laboratory to remeasure the relative neutron fission cross sections of these nuclides.¹ Accurate knowledge of the relative masses of active material on the foils is essential to these measurements. The foils being used for the measurements are 1 in. diam vacuum-evaporated UO_2 or NpO_2 on 0.005 in. Pt backing.²

It would seem a simple matter to obtain the weight of either of these compounds on a plate, by weighing the plate before and after deposition and taking the difference. However, the weights of material thus obtained we found to be always somewhat too high, even for vacuum evaporated foils prepared from pure starting materials. The inaccuracy apparently arose not from weighing error, but from some unknown component in the deposit. (One normal uranium foil was shown by x-ray diffraction to be UO_2 , had no more than 0.5% metallic impurities, and was quantitatively analyzed for uranium, but the total of all the material known to be on the foil still left 7% unaccounted for.)

A possible approach to finding the foil weights is to make all the desired fission measurements, then dissolve off the material and quantitatively analyze it. Foil weights known to 0.3% are attainable for uranium, using this technique, and this may be done eventually. White³ reports his most consistent data were obtained when masses were based on destructive analysis. It is doubted that the accuracy of the analysis of the neptunium foils would be improved, considering the local state of the art.

A non-destructive method of assay of the foils is α counting, either 2π or low geometry. Both require a knowledge of the disintegration rate/mass of active

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material; both require a knowledge of the efficiency of the counter. Use of the 2π counter for foil assay requires a knowledge of the back-scattering of particles from the backing and deposit, which for thin foils increases the counting rate to more than 50% of its disintegration rate.⁴ The loss in count rate due to self-absorption in the foil must also be known. The low-geometry counter is generally preferred for accurate work, since the geometry can be calculated from the dimensions of the aperture and foil and the distance between them. It has the additional advantage of admitting only particles emitted normal to the foil, thus rejecting essentially all back-scattered particles; and because the mass of material the particles travel through is small, the self-absorption is slight. The low-geometry counter is, however, sensitive to the size and shape of the foil, especially at positions close to the aperture, requiring uniform foils of known diameter for accurate counting. And needless to say, if the calculated geometry is to be reliable, the pertinent dimensions must be well known.

The principal difficulty arising when assaying a foil by α counting is that very seldom is the foil material isotopically pure, with some of the principal α components near or below the limits of detection of the mass spectrometer. It is not usually wise, therefore, to attempt to calculate the disintegration rate using only the mass spectrometric data and the published half-lives. Much of the uncertainty can be eliminated if the α spectrum of a mixture of isotopes is used together with the mass spectrometric analysis to calculate the disintegration rate. Even this refinement is not very useful, however, when one of the principal α components is very complex, as in the case of highly enriched ^{235}U .

Our 3 nuclides include several different degrees of difficulty of measurement of the specific activity. Neptunium is isotopically pure. Measurement of its specific activity, had it been within the scope of this effort, would have amounted to a redetermination of the half-life, and this has apparently been well done previously.⁵ Assay of the neptunium foils then consisted of determining the disintegration rate of the foils as well as possible and dividing by the literature value for the specific activity. Although the ^{238}U specific activity should be calculable using a combination of the mass spectrometric analysis and the α -pulse analysis, the ^{235}U is not, and since it was desired that the ^{235}U and ^{238}U foils be as comparable as possible, the foils were assayed by parallel methods. The specific α activities of the ^{235}U and ^{238}U were measured directly by depositing known amounts on platinum and counting in the 2π counter.

EXPERIMENTAL METHODS

Instrumentation

1. The 2π Counters. The 2π counters are loop-anode, methane-flow proportional counters, operated in the α -plateau region, which has a slope of 0.25%/100 V. The bias of the counters cuts off all α particles below ~ 1 MeV. These counters have been in routine use continuously since 1950, with long-lived standards counted in them daily. Experience has shown them to be very stable. (Improved electronic components have been introduced from time to time.) All the counters count with the same efficiency, to 0.2-0.3%. The self-absorption measurement counts (Figs. 4 and 5) were taken on 2 of the most divergent counters and averaged. A schematic diagram of a typical 2π counter chamber is shown in Figure 1.

2. The Low Geometry Counters.

LG-1

The oldest of our low geometry counters, herein designated LG-1, was a crude arrangement, by modern standards. The detector was a Dumont 6292 photomultiplier tube on whose surface a viscous silicone grease had been spread, then powdered

ZnS applied generously. There was provision for the introduction of apertures between the detector and the sample. Variable distances from the source to the aperture were obtained by screwing different length "posts" into the bottom of the tank. Conventional hard-tube electronic components were used. Figure 2 is a diagram of the counting chamber of LG-1-2.

This counter had a flat plateau above 900 V tube voltage, and was stable over the 8 years it was in use. Since the critical distances were not well known and the tube surface was probably non-uniform, the calculated geometry was never used. The counter was calibrated by other techniques.

LG-2

The tube surface of LG-1 eventually became contaminated, and a new surface of ALPH-A-CAL,⁶ a fairly uniform ZnS decal, was applied. The counter, now designated LG-2, had a somewhat higher counting efficiency, within 1-2% of its calculated geometry.

LG-3

Since this is a new counter, and the only one whose calculated geometry was used to measure disintegration rates, it will be described more fully than the others. It is a versatile counter, intended to use the existing solid-state detectors, or better ones as they are developed. Three lithium-drifted silicon detectors, ranging from 0.8 cm diam and 24 keV resolution to 2 cm diam and 78 keV, may now be interchanged. Foils up to 3 in. in diameter may be positioned 3 in. to 18 in. from the aperture. There is provision for α - γ coincidence measurements.

All shelf-aperture distances are known to < 0.001 in., while the diameters of the apertures are known to 0.0001 in. The foils are screwed down into position on the shelf inserts with a six-fingered ring which grips the perimeter of the mount and allows visible positioning of the foil within a recessed circle. Geometries of the counter were calculated by a machine code using the equations of Curtis.⁷ A diagram of LG-3 is shown in Figure 3.

The electronic components are: LASL Model 570 preamplifier, Ortec Model 410 Multi-mode amplifier, Ortec Model 408 biased amplifier, Hamner NC-10 discriminator, and a Hamner NS-10 scaler. Pulses may be pulse height analyzed and/or counted by the scaler. When the system is operated using only the amplifier, the threshold of the scaler corresponds to channel 10 on the 400 channel analyzer, or an α energy of 670 keV. A linear extrapolation of the pulse height distribution to zero energy indicates that $\leq 0.1\%$ of the pulses are lost below the threshold of the counter, even for foil thicknesses ~ 0.5 mg/cm². The scaler and the analyzer count rates are the same, but for very weak samples the integrated analyzer counts are preferred.

FOIL COUNTING

Corrections Applied to the 2π Counts

1. Back-Scattering. Ever since Cunningham and co-workers⁸ discovered they were troubled with back-scattering, researchers have been measuring, or trying to measure it. Measured as well as calculated values have ranged roughly from 3-4% of the theoretical count rate of 50%. Two are cited as examples.^{9,10}

One of us¹¹ measured the counting efficiency of a 2π counter for ^{233}U by the following technique: eight 1-2 μg deposits of ^{233}U were vacuum evaporated on Zapon films (the Zapon was 10 $\mu\text{g}/\text{cm}^2$), and counted in 2 different 4π proportional counters. It was calculated that the loss in the mount and film was less than 0.1%, and was neglected. The Zapon films were then laid down on polished platinum plates, melted, and ignited. This burned off the Zapon and deposited the ^{233}U on the platinum in an invisible, adherent deposit. The counting efficiency obtained

by this method was 0.5174 ± 0.0015 . (At the same time, using the same technique a counting efficiency for ^{241}Am of 0.5210 ± 0.0020 was measured.)

The counting efficiency of the 2π counters was measured in a different way as a by-product of the current experiment. The normal uranium foil at 0.714 mg U/cm^2 (see Fig. 4), after being counted both in the 2π counters and in LG-1, was dissolved from the foil and quantitatively analyzed for uranium. This is the foil mentioned in the Introduction. The disintegration rate of the foil was calculated by multiplying the weight of uranium found (corrected for 0.2% left on the foil) by the disintegration rate of normal uranium, $1501 \pm 3 \text{ d/min mg.}^{12}$ Its 2π counting rate, weightless, was calculated by correcting the foil's observed count rate for its observed loss (Fig. 4). The ratio of the corrected 2π count rate to the calculated disintegration rate is the counting efficiency of the 2π counter, and was found to be 0.5176 ± 0.0015 .

The agreement between the 2 methods is no doubt fortuitous; however, the average, 0.5175, corresponding to 3.5% back-scattering, will be used as required in this work.

2. Self-Absorption. It was intended to correct the 2π counts for self-absorption using an experimental relationship between foil thickness and counting loss in the 2π counter. As it turned out, these data were not used directly to correct the fission foils, but are included because of the notable lack of such data in the literature.

A set of 6 ^{234}U (95% enrichment) foils were prepared by electroplating 2-6 μg in $7/8$ in. circles on $1-7/8$ in. Pt plates. These were all counted in two 2π counters and in LG-1. An average ratio $2\pi/\text{LG-1}$ was calculated. Then various thicknesses of UO_2 (normal uranium) were vacuum evaporated in 1 in. circles over 5 of the ^{234}U deposits. The foils were weighed before and after the normal uranium deposition. This weight was used in calculating the abscissa of Fig. 5. (We know that there was not this much uranium on the foils by several percent, but there was present absorbing material at least equivalent to the observed weights.) A normal uranium sister foil was evaporated simultaneously with each deposition over the ^{234}U . Because normal uranium has an appreciable count rate of its own, the sister foil, usually very similar in mass, was used to correct ^{234}U normal uranium foil for the increment due to the normal uranium.

All foils were then counted again in the 2π counters and LG-1. Both the 2π and low geometry count rates of the ^{234}U + normal uranium foils were corrected for the normal uranium contribution, and the ratio

$$\frac{2\pi \text{ (corrected)}/\text{LG-1 (corrected), } ^{234}\text{U} + \text{normal uranium foil}}{2\pi/\text{LG-1, } ^{234}\text{U foil}} \quad (1)$$

was computed for each foil. These values were plotted versus the calculated weights of U/cm^2 from the measured weights of the deposits, as previously explained. This relationship is shown in Fig. 5, where a straight line least squares fit of the data gave

$$\text{Ratio (1)} = 1.0003 - (0.1622 \pm 0.0062 \times \text{mg U/cm}^2) \quad (2)$$

Figure 4 shows the relationship between

$$\frac{2\pi/\text{LG-1, normal uranium foil}}{2\pi/\text{LG-1, average of 6 } ^{234}\text{U foils}} \quad (3)$$

and the thickness, calculated as above. Also shown are the actual weights of uranium on the foils, determined by α counting in LG-1, using the calibration point provided by the analysis of the 0.714 mg/cm^2 foil.

$$\text{Ratio (3), upper line, Fig. 1} = 1.00 - 0.0677 \pm 0.0067 \times \text{mg U/cm}^2 \quad (4)$$

$$\text{Ratio (3), lower line, Fig. 1} = 1.00 - 0.0735 \pm 0.0070 \times \text{mg U/cm}^2 \quad (5)$$

The vertical error bars on both graphs represent $\pm 1\sigma$ counting statistics, propagated through all operations. The horizontal errors (weighing) are believed to be quite small, $< 50 \text{ } \mu\text{g}$. However, the effective weight, in terms of α absorption, may be quite uncertain, contributing to the scatter in the points. This may account for the fact that the slope of Line (4), Fig. 4, which should be half that of Line (2), Fig. 5, is actually somewhat less.

Assay of the Fission Foils

1. ^{238}U . The ^{238}U used for the fission foils had the following composition:

	^{234}U	^{235}U	^{236}U	^{238}U
Atomic %	< 0.001	0.100 ± 0.008	< 0.005	99.899 ± 0.008

(The errors here represent $\pm 2\sigma$)

The specific α activity of this material was determined as follows: approximately 5 g of the metal was cleaned with acetone, weighed, dissolved, and diluted to a known weight of solution. The solution was quantitatively analyzed titrimetrically with an accuracy of $\pm 0.07\%$. The analysis showed the metal to have been 99.52% uranium, a credible value.

All subsequent dilutions and aliquots were made by weight. Small volumes of solutions were weighed by difference in micropipets supported horizontally on a light wire "cradle". No loss due to evaporation was observed during the weighing time.

Two dilute solutions were prepared from the parent solution, and several 10-20 $\mu\text{g U}$ aliquots of each were stippled on 1-7/8 in. platinum plates, dried, and ignited. These were counted in the 2π counter, and the specific activity was calculated to be $412.7 \pm 1.8 \text{ counts/min mg}$. Our sponsor¹⁵ measured the effective thickness of one of the stippled foils (^{235}U) by comparison fission counting (thermal) against a vacuum-evaporated ^{235}U foil. By comparison of the low-energy tailing of their fission fragment spectra, he concluded that the stippled foil had an effective thickness of $90 \text{ } \mu\text{g U/cm}^2$. A foil of this thickness is losing approximately 0.8% from self-absorption, therefore the specific α activity 2π was corrected by that amount, to give $416.0 \pm 1.8 \text{ counts/min mg}$.

The specific disintegration rate of this material was calculated by dividing the specific 2π activity by 0.5175 (see the section on Back-Scattering) to obtain $803.9 \pm 3.5 \text{ dis/min mg}$.

All of the ^{238}U foils were counted 2π ; one was counted in LG-3. The 2π count rates were corrected to weightless, using the ratio

$$\frac{2\pi/\text{LG-3, thin } ^{234}\text{U foil}}{2\pi/\text{LG-3, } ^{238}\text{U foil}} \quad (6)$$

for the correction factor. The foil weights obtained from counting are given in Table 1. A comparison is made also with the weights of U calculated from the actual weights.

2. ^{235}U . The ^{235}U has the following composition:

	^{234}U	^{235}U	^{236}U	^{238}U
Atomic %	0.065 ± 0.013	99.727 ± 0.022	0.08 ± 0.022	0.128 ± 0.010
	0.066 ± 0.002^a			

^aFrom α pulse-height analysis

The ^{235}U foils were assayed in a manner very similar to those methods used for the ^{238}U foils. The specific α activity determination was completely analogous, except that the metal was not available, since the material was already in solution. The titrimetric analysis was again taken as the primary standardization.

The low geometry count on Foil 3 was taken in LG-2 instead of LG-3, but as can be seen from the Np data, it would have counted essentially the same in LG-3. The specific α activity 2π was found to be 6912 ± 16 counts/min mg U; the disintegration rate was 13356 ± 31 dis/min mg. The weights obtained from α counting, compared with actual weights, are given in Table 2.

3. ^{237}Np . The neptunium was isotopically pure; minor α contaminants, consisting of ^{239}Pu and ^{238}Pu - ^{241}Am , which totaled $< 0.1\%$ by α activity, were neglected.

Concentrating on one of the Np foils (Foil 1) for intensive measurement, we measured its disintegration rate by several different methods.

Calibration of LG-1 to 2π Through ^{235}U (Foil 3)

This involved counting the Np and ^{235}U foils in the same position in LG-1, assuming the ^{235}U self-absorption correction to correct to 2π weightless, and dividing by 0.5175 to obtain the ^{237}Np disintegration rate. The rate found was 4.711×10^6 dis/min.

Standardization of LG-1 Using the Disintegration Rate of Normal U

The calculated disintegration rate (mg U $\times 1501 \pm 3$ dis/min mg ^{12}C) of the normal uranium foil at 0.714 mg/cm² which had been destroyed for chemical analysis was used to calculate the counting efficiency of LG-1 (see again the section on Back-Scattering). The disintegration rate of the ^{237}Np foil obtained by this method was 4.712×10^6 dis/min.

Calibration of LG-2 by Cross-Calibration With 2π

LG-2 was calibrated with a set of very thin vacuum-evaporated ^{239}Pu standards, 1 in. in diam, stepping down from 2π . In this counter ^{237}Np (Foil 1) gave 4.785×10^6 dis/min.

LG-3 Calculated Geometry

In LG-3 the foil's count rate was 4.782×10^6 dis/min.

The average of these four methods gives 4.748×10^6 dis/min. This was divided by 1.562 ± 7 dis/min mg^{1/2} to give a mass of 3.039 mg Np on the foil. Combining the estimated errors in counting Foil 1, the half-life error, and the errors in the relative counting rates, a standard deviation of 0.9% was obtained for the 3 foils. This was arbitrarily increased to 1%. The σ can be decreased by counting all 3 longer in the low geometry counter. Foil weights, again compared to actual weights, are presented in Table 3.

SUMMARY

Sets of ^{238}U , ^{235}U , and ^{237}Np foils have been assayed by a combination of 2π and low geometry counting. The weights of active material on the foils are believed to be known to 1-1.6%. Destructive assay or refinement in measurement techniques may result in reduction of the uncertainties.

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Table 1

Mass of ^{238}U , mg U on Foil

Foil number	Calc from actual wt	From 2π counts, corr. for 4.2% self-abs.	From LG-2 count ^a	Ave of α -counting methods	$\frac{\text{U, calc from wt}}{\text{U, ave from } \alpha \text{ cts}}$
1	2.797	2.683 ± 0.030	2.735 ± 0.030	2.709 ± 0.043	1.032
2	2.778	2.694 ± 0.030	2.746 ± 0.030	2.720 ± 0.043	1.021
3	2.786	2.692 ± 0.030	2.744 ± 0.030	2.718 ± 0.030	1.025

^aFoils 1 and 2 calculated from Foil 3, using relative 2π count rates.

Table 2

Mass of ^{235}U , mg U on Foil

Foil number	Calc from actual wt	From 2π counts, corr. for 4.5% self-abs.	From LG-3 count ^a	Ave of α -counting methods	<u>U, calc from wt</u> <u>U, ave from α cts</u>
1	2.486	2.368 ± 0.024	2.399 ± 0.024	2.384 ± 0.034	1.043
2	2.479	2.374 ± 0.024	2.406 ± 0.024	2.390 ± 0.034	1.037
3	2.453	2.358 ± 0.024	2.390 ± 0.024	2.374 ± 0.034	1.033

^aFoils 1 and 2 calculated from Foil 3, using relative 2π count rates.

Table 3

Mass of ^{237}Np , mg Np on Foil

10	Foil number	Calc from actual wt	LG-1 rel. to ^{235}U	LG-1 rel. to normal U	LG-2 rel. to 2π	LG-3 Calc Geom	Average ^a	<u>U, calc from wt</u> U, ave from α cts
	1	3.113	3.016	3.017	3.063	3.061	3.039 $\pm$ 0.03	1.024
	2	3.128					3.022 $\pm$ 0.03	1.035
	3	3.130					3.028 $\pm$ 0.03	1.034

^aFoils 3 and 4 calculated from Foil 1, using relative LG-1 rates.

FIGURE 1. METHANE-FLOW 2π PROPORTIONAL COUNTER CHAMBER.

FIGURE 2. SCHEMATIC: LOW GEOMETRY COUNTER, LG-1 AND 2.

FIGURE 3. SCHEMATIC: LOW GEOMETRY COUNTER, LG-3.

FIGURE 4. COUNTING RATE, 2π RELATIVE TO 2π WEIGHTLESS, OF VARIOUS THICKNESSES OF NORMAL UO_2 FOILS.

$\frac{2\pi(\text{CORR.})/\text{LG-1}(\text{CORR.}), {}^{234}\text{U} + \text{NORMAL U FOIL}}{2\pi/\text{LG-1}, {}^{234}\text{U FOIL}}$

FOIL THICKNESS, MG U/CM², CALCULATED FROM FOIL WEIGHT

FIGURE 5. RESIDUAL COUNT RATE, 2π , OF ELECTROPLATED ${}^{234}\text{U}$ FOILS OVERLAID WITH VARIOUS THICKNESS OF VACUUM-EVAPORATED NORMAL UO_2 .

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