2. Instrumental Parameters - dQ, alpha & lor

Overview

In this tutorial you will learn how to extract instrumental parameters from reciprocal- and real-space data. As with all refinements, the correct modelling of instrumental parameters is essential if you are going to extract fully quantitative and accurate values. For PDF data we need to not only know the affect of instrumental parameters from the reciprocal space data, but also the parameters used to generate your PDF data.

Required files

- XPDF_Si_Bragg.xy
- Si_NIST_640c.cif
- XPDF_Capillary_Bragg.xy
- XPDF_Si_Qmax20_dofr.xy
- XPDF_Si_Qmax25_dofr.xy
- XPDF_Si_Qmax38_dofr.xy
- XPDF_Si_Qmax38_withSoperLorch_dofr.xy

Tutorial instructions

First we will perform a Rietveld refinement of a Si standard to determine some instrumental parameters.

1. Start a new refinement with the file "XPDF_Si_Bragg.xy" (*TOPASforBragg > 1. Select Diffraction File > 1. Select Diffraction File*) and save the input file as "Si_Bragg.inp".
2. Terminate the refinement at 25° 2 (*TOPASforBragg > 1. Select Diffraction File > 1b. file preparation > end x-value*).
3. Add a Chebyshev background (*TOPASforBragg > 2. Background > i. Chebyshev Background*).
4. Add a background from file, and enter the filename "XPDF_Capillary_Bragg.xy" (*TOPASforBragg > 2. Background > vi. background from file*).
5. Add the X-ray source as 'synchrotron energy (area detector)' with an energy of 76.69 keV and sample-to-detector distance of 200 mm (*TOPASforBragg > 3. Radiation Source > 3a. X-ray Sources > Synchrotron Energy (Area detector)*).
6. Add a Rietveld refinement phase using Si_NIST_640c.cif (*TOPASforBragg > 5. Rietveld Refinement > 5c. new phase from .CIF file > ii. Read a .CIF File*) and allow the thermal parameter to refine (`beq @ 1`).
7. Enter a phase name of "Si" (*TOPASforBragg > 5. Rietveld Refinement > 5c. new phase from .CIF file > ii. Phase Name*).
8. Add a 'Bragg dQ + alpha.Q' peak shape (*TOPASforBragg > 5. Rietveld Refinement > 5c. new phase from .CIF file > iii. Peak Type > Bragg dQ + alpha.Q peak shape*).
9. This is a standard, so there is no need to refine the lattice parameters.
10. Turn on `do_errors`, run the refinement and look at the results. The refined values for `dQ`, `alpha` and `lor` are instrumental parameters for our PDF data. The refined `bkg_file` scale factor gives us an indication of the scaling of the background to our data (this may be useful when processing data to PDFs).

Next we will try to extract the same numbers from our PDF data.

1. Start a new refinement with the file "XPDF_Si_Qmax38_dofr.xy" (*TOPASforPDF > 1. PDF Data > Select PDF Data File*) and save the input file as "Si_PDF.inp".
2. Start the refinement froman *r* of 1 Å (*TOPASforPDF > 1. PDF Data > file preparation > start r value*).
3. Add a 'dQ damping with Lorentzian contribution' with approximate starting values of 0.05 and 0.5 (*TOPASforPDF > 2. Instrumental parameters > dQ damping with Lorentzian contribution*).
4. Add a 'Q-dependent broadening convolution' with an approximate starting value of 0.001 (*TOPASforPDF > 2. Instrumental parameters > Q-dependent broadening convolution*).
5. Add a new phase from 'Si_NIST_640c.cif' (*TOPASforPDF > 3. Phase information > 3b. add new phase from CIF*).
6. Replace 'beq 1' with a 'beq spherical with min r cutoff' peak shape, with an rcut value of 2.7 (*TOPASforPDF > 3. Phase information > beq peak shape functions > beq spherical with min r cutoff*).
7. Turn on `do_errors`, run the refinement and look at the results.
    a. How do the refined results compare with those from the Bragg data?
    b. How do the errors compare?
    c. Is it best to refine these values from PDF or Bragg data?

Now we will perform a joint refinement on both datasets at the same time. This will use a range of shortcuts to make this type of refinement easier.

1. Save your previous input file "Si_PDF.inp" as "Si_Joint.inp".
2. Declare the parameters `dQ`, `lor` and `alpha` as global parameters towards the top of the file (before where the `xdd` filename is declared) and fix them at the values determined in your Bragg refinement.

```
prm !dQ    0.05275
prm !alpha 0.00449
prm !lor   0.37618
```

3. Change the `dQ_lor_damping` and `convolute_alpha` instrumental functions so they use these declared parameters.

```
        dQ_lor_damping( ,dQ, ,lor)
        convolute_alpha( ,alpha)
```

4. At the bottom of the file, add the following block of code (note thew code is likely wider than is displayed on this page!). The `for xdds` and `for strs` can be used to declare the same information across multiple datasets and structures respectively.

```
for xdds {
        for strs {
                a 5.43119
                b 5.43119
                c 5.43119
                al 90
                be 90
                ga 90
                space_group "Fd-3m"
                site Si1    x 0.000000      y 0.000000      z
0.000000      occ Si   1.0                beq_rcut_spherical(!rcut,
2.7,beqcut,0.1,beqlo,0.1,beqhi,2.0,beqradius,20)
        }
}
```

5. This replaces the need to declare the same information below where you delare `str` for the PDF data, so now all this needs to include is the `str` keyword and nothing else.
6. Copy everything from the xdd line down to the end of the file section from your Bragg refinement into this input file, placing it below the PDF data.
7. The details of the structure from the Bragg data are not needed as they are already declared within `for strs`. Delete everything apart from the lines for `str`, `scale` and the `pkshape_dQ_alpha`. Edit the `pkshape_dQ_alpha` so it is using the global parameters declared at the top of the file (`pkshape_dQ_alpha( ,dQ, ,alpha, ,lor)`).
8. Run the refinement and look at the results.
9. Make it easier to turn on/off the different datasets by declaring the following parameters at the top of the file, and adding e.g. '`#ifdef Bragg`' or '`#ifdef PDF_Qmax38`' before and '`#endif`' after each of the `xdd` blocks (everything from `xdd` to below the line within the `str`) within the .inp.

```
#define Bragg
#define PDF_Qmax38
```

Experiment with turning on/off these data and running the refinements.

10. You have also been supplied with the datasets XPDF_Si_pdfgetX3_Qmax20.xy and XPDF_Si_pdfgetX3_Qmax25.xy, which are the same data but terminated to different Qmax. Use what you have learnt to add these data to the refinement in a way that allows you to easily turn them on/off using #define. For these data, you will also need to define a Qmax Sinc function convolution with a fixed Qmax (e.g. `convolute_Qmax_Sinc( ,20)`), or the resolution will not match. You may also need to refine a scale factor.
11. Run the refinement and see how it converges.
    a. Can you get all of the data to fit at the same time?
12. A final file, XPDF_Si_Qmax38_withSoperLorch_dofr.xy, is also supplied. This was processed with GudrunX and has had a Soper-Lorch function applied with a width (`d_zero`) of 0.1. Make another copy of the PDF block of information for this file. You will need to add a Soper-Lorch function convolution to get this data to fit along with the rest of your data.
    a. Can you get all of the data to fit still?

Final comments

The proper handling of instrumental parameters is essential when refining information accross multiple files and for extracting meaningful data from both Bragg and PDF data. The use of a good standard (e.g. Si) is necessary for this to work. For PDF data, not only the traditional instrumental resolution needs to be considered, but also PDF processing parameters such as Qmax and whether a Soper-Lorch function has been applied.