Rapid Microdetermination of Dysprosium in Terbium Oxide by Neutron Activation and γ-Ray Spectrometry

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A neutron activation analysis method has been devised to evaluate the dysprosium content of high-purity terbium-oxide powder. Since the activation cross section for terbium is large (more than 22 barns[1] for thermal neutrons), terbium may interfere with the determination of dysprosium unless the matrix is removed. From the calculation based on the data[2] for thermal neutron activation and γ-ray spectrometry, it is seen that the most abundant γ-ray from 0.1 g. of activated terbium is in the same order as that from 1 μg. or more of activated dysprosium when both elements are irradiated at the same flux density of thermal neutrons. This implies that the non-destructive determination of dysprosium in terbium oxide may be interfered with terbium if the dysprosium content is less than 10 p. p. m. The possible interferences from terbium are γ- and X-rays of ^{160}Tb. The energies in MeV. are: 0.96(35%), 0.88(33%), 0.30(30%), 0.05 (~30%), etc.

Experimental

Varying amounts (7~25 mg.) of the sample to be analyzed were sealed in separate polyethylene capsules (1 cm. in diameter and 2 cm. in length) and placed by turns in the Japan-Research-Reactor-1 for 60 sec. to be irradiated with neutrons at a flux of 3×10^{11} neutrons per cm^2/sec. Then, after a 15-sec. interval, the decay of the activities produced was determined by use of a 2 in. thick, well-type, thallium-activated sodium-iodide crystal attached to a 256-channel pulse-height analyzer. In each successive γ-spectra obtained, the height of the photopeak of 0.108 MeV. γ-ray from ^{165m}Dy was measured, and a decay curve was made by plotting the log peak height vs. the time. Identification was made from the γ-ray energy and the half-life. Dysprosium standards were made so as to contain known amounts of dysprosium ranging from 0.05 to 5 μg.

Results and Discussion

Typical γ-ray spectra obtained are shown in Figs. 1 and 2. It can be seen from these figures that the γ-ray spectra for the irradiated terbium-oxide sample consist mainly of those of ^{165m}Dy; that is, the Compton continuum for the γ-rays of ^{160}Tb is negligible. The half-life of the photopeak which appeared at about 0.108 MeV. in the spectra for the sample was equal to that of the photopeak at the same position in the spectra for the dysprosium standard solution.

Table I shows the results of analyses where nine aliquots have been taken from one unknown sample. Since the observed dysprosium contents are nearly constant in the range of the sample amounts below 24 mg., it can be concluded that the self-shielding effect is negligible in this range. Hence, the average

1) D. J. Hughes and R. B. Schwartz, *U. S. Atomic Energy Comm., BNL-325*, 2nd ed. (1958).
2) M. Okada, Paper presented at the 4th Japan Conference on Radioisotopes, Kyoto, October, 1961.

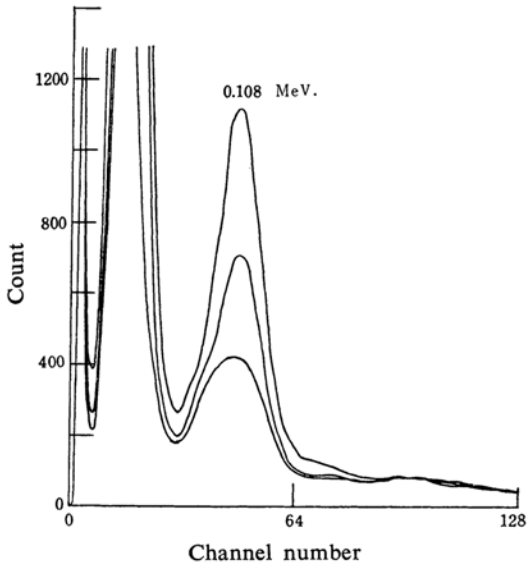

Fig. 1. Spectra for γ-rays of an activated terbium oxide sample (No. 2 in Table I).
Time interval between the 1st (the highest) and the 2nd spectra is 65 sec., and that between the 2nd and the 3rd spectra is 70 sec.

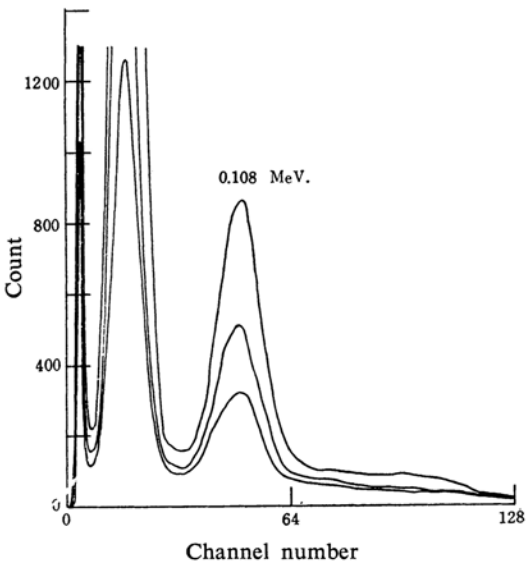

Fig. 2. Spectra for γ-rays of an activated dysprosium standard solution containing 0.22 μg. of dysprosium.
Time intervals are the same as those in Fig. 1.

(48 p. p. m.) can be taken for the dysprosium content. From the five values for "dysprosium found" shown in the lower part of Table I, the coefficient of the variation of single analyses can be estimated at 5.2% for 24.4_4 mg. sample.

TABLE I. RESULTS OF ANALYSES OF A TERBIUM OXIDE POWDER SAMPLE

No.	Sample wt. used mg.	Dysprosium found p. p. m.
1	6.96	49
2	11.1	50
3	11.1	45
4	19.4	50
5	24.5	46
6	24.4	52
7	24.5	49
8	24.4	48
9	24.4	45

The production of ^{165m}Dy through ^{165}Ho (n, p) and ^{168}Er(n, α) reactions was neglected in this experiment because their cross sections were expected to be very small and because the amounts of holmium and erbium in the sample were expected to be less than that of the dysprosium.

The radioactive nuclides which were produced by neutron irradiation of the samples and which decay with a γ-ray energy about 0.108 MeV. are ^{183m}W, ^{m}Yb(6 sec.), ^{165m}Dy, ^{161}Gd, ^{79m}Se, ^{151}Nd, ^{188m}Re, ^{155}Sm, ^{149}Nd, ^{165}Dy and ^{153}Sm. The radionuclides which can only slightly be formed upon 60 sec. irradiation with thermal neutrons are omitted. In these eleven nuclides, the ratio of the half-life of ^{165m}Dy to the nearest half-life is 1 : 3.1. Therefore, only a few successive spectra per sample are enough to identify dysprosium so long as the interfering activity of the nearest half-life is negligible. This was true in the sample tested in this work.

It is a good point of this method that the procedures required are simple and can be carried out on a very low-level radioactivity. From Fig. 1, which represents a sample of 48 p. p. m. dysprosium content, it can be deduced that one-tenth of this concentration of dysprosium would produce an adequate size of photopeak. Contamination can hardly become a problem in this method because no chemical procedure is employed for the sample to be analyzed.

Summary

Dysprosium in terbium oxide powder was determined by a simple method involving 1 min. irradiation with pile neutrons of 3×10^{11} neutrons per cm^2/sec. and repeated γ-ray spectrometry using a 2 in. thick sodium-iodide crystal attached to a 256-channel pulse-height analyzer. Both interferences and the self-shielding effect were negligible. The coefficient of variation of single analyses was 5.2% for a 24 mg. sample of 48 p. p. m. dysprosium content.

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