

X-ray diffraction (XRD) methods described cannot be used to identify the amphibole species present, to distinguish serpentine from chlorite, or to quantify serpentine or quartz, because of interferences from chlorite and talc, respectively.

Unequivocal identification and quantification of the amphibole species present on the basis of the tremolite 8.38 Å (110) peak and the anthophyllite 8.26 Å (210) peak is impossible. Examination of the powder diffraction file published by the Joint Committee on Powder Diffraction Standards (JCPDS) for amphiboles (52 amphibole cards in the JCPDS file) reveals that all 52 have their (110) or (210) diffraction peaks within ± 0.2 Å of each other. For $\text{CuK}\alpha$ radiation, a change in d -spacing of 0.2 Å at $10.5^\circ 2\theta$ (the region step scanned is 10° – $11^\circ 2\theta$) is equivalent to a 2θ angular change of only 0.25° . Slight compositional and/or structural variations in the amphiboles readily cause small shifts in the position of the (110) and (210) peaks. Similarly, very small changes in the position and/or nature of the sample surface will cause small peak shifts. As a result of general peak overlap among all amphiboles, unpredictable peak shifts, and mismeasurement of peak position, it is not possible to identify any specific amphibole on the basis of the (110) or (210) peak, even if the sample is 100% pure amphibole of one variety. It is clearly impossible to identify and/or quantify trace to minor amphiboles by the methods described in this paper. Bulk chemical analyses for major, minor, and trace elements has questionable value in the identification of an amphibole present in trace to minor amount.

The authors used the serpentine (004) peak at 3.66 Å ($24.32^\circ 2\theta$ for $\text{CuK}\alpha$) for identification of serpentine. Chlorite has a major diffraction peak (004) in this region. It is well known that chlorite and serpentine are difficult to differentiate by all known methods of characterization but that their (004) XRD peaks are separate enough to generally allow unambiguous determination of the presence of both phases when they are present in quantities sufficient to give definable peaks. However, the (004) peak of chlorite varies between 3.53 and 3.60 Å (24.73° – $25.23^\circ 2\theta$ for $\text{CuK}\alpha$) depending on the particular compositional and structural variety present. To attempt quantification of trace serpentine in talc by using the (004) peak is not possible since talcs almost invariably contain chlorite far in excess of serpentine, and chlorite will therefore interfere. The authors acknowledge that Stanley and Norwood were confronted with the chlorite-serpentine interference problem, but they still claim to have been able to use XRD for identification and quantification of trace serpentine in the presence of chlorite (samples 12 and 15).

In addition, if any of the clay minerals kaolinite, halloysite, or dickite are present, they will also interfere with the XRD identification (and quantification) of serpentine. The XRD peaks of serpentine, chlorite, kaolinite, dickite, and halloysite are typically broad and often poorly resolved, further complicating identification and almost certainly

eliminating an XRD if any of

An additional halloysite has therefore cannot

The authors ($\text{CuK}\alpha$) for quartz large peak and problem involving using a weak peak with by a strong diffractogram shoulder of the overlapping the minor amount possible in the

The interference apparent when form $y = a + b$ mineral content content with the of fit, the error detection limit quartz, and trace errors for each 1.4% for anthophyllite prepared (5%) Using the authors for the 5% stated detection (5%), the calculations of tremolite. Line ranges are not the data is quartz interferences are are distinctly equation to fit

I also wish and Norwood is an error in the electron micro 0.5 X 0.02 μm .

The entire and amphibole

eliminating any possibility of quantifying trace amounts of serpentine by XRD if any of these interfering phases are present.

An additional complication is presented by halloysite if it is present. Halloysite has a tubular structure similar to that of chrysotile and therefore cannot be distinguished from chrysotile by electron microscopy.

The authors used the quartz (211) peak at 1.54 Å (60.08° 2θ for CuKα) for quantification of quartz in spite of the fact that talc has one large peak and several small peaks in this 2θ region that can interfere. The problem involves quantification of trace to minor amounts of quartz by using a weak quartz peak [(211), relative intensity 15] that is interfered with by a strong talc peak [(060), relative intensity 55]. A recent X-ray diffractogram of very pure talc run in this laboratory had the low-angle shoulder of this talc peak extending to 59.7° 2θ, for example, completely overlapping the (211) quartz peak position. Quantification of trace to minor amounts of quartz on the basis of its (211) peak is clearly not possible in the presence of major amounts of talc.

The interference and identification problems discussed above become apparent when examining errors associated with the linear equations of the form $y = a + bx$ given by the authors to correlate peak area with percent mineral content (table 3). From table 3, comparing the known mineral content with the calculated mineral content using peak areas and equation of fit, the error for each data point can be calculated. The stated detection limits of 2.0, 0.7, 1.4, and 0.1% for anthophyllite, chrysotile, quartz, and tremolite, respectively, have very little meaning when the errors for each data point are examined. The detection limits of 2 and 1.4% for anthophyllite and quartz are less than the lowest standard prepared (5%) and are therefore strictly theoretical rather than observed. Using the authors' calibration equation for the quartz, the calculated value for the 5% standard is an overestimation of the true level by 56%. At the stated detection levels for chrysotile and tremolite (0.7 and 0.1%, respectively), the calculated values from the calibration equations are overestimations of the true levels by at least 100% for chrysotile and 500% for tremolite. Linear calibrations for these types of samples and concentration ranges are not ordinarily expected, and the use of linear equations to fit the data is questionable, especially in view of the previous discussion of interferences and invalid procedures. For example, the data for chrysotile are distinctly nonlinear (figure 2) but the authors still use a linear equation to fit the data.

I also wish to point out that the quotation from the paper by Stanley and Norwood is a paraphrase rather than a direct quotation. Also, there is an error in the quoted fiber size of 0.5 X 0.2 μm requiring transmission electron microscopy—the size stated by Stanley and Norwood is 0.5 X 0.02 μm.

The entire paper reflects a strong bias to assume that all serpentine and amphibole minerals are asbestiform. The fact that the authors added