



Research Techniques and Procedure

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Preparation

- ▶ Using the glove box
 - ▶ Why it is important
- ▶ Filling capillaries
 - ▶ Liquid Consistency
 - ▶ Techniques
- ▶ Keeping instruments clean



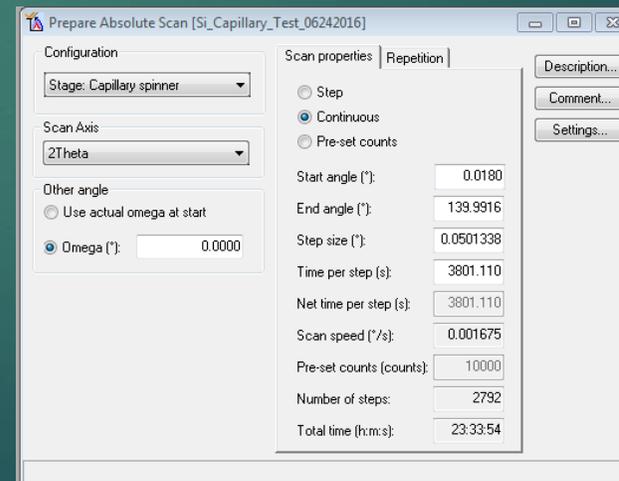
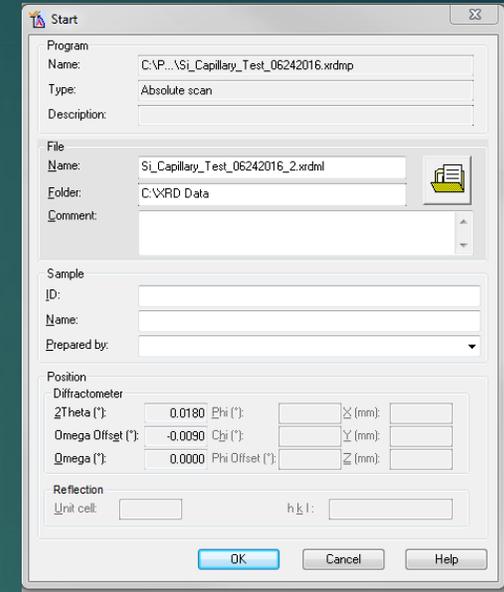
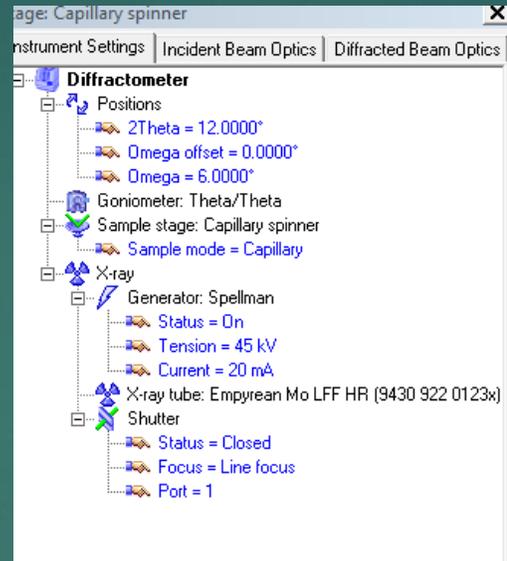
Experiment Set Up

- ▶ X-Ray Diffractometer
- ▶ Alignment of capillary
 - ▶ Spinner Stage
 - ▶ Precision
- ▶ Program set up
- ▶ Machine Inspection
 - ▶ Make sure doors lock
 - ▶ Periodically check machine

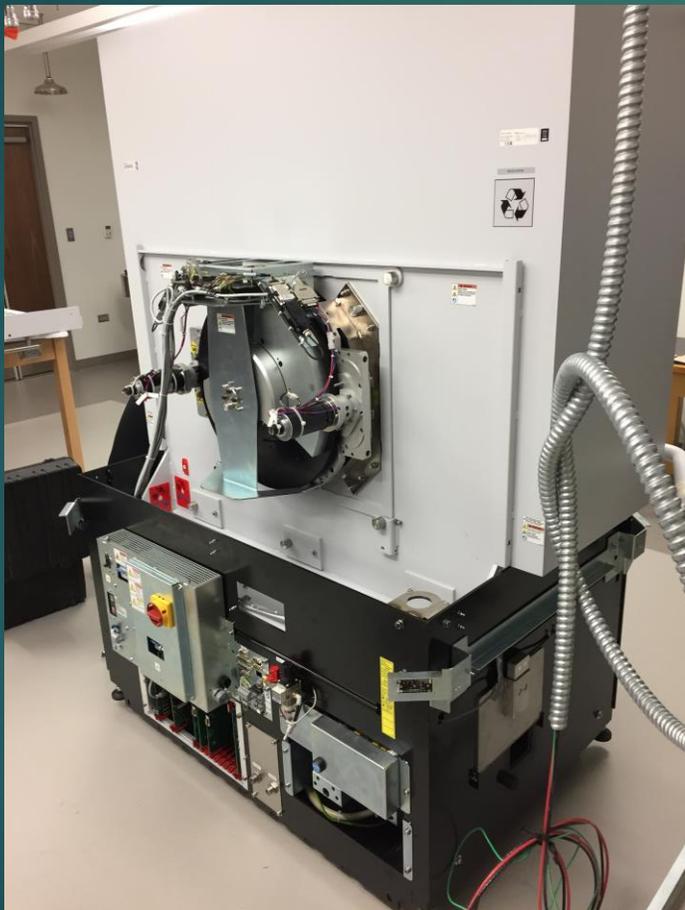


Program Set Up

- ▶ Setting up scan
- ▶ Things to note



Machine Internals

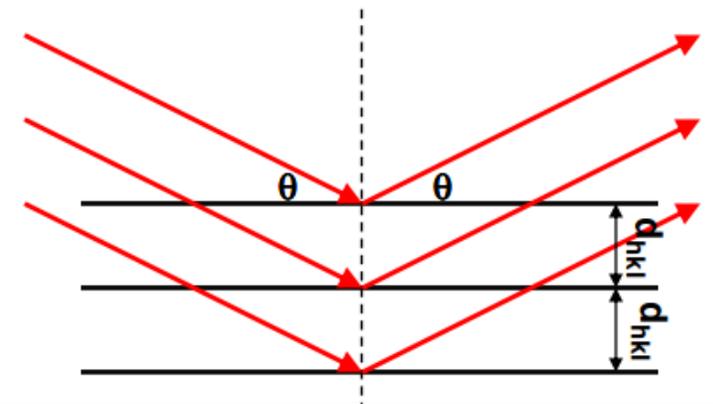


Experiment

- ▶ X-Ray Diffraction
 - ▶ Waves travel different distances
 - ▶ Distance traveled as an integer of wavelength
 - ▶ In phase X-Rays give a signal
 - ▶ We have a known wavelength
 - ▶ Signals are turned into peaks

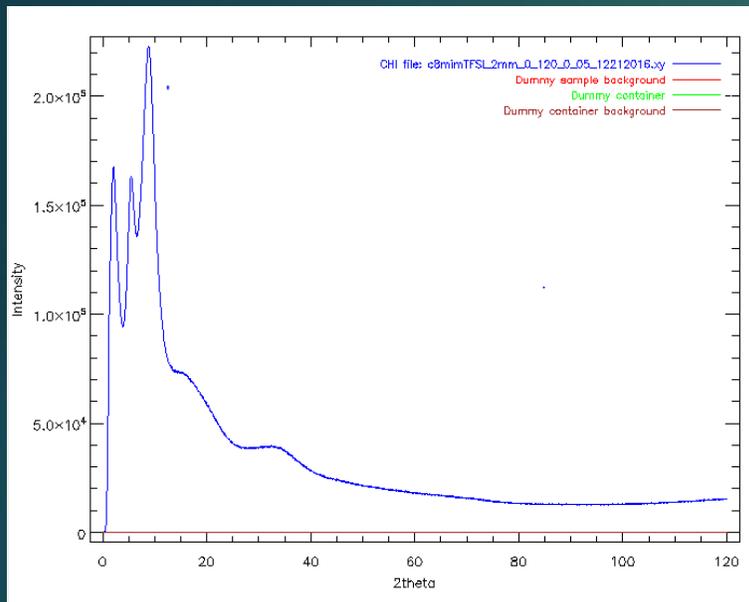
$$\lambda = 2d_{hkl} \sin \theta$$

$$d_{hkl} = \frac{\lambda}{2 \sin \theta}$$

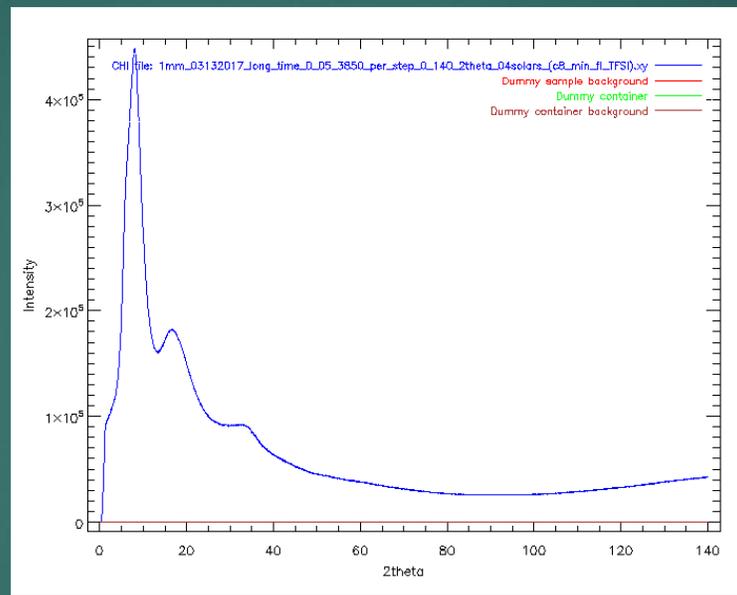


<http://xray.tamu.edu/pdf/notes/intro2xrd.pdf>

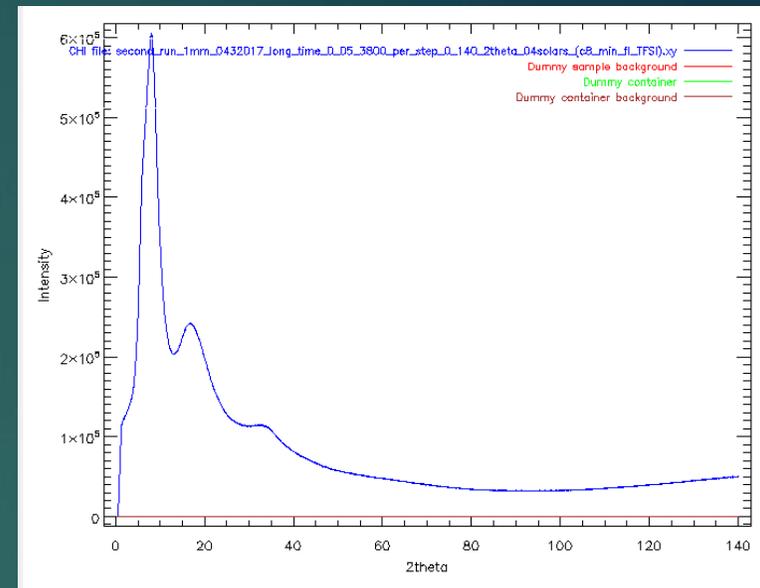
Results



Graph 1. c8mim_TFSI from 0-120 2theta



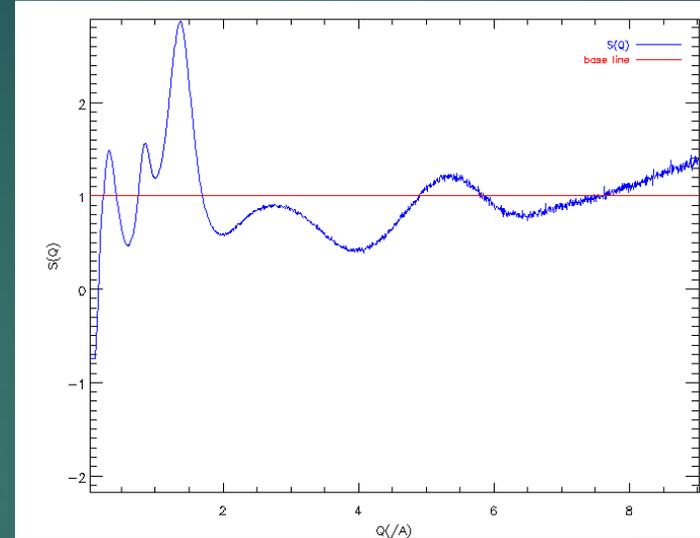
Graph 2. c8mim_TFSI_fl from 0-140 two theta



Graph 2. c8mim_TFSI_fl from 0-140 two theta recalibrated

Results Cont.

- ▶ Very interesting Results
- ▶ Why the c8mim_TFSI_fl graphs differ
- ▶ The difference between liquids
- ▶ Using pdfgetx2 and labview
- ▶ Further analysis



The screenshot shows the PDFgetX2 v1.0 software interface. The title bar reads 'PDFgetX2 v1.0 build 20050312'. The main window contains several sections:

- File Switch --> Help**: A menu bar.
- History -->**: A dropdown menu.
- Title:** X-ray PDF
- Producer:** Gyti Binstribas
- Working Directory:** C:\Users\Gyti Binstribas\Documents\Research\
- Data File Format:** CHI
- Sample:** c8mimTFSI_2mm
- Container:** 2mm_02082017_Jc
- Detector #:** 1 and 2, both with 'Unknown' settings for X Column, Det. Column, and Det. Err. Col.
- Method:** Generic (selected), with options for I(Q) Simu. and SetUp: Elastic.
- Sample Self-Absorption:** Checked.
- Multiple Scattering (2nd only):** Checked.
- Fluorescence:** Type: Constant, Scale: 2000.000.
- X-ray Polarization:** Checked.
- Compton Scattering:** Checked, profile: empirical form.
- Rulandwin Func. Width:** 0.017000.
- Bret-Dirac Factor:** Expo: 3.
- Energy Dependent:** 1/E Linear.
- Laue Diffuse Scattering:** Checked, Weighting Function Type: caef>2.
- Auto Scale:** manual, High Q Range: 51, % calc: 0.00028430947.

At the bottom, there are buttons for 'Reset Data', 'Get I(Q)', 'Calc Corrections', 'Get S(Q)', 'Get G(I)', 'Plot Data', and 'Advanced ->'. A user message at the very bottom states: 'User Message: [Wed Apr 05 22:48:47 2017] successfully read history file: C:\Users\Gyti Binstribas\Documents\Research\c8mim history file 5'.

Helpful Links

- ▶ Basic X-Ray Powder Diffraction (XRPD)
- ▶ <http://xray.tamu.edu/pdf/notes/intro2xrd.pdf>
- ▶ X-Ray Powder Diffraction
- ▶ http://serc.carleton.edu/research_education/geochemsheets/techniques/XRD.html
- ▶ Pdfgetx2 manual