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The Counting Efficiency of Pa233

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February 27, 1951

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ABSTRACT

The counting efficiency of Pa^{233} has been determined by comparison of the counting rate of Pa^{233}, under stated conditions, with the disintegration rate of U^{233} produced by Pa^{233} decay.

INTRODUCTION

The 27.4 day Pa^{233} is intermediate in the beta-decay chain leading from Th^{233} to U^{233}. It has been deemed desirable to determine the counting efficiency of Pa^{233} - that is, the ratio of the observed counts to the actual disintegrations under specified counting conditions. Pa^{233} is a beta-emitter, with a number of gamma rays and so many conversion electrons that its beta-particle spectrum is masked by electrons. It decays to form U^{233}, which is an alpha-emitter with 1.62×10^5 year half-life.[1] The counting efficiency was determined by counting Pa^{233},

1. G. T. Seaborg and I. Perlman, Rev. Mod. Phys. 20, 585 (1948).

then allowing it to decay to U^{233}. Since the alpha-particles of U^{233} can be counted under conditions where their counting efficiency is known, the number of atoms of U^{233} present can be calculated. This is

equivalent to the number of atoms of Pa^{233} that have decayed. Knowing the number of atoms of Pa^{233} initially present and the geometric arrangement for counting, the number of counts that would have been observed had each disintegration given rise to one count can be calculated. The ratio of the counts observed to the calculated number of disintegrations is the counting efficiency.

EXPERIMENTAL

The protactinium was obtained from decay of Th^{233}, produced by neutron irradiation of 6 g of thorium (as $Th(NO_3)_4 \cdot 2H_2O$) in the Oak Ridge pile. The protactinium was separated from the thorium target material, purified, and again separated from its uranium daughter activity by extraction into di-isopropyl ketone (DIPK). Protactinium forms an extractable complex with the ketone in hydrochloric acid concentrations of 6 to 12 molar, while thorium and uranium can be extracted only to a slight extent in 6 M acid. Table I shows the ratio of activity in the DIPK phase to that in the aqueous phase for tracer thorium, protactinium, and uranium.

The thorium nitrate was dissolved in 6 M HCl and refluxed during periodic addition of formic acid, to destroy the nitrate ion. When the nitrate was judged destroyed, the solution was distilled to decrease the volume and to bring the hydrochloric acid concentration to approximately 6 molar. The protactinium was extracted from the thorium into DIPK and the DIPK phase washed twice with 6 M HCl to remove traces of thorium and uranium. The protactinium was then extracted from DIPK into 2 M HCl. This procedure was repeated twice in order to purify thoroughly from

uranium, and analysis of the hydrochloric acid solutions used to wash the DIPK phase last showed the protactinium free from all but a neglible amount of U^{233}. The protactinium was extracted into an aqueous phase consisting of 2 M HCl, the acid concentration brought to 6 M, and the protactinium stored in a 10 ml volumetric flask and allowed to grow its U^{233} daughter. After a period of growth, amounting to approximately two weeks, the uranium was separated from the undecayed protactinium by extraction of the protactinium with DIPK, leaving the uranium behind in the aqueous phase. All operations on the protactinium up to this point were carried out in a Berkeley box, for safety in handling the high level of activity used. Three extractions with equal volumes of DIPK were necessary before the Geiger activity of the aqueous phase was low enough to allow removal from the Berkeley box. The total Geiger activity of Pa^{233} was measured by removal of two separate aliquots of 0.100 ml each from the original 10 ml solution of Pa^{233}. These aliquots were diluted to 250 ml and 0.100 ml samples were evaporated on platinum counting disks for measurement in a Geiger counter. The two separate aliquots gave the same Geiger count within 4 per cent. The decay of the counting samples was followed and no significant deviations from the 27.4 day half-life of Pa^{233} were noted over a period of 40 days. The original activity of the Pa^{233} was obtained by extrapolation of the decay curve to the point where purification was completed.

The U^{233} formed by decay of the Pa^{233} was measured by counting the alpha activity directly in a 25 per cent aliquot of the uranium fraction.

Only one type of Geiger tube was used, and end-window tube trade named Amperex filled with argon-chlorine mixture with a window diameter of 2.75 cm and a window thickness of 3.5 mg/cm^2. The counts were taken inside the customary lead chamber at five standard shelf positions and averaged over six different counters. The average geometry at each position has been determined[2] and is shown in Table IV.

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2. H. Neumann, Univ. of California Radiation Laboratory Classified Report UCRL-840 (August 1, 1950).

The samples were mounted on platinum counting disks and were counted uncovered. To make comparison possible, samples were mounted also on 1.1 mg/cm^2 mica backing and the ratio of counts observed for various backing materials to those for mica backing was measured by placing various backing materials beneath the mica. The backscattering from the mica was assumed to be neglible, and the ratio between the count of Pa^{233} on the various backings to the count on mica backing is considered the backscatter factor. Values of this factor determined are given in Table II.

The alpha particles of U^{233} were counted in an argon ionization chamber at 52 percent geometry. It was necessary in some cases to count samples below the counting plateau, due to the high level of associated beta activity from unseparated Pa^{233}. In these cases, a correction was applied to the data by comparison with a plateau curve determined using a U^{233} standard.

Absorption data was obtained by interposing thin aluminum or mica sheets between the sample and the Geiger tube. To obtain the absorption curve of Pa^{233} at zero absorber, use was made of a windowless counter, trade named "Nucleometer." Absorption curves for Pa^{233} are shown in Fig. 1. The corrections for absorption were made using the Nucleometer curve, since extrapolation of low-geometry data is quite uncertain.

RESULTS

Three separate determinations of the counting efficiency were made. The procedure for the first two determinations was identical, except for slightly different decay periods. The protactinium was purified from thorium and uranium, allowed to decay two weeks, then the U^{233} produced by the decay separated by four extractions in the Berkeley box, followed by six or seven additional extractions, for which the U^{233} lost was recovered by recycling. The third determination was made doing no further operations after removal from the Berkeley box. The data necessary for calculation of the results are included in Table III, where the disintegrations of U^{233} have been corrected for a 7 percent loss in the first four extractions, and for counter geometry. The value adopted is the average of the three determinations, 6.3×10^{-3} counts (on the fifth step off platinum backing) per disintegration.

The average number of counts per disintegration at various geometries is shown in Table IV. Corrections for backscattering, for absorption in air, for absorption by the counter window, and for counting geometry give an average of 2.7 electrons per disintegration. The large deviation

from the mean at the first step position is presumed due to uncertain geometry.

This is in essential agreement with Seaborg and co-workers,[3] who

3. G. T. Seaborg, J. W. Gofman and R. W. Stoughton, University of California Radiation Laboratory Report A-192 (June, 1942).

had previously determined by a different method that Pa^{233} emitted 2.3 electrons per disintegration.

The values of the counting efficiency are consistent to 5 percent, but the absolute accuracy is expected to be no better than 9 or 10 percent. Since the variation among different counters checked was 5 percent, it is felt that to claim greater accuracy than would necessitate specifying the particular Geiger counter to be used. The major portion of the uncertainty is thought to be in the counting determinations, and in the determination of the counting geometry.

Table I

Element	DIPK 3M HCl	DIPK 6M HCl	DIPK 9M HCl	DIPK 12M HCl
Th	< 0.001	0.002	0.003	0.004
Pa	0.016	37	93	296
U	-	0.016	-	0.4

Table II

Det.	c/m Pa^{233} Amperex Tube Step 5	Decay Period	% of Ofig. Pa Decayed	U^{233} Disinte-grations Produced	d/m Pa^{233}	Counting Efficiency Step 5 c/d
1	5.63×10^{8}	14.0day	29.7	1.17×10^{4}	8.52×10^{10}	6.61×10^{-3}
2	6.23×10^{8}	14.8day	31.2	1.47×10^{4}	1.02×10^{11}	6.12×10^{-3}
3	5.88×10^{8}	15.0day	31.6	1.37×10^{4}	9.36×10^{10}	6.28×10^{-3}

Ave. 6.33×10^{-3} c/d
or 158 d/c

Table III

Backing Material	Backscatter Factor
1.1 mg/cm^2 mica	1.0
1.1 mg/cm^2 mica on cardboard	1.1
Glass 40 mg/cm^2	1.15
Steel 220 mg/cm^2	1.32
Pt 100 mg/cm^2	1.55
Ag 130 mg/cm^2	1.4
Pb 48 mg/cm^2	1.56
Lucite 175 mg/cm^2	1.02
Aluminum 680 mg/cm^2	1.1

Table IV

Amperex Tube - 3.5 mg/cm^2

Shelf	Distance from window to sample		Disintegrations per count		Geometry %	Absorption Correction Factor	Electrons/Disintegration
	Cm	Total Absorber mg/cm^2	Pt Backing	Mica Backing			
1	0.38	4.0	4.5	6.97	12.6	3.3	3.78
2	1.97	5.9	17.8	27.6	4.9	3.8	2.81
3	3.56	7.8	45.0	69.7	2.3	4.5	2.81
4	5.15	9.7	91.8	142	1.4	5.2	2.62
5	6.74	11.6	158	245	0.9	6.0	2.73

Nucleometer: 0.65 counts/disintegration average of last four steps 2.74

This work was performed under the auspices of the U. S. Atomic Energy Commission.

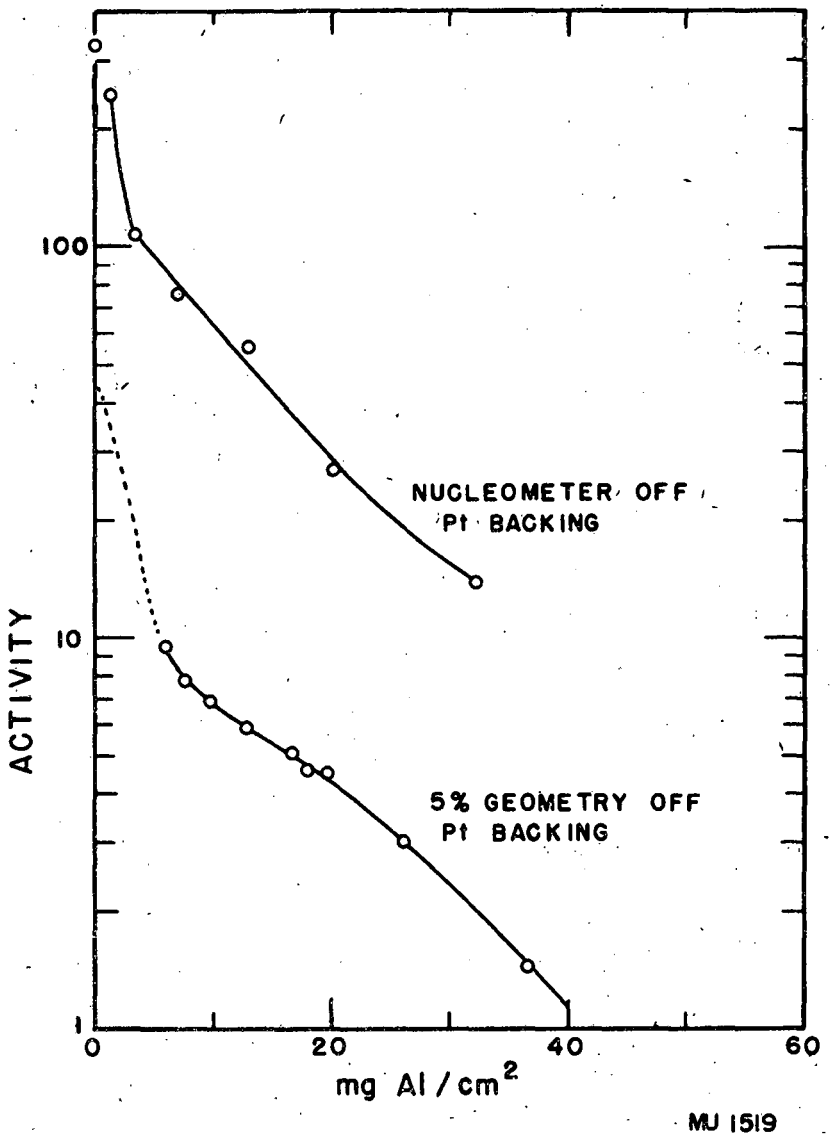

Fig. 1

Absorption Curves for Pa^{233}