Basic Powder XRD methods at RECX

Phase identification, Quantitative Analysis and Lattice parameter refinement

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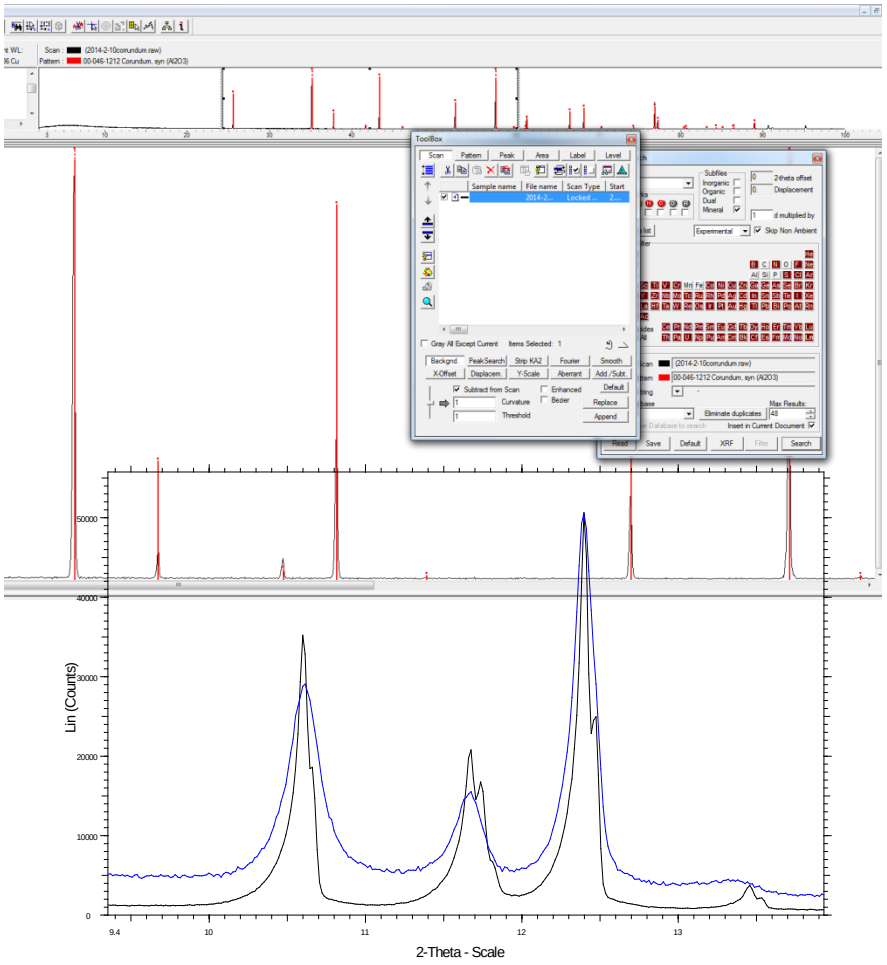
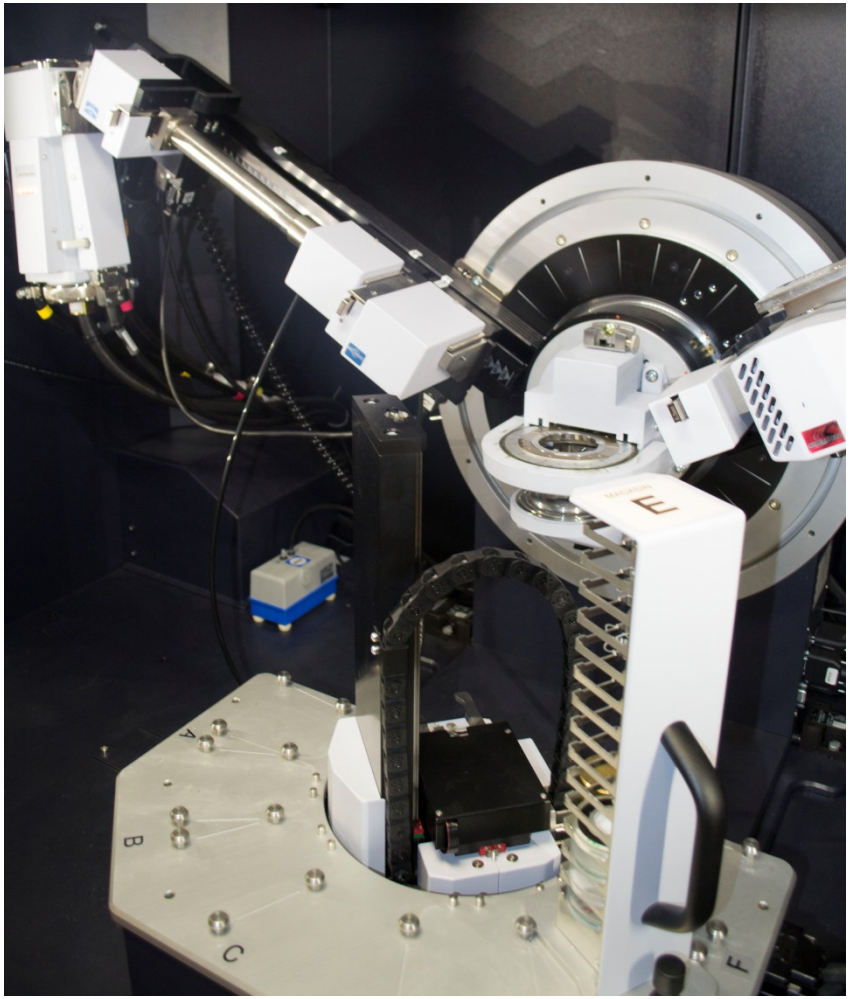
What can you get from a Powder Diffraction Pattern?

- Peak positions
- Peak Intensities
  - Phase identification
  - Quantitative Analysis (QPA)
  - Lattice parameter refinement
- Peak Shapes
  - Crystallite size
  - strain



Diffractometer and Data collection

- Any diffractometer!
  - automated routine instruments are the most common choice
    - Bruker D8 Discover in Oslo
    - Bruker D8 Advance in Trondheim
  - Capillary instruments may be used to reduce preferred orientation
  - More complicated applications require better data



How Can We Help You?

- Data collection
  - And advice on data collection methods for specific samples
  - We have experience of a range of difficult sample types
- Training
- Phase identification and analysis
  - Subject to “man hour” costs

Phase Identification

- Diffraction pattern is a unique fingerprint of the crystal lattice which produced it
- Phases from a database are matched to the observed pattern using line position and intensity
- Sample height correction is important
  - Back loading sample holders can help
  - Capillaries give best reproducibility
- ICDD and COD databases available at both RECX PXRD nodes
- Databases are integrated into the EVA pattern analysis software for automatic searching

Quantitative Analysis

- The peak intensities are related to the weight fractions of phases in a mixture
- Once the phases in a sample are known they can be quantified
- Traditional method:
  - Identify unique, non-overlapping peaks for each phase
  - measure the intensities
  - Calculate phase fractions

Quantitative Analysis

- Modern Method
  - Full profile Rietveld refinement
- Advantages
  - More accurate- uses all peaks
  - Easy to do with modern software and can be automated
  - Unknown phases can be included (PONKCS method)
- Requirements
  - Need structural data for the phases (unless using PONKCS)
  - Need to use Rietveld software (relatively straightforward today)
  - Preferred orientation should be avoided

QPA demo

- Si and LaB6

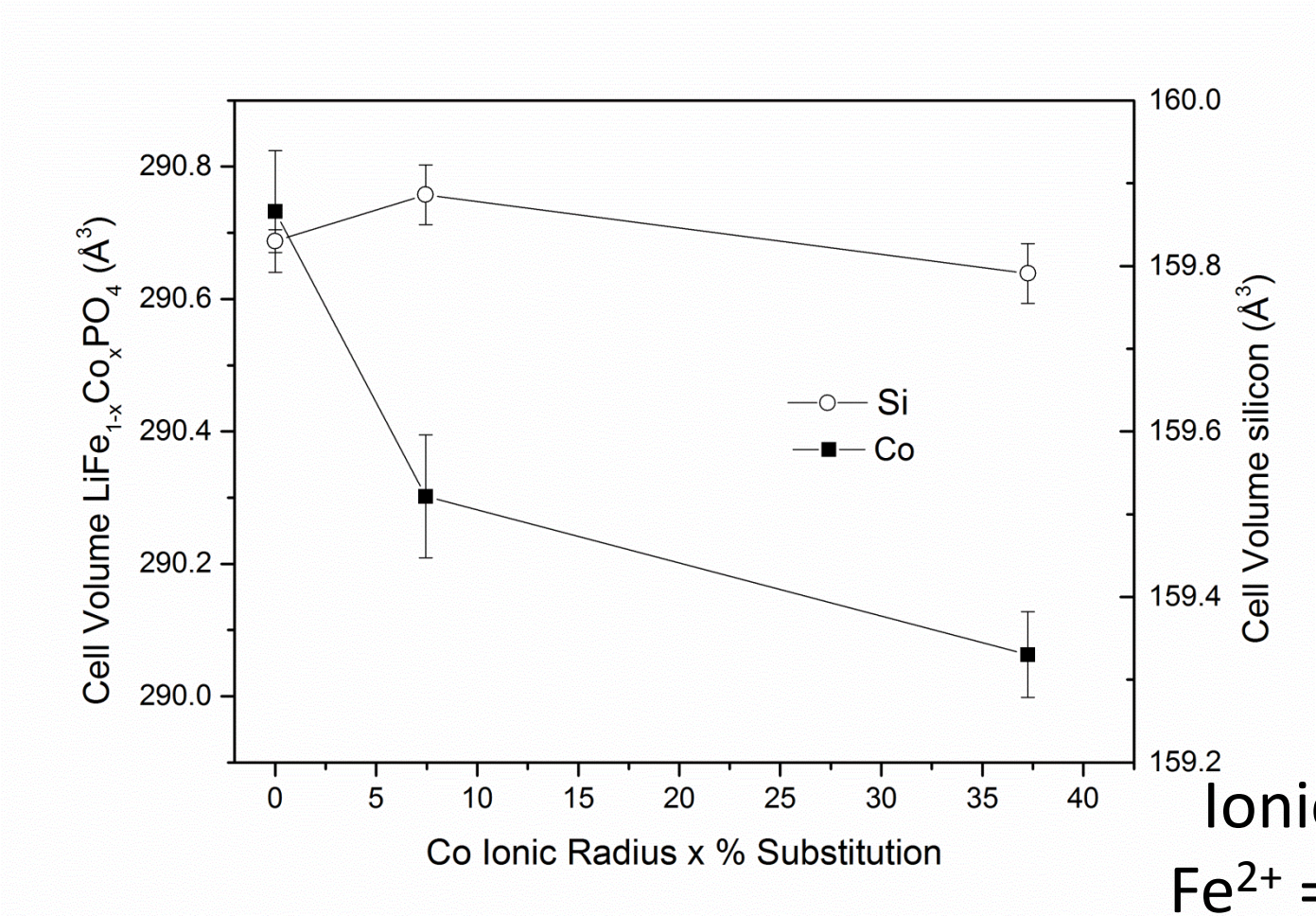
Lattice refinement

- Substitution of atoms in a lattice changes peak positions and intensities
- We can study the degree of substitution by studying the size of the crystal lattice (from peak positions)
- A starting “parent” unit cell is required
- Least squares fitting methods are used
  - Old:
    - Get parent and substituted peak positions
    - Fit numerically
  - New
    - Fit parent unit cell against substituted data with full pattern refinement
    - Full Rietveld method (peak intensities from structure) or Structureless methods (dummy intensities) can be used (Pawley and Le Bail methods)

Structureless Lattice Refinement Demo

- Co in LiFePO4

Example Data From Lattice Parameter Refinement



Ionic Radii

Fe^{2+} = 78 pm

Co^{2+} = 74 pm

Data: Ingunn Sandberg Masters Project, 2014

Summary

- A lot of data can be extracted from quick, routine powder XRD data collections
  - Phase identification
  - Quantitative analysis
  - Lattice parameters
  - Crystallite size (approximate)

RECX can help you to do this!