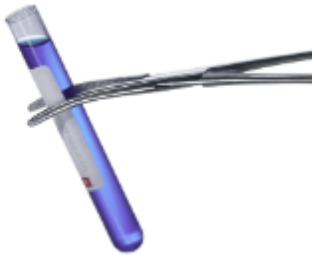
Lakehead UNIVERSITY | Centre for Analytical Services

Technical Bulletin



X-Ray Diffraction (XRD)

Uses of X-Ray Diffraction

The use of X-ray diffraction in analytical techniques has many very versatile applications:

- Environmental: e.g. dust, pipework precipitates, asbestos
- Mineral Collectors and Dealers
- Mining and Exploration companies
- Metals and Alloys
- Soils and Clay
- Clinkers and Cement
- Ceramics
- Raw materials
- Chemicals
- Nanomaterials
- Pharmaceuticals
- Forensics
- Environments

Basic Introduction to XRD

X-ray Powder Diffraction (XRD describes the interaction of X-rays with a crystalline substance that results in a diffraction pattern. The diffraction pattern that the powder emits is analogous to a "fingerprint" of the substance. XRD allows us to identify and characterize polycrystalline phases. To increase certainty, XRD data is often used in combination with other mineral analytical techniques such as SEM (Scanning Electron Microscope) and optical microscopy.

In X-ray diffraction work, high energy electrons strike a pure anode in a sealed vacuum tube which produces X-rays. Copper X-ray tubes are most commonly used for the diffraction of inorganic materials. $K\alpha_1$, $K\alpha_2$ and K_β radiation are produced by this technique.

- The $K\alpha_1$ is used for the analysis of patterns.
- The K_β radiation if removed by a filter.
- The $K\alpha_2$ radiation is removed electronically during analysis of patterns.

A variety of scatter, soller and receiving slits help produce a parallel focused incident beam of X-rays. The incident X-rays strike the sample and diffracted X-rays are produced and measured, producing a diffraction pattern. This diffraction pattern is the "fingerprint" of the phase (or the "fingerprint" of various phases that could be present in the sample). The pattern is used in a search/match program to correctly identify the phases present in the sample. The search/match software is specific to the XRD, however, all search/match software utilizes standard diffraction data sets (see below).

Air Sensitive XRD

Samples which degrade easily when exposed to oxygen or humidity are carefully prepared for x-ray diffraction in a glove box (inert atmosphere). The sample is mounted onto a specialized air sensitive sample holder which is covered and protected with a thin layer of Kapton foil. This allows

the sample to be analyzed without any contact with air or humidity. *Lakehead University is the only analytical laboratory in Canada to offer this specialty service.*

Multi-Phase Semi-Quantitative Analysis: affordable and fast results for prospectors and those involved in mineral exploration

Semi-quantitative analysis calculates the estimated mass fractions of the accepted phases present in the sample. The scale factor and the RIR (=Reference Intensity Ratio) values (also called I/Ic values) from the standard database (ICDD, ICSD) are used to perform the calculation. The result is a list of phases present in the sample along with an estimation of their quantitative abundances.

This technique is very suitable for geologists looking for a quick, inexpensive technique to identify the mineral phases and their abundances in a rock. It is an alternative to thin section and petrographic methods, which can be costly, time consuming and sometimes not necessary. In addition, it correctly identifies the alteration minerals that are present in the rocks, which can be difficult if not impossible to identify by petrographic methods.

Quantitative Phase Analysis utilizing Rietveld Refinement Technique: used to describe materials' structure

The Rietveld method is used in the characterization of crystalline materials. The height, width and position of the peaks in an XRD scan can be used to quantify a phase (or multiple phases) and give valuable information about the material's structure. Some of the advantages of using the Rietveld Refinement method for quantitative analysis are 1) the calibration constants are calculated from reliable structural data, and 2) all of the reflections are included in the calculations.

Overall Advantages of XRD

- Fast analysis (automated analysis also available with sets of 30 samples)
- Non-destructive
- Lower cost than most analytical applications
- Extremely small samples are required
- Identifies different phases with the same composition
- XRD can identify phases that can't be identified by other means (ex. Soft, altered rocks that can be difficult to prepare a thin section)

Lakehead University's Instrumentation Laboratory recently acquired the PANalytical X'Pert Pro MPD and X'Pert data collector and X'Pert Highscore Plus software for total X-ray powder pattern analysis. Standards for XRD analysis include the International Centre for Diffraction Data (ICDD) sets 1-50, and the Inorganic Crystal Structure Database (ICSD).

References

Klug, H.P., and L.E. Alexander. 1974. X-ray diffraction procedures for polycrystalline and amorphous materials. 2 ed. Wiley, New York.

Young, R.A. 1993. The Rietveld Method, Oxford University Press.

James Connolly (2007) (website)

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