LAB TFY 11 Fall 2010: SAXS/WAXS (NTNU)

The SAXS instrument has been introduced in the lecture-notes. During the experiments, clay powder samples with different water contents will be placed in a sample holder which will be shown in the lab.

In the laboratory the following should be done:

Before starting on the list, prepare the samples needed in 3) and 6).

1) Calibrate the sample to detector distance by measuring the Bragg peaks of a known calibration sample given to you in the lab. The Bragg peaks of the calibration sample are found by Googling. Remember to set the calibrated distance and beamcenter in the Bruker software on the X-ray machine. Use as thin calibration sample as possible. Since the X-ray machine has been down recently, we do not know how thin the sample can be and still give a signal on the detector within reasonable counting time.

2) Install a sample of Na-fluorohectorite synthetic clay powder at Room Temperature and Relative Humidity of the lab. Place the sample using Scotch tape as shown in the lab. Use about 1 mm sample thickness (pathlength for the X-rays). Place the sample in the beam using the nanography option in the software. Measure the WAXS scattering including the 001 layer scattering peak at room temperature and room relative humidity. Record a diffractogram taking 15 minutes. Clays have been introduced in the lab lecture. Measure and calculate the stacking d-spacing for this sample under these conditions.

3) Repeat 2) but this time under high humidity conditions (i.e add water to the powder) before closing the Scotch tape. Measure and calculate the stacking d-spacing for this sample under these conditions.

4) Place the sample in a 1 mm capillary and repeat 1). Record peak position and peakwidth (PseudoVoigt analysis with unknown instrumental resolution function and sample contribution).

5) Place the sample in a 2mm capillary and repat 1). Record peak position and peakwidth (PseudoVoigt analysis with unknown instrumental resolution function and sample contribution).

6) If time allows: Repeat 2) but this time under moderate humidity conditions slightly above the room humidity conditions (i.e add abit water to the powder) before closing the Scotch tape. Measure and calculate the stacking d-spacing for this sample under these conditions

After the lab:

Use the Bruker software on the X-ray machine for integrating the diffractograms. This software can also be installed on your own computer. Use fex. Origin for the data analysis. Integrate the diffractograms and export/import the integrated curves to Origin (or Matlab). Subtract backgrounds from all the integrated curves. Backgrounds are the parts of the curves far from the Bragg scattering.

Fit Pseudo-Voigt functions to the pure intercalation Bragg peaks for clay samples as well as for the calibration sample. Why do we want as thin calaibration sample as possible? Is there a difference in the peak widths for the 2 mm capillary sample compared to the 1 mm capillary and the calibration sample? If so, why? Discuss this in terms of instrumental resolution functions.

For the data recorded in 6), are there any observable random intercalation Hendricks-Teller peaks? If so, subtract the pure state Bragg peaks from the data, and plot the remaining random intercalation Hendricks-Teller peak. Briefly discuss the Hendricks-Teller theory.

Include all these analysis in the report, and discuss shortly the fitting analysis "theory" as described in the associated lab lecture.
In the report, also discuss properties of the incoming X-ray beam, i.e. the workings of this particular X-ray source compared to other sources. How is the "monochromation" and collimation done on the particular X-ray instrument that we use in this lab?