AN X-RAY BACKGROUND METHOD FOR THE DETERMINATION OF TOTAL IRON IN TRIOCTAHEDRAL MICAS

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The use of Cu K_α radiation in diffraction work on iron bearing materials results in the excitation of fluorescent radiation from the iron. This fluorescent radiation shows up as a high background intensity on a diffraction trace and is most noticeable above 20° 2θ. It is generally considered to be a nuisance, but under certain conditions the background intensity can be used to estimate the total amount of iron present in the sample. These conditions are:

1. No other element with a mass absorption coefficient close to iron is present in appreciable quantities.

2. Matrix effects are constant.

3. A series of analyzed standards is available.

A group of trioctahedral micas is used to illustrate this method. In trioctahedral micas, there can be nearly complete substitution of Fe^{2+} for Mg^{+1}, resulting in the replacement of an element with a low mass absorption coefficient by one with a much higher mass absorption coefficient for Cu K_α radiation. Other substitutions can occur but are generally of much less importance. The relative amounts of other elements thus stays about the same, as does the type of silicate lattice; therefore, matrix effects remain constant and can be ignored. Condition number one is commonly met (Foster, 1960), as is the case with most rock forming silicates, so if a few standards with total iron analyses are available for comparison, background intensity can be used to estimate total iron content.

The following experimental conditions were used to obtain background intensities:

Diffractometer: Tem-Pres Model XD-1
Radiation: Ni-filtered CuKα
Warm-up time: 1 hr
Tube settings: 25 kv., 10 mA.
Goniometer setting: 50.5°2θ
Time constant: 1 sec
Full scale deflection: 10,000 counts/min
Sample preparation: Micas were ground to less than 63 μ in a Wig-L-Bug amalgamator and sedimented onto a porous tile plate by filtering out the water with the aid of a vacuum.
Method: Background intensity was recorded for one minute and an average value used.

Because the Tem-Pres diffractometer has no absolute counting circuit, the background for iron free fluorphlogophite was arbitrarily set near zero. Background intensities for the other micas are thus relative to this
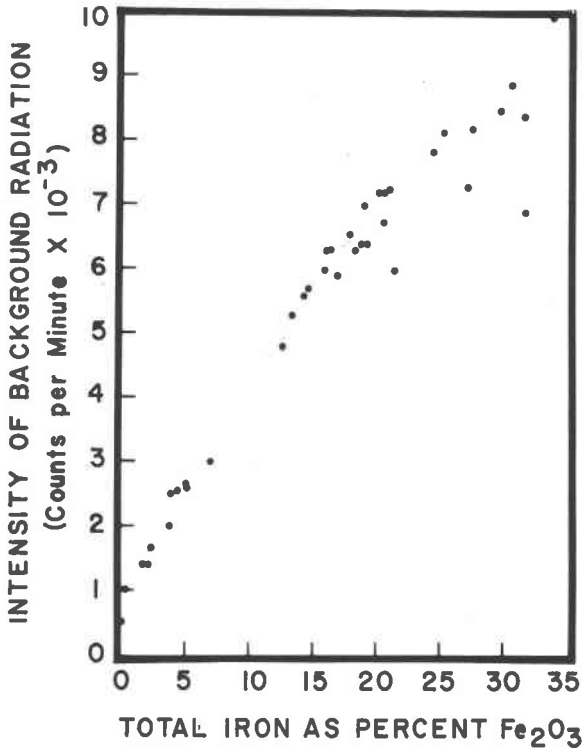
Fig. 1. X-ray background intensities of thirty-eight trioctahedral micas plotted as a function of total iron content.

arbitrary baseline and are not absolute. Since background intensity will vary among diffractometers and with instrument settings (Nash, 1964), unknowns must be referred to a set of standards and this factor is not important. The angle 50.5° 2θ was selected because no diffraction peaks occur in this part of the pattern and this is a portion of the diffraction trace that exhibits a high background when appreciable iron is present. Furthermore, background intensity is nearly constant for several degrees on either side of this value so erroneous settings of the angle 2θ should not materially affect the results.

X-ray background intensities of thirty-eight trioctahedral micas that had been analyzed for total iron by the method of Shapiro and Brannock (1962) show a nearly linear direct relationship between background intensity and total iron content (Fig. 1). The deviation from linearity at high counting rates is probably due to coincidence loss in the geiger tube. Such losses become appreciable at about 6000 counts/min (Klug and Alexander, 1954, p. 282). The close grouping of the points to the general

trend indicates that condition number one is most likely met and background intensity can indeed be used to estimate total iron content in trioctahedral micas.

This background intensity method should be applicable to other mineral groups where isomorphous substitution of iron for another element with a much smaller mass absorption coefficient takes place. Nash (1964), in an X-ray study of the SiO_2 content of glasses, found a similar direct relationship between background intensity and total iron content of the glasses so isomorphous substitution may not even be necessary. It might also be possible to apply the same method to mineral groups where isomorphous substitution of two other elements with widely different mass absorption coefficients takes place.

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A TECHNIQUE FOR MOUNTING, SECTIONING AND POLISHING
PARTICLES SMALLER THAN 30 MICRONS
IN DIAMETER[1]

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Quantitative analysis of individual particles with the electron microprobe requires the creation of flat, well polished surfaces in order to reduce geometrical variations of X-ray absorption, surface charging and other phenomena observed when particles are mounted directly on surfaces. In our laboratory, particles as small as 8μ in diameter are routinely prepared for reflected light microscopy and electron microprobe analysis by mounting them in bakelite plugs. We use a cover glass and an electron microscope grid to accurately determine the particle's position.

The particles selected for sectioning are transferred to a circular cover glass of the same diameter as the bakelite mold and a 100-mesh copper electron microscope grid is positioned over the particle. The grid may be tacked to the cover glass with a small drop of plastic cement. The cover glass is placed, with the particle up, at the bottom of the mold assembly of the bakelite press (a circular piece of thin cardboard may be placed

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