Powder X-ray Diffraction Method (XRD)

X-ray diffraction analysis is a non-destructive analysis in which a sample is scanned on an X-ray diffractometer to determine atomic scattering of an X-ray beam which can be interpreted to reveal crystallographic information about the material. For unknown materials, the method can be used to identify mineral and crystalline components by comparison with a database of reference materials.

X-ray Equipment

Powder XRD scans were collected on PANalytical “X’Pert Pro – MPD X-ray Diffractometer using the following settings: theta/theta geometry, Cu long-fine-focus X-ray tube (Ni filtered), “X’celerator” solid state “strip” detector, X-ray tube setting: 45 kV, 40 mA, 15 mm beam mask, ½° anti-scatter slit, ¼° divergence slit, ½° receiving anti-scatter slit, ¼° receiving divergence slit, step size 0.033 degrees in continuous scan mode, scan range of 5 to 80 degrees two-theta, sample spinner on, scan rate was 1 degree per minute.

Parallel beam scans of rock chip surfaces were collected on a Rigaku SmartLab Diffractometer with the following settings: Cu K alpha sealed tube source 40 kV 40 mA, One dimensional solid state Rigaku Dtex detector, with Ni foil Cu K beta filter, 5 degree source and detector Soller slits, 10 mm wide incident beam slit, 2/3 degree incident bean divergence slit, data collected at 6 degrees/minute, 0.02 degrees step size, and data collection range 3-150 degrees two theta.

Sample Preparation

Sample preparation was limited to grinding using a Spex mixer mill with zirconium vial set for select samples. All powders, both unprocessed and milled were passed through a 100 mesh sieve (150 um) to remove large particles prior to mounting. Three sample mounts were employed dependent on the amount of powder available. Samples with 1 gram of material were back packed into a Panalytical random powder mount. Samples with mass between 500 mg and 1 gram were top loaded into a Panalytical random powder mount with a zero background plastic insert. Samples with less than 500 mg were sprinkled onto a silicon zero background wafer and then placed into a Panalytical universal holder.

Qualitative Mineral Identification

Minerals are identified based on peak position and intensity in the XRD scan compared to the mineral reference data. The US Geological Survey subscribes to International Center for Diffraction Data (ICDD) PDF-4/Minerals 2022 database which contains more than 23,000 entries.

A typical detection limit for a mineral is approximately 3 weight percent; however the limit varies from one mineral to the next depending upon several factors including: crystallinity, interference with other minerals, atomic substitutions, and other factors. Any real world sample will differ slightly from the reference mineral listed in the database. Thus there will be slight differences in peak position (two-theta) and intensity that result from any elemental substitutions, crystal defects, differences in sample preparation or data collection conditions.

In some cases, a reflection may be observed in a pattern, but not assigned to a phase, indicating that at least one more phase is present in the material. Based on the position and character of the reflection (intensity and shape), the phase may be limited to a small group of minerals. These are typically reported as a “questionable” phase and require further analysis and data to confirm their presence.

Whole Pattern Fit Model

Semi-quantitative mineral estimates are calculated using ICDD (2021). JADE Pro v8.4 (Computer Software), ICDD, Livermore CA, USA which simultaneously calculates a whole pattern fit and a Rietveld refinement of the minerals. Reference minerals are selected from the ICDD database, some of which are “structure” references that represent perfect crystals of the mineral and other entries are real world mineral specimens. Each card contains a full crystallographic description of the mineral. A calculated model of the observed pattern is produced by non-linear, least-squares optimization. The calculations, performed by the software, involve the application of various parameters to improve the fit of the model to the observed data. Modeling parameters include background reduction, profile fitting, and lattice constants which iterates minimizing a residual error between the calculated x-ray diffraction pattern from reference cards compared to the measured scan of the sample. All data are normalized to 100% based on the identified minerals. A full description of the Whole Pattern Fit algorithm is available from ICDD.

Limitations of the X-ray diffraction data

X-ray diffraction analysis identifies the crystalline portion of the sample. It does not identify any amorphous (inorganic or organic) phases present with the exception that, with the addition of an internal standard, the total amorphous content of the sample can be calculated. Internal standards were not employed in this study. The mineral contents in the table are reported as major (>15%), minor (5-15%), trace (<5%), and “?” questionable (presence not confirmed) and represent only the crystalline portion of the sample.