electron-diffraction pattern (Fig. 3 of KR) shows, besides strong $hk0$ reflections with $h-k=3n$, weaker ones with $h-k\neq3n$. This fact may be explained by postulating that the thin platy crystal used was only some ten layers thick, each iowaite layer being 8.1 Å thick. This number of layers is far from infinity, which is assumed in the idealized diffraction theory. The spots in reciprocal space will, therefore, be elongated along c^*; it also follows that reflections with $l=\pm1$ will appear on the electron-diffraction pattern.

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ON THE VALENCE OF IRON IN TRIPUHYITE: A MÖSSBAUER STUDY

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Abstract

Mössbauer spectra show iron in tripuhyite is ferric, giving the formula $FeSbO_4$.

In recent years, the use of Mössbauer spectroscopy has developed into a remarkable tool in many branches of physics, chemistry and biology. One of the most important parameters studied is the so-called isomer shift, also referred to as the "chemical shift". The isomer shift is the position (in energy) of the center of gravity of the spectrum. The relevant

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information in the present context, obtained from the isomer shift, concerns the solid state or chemical aspect. It turns out that when the same atom can appear in different valence states, these can in many cases be characterized by the different range of values of the isomer shift, since the nuclear factor involved does not depend on the valence state (Wertheim, 1964).

The problem of the valence of iron in the mineral tripuhyite was discussed in detail by Mason and Vitaliano (Mason, 1953). The usually ac-
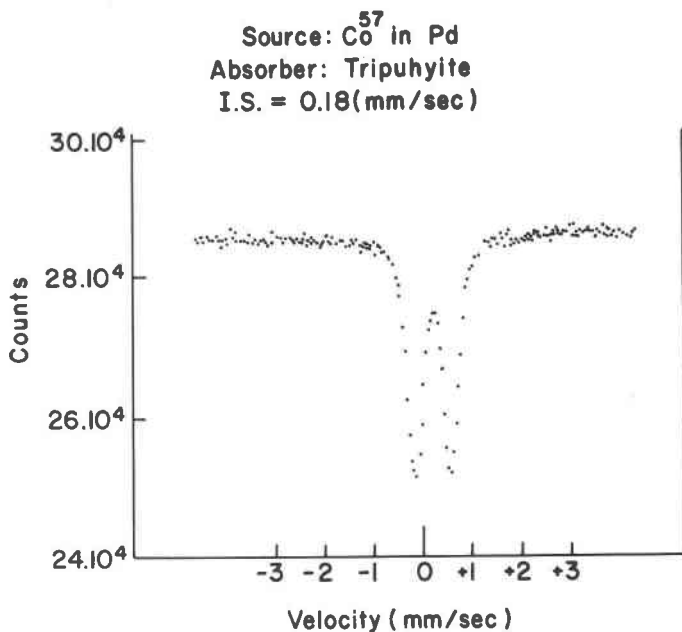

Fig. 1. Unretouched spectrum of tripuhyite at room temperature. Positive velocities correspond to source approaching absorber.

cepted formula for this mineral is $Fe_2Sb_2O_7(2FeO \cdot Sb_2O_5)$ (Hussak, 1897; Donnay and Nowacki, 1963), and on this basis one expects the iron to be in the ferrous state. Mason and Vitaliano performed X-ray studies and chemical analysis of tripuhyite from El-Antimono (Mexico) and compared them with previous studies on tripuhyite from Brazil (Hussak, 1897) and also on flajolotite ($FeSbO_4$). They concluded that the ideal formula of tripuhyite was, in fact, $FeSbO_4$, so that the iron is in the ferric state.

For our Mössbauer experiment, we used a sample of tripuhyite from

Djebel Nader[1] (Constantine, Algeria). The spectra were taken on a constant velocity, automatic recording Mössbauer spectrometer.[2] The absorber (50 mg/cm^2) and the source (Co^{57} in Pd) were both at room temperature. A typical spectrum is reproduced in Figure 1. The isomer shift is (I.S.): 0.18 ± 0.02 mm/sec for the above source. There is also a small quadrupole splitting: $\Delta E_Q = 0.72 \pm 0.02$ mm/sec, which is expected since the point symmetry at the site of the Fe nucleus is lower than cubic (Mason, 1953). The value of the I.S. versus Co^{57} in stainless steel would be: 0.46 mm/sec,[3] which is typical for the ferric (Fe^{3+}) ion (Goldanskii, 1964, p. 25). We therefore conclude, that the Mössbauer measurement strongly supports the previous assertion of Mason (1953) that iron in tripuhyite is in the ferric state, and the chemical formula should probably be: FeSbO$_4$.

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[1] Supplied by the American Museum of Natural History, New York, N.Y.

[2] Manufactured by "Elron", Electronic Industries, Haifa, Israel.

[3] Relative Isomer Shifts for various sources are given by Muir, 1966.