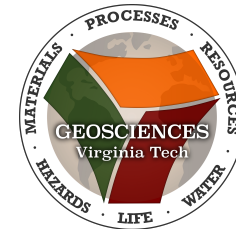VIRGINIA TECH™

Mineral Identification and Quantification by Powder X-ray Diffraction

F. Marc Michel

Associate Professor

Department of Geosciences
Hamlett Junior Faculty Fellow
Leader, Division of Nanoscience
Deputy Director, NanoEarth
Virginia Tech, Blacksburg, VA USA

marc.michel@vt.edu

What's in my sample?

Virginia Tech Crystallography Lab (VTX)

Equipment & Services:

- Panalytical Empyrean Nano Edition

- Equipped for wide variety of experiments (pXRD, SAXS/WAXS, GI-XRD, XRR, xPDF)

- **Powder x-ray diffraction (pXRD)** is widely used in Earth and environmental sciences

Should you use powder diffraction (pXRD)?

If you want to know…

- The **identity** of the mineral(s) present

- The **proportions** of different minerals are present

- Important **structural details** (e.g., presence of elemental impurities, strain, defects, etc.)

Powder diffraction most used for samples that are…

- **Crystalline** and **nanocrystalline** materials

- **Natural** and **synthetic**

- **Dry** (particle sizes <1 μm, amount >20 mg)
  - Also solid/pressed pellets, filter papers, dense suspensions, etc.

Mineral Identification: "Fingerprinting"
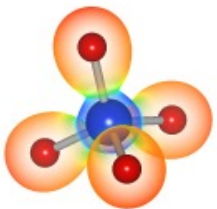

Arch Loop Whorl
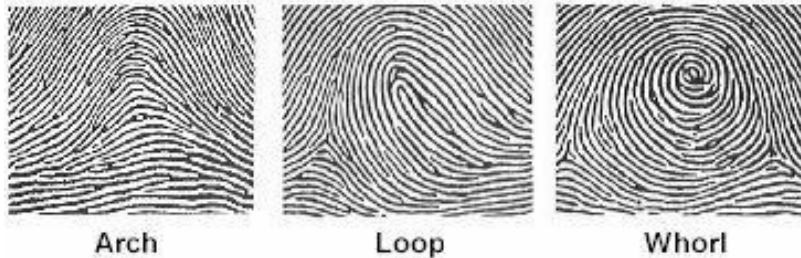
Standard approaches:

- Calculate patterns from known structures and compare (free way)

Crystallography Open Database

http://www.crystallography.net/cod/
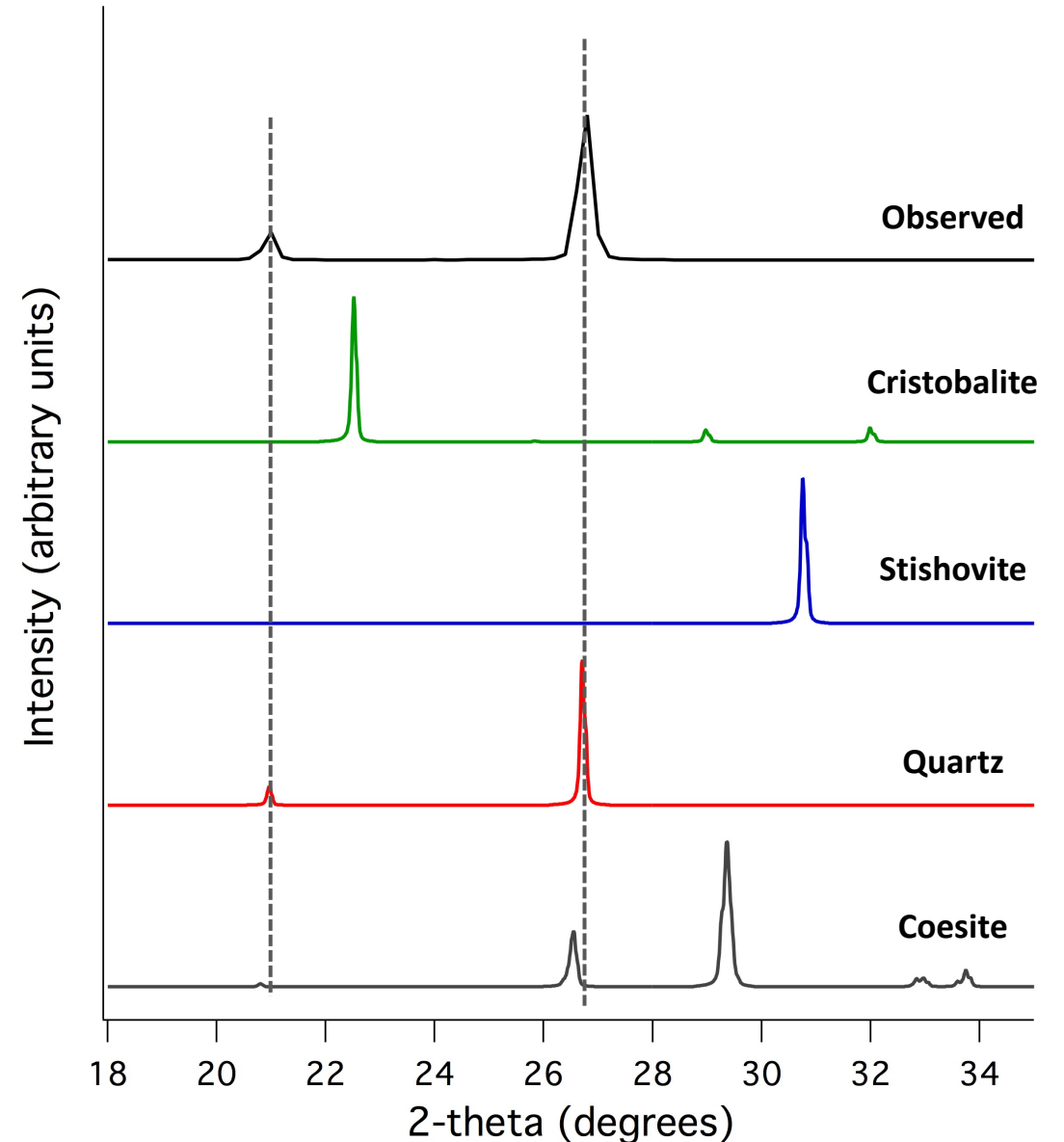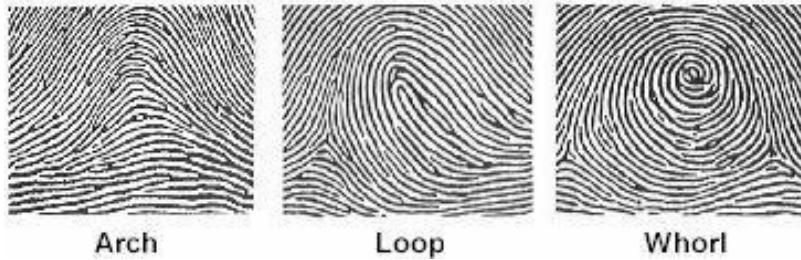

VESTA

Visualization for Electronic and STructural Analysis

www.jp-minerals.org

Observed

Cristobalite

Stishovite

Quartz

Coesite

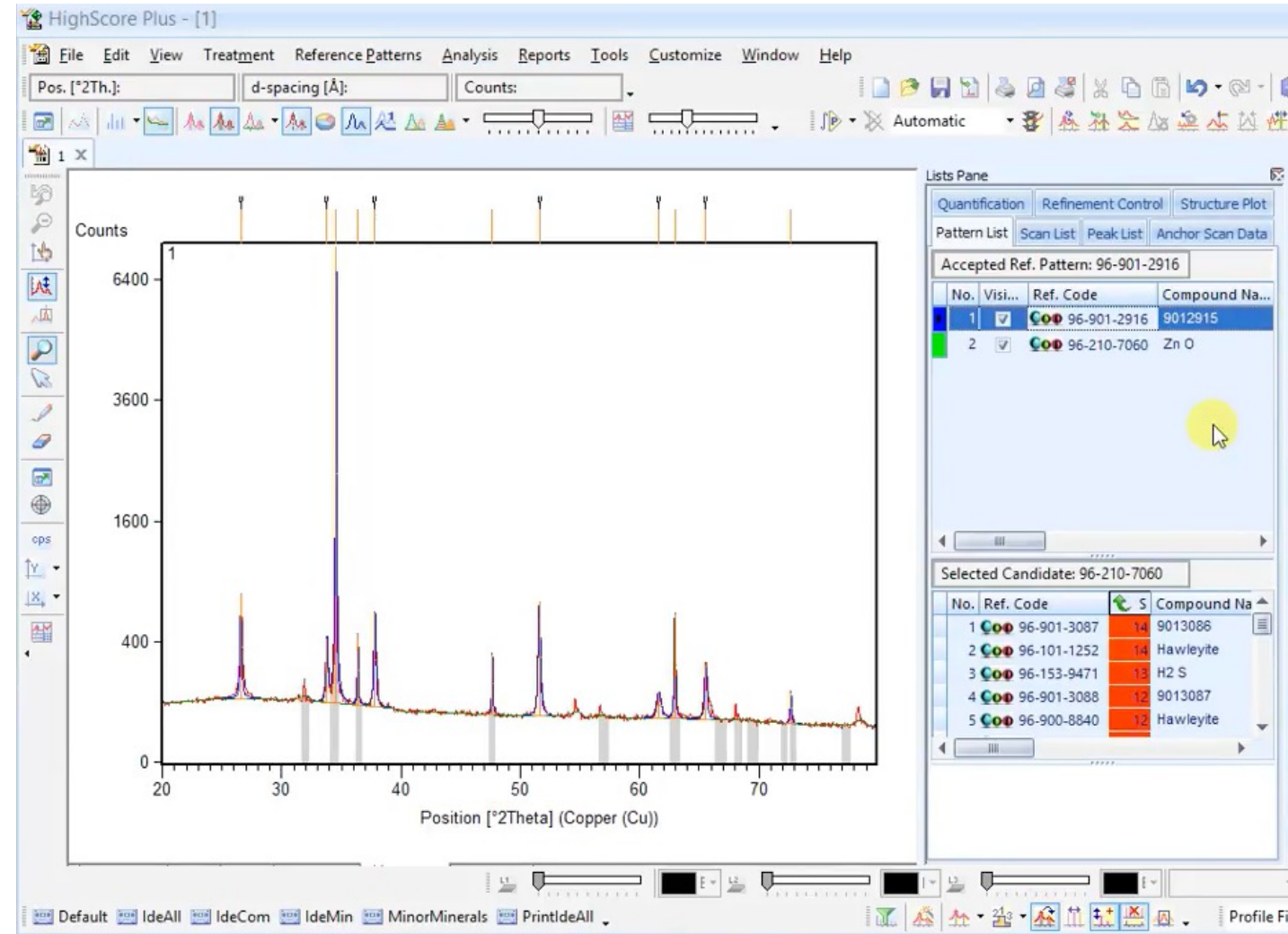Intensity (arbitrary units)

2-theta (degrees)

Mineral Identification: "Fingerprinting"

Arch   Loop   Whorl

Standard approaches:

- Calculate patterns from known structures and compare (free way)

- Automated search-match (usually requires licensed software/databases)

- Always add pinch of intuition

Fitting & Quantification

Standard approaches:

- Full-profile fitting using Rietveld analysis
  - Absolute mineral abundances
  - Structural details (e.g., lattice parameters, site occupancies, microstrain)

- Open-source software available (e.g., GSAS-II)

- Other analysis methods available – specific to samples and questions
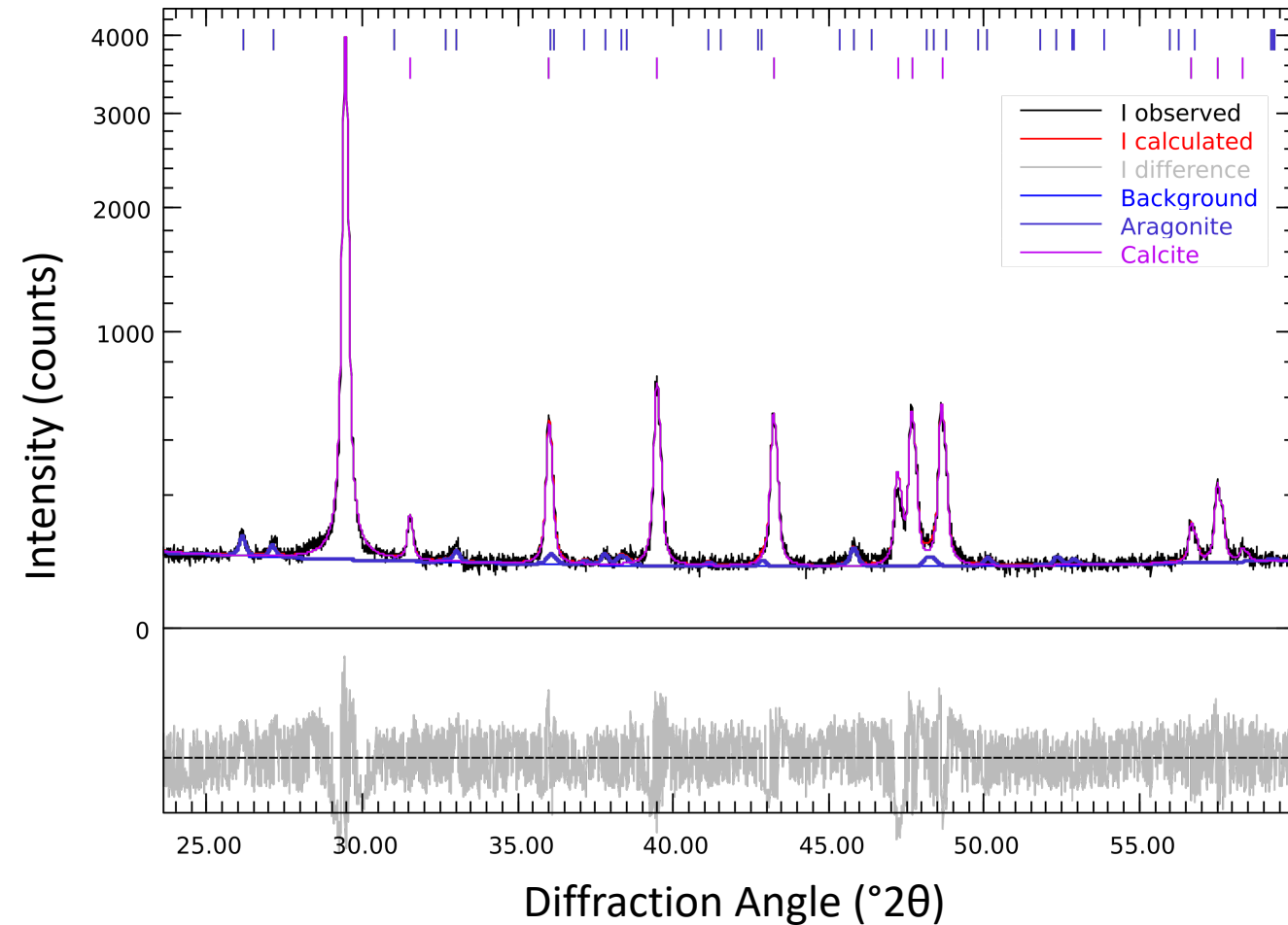
Diffraction of Nanoparticles
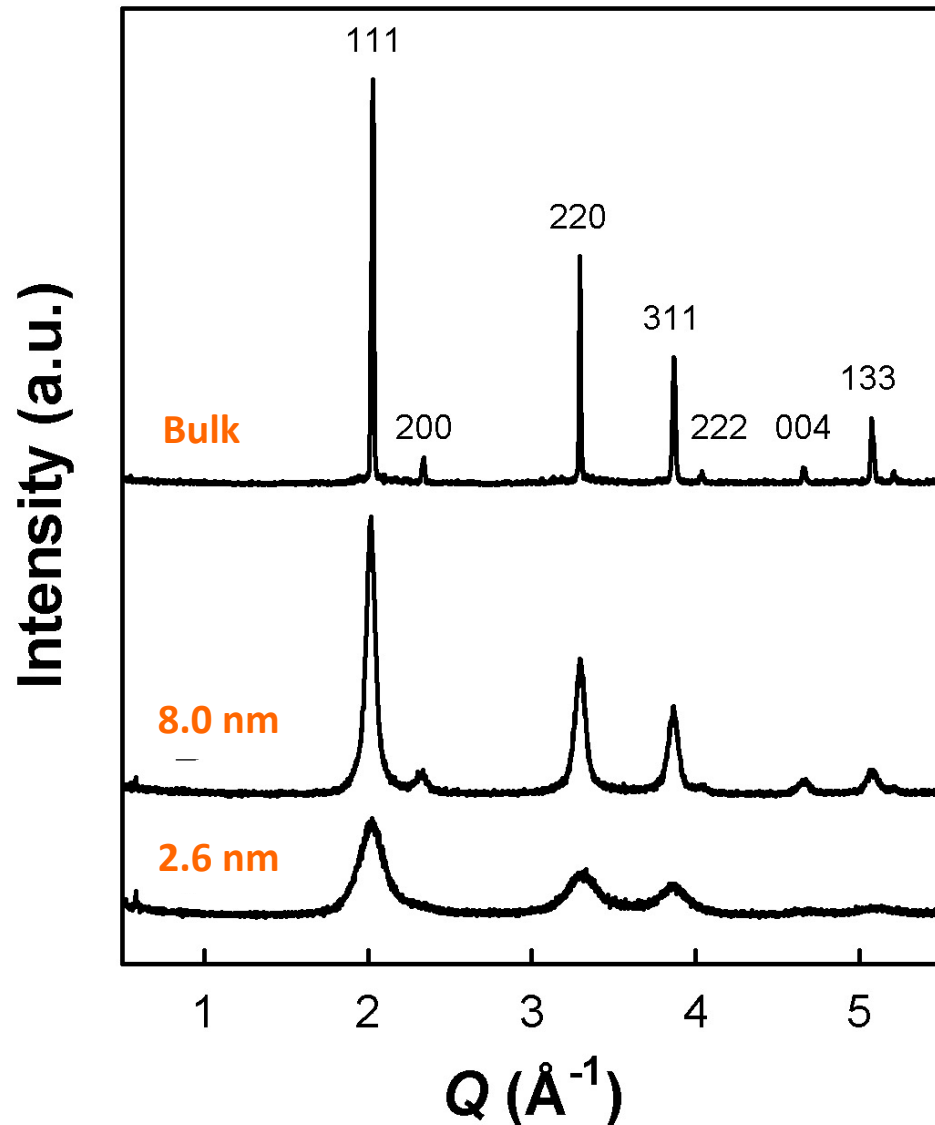
The "problem"…

- Crystallite size *inversely* related to peak width

- Decreasing size increases broadening often leading to strong peak overlaps, which can limit:
  - Phase ID & detection of minor phases
  - Extracting structural details

- Provides info on average crystallite size and shape (typically <200 nm)
  - Quick way: Scherrer analysis
 - Best way: Rietveld analysis

Partially Disordered to Amorphous Samples
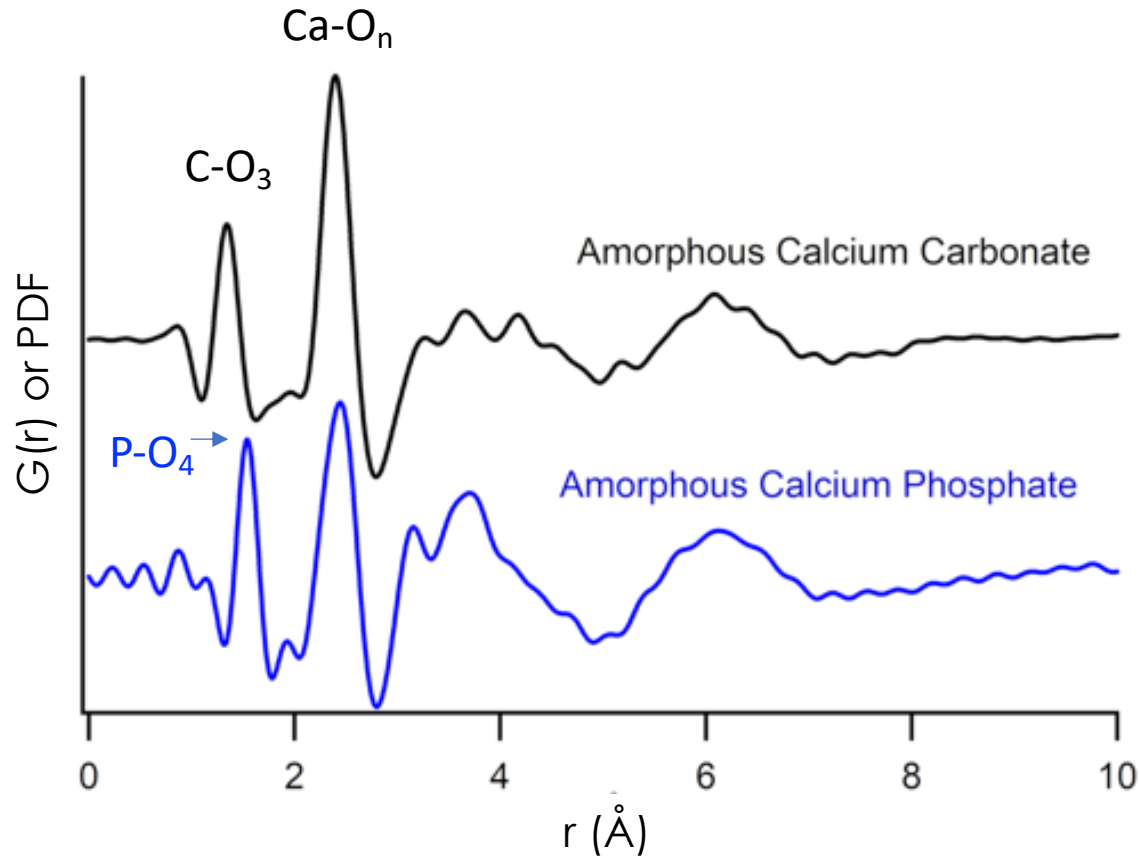
The "problem"…

- Conventional diffraction limited for disordered materials

- Bragg scatter (sharp peaks) replaced by diffuse scatter (weak, broad intensity)

- Alternative structure analysis methods provide information on local atomic structure

  - Total scattering for pair distribution function (PDF) analysis
  - Real-space fitting methods

Virginia Tech Crystallography Lab (VTX)
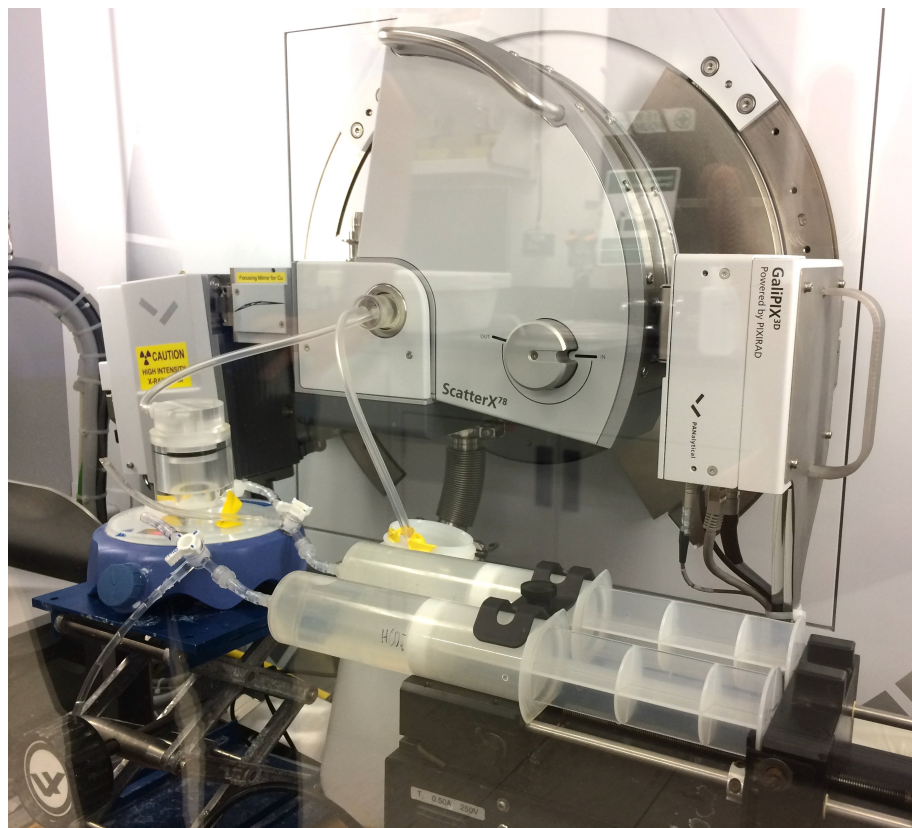
*In situ* small-angle x-ray scattering (SAXS)
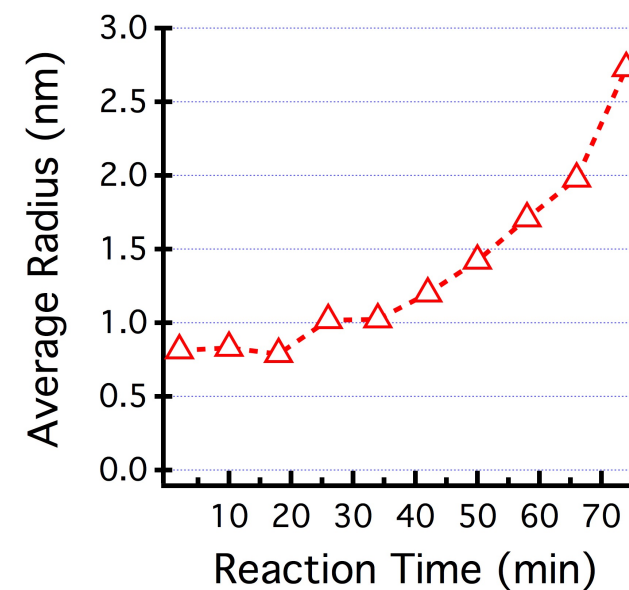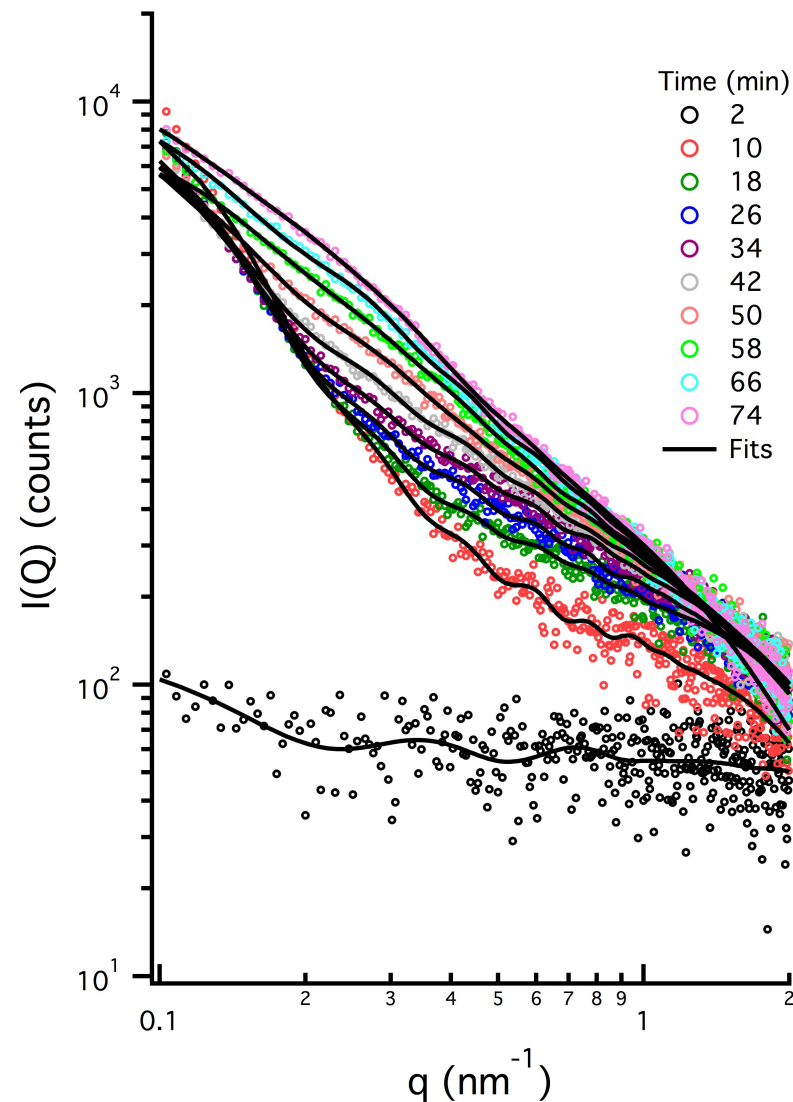
Virginia Tech Crystallography Lab (VTX)
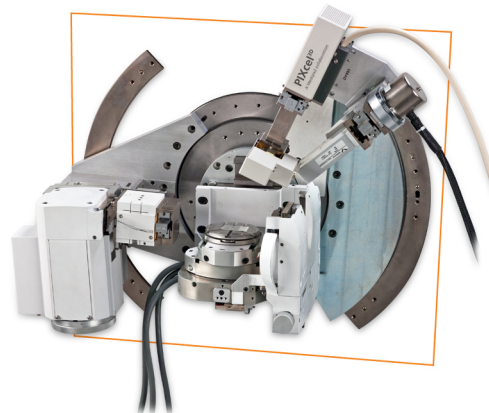
Equipment & Services:

- Panalytical Empyrean Nano Edition

- Equipped for wide variety of experiments, including some *in situ*

- **NanoEarth users can request analyses**
  - Samples mailed in or dropped off
  - Experiments performed by trained personnel
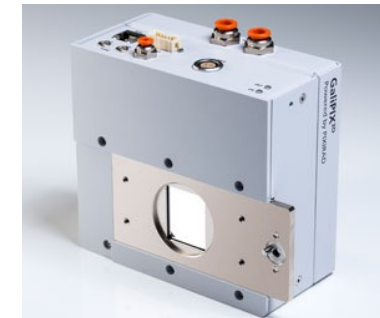 - Offer assistance with data analysis
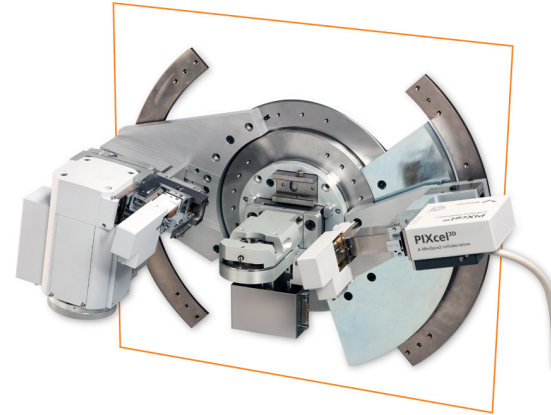
Powder Diffraction
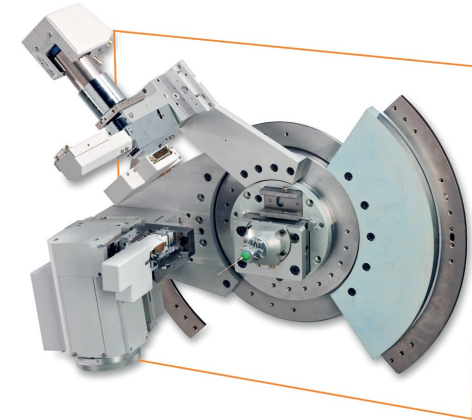
Pair Distribution Function Analysis

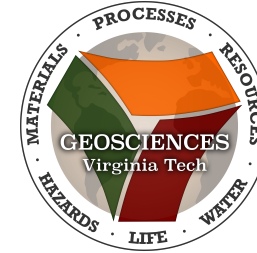Reflectivity and Grazing–Incidence Diffraction

Area Detector (CdTe)

Small–Angle & Wide–Angle Scattering

Thank you!

NSF Geobiology
& L-T Geochemistry
CAREER 1652237

NanoEarth https://nanoearth.ictas.vt.edu/

VTX Lab https://sites.google.com/vt.edu/vtnano/resources/facilities

Michel Environmental Nanoscience Group
https://sites.google.com/vt.edu/vtnano/home

marc.michel@vt.edu