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Compton-Scattered Tungsten X-Rays as a Measure of Mass Absorption Coefficients in Rocks

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Abstract

The intensity of Compton-scattered $WL\alpha_c$ X-rays may be used to estimate mass absorption coefficients (μ) analogous to Reynolds' (1963) method for $MoK\alpha_c$ radiation. This procedure depends on removal of two interfering sources of radiation: (1) Rayleigh-scattered $WL\alpha$ X-rays at 1.476 Å may be eliminated by use of a Ni filter (absorption edge = 1.488 Å). (2) Fluorescent $NiK\beta$ radiation generated in any Ni-bearing rock occurs at the same wavelength (1.500 Å) as the $WL\alpha_c$ peak. The constant relative intensity of $NiK\alpha$ and $NiK\beta$ permits determination of an empirical correction factor (independent of sample Ni content) for this interference. Application of these procedures with a linear calibration curve (μ *vs* $WL\alpha_c$ intensity) generally yields a μ within 5 percent of that calculated from chemical composition.

Introduction

Reynolds (1963) showed that the mass absorption coefficient (μ) of common rocks and minerals is an approximately linear function of the reciprocal intensity of Compton-scattered X-rays. This discovery provided a simple and rapid method of estimating μ for X-ray fluorescence analysis of geological materials of unknown chemical composition.

Reynolds' original measurements were made using a molybdenum target tube and the $MoK\alpha$ Compton-scattered peak ($MoK\alpha_c$). Reynolds (1967) subsequently demonstrated several refinements of the method, including selective filtration of the interfering, Rayleigh-scattered $MoK\alpha$ peak (thus enhancing the intensity ratio of $MoK\alpha_c/MoK\alpha$). He also stated (Reynolds, 1967, p. 1494, note 1) that long-wavelength X-rays, specifically the *L*-series lines from a tungsten target, are not suitable for determining μ from geological materials by the Compton-scattered method. We report here an adaptation of Reynolds' procedures and show that $WL\alpha$ Compton-scattered radiation is, in fact, a reliable measure of μ. This method may be useful to workers who lack a Mo tube as used by Reynolds, but have a W tube available.

All of our analyses were made with a General Electric XRD-5 spectrometer equipped as follows: (1) W target tube operated at 50 kv and 45 ma; (2) LiF (200) analyzing crystal; (3) primary and secondary collimators with slit spacing of 1.59 and 0.25 mm, respectively; and (4) a G. E. No. 6 SPG sealed proportional counter. Correction for a deadtime of 9.8 μsec, as determined by the method of Norrish and Chappell (1967), was applied to all intensity readings. Rock powders were pressed into aluminum dishes to yield samples at least 2 mm thick. This depth yields the equivalent of an infinitely thick sample for all X-ray wavelengths and values of μ of interest here.

The XRD-5 spectrometer sample chamber provides an angle of 90° between the incident and scattered beams. For this geometry, the wavelength increase of Compton-scattered radiation relative to its corresponding Rayleigh-scattered peak is 0.024 Å, independent of target material (Reynolds, 1963, p. 1136). Thus, the $WL\alpha$ peak at 1.476 Å will have a companion $WL\alpha_c$ peak at 1.500 Å. This proximity of the Rayleigh-scattered $WL\alpha$ radiation provides one of the two sources of interference to the accurate measurement of the $WL\alpha_c$ peak.

Following the example of selective filtration shown by Reynolds (1967), we eliminated the $WL\alpha$ interference by use of a Ni filter (absorption edge = 1.488 Å). A 0.025 mm thick Ni foil placed over the exit of the primary collimator eliminated the

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W$L\alpha$ radiation and decreased the W$L\alpha_c$ peak to about 50 percent of its unfiltered intensity.

The second source of interference with the W$L\alpha_c$ peak is Ni$K\beta$ radiation of precisely the same wavelength ($\lambda = 1.500$ Å). This interference will arise in any Ni-bearing sample, including many mafic and most ultramafic igneous rocks, with the result that the apparent W$L\alpha_c$ peak intensity will contain a component of fluorescent Ni$K\beta$ X-rays. Obviously, the measured intensity of W$L\alpha_c$ will be too large in proportion to the Ni content of the sample, all other factors being equal. It is possible to allow for the Ni$K\beta$ component of the composite W$L\alpha_c$ + Ni$K\beta$ peak by determining a correction factor as follows.

Using the Ni filter described above to eliminate the Rayleigh-scattered W$L\alpha$ peak, measure the intensity of W$L\alpha_c$ radiation scattered from Ni-free synthetic quartz. (We have established by scanning and counting across the Ni$K\alpha$ peak region that no detectable Ni X-rays are generated in the Ni filter, nor in the tube target itself.) Under identical operating conditions, measure the total intensity of the filtered composite W$L\alpha_c$ + Ni$K\beta$ peak ($\lambda = 1.500$ Å) and the net intensity of the filtered or unfiltered Ni$K\alpha$ peak ($\lambda = 1.659$ Å) from a sample of the same quartz spiked with a known amount of Ni (~500 ppm). The net intensity of Ni$K\beta$ in the spiked sample is obtained simply by subtracting the total W$L\alpha_c$ intensity (without background correction) for the pure quartz from the total intensity of the composite W$L\alpha_c$ + Ni$K\beta$ peak in the spiked sample. Because the intensity ratio of $K\beta/K\alpha$ is constant for any element, correction factors may then be calculated as intensity ratios: Ni$K\beta_{\text{fil}}$/Ni$K\alpha_{\text{unfil}}$ = 0.0881 ± 0.0011, and Ni$K\beta_{\text{fil}}$/Ni$K\alpha_{\text{fil}}$ = 0.202 ± 0.003. The foregoing values represent the mean ± its standard error; subscripts "fil" and "unfil" indicate that the peak intensity was measured with and without the Ni filter, respectively.

To apply these factors to an unknown sample, the product of 0.0881 times the unfiltered net intensity of Ni$K\alpha$ (measured from the unknown sample) is subtracted from the filtered composite peak:

$$\text{total } WL\alpha_c = \text{total } (WL\alpha_c + NiK\beta)_{\text{fil}} - 0.0881 \times \text{net } NiK\alpha_{\text{unfil}}.$$

This procedure may thus be used to construct a calibration curve of μ *vs* W$L\alpha_c$ intensity for standard samples and to determine the Compton-scattered intensity of unknown samples. If the Ni-spiked quartz sample is routinely analyzed, it may also be used as a standard for determination of the Ni contents of the unknown samples as a byproduct of this correction procedure.

TABLE. 1. Calculated Values of $\mu_{1.5\text{Å}}$ and Measured Ni Concentrations of U.S.G.S. Silicate Rock Standards

SAMPLE	$\mu_{1.5\text{ Å}}$	Ni (ppm)*
G-2 (granite)	39.55**	10.0 ± 0.1
GSP-1 (granodiorite)	43.75**	10.8 ± 0.2
AGV-1 (andesite)	48.54**	15.3 ± 0.2
W-1 (diabase)	60.72***	70.3 ± 0.2
BCR-1 (basalt)	63.75**	10.1 ± 0.3

**Mean and standard error of the mean for 15 determinations.*

***Chemical analyses from Flanagan (1969), Table 1, pp. 84-86, 89; average of analyses 3, 4, 19, 23, 25, and 26.*

****Chemical analysis from Fleischer (1969), Table 1, col. 3b, p. 65.*

We have followed the above methods to construct calibration curves based on the five U. S. Geological Survey silicate rock standards listed in Table 1. For each rock, μ was calculated at 1.5 Å from the chemical analyses cited and the tabulated data of Lieb-

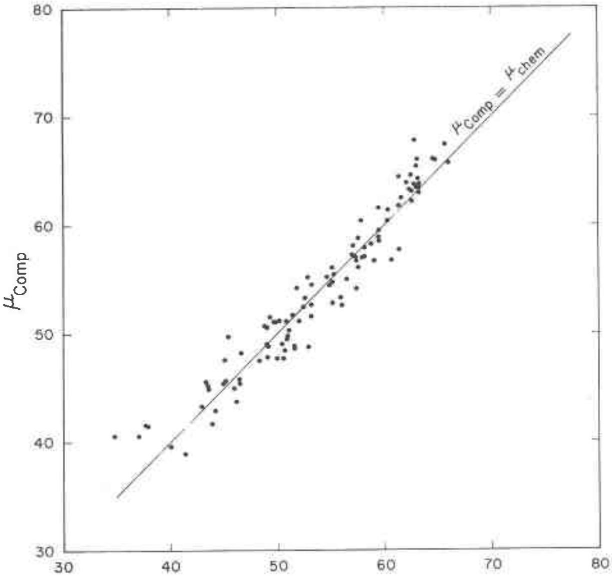

FIG. 1. Comparison of mass absorption coefficients calculated from chemical analyses (μ_{chem}) with those derived from a calibration curve using the intensity of the W$L\alpha_c$ peak (μ_{Comp}). The line labeled $\mu_{\text{Comp}} = \mu_{\text{chem}}$ corresponds to exact agreement by the two methods.

hafsky *et al* (1960, Appendix IV) for μ of individual elements. The correlation coefficient of the data for each determination of the calibration curve was always 0.985 or greater. Using these calibration curves, we have determined in duplicate the value of μ (at 1.5 Å) of 103 igneous rocks of known chemical composition. The samples included a variety of rock types—ankaramites, tholeiitic and alkali basalts, andesites, dacites, trachytes, and Fe-rich melarhyolites—with μ values ranging from 34.8 to 68.2. Ni contents, using the Ni-spiked quartz as a standard, were in the range 3.7 to 309 ppm. In the most Ni-rich samples, the Ni$K\beta$ peak contributed 15–20 percent of the composite W$L\alpha_c$ + Ni$K\beta$ peak.

Figure 1 compares the mean μ from the duplicate W$L\alpha_c$ determinations (μ_{Comp}) with the value calculated from the known chemical composition (μ_{chem}). For 85 of the 103 samples, the agreement is within ±5 percent or better.

Also listed in Table 1 are Ni concentrations in the five U. S. G. S. rock standards. These values were calculated from the simple relationship given by Reynolds (1963, eq. 2, p. 1134), using the values of μ at 1.5 Å given and the measured intensities of the Ni$K\alpha$ peaks. (The Ni content and μ value of the spiked quartz standard are 520 ppm and 29.90, respectively.)

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