Unknown Mineral Identification in Geological Samples

Unknown identification is a common problem that doesn't always have a simple solution. Phase identification of inorganics such as geological materials is important for grade control of ores during exploration of mineral deposits, for determining corrosion products in order to mitigate corrosion processes, and for offering clues to the formation mechanisms of rocks for geologists. X-ray diffraction (XRD) is the standard method for qualitative or quantitative unknown identification of polycrystalline materials, but further information can provide valuable insights.

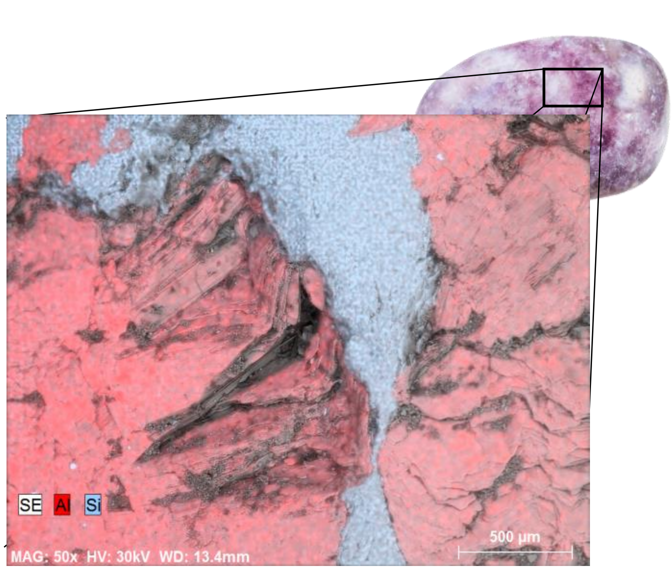

Figure 1. Top right: Optical image of a polished geological sample. Bottom left: backscattered electron image of the sample surface with EDS data represented by false colors. Red areas indicate Al-rich areas, blue areas indicate Si-rich areas.

For instance, when a more comprehensive picture is needed for an unknown material's identity, history, and predicted properties, scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS) can be used to complement XRD work. SEM visually characterizes the microstructures; EDS yields the elemental compositions and XRD identifies the crystalline structures. In this case study, all three techniques were used to identify the minerals present in the geological sample seen in Figure 1. First, the microstructure was imaged in the SEM using backscattered electrons and low vacuum mode to avoid charging effects without coating the sample. A typical region demonstrating at least two distinct microstructures is shown in the lower left of Figure 1. Simultaneously, the distributions of aluminum and silicon were recorded with EDS (mapped in red and blue, respectively.) Next, high quality spectra were recorded from each region to determine the quantitative elemental compositions. The major components in the Al- rich phase were Al, O, and Si, with trace amounts of F, K, and Mn. The Si-rich phase was a 1:2 ratio of Si:O, with trace amounts of Al and K, strongly suggesting SiO_2. However, there are several chemically identical forms of silicon dioxide; crystobalite, quartz, and glass all share the same chemical fomula.

An XRD pattern measures the crystalline structure of a material, which can be used to fingerprint unknowns and distinguish between different phases of the same chemical. The sample's XRD pattern was measured using a Rigaku SmartLab XRD with a Cu Kα x-ray source run at 40 kV and 44 mA in the Bragg-Brentano geometry. The diffraction pattern is shown below in Figure 2. The peak positions and relative intensity ratios were compared to those of reference materials in powder diffraction file library in order to positively identify the crystalline structures. The sample was found to contain three different materials listed in Table 1. The peak positions of the matching phases are indicated with the experimental pattern in Figure 2.

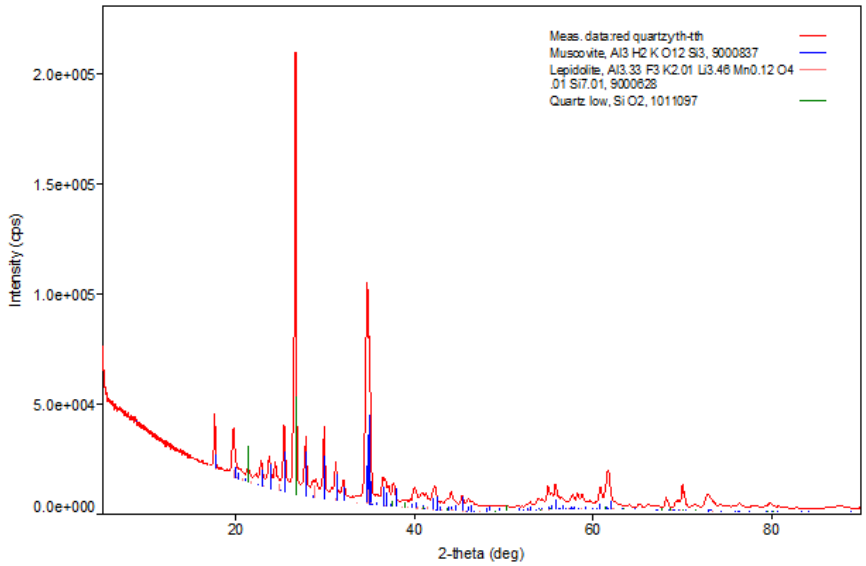

Figure 2. X-ray diffraction pattern of the geological sample. Blue, pink, and green vertical lines indicate the predicted angles of lepidolite, muscovite, and quartz diffraction peaks, respectively.

The regions with layered microstructures seen by SEM are a likely a mixture of lepidolite and muscovite, since both materials are layered phyllosilicate minerals in the mica family and are known to intermix. The EDS measurements of the elemental composition show that it contains aluminum, fluorine, and manganese. Manganese is responsible for the pink to purple color in lepidolite, which is consistent with the sample's optical appearance. Similarly, the smooth phase was identified as quartz from the EDS elemental composition measurement. Our findings are supported by geological observations of muscovite, lepidolite, and quartz occurring together in granitic pegmatites. As demonstrated with this geological sample, the combined analysis using XRD, SEM, and EDS can provide a much more complete understanding of a material than any one technique could alone.

Table 1 Possible Formulas of Phases Identified by XRD

Phase name	**Possible Formula**
Muscovite	$KAl_3Si_3O_{10}(OH)_2$
Lepidolite	$K(Li,Al)_3(Si,Al,Mn)_4O_{10}(F,OH)_2$
Quartz	SiO_2

Address: 10025 Valley View Road, Ste 150, Eden Prairie, MN 55344, U.S.A.
Tel: +1 (952) 941-2199, Fax: +1 (952) 746-8086, Email: natlab@ebatco.com
www.ebatco.com