

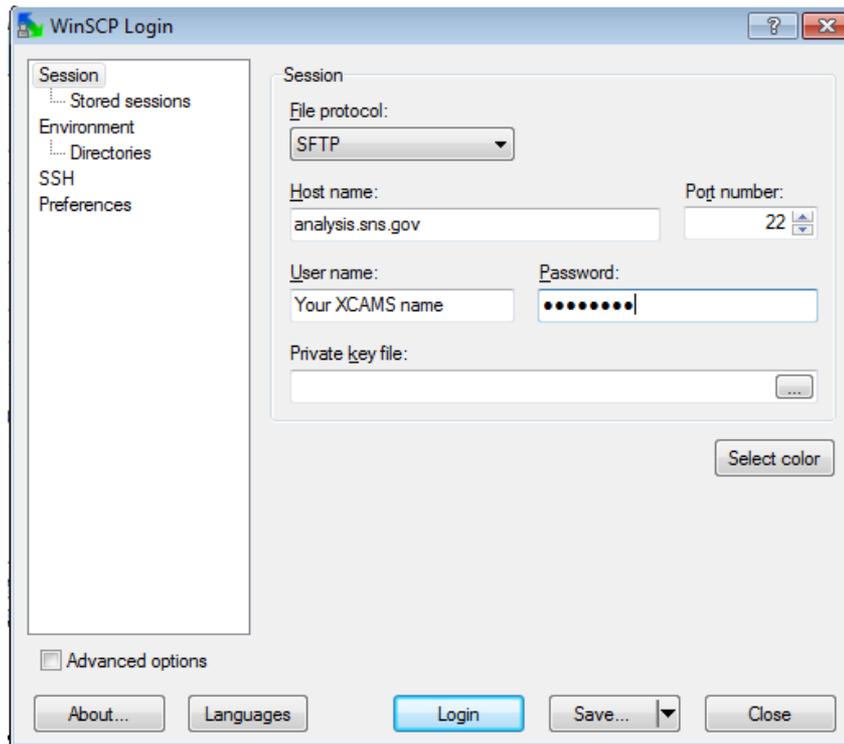
How to reduce NOMAD data

04/28/2014

The data reduction is done automatically and it will be started by your local contact. You can find your data at [/SNS/NOM/IPTS-XXXX/shared/autoNOM](#) where XXXX is the IPTS number of your experiment

Remotely access your data

1. Go to <http://analysis.sns.gov/> and chose connection option appropriate for you OS (NX Client for Windows machines). NX provides the same computing environment as the beamline computer.
2. Go to <http://winscp.net/eng/download.php> and download the “**Installation package**” of WinSCP version for you OS. WinSCP allows you to copy files to your local computer.
3. Install and run the program
4. Use your XCAMS account (the account that you used to create your proposal) credentials to set it up



5. Use WinSCP help file to guide your trough the interface

Relevant equations or what happens during data reduction

$$I_{coh} = \frac{I_{sample} - I_{background}}{I_{vanadium}}$$

where, I_{sample} scattering from the sample, $I_{background}$ is background (container + empty instrument) scattering and $I_{vanadium}$ the corrected (background absorption, mult. scattering) scattering from vanadium

$$S(Q) - 1 = \frac{I_{coh} - I_{poly}}{I_{poly}}$$

and

$$I_{poly} = \begin{cases} \frac{\rho\sigma d^2}{\rho_v\sigma_v d_v^2} p & (hyd = -1) \\ a_0 + a_1q + \dots a_nq^n & (hyd = 0) \\ xL(q_0) + (1-x)G(q_0) & (hyd = 2) \end{cases}$$

absolute normalization (requires knowledge of sample properties and is not done in autoreduction)

least-square polynomial fit within q-region [$q_{min} < Q < q_{max}$]

non-linear least squares fit to pseudo-Voigt function. Used for samples containing hydrogen or deuterium

$S(Q)-1$ data is provided in *SQ.dat files. GSAS files are $I_{coh} (TOF) / \Delta TOF$ and FullProf files $I_{coh} (TOF)$

$$g(r) - 1 = A \int_{Q_{min}}^{Q_{max}} (S(Q) - 1) Q \sin(Qr) dQ$$

'lower case' $g(r)$ usually used by liquid community

$$A = \frac{1}{2\pi^2 r \rho \sum b^2}$$

$$G(r) = r(g(r) - 1)$$

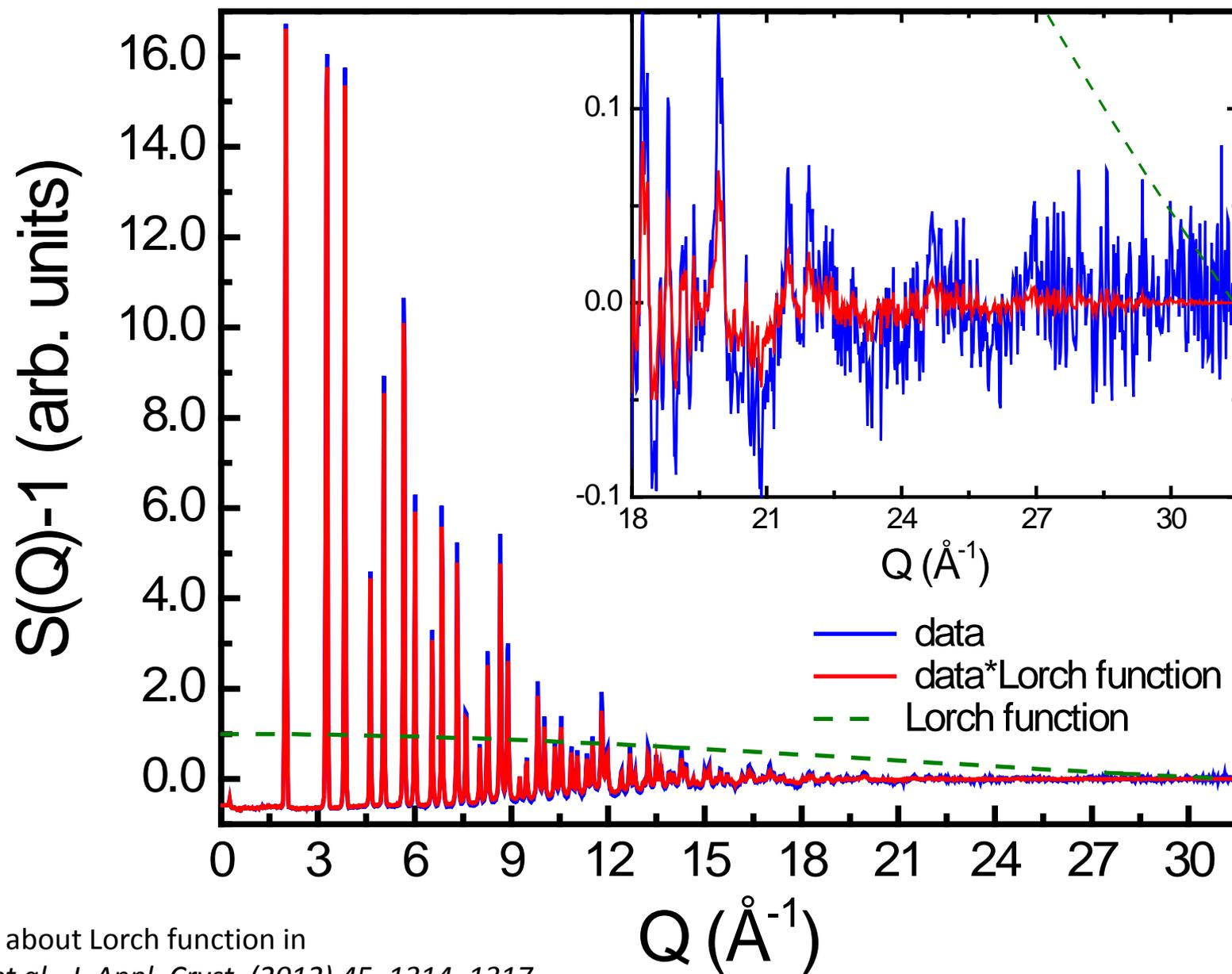
'upper case' $G(r)$ or PDF usually used by crystallographers

In autoreduction, no absolute normalization is done and A is arbitrary.

What the files do I get from autoreduction (XXXX is the run number):

- **los.txt** is the logbook of your experiment (updated automatically)
- **NOMXXSQ.dat** – ASCII file of $S(Q)-1$. You can plot it in *gnuplot* or any other software
- **NOMXXrgrftf.gr** – $G(r)$ or PDF of your samples, ready for analysis for PDFgui software package
- **NOMXXrgrftl.gr** – the same, but convoluted with the Lorch function (see below)
- **NOMXXftl.dat, NOMXXftf.dat** – small $g(r)$
- **NOMXXtof.gsa** – Diffraction pattern in GSAS format
- **NOMXXtof-n.dat** – where n is the bank number, diffraction pattern in FullProof format
- **NOMXXtof.getN** – Diffraction pattern in PDFgetN format

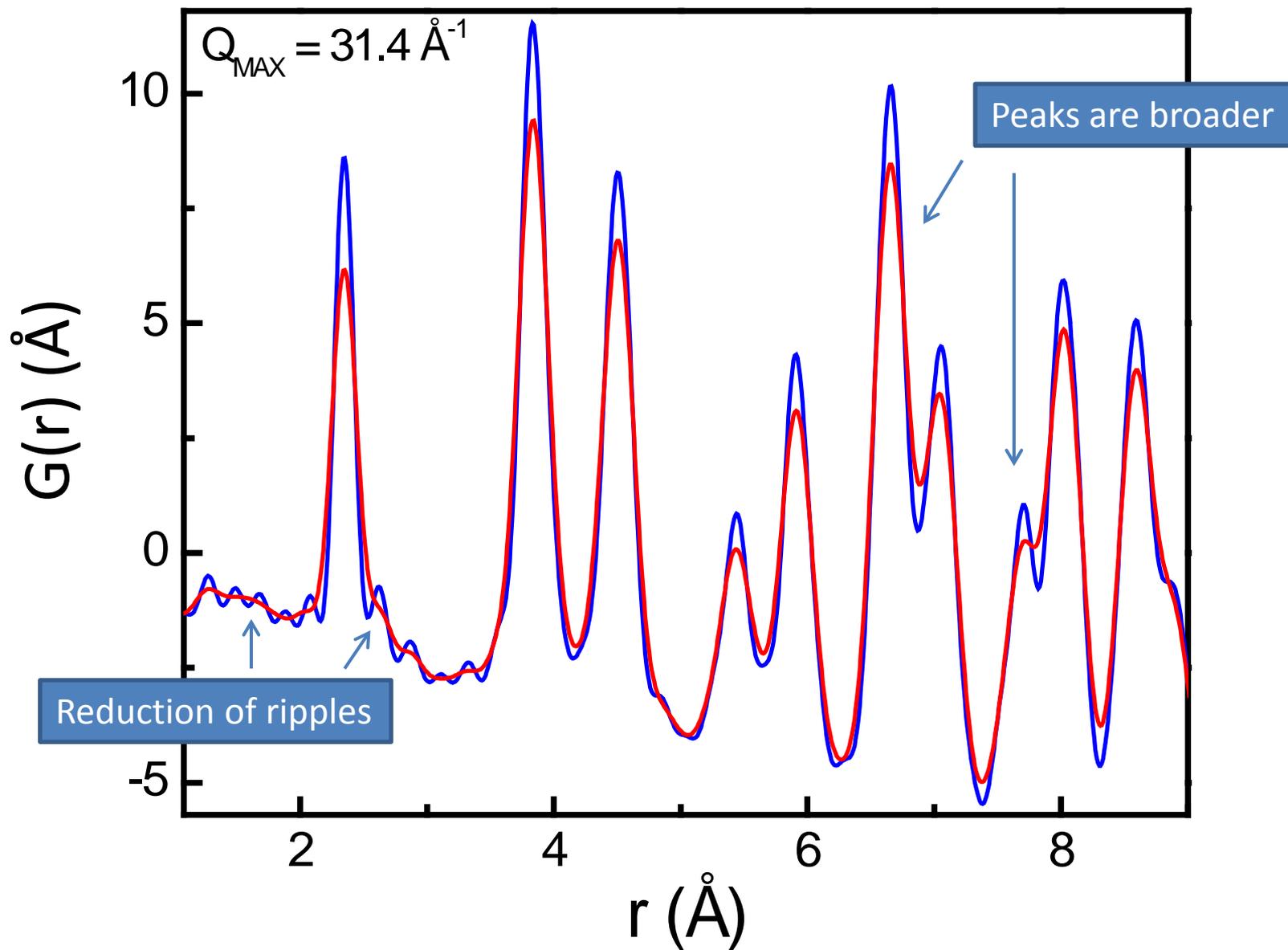
Multiplication with a Lorch function reduced the influence of high Q noise at the expense of real space resolution (multiplication in $Q = \text{convolution in } r$)



See more about Lorch function in
A. Soper et al., J. Appl. Cryst. (2012) 45, 1314–1317

Reduction of noise at high Q leads to smoother PDF data, however it lowers the resolution in real space:

— data (fft)
— data*Lorch function (ftl)

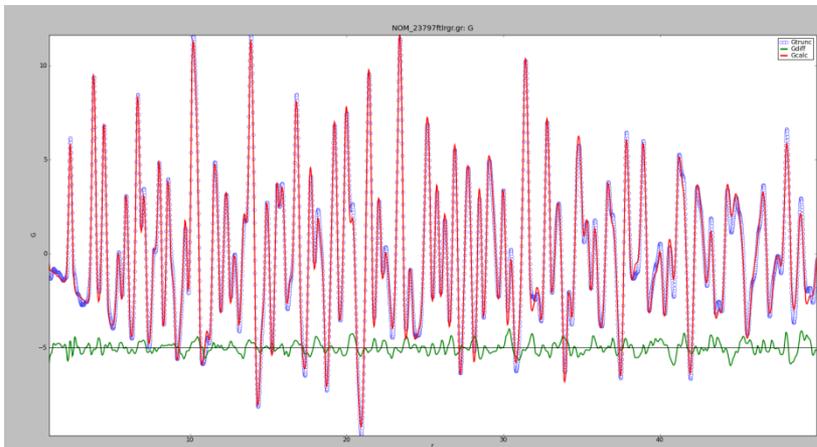


What effect the application of Lorch function has on results of PDF refinements?

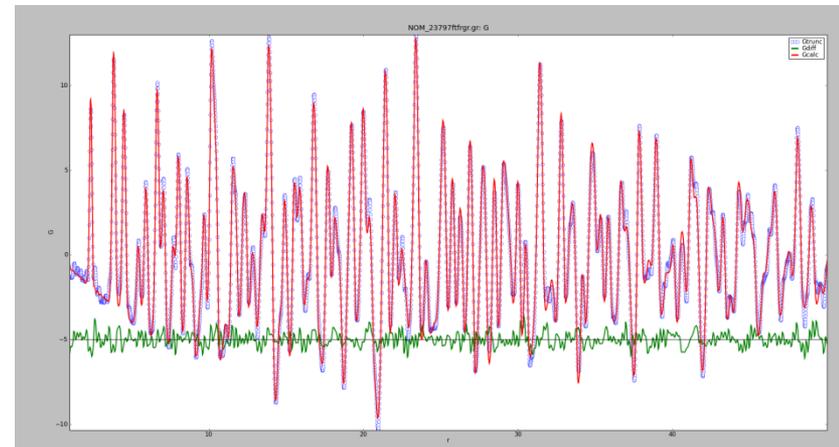
Results of the refinements for ftl and ftf for NIST standard Si powder $a=5.430940 \text{ \AA}^*$
(mass: 100 mg, $T=300 \text{ K}$, counting time: 26 minutes, $Q_{\text{MAX}} = 31.4 \text{ \AA}^{-1}$, $r = 1-50 \text{ \AA}$)

	FTL with Lorch	FTF w/o Lorch
R_w	0.0826	0.0989
$a \text{ (\AA)}$	5.43023 (2)	5.43002 (2)
$U_{ii}(\text{\AA}^2)$	0.0112125 (1)	0.00963896 (1)
delta1	1.17653 (6)	1.63703 (4)

Fit of data with Lorch function (ftl)

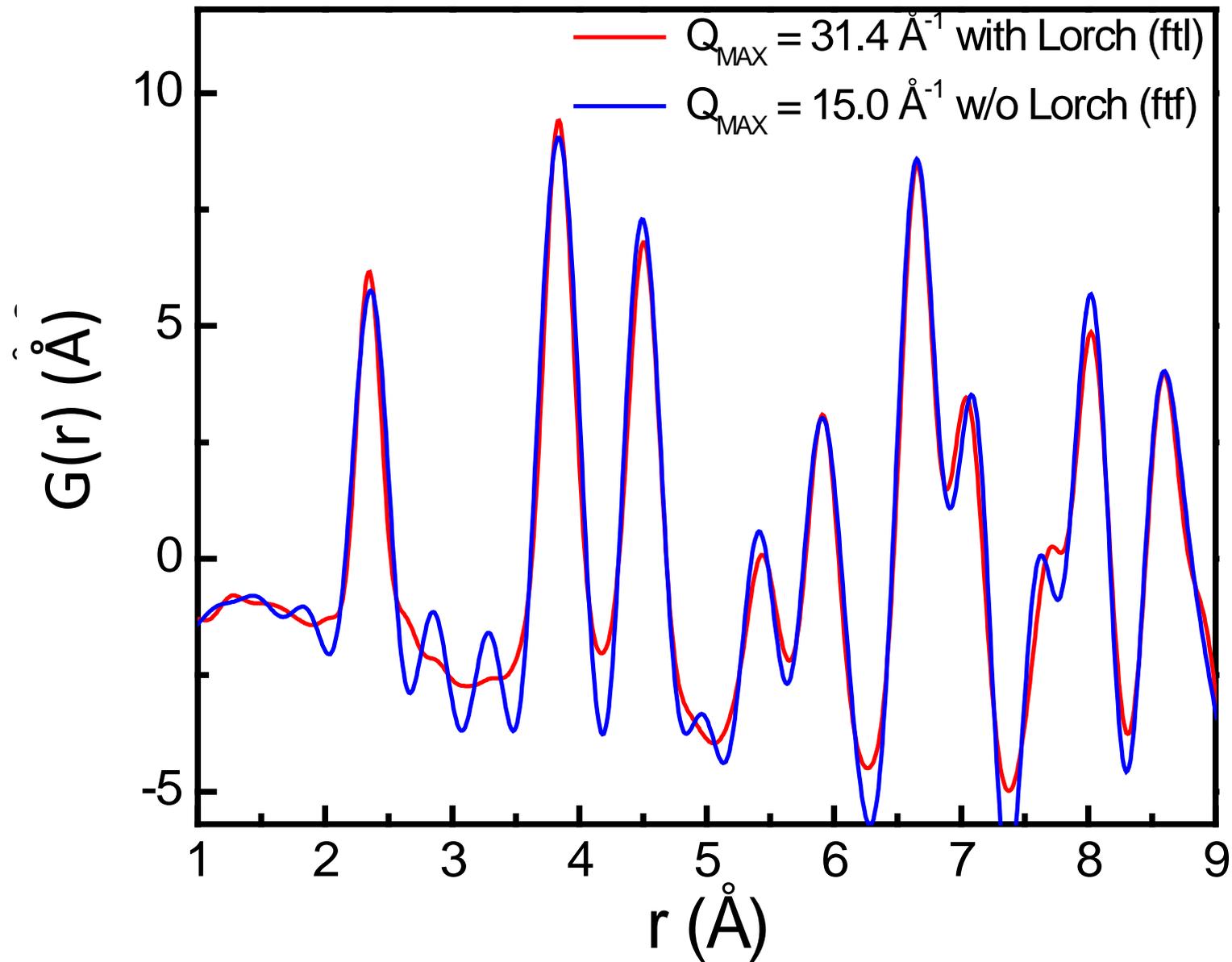


Fit of data w/o Lorch function (ftf)



(*) it is known that lattice constant values derived from Rietveld and PDF refinements deviate (see more details in X. Qiu, E.S. Bozin, P. Juhás, Th. Proffen and S. J. L. Billinge, *Reciprocal space instrumental effects on the real space neutron atomic pair distribution function*, *J. Appl. Crystallogr.* 37, 110-116 (2004))

The broadening in real space introduced by the Lorch function is roughly equivalent to termination at $Q_{\text{MAX}}/2$. Note, however, that ripples are present for $Q_{\text{MAX}}=15 \text{ \AA}^{-1}$ w/o Lorch

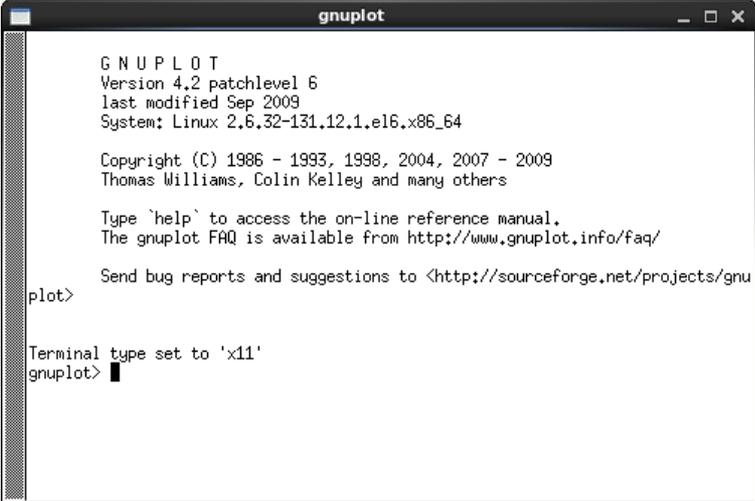


What do I do with the data?

- **.gr** files are ready to be used in PDFgui software (<http://www.diffpy.org/>). Tutorial video (https://www.youtube.com/watch?list=UUCyTfdry5UJ-iiWaS2gTm_A&v=tTQ3oidEHjY). Make sure to join Google-group for more info and useful tips (<https://groups.google.com/forum/#!forum/diffpy-users>)
- **.gsa** files are to be used with GSAS-EXPGUI software (<https://subversion.xor.aps.anl.gov/trac/EXPGUI/wiki/InstallWindows>). The Instrument parameter file can be found at /SNS/NOM/shared/GSASandPDFgetN folder. Check tutorials here ([http://www.aps.anl.gov/Xray Science Division/Powder Diffraction Crystallography/EXPGUItricks.html](http://www.aps.anl.gov/Xray_Science_Division/Powder_Diffraction_Crystallography/EXPGUItricks.html))
- **.dat** files are to be used with FullProof package (<https://www.ill.eu/sites/fullprof/index.html>). Instrument parameter file for each bank can be found at /SNS/NOM/shared/GSASandPDFgetN folder.
- **.getN** files are to be used with PDFgetN software. **Important! Do not use on-line version. Use PDFgeN-SNS version available at /SNS/NOM/shared/GSASandPDFgetN/PDFgetN-SNS_users.zip.** You can also find parameter file, manual and settings for the program in the same folder. Remember that PDFgetN-SNS only works for samples measured in standard vanadium cans.

How to plot your data on analysis or nomad computer

1. In terminal type **xterm -e gnuplot &**. The window will appear:



```
gnuplot
GNUPLOT
Version 4.2 patchlevel 6
last modified Sep 2009
System: Linux 2.6.32-131.12.1.el6.x86_64

Copyright (C) 1986 - 1993, 1998, 2004, 2007 - 2009
Thomas Williams, Colin Kelley and many others

Type 'help' to access the on-line reference manual.
The gnuplot FAQ is available from http://www.gnuplot.info/faq/

Send bug reports and suggestions to <http://sourceforge.net/projects/gnu
plot>

Terminal type set to 'x11'
gnuplot> █
```

2. To plot your data type **plot 'file name'**. Use comma to separate datasets (*w l* is short for *with lines*)

```
gnuplot> plot 'NOM_5175SQ.dat' w l, 'NOM_5197SQ.dat' w l, 'NOM_5191SQ.dat' w l █
```

3. To zoom in, hold left mouse button to draw a rectangle. To auto scale type “a” in the plot window).

4. Refer to the gnuplot help file online to learn how to change font size, save figure and etc.

How to sum your data after experiment is done

1. Open terminal (Right mouse click on Desktop – Open in Terminal) :



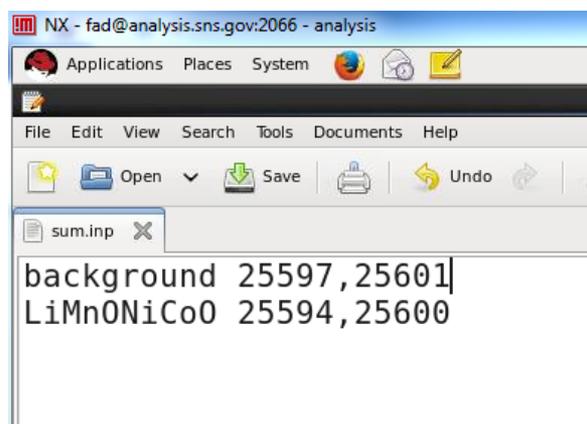
2. Type in terminal your autoNOM folder destination :

```
cd /SNS/NOM/IPTS-XXX/shared/autoNOM
```

where, 'XXXX' is your IPTS number

3. Make sum.inp file : **gedit sum.inp &**

4. Edit the file, providing the run numbers for the background and all samples measured (ranges of scan numbers can also be specified as 25600-25603)



5. Save the file and type :

```
python ~zjn/pytest/sumscans.py -f sum.inp
```

6. You will go through several idl plots (ask your local contact about their meaning), just keep pressing “Return” to continue

7. After it is done, the usual reduced files of PDF are generated with the prefix “NOM_9999_”. For example, file “NOM_9999_sample1_ftfrgr.gr” is the FTF G(r) for all run numbers listed for “sample1” in the file sum.inp. NOM_sample1.gsa is the summed GSAS file.

8. You can have multiple sum.inp files: sum1.inp, sum2.inp and etc., for different sample environment setups. To run them simply change the name:

```
python ~zjn/pytest/sumscans.py -f sum1.inp
```

9. **Important!** If you want to run several sum.inp files in one folder, delete background files generated by the previous sum file: **rm backMTc.dat backMTcg.dat**

10. You can edit parameters of your summed PDF by editing sumsample1_pdf.pro file, and running it with idl :

```
idl sumsample1_pdf.pro
```

How to change parameters of your PDF

1. If you summed the files, open the macro (in this example sample name is C):

```
gedit sumC_pdf.pro &
```

2. In the file, you can change parameters after “creategr” command:

```
creategr,suma,sumb,back='backMTc.dat',norm='norm25876.dat',$  
hydro=0,qminpla=10,qmaxpla=50,qmaxft=!pi*10,sc=9999,inter=1,use=1,maxr=50,$  
comment='neutron, Qmax=31.414, Qdamp=0.017659, Qbroad= 0.0191822'$  
,ignq=11,qual='_C_',error=esuma
```

Where, hydro is the correction coefficient for hydrogen correction, qminpla/qmaxpla are the limits in q for least-square polynomial fit for S(Q), qmaxft is Qmax, maxr is the maximum distance PDF is calculated for and qual is qualifier to avoid overwriting existing files (for example you the same PDFs at various Qmax, i.e. qaul='Qmax12' or 'Qmax25').

Important: if you change qmaxft, don't forget to change Qmax in the comment line. Qmax will read by PDFgui software, not qmaxft

3. Make necessary changes. Save the file and type in the terminal:

```
idl sumC_pdf.pro &
```

4. If you see an error message 'Permission denied' type **chmod 666 *** and try again