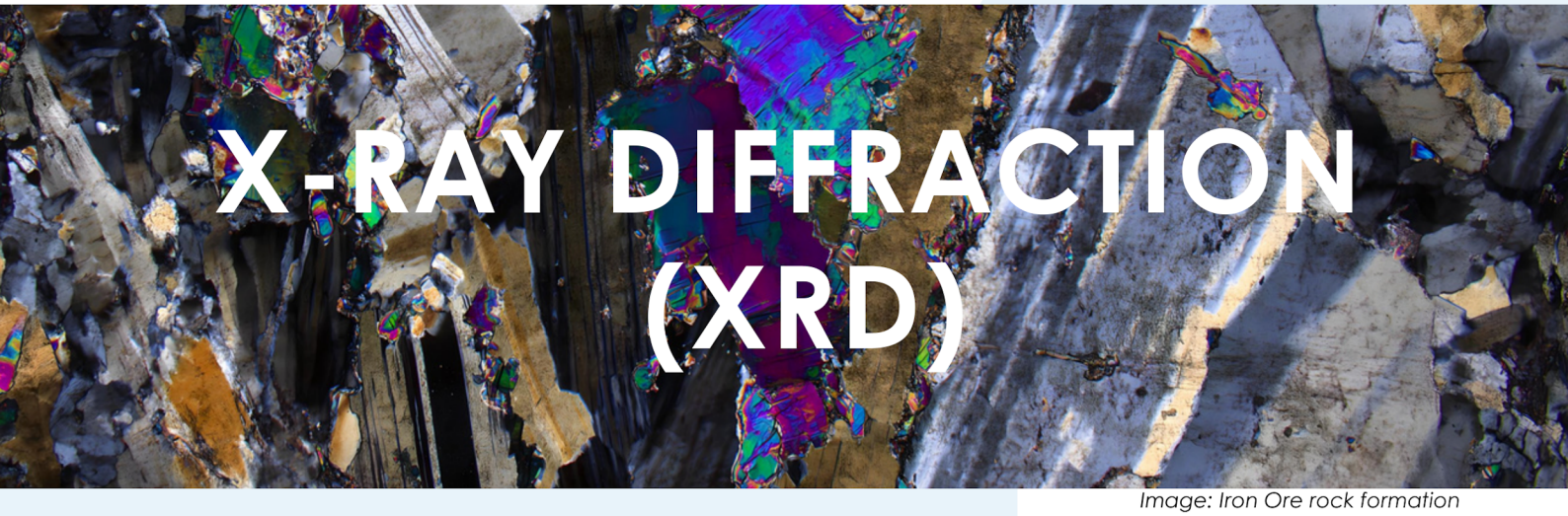
microanalysis australia



X-RAY DIFFRACTION (XRD)

Image: Iron Ore rock formation

XRD

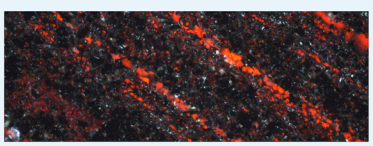
X-Ray Diffraction (XRD) is a technique for examining the arrangement of atoms in a crystal lattice. This is a comparative technique where the pattern obtained for a sample is compared against a standards database. Elemental composition is not determined by XRD but can be inferred from the results. XRD analysis can be performed on any crystalline solids ranging from single minerals and crystals, pre-pulped assay samples, and whole rocks or drill core. The ultimate goal of XRD in all these cases is to identify phases present and their abundances.

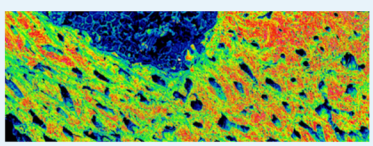
Other information that can be gleaned from an XRD trace includes the degree of weathering or alteration, crystallite size, substitution, degree of disorder and the amorphous content.

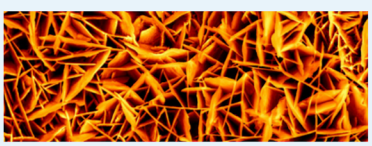
In real world samples it is common to have over a dozen different mineral phases at varying concentrations. The use of elemental assay, information of the sample's history and location, and complimentary techniques can all assist analytical outputs by strengthening confidence in phase identifications.

Image: Sample preparation for XRD analysis

5 Alvan Street, Mount Lawley 6050 P:08 9225 5810 E:admin@microanalysis.com.au ABN:65 133 060 787







XRD techniques vary from qualitative to semi quantitative through to fully quantitative Rietveld analysis. Each different technique will deliver different levels of accuracy and precision. For each technique a careful balance is reached between sample preparation processes, analytical instrument choice, analyst time, and overall project goals and costs.

One of the most challenging areas for phase identification is clay speciation. Due to the poor crystallinity and irregular ordering of some clay groups, identification may require further work involving glycolation, heat treatment and other techniques. Microanalysis are proud to offer this service.

Microanalysis operates several automated powder X-ray Diffractometers. We have several diffraction databases including the latest complete International Centre for Diffraction Database (ICDD). Our standard holders take approximately one cubic centimetre of powdered sample but we also have holders that allow measurements of milligram quantities of sample.

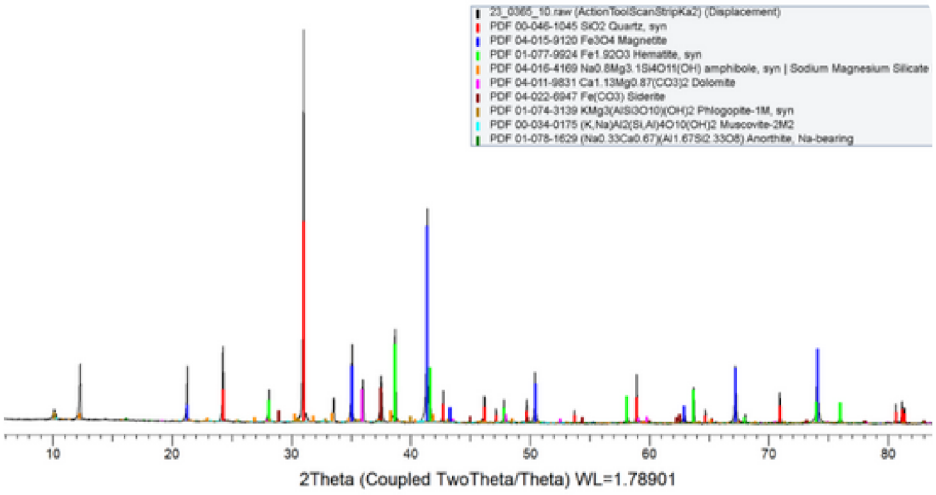


Image: XRD analysis of mineral composition

The techniques we frequently utilise are:

- Qualitative XRD where phases are identified with abundances listed as major, minor, and trace.
- Semi-quantitative XRD where phase identification includes percentage concentrations calculated using the normalised reference intensity ratio method. The intensity of the 100% peak divided by the published I/Ic value for each mineral phase is summed and the relative percentages of each phase calculated based on the relative contribution to the sum. An estimation of the amorphous content can also be given.
- Quantitative XRD analysis using an internal standard. Rietveld analysis offers the highest confidence possible in the concentration of phases and amorphous content.
- Crystalline silica determinations for alpha quartz, cristobalite, and tridymite. A full phase identification checks for overlaps that may be present from the phases present. A specific peak scan is performed with this result compared against a five point calibration curve to produce a value of each crystalline silica phase present.
- Clay speciation. A semi-quantitative analysis is performed followed by gylcolation and heating to determine specific clay groups.
- Respirable free silica determination. A semi-quantitative analysis is performed on respirable size fractions for crystalline silica and its polymorphs (cristobalite and tridymite), and is combined with particle size determinations and additional scanning electron microscopy.
- Comparative XRD. If there is no entry in the databases for a given compound we can construct a pattern from a suitable reference standard for comparative analysis.
- Specific mineral semi-quantitative analysis. Phase(s) of interest are concentrated through solvent washing, acid washing, heavy liquid separation, magnetic separation or other techniques to improve confidence in phase identification or to enhance XRD features to study substitution.