X-ray Diffraction (XRD)

Purpose
Routine XRD-mineralogy profiles can provide qualitative and semiquantitative records of shifts in the source of sedimentary components to a lake sequence. XRD mainly displays information on autocthonous and authigenic minerals, but can give some indication of the abundance of amorphous silica phases. Set up with routine data collection, XRD is a rapid, accurate technique which can process 40 samples per day using an automated sample changer.

Principle
Each mineral is defined by a crystal lattice with characteristic diffraction properties resolved by x-rays. The Ångstrom d-spacing of certain crystallographic lattice directions show up as relative peak (area) heights on the diffractogram (usually in mm) in a fixed relationship to the 2θ (two-theta) angle of the scintillator counter as defined by Bragg's law of diffraction.

Using calibrated peak-area intensities of the major peak, the proportion of mineral species in a profile can be given with about ± 5%, at least for minerals which constitute more than 5% of the bulk sample. Generally, corundum is used as the reference mineral for intensity and for d-spacing. XRD however does not replace the important checks with smear slides which can better determine mineral shapes, sources, and origins.

Procedure Summary

Samples are ground to a fine (< 63 μm) powder in an agate mortar and pestle in acetone or alcohol. If quantitative data is desired, a known volume of spike (generally corundum) is added to each sample.

The slurries are pipetted from the mortar onto an instrument-specific plastic or metal slide. It is important to completely cover the plastic slide's stage, as plastic will contribute a broad peak at 23 two-theta which can ruin some analyses.

Specify:
scan angle (typically ca. 5º to 65º for a bulk sediment),
speed (typically 1 second dwell time),
and rate (typically .005 steps/degree).

Data is acquired both as a graph plot and in digital form as IBM ASCII text files which will need further reduction.

Unknown peaks may be searched by a software program (Jade), or using a common sedimentary minerals manual (copy in core lab). Searches can be limited using Jade's chemistry filter.

Quantitative analysis requires an involved procedure using Jade software. Briefly, after removing the baseline and filtering the data, peak heights are adjusted to match those of the sample's scan. Jade will then calculate the weight percent of each mineral as a part of the "simulate pattern" command, taking into account varying refractive index ratios of each mineral. This value is not known for all minerals, hence, acquiring quantitative data for some mineral assemblages will require initial calibration studies.

Identifying clay minerals requires special preparation procedures. Consult Maurice Tucker's textbook in Sedimentology for a clear description.

Equipment

LRC uses two different facilities, depending on throughput and financial considerations. We use either a new Rigaku MiniFlex XRD unit located in Pillsbury Hall which has a six-sample changer, and an older Siemens XRD unit with a 40 slide capacity automatic sample changer located in the Characterization Facility (http://www.charfac.umn.edu). Contact Rick Knurr (knurr001@umn.edu) or Marc Hirschmann (hirsc022@tc.umn.edu) for training and instrument reservations for Pillsbury Hall, or LRC Core Facility Staff for use of the Siemens unit.

Powder sample preparation materials are located in the Core Lab. Bring a jump drive to the XRD facility collect data.