

Powder X-Ray Diffractometer (XRD)

Instrumentation and Sample Preparation

Instrumentation / Model:

- Rigaku Miniflex 600 (Figure 1)
- Uses a 600 W X-ray generator with a cobalt anode (X-ray tube)
- Produces powder-diffractograms that are unique to each crystal structure of a solid.

Sample Preparation:

1. Solid sample is crushed and ground into a fine paste using a pestle and mortar and a solvent (acetone or water).
2. Sample is milled into a fine powdered paste using an XRD-Mill McCrone.
3. Sample is dried, ground and pressed flat into an XRD sample holder. (Figure 2)



Figure 1: Miniflex 8-Sample Holder



Figure 2: Miniflex XRD Instrument

How does it work?

X-Ray Generation:

A cathode generates electrons that are propagated along the x-ray tube in a vacuum. A cobalt plate acts as the anode where electrons hit the surface and cause excitation.

De-excitation occurs in the form of heat radiation (99%) and X-ray emission (1%).

Bragg's Law:

$$n\lambda = 2d \sin \theta$$

Within a lattice, there are planes that are separated by distance d . Where the incident wavelength (or integer multiple, $n\lambda$) is equal to the path length ($2d \sin \theta$), Bragg's law is satisfied and a diffraction maximum is observed. (Figure 3)

Detection:

Diffraction maxima are plotted on a diffractogram of 2θ vs. intensity – and as each diffraction maximum is unique to each mineral – so is the diffractogram.

A diffractogram acts as the 'fingerprint' of each mineral. (Figure 4)

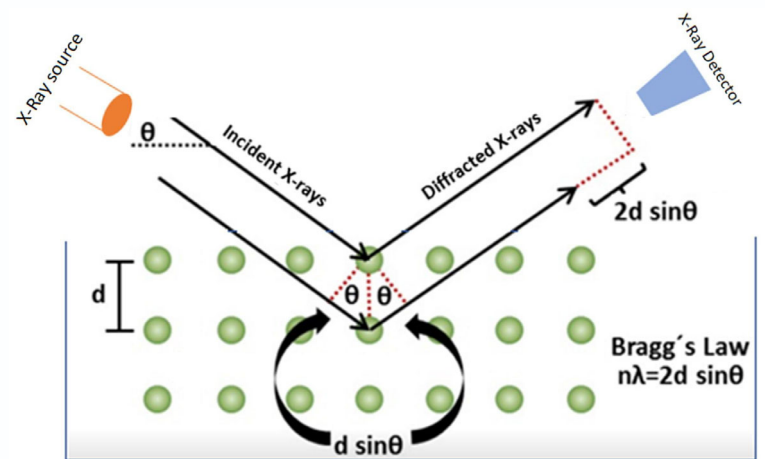


Figure 3: Diagram of the Theory of Operation of an X-Ray Diffractometer

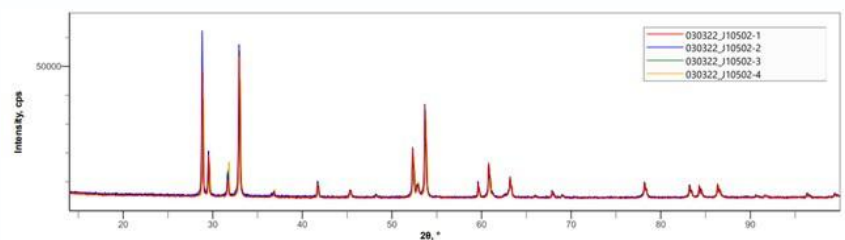


Figure 4: Example of LCM Analysis of NdPr Metal-Making Bath Chemistry

What does it detect?

Minerals and Alloys:

Each mineral has unique values for d which can be measured as a diffractogram.

A diffraction pattern can be measured from an unknown sample which can be pure or a mixture of different minerals. These are compared to a crystallography open database (COD) of known materials, and the relative compositions of the sample can be measured.

At less common metals, powders of samarium cobalt, neodymium iron boron and NdPr metal-making bath chemistry samples, and customer specified alloy samples, are analysed.

Analysis is rapid (10 minutes) and is non-destructive. The Miniflex has an 8-sample holder for rapid, sequential analysis.