



Ebatco Nano

A Bimonthly Newsletter

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Nano Brief

Thank you to all who attended the training course, “Contamination, Impurity, and Unknown Material Identification”! It was a big success and we hope to continue providing advanced and practical training courses on a variety of topics in the near future.

Ebatco will be exhibiting at these upcoming events:

- October 23rd – October 24th, MD&M 2019, Booth #1040, Minneapolis Convention Center, Minneapolis, MN
- November 10th – 14th, ISTFA 2019, Booth #806, Oregon Convention Center, Portland, OR
- December 1st – 6th, Materials Research Society Fall Meeting & Exhibit, Hynes Convention Center, Boston, MA

Please stop by our booth to discuss the incredible world of surface sciences, nanotechnologies, nanomaterials, and nano/micro scale material and device characterization with our staff scientists. We hope to see you there!

Ebatco

In order to better support the materials analysis needs of our customers, Ebatco has acquired a Rigaku SmartLab X-Ray Diffractometer. The SmartLab XRD is designed for quickly switching between parallel beam and Bragg-Brentano modes, allowing us to provide everything from high resolution single crystal measurements to powder diffraction services with a minimum of instrumental setup time. XRD is an ideal method for measuring quantitative composition, film thicknesses, residual stress and strain, lattice parameters, polymer crystallinity, and identifying unknown materials. XRD is a fast and non-destructive method for measuring structural properties of a huge range of materials, such as metals, alloys, ceramics, geological samples, pharmaceuticals, oxides, and thin films.



The Rigaku SmartLab X-ray Diffractometer

Case Study

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.

For instance, when a more comprehensive picture is needed for an unknown material's identity, history, and anticipated properties, scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS), could 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 precisely. An accelerating voltage of 30 kV was used to ensure all elements present would be detected. The major components in the aluminum rich phase were Al, O, and Si, with trace amounts of F, K, and Mn. The silicon-rich phase was a 1:2 ratio of Si:O, with trace amounts of Al and K, strongly suggesting SiO₂. However, there are several chemically identical forms of silicon dioxide; cristobalite, quartz, and glass all share the same chemical formula.

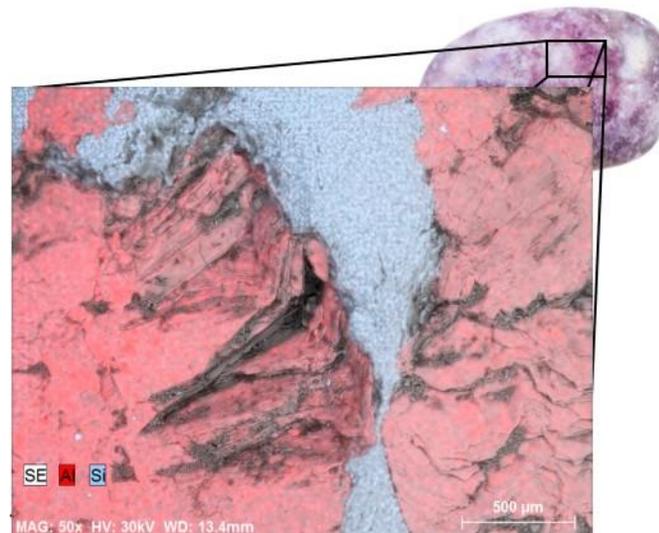


Figure 1. Top right: Optical image of a polished geological sample. Bottom left: backscattered electron image of the sample surface with EDS data represented by false colors. Red areas indicate aluminum-rich areas, blue areas indicate Si-rich areas

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 K α x-ray source run at 40 kV and 44 mA in the Bragg-Brentano geometry. The sample is polycrystalline and did not require any sample preparation for qualitative phase identification. The resulting 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.

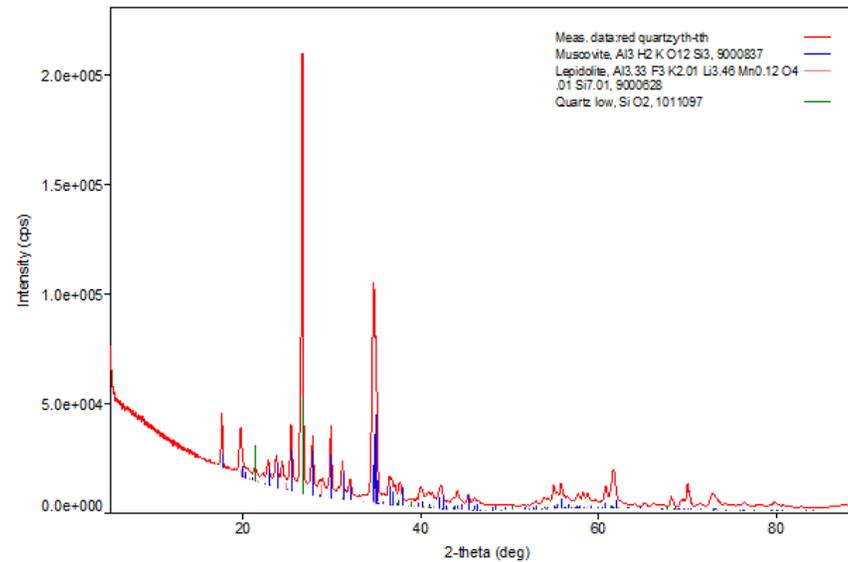


Figure 2. X-ray diffraction pattern of the geological sample. Blue, pink, and green vertical lines indicate the predicted angles of lepidolite, muscovite, and quartz diffraction peaks, respectively.

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.

Table 1 Possible Formulas of Phases Identified by XRD

Phase name	Possible Formula
Muscovite	$\text{KAl}_3\text{Si}_3\text{O}_{10}(\text{OH})_2$
Lepidolite	$\text{K}(\text{Li},\text{Al})_3(\text{Si},\text{Al},\text{Mn})_4\text{O}_{10}(\text{F},\text{OH})_2$
Quartz	SiO_2

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.

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