Reports and Letters

Quantitative Analysis for Thorium by X-ray Fluorescence

Techniques are being developed by the U.S. Geological Survey for the quantitative analysis of rocks, minerals, and ores for thorium by fluorescent x-ray analysis. This work is part of a program that is being conducted on behalf of the Division of Raw Materials of the U.S. Atomic Energy Commission.

The method is a modification of the thorium method described by I. Adler and J. M. Axelrod [*Anal. Chemistry* **27,** 1002 (1955)]. It differs from their method in that (i) selenium is used instead of thallium as the internal standard and (ii) the samples are not briquetted.

The equipment consists of an unmodified Norelco x-ray spectrometer and plastic sample holders. These sample holders fit into the sample holder supplied by the manufacturer. In the top of the plastic blocks there is a rectangular recess 34 by 21 by 1 mm.

Standards are prepared by grinding together for ½ hr in an agate mortar 2 g of reagent-grade ThO_2 and a matrix of ground silica and Fe_2O_3, 3:1 by weight; 400 mg of silicon carbide No. 320; and 40 mg of 10-percent elemental selenium. The silicon carbide aids in the grinding process by further reducing the particle size of the other constituents. The powder is packed in the plastic sample holder. The sample is then inserted into the conventional Norelco x-ray spectrometer, and three positions are measured—$ThL\alpha_1$, $SeK\beta$, and a nearby convenient background position. This background position is determined by scanning over the spectrum region of the $ThL\alpha_1$ and $SeK\beta$ lines. The position is chosen by visual inspection of the graph. Using a LiF crystal, 29.5° was found to be a satisfactory background position in most cases.

The background is subtracted from the intensities of the $ThL\alpha_1$ and $SeK\beta$ lines. The ratio of the intensities of $ThL\alpha_1$ to $SeK\beta$, in counts per second, is plotted against a percentage of thorium (as the ordinate). The resulting curve is a straight line up to about 0.7 percent of thorium.

Above about 0.7 percent of thorium, the curve deviates from linearity because of coincidence losses in the counting circuits and poor resolution. This causes some $ThL\alpha_1$ radiation to be counted as $SeK\beta$. Coincidence losses can be overcome by the use of apertures to reduce the counting rate to about 500 counts/sec. Adler has shown that better resolution, but poorer sensitivity, can be obtained by using a quartz crystal instead of LiF.

Samples are prepared in much the same fashion. To a 2-g sample, 40 mg of 10-percent selenium in silica and 400 mg of SiC is added. This mixture is ground for ½ hr and is then measured in the same way as the standards. In each case, the background count is subtracted from the $ThL\alpha_1$ and $SeK\beta$ before computation.

The x-ray fluorescent analyses are compared with quantitative spectrographic analyses and with radiochemical analyses of the same samples in Table 1.

One sample was packed into the holder and counted ($ThL\alpha_1$) six times. This process was repeated six times, yielding 36 measurements. Variance was calculated on these replicate analyses. The actual quantity used was the time in seconds required to count 12,800 counts. The sample concentration was 0.215 percent of thorium. The standard deviation owing to packing, or more probably to sampling, was 0.72 sec. The error, or precision standard deviation, was 0.32 sec. The total time for 12,800 counts was about 40 sec. Sampling and packing doubled the error caused by recounting alone.

The error variance was calculated both on routine samples (ores) and on synthetic standards. Ores ranging from 0.4 to 0.8 percent of thorium had a standard deviation of 0.026 percent of thorium, based on 18° of freedom. Ores in the range of 0.01 to 0.05 percent of thorium had a standard deviation of 0.0026 percent of thorium based on 12° of freedom. Synthetic standards in the range of 0.1 to 0.3 percent of thorium had a standard deviation of 0.0042 percent of thorium based on 6° of freedom. In the range from 0.02 to 0.3 percent of thorium the standard deviation was 0.0026 percent thorium with 6° of freedom.

Table 1. Accuracy of fluorescent x-ray analysis for percentage of thorium

Sample	Quantitative spectrographic	Radiochemical	Synthetic spectrographic standard	Average of quantitative spectrographic and radiochemical	Fluorescent x-ray analysis
A, synthetic			0.10		0.10
B, synthetic			0.02		0.023
C, synthetic			0.032		0.037
D, synthetic			0.32		0.324
E, synthetic			0.75		0.71
F, ore	0.63	0.61		0.62	0.64
G, ore	0.49	0.47		0.48	0.48
H, ore	0.66	0.66		0.66	0.80
I, ore	0.34	0.38		0.36	0.43
J, ore	0.52	0.70		0.61	0.75
K, ore	0.43	0.51		0.47	0.57
L, ore	0.12			0.12	0.12
M, ore	0.17	0.17		0.17	0.18
N, ore	0.70	0.66		0.68	0.75
O, ore	0.48	0.49		0.485	0.48

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Pathways of Glucose Metabolism in Corneal Epithelium

It has been previously shown that in the corneal epithelium the direct oxidative pathway, or the hexose monophosphate shunt, is an alternate route for glucose metabolism (*1*). It appears that in ocular tissues, such as the corneal epithelium and especially in lens (*2*), the shunt mechanism accounts for most of the CO_2 produced from glucose. To approximate the significance of the alternate pathway in the corneal epithelium, a comparison was made of the incorporation of C^{14} from 1-C^{14} and 6-C^{14} glucose into CO_2 (*3, 4*) and lactic acid (*5*).