UNIVERSITY OF TRENTO - Italy
Department of Industrial Engineering

Rietveld-PDF analysis

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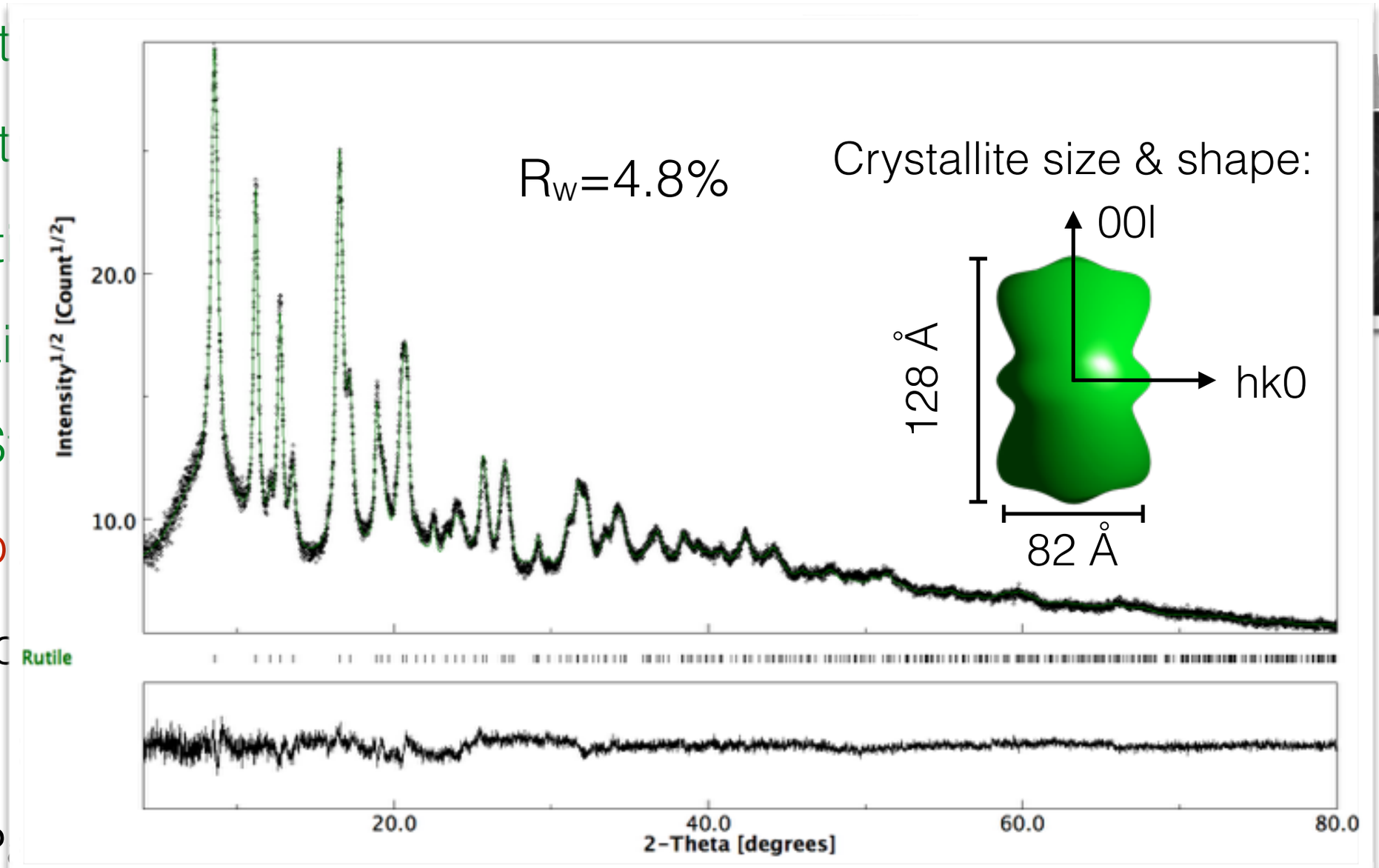
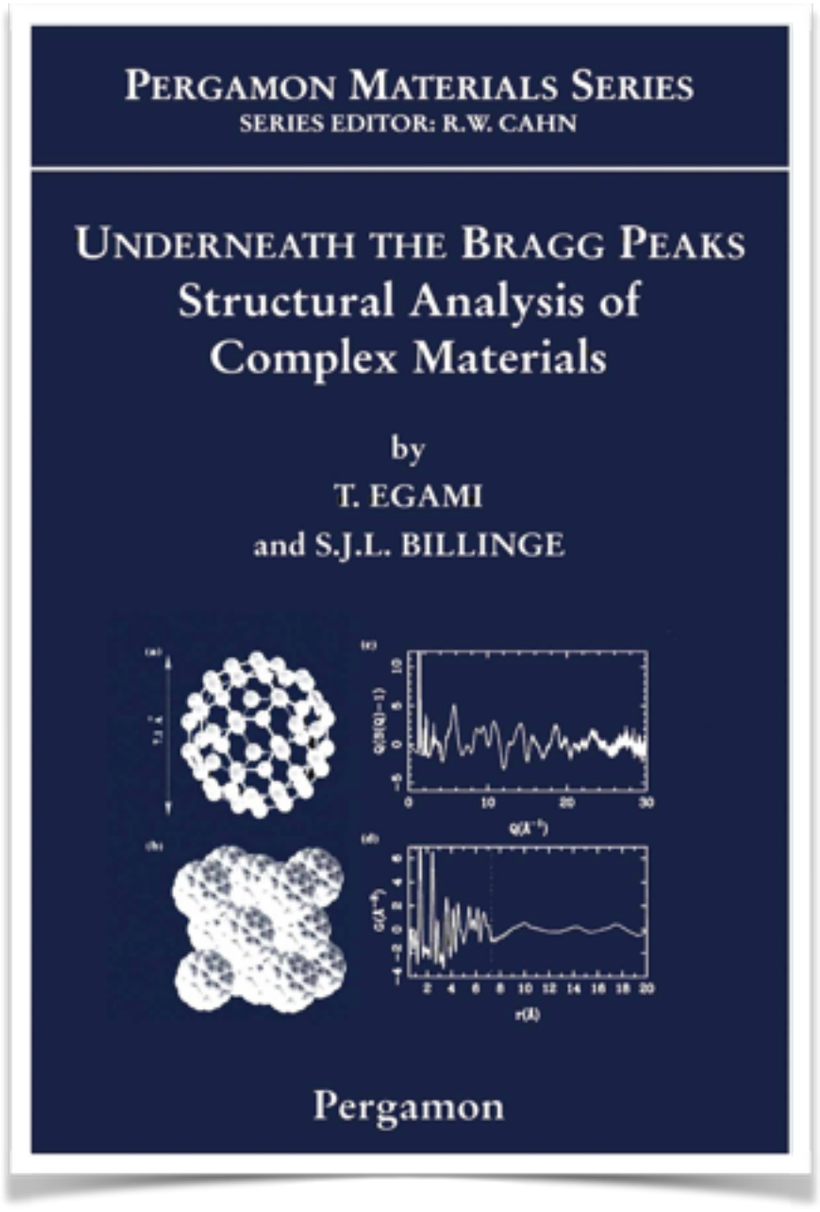
Analysis of nano-materials

- Crystal st
- Crystal st
- Quantitat
- Size-Strai
- Texture/S
- Amorpho
- Pseudo
- Debye
- PDF (P



- requires single phase
- normalization and background removal
- difficult to get domains info
- how to get structural informations out of it

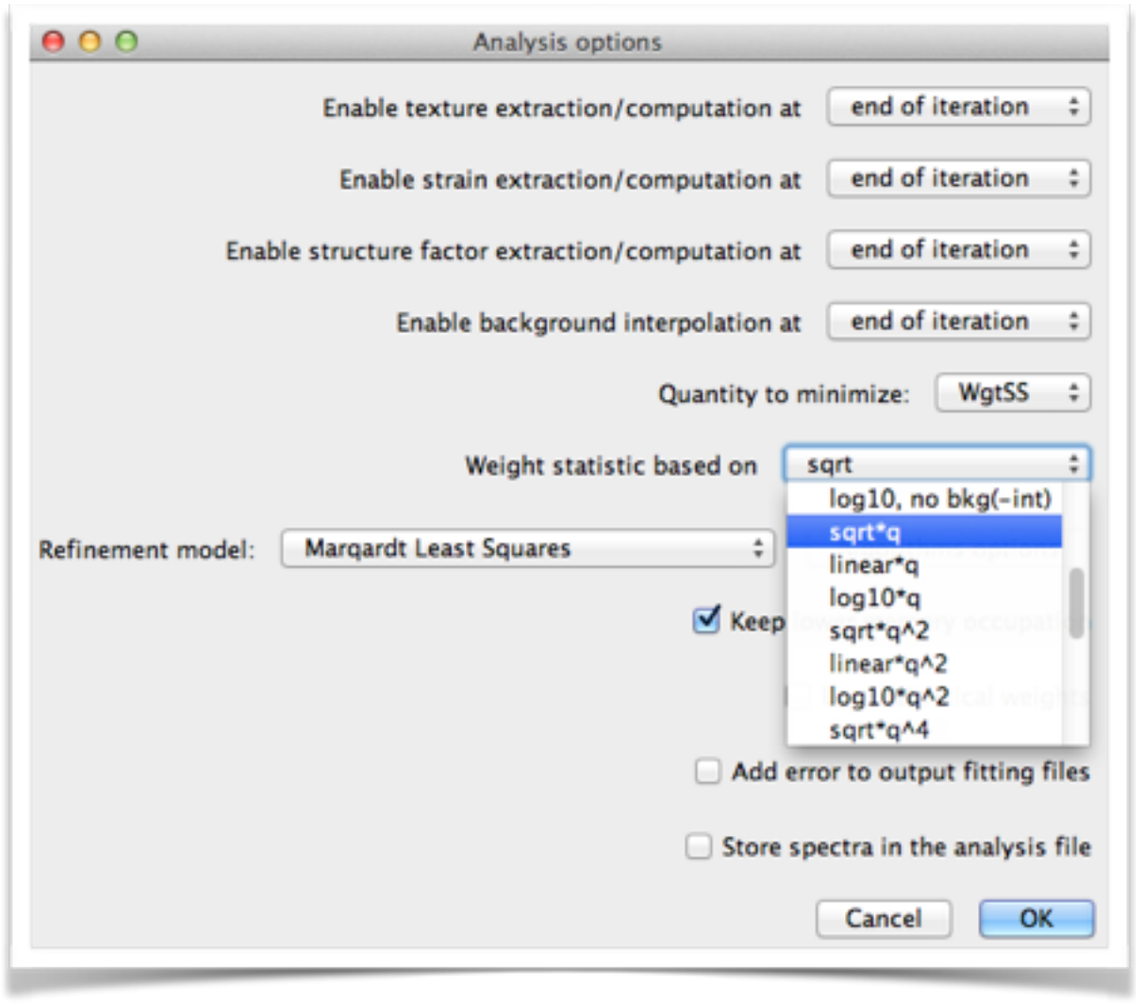
PDF in the real or reciprocal space?



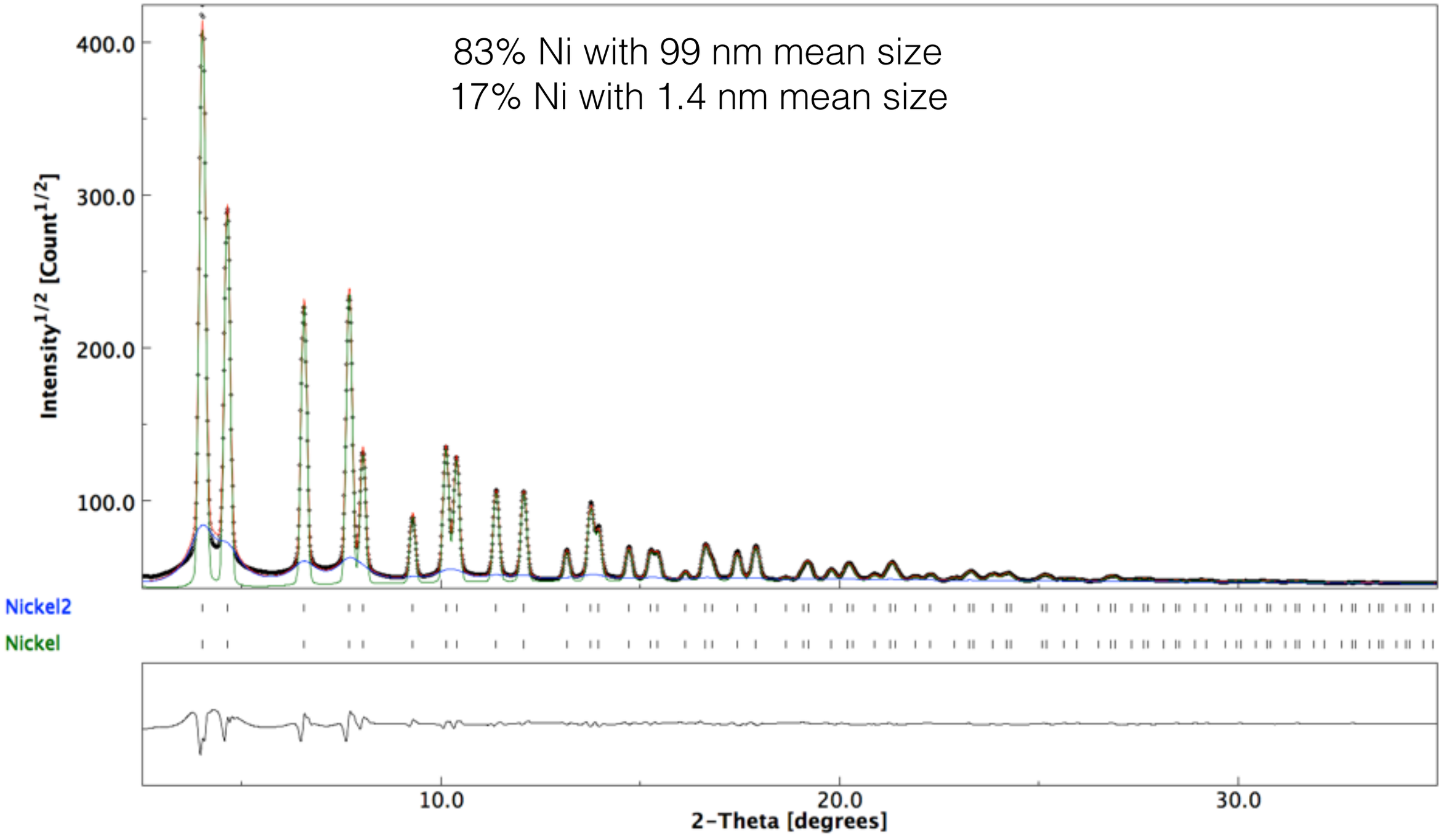
This approach has been applied, in exact analogy, to the PDF (Proffen and Billinge, 1999; Billinge, 1998) in the program PDFFIT. We highlight here the similarities and differences with conventional Rietveld. The main similarity is that the model is defined in a small unit cell with atom positions specified in terms of fractional coordinates. The refined structural parameters are exactly the same as those obtained from Rietveld. The main

PDF with the Rietveld (reciprocal space)?

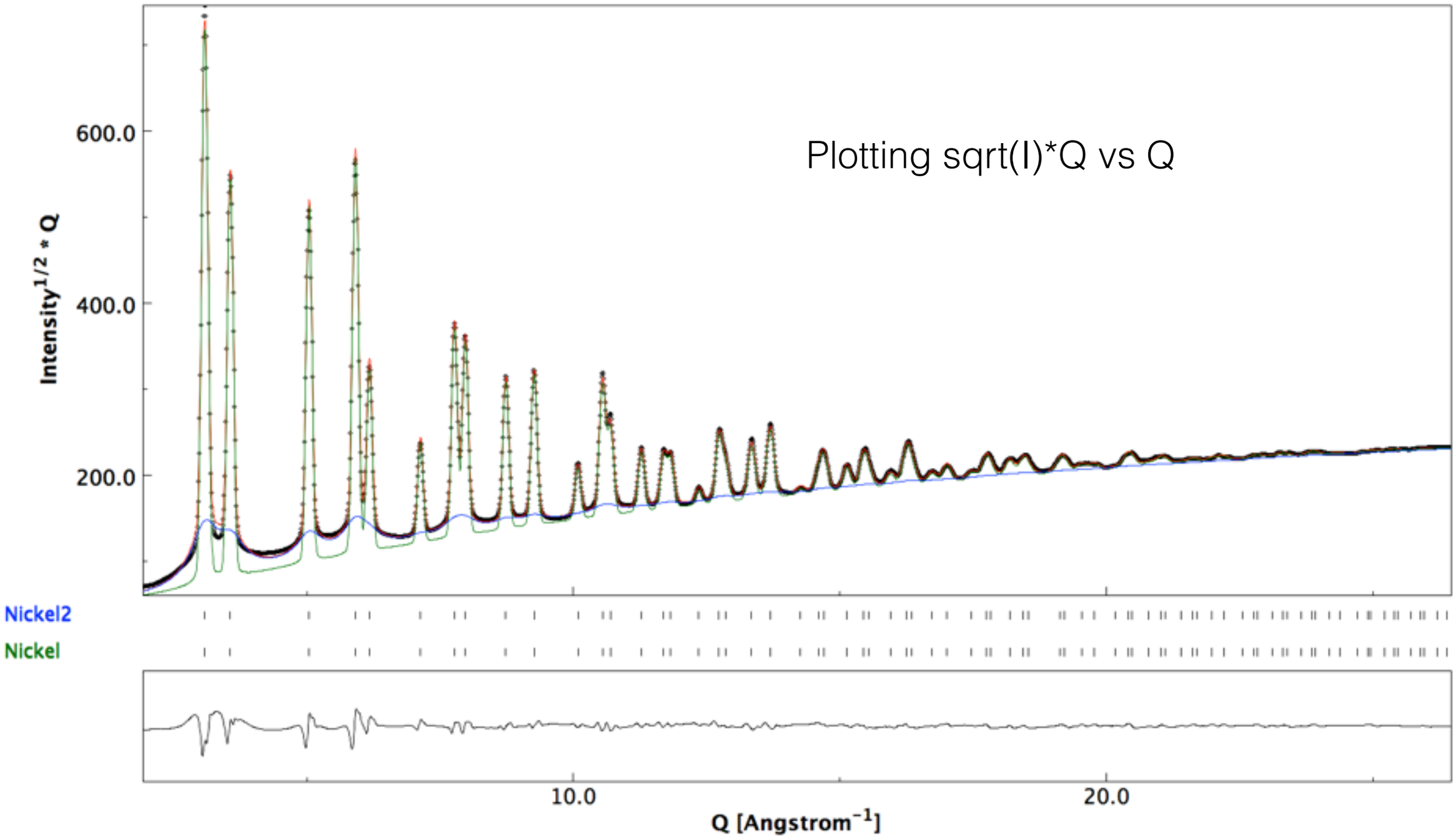
- Use large range in Q (10 $Å^{-1}$ or larger)
- Use proper statistic modifiers during refinement
- More sensitivity to light atoms and small distorsions (like PDF)
- Add diffuse computation (?)



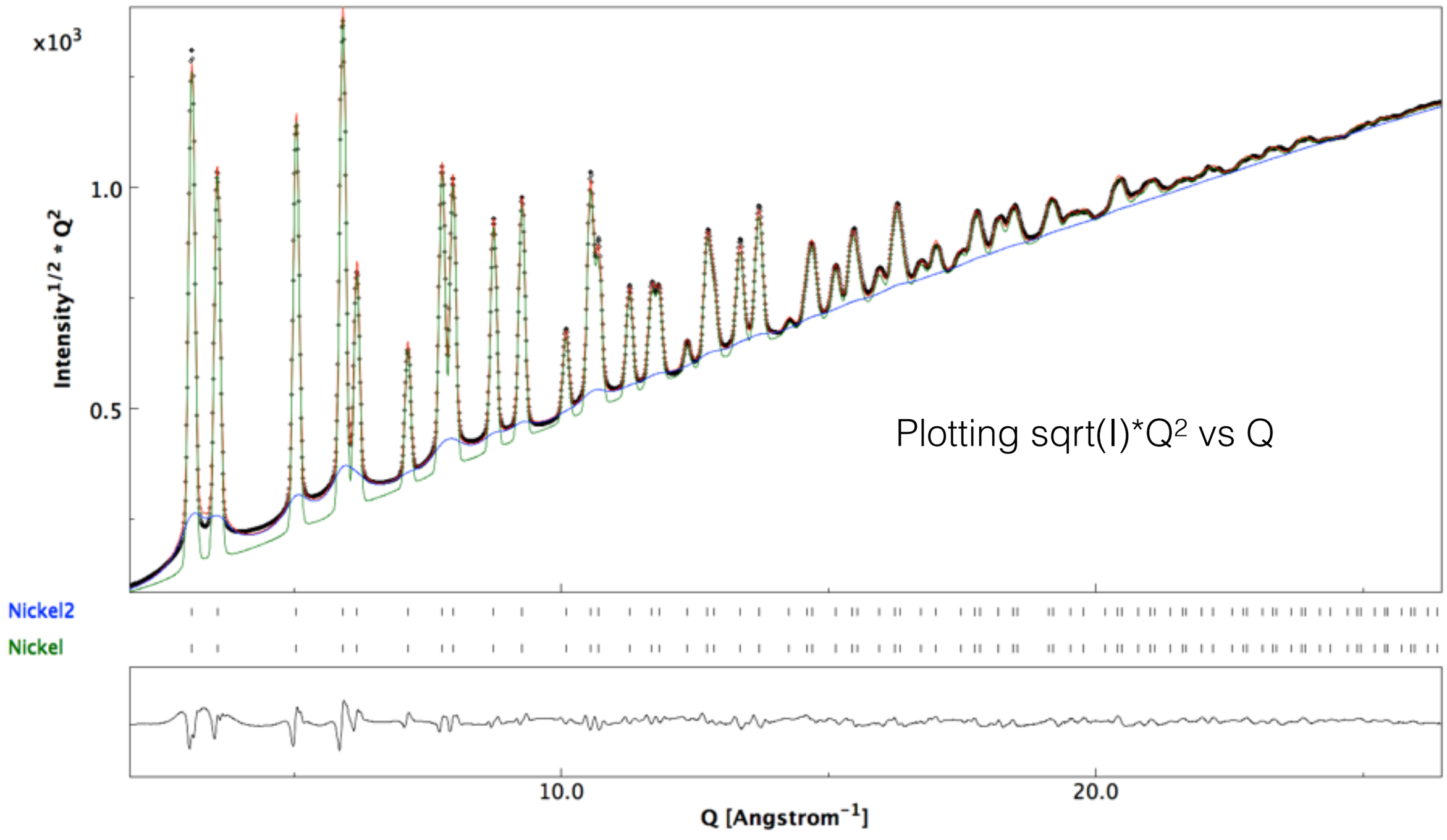
PdfgetX3 examples by Maud: Ni



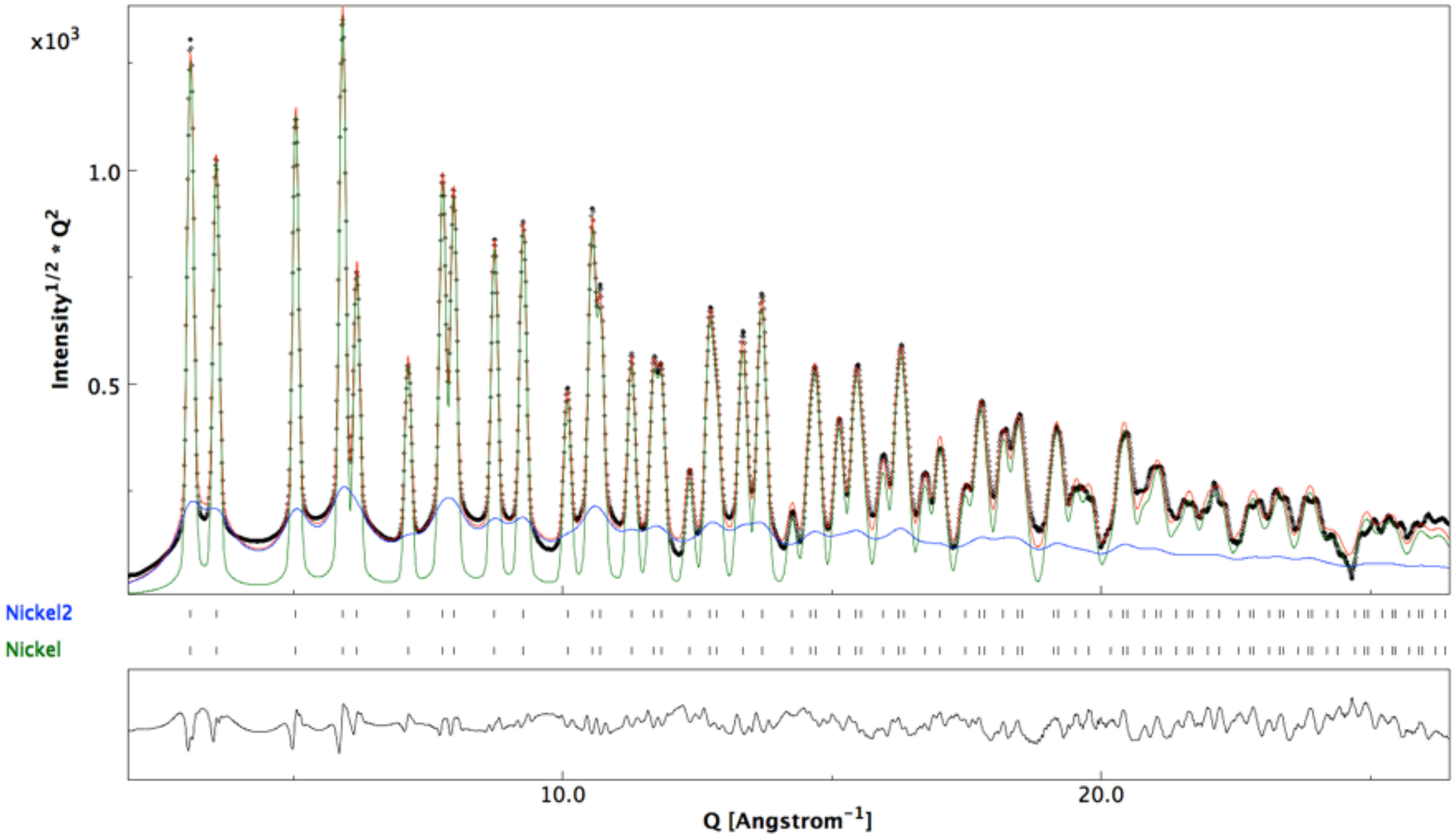
PdfgetX3 examples: Ni



PdfgetX3 examples: Ni



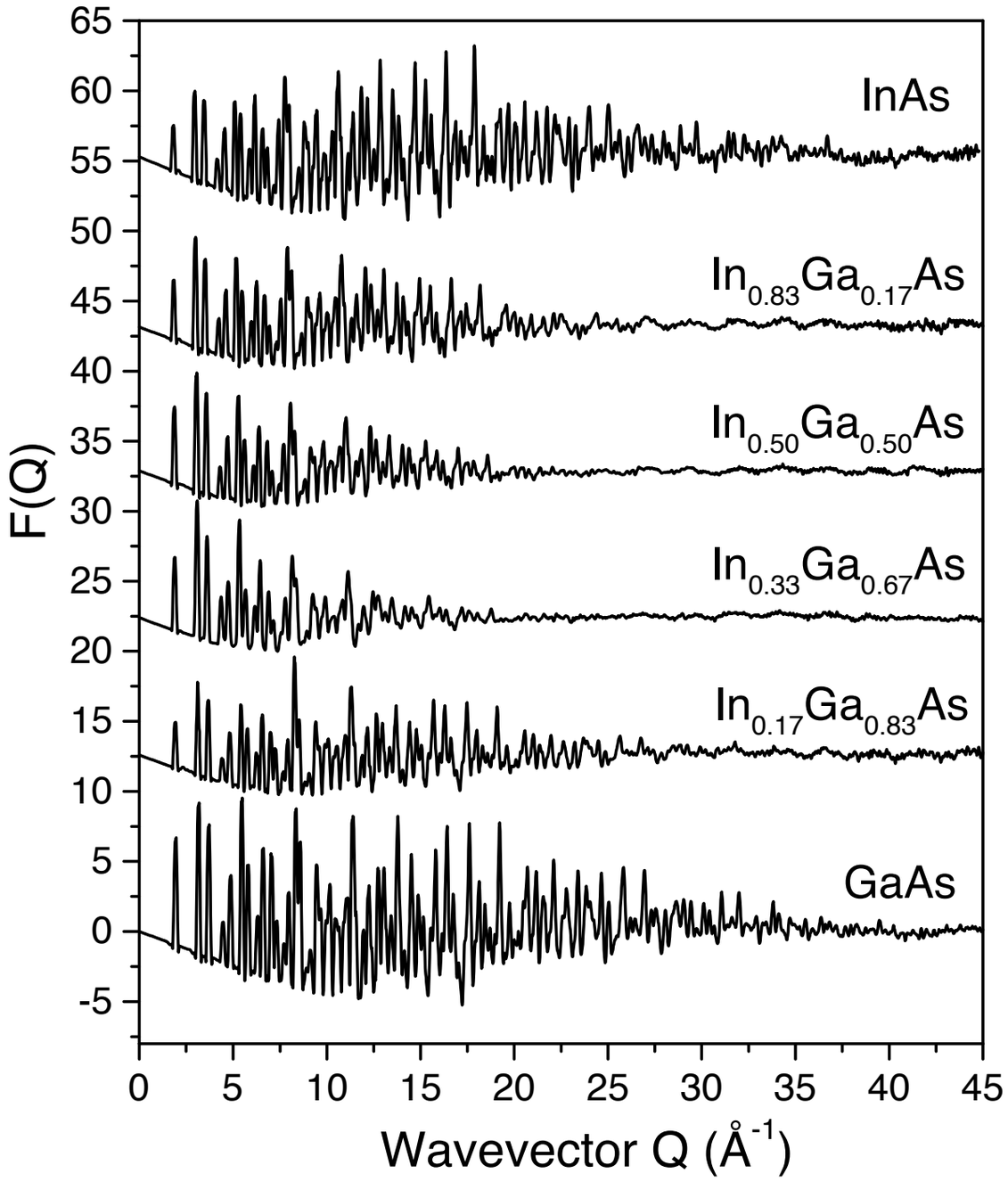
PdfgetX3 examples: Ni

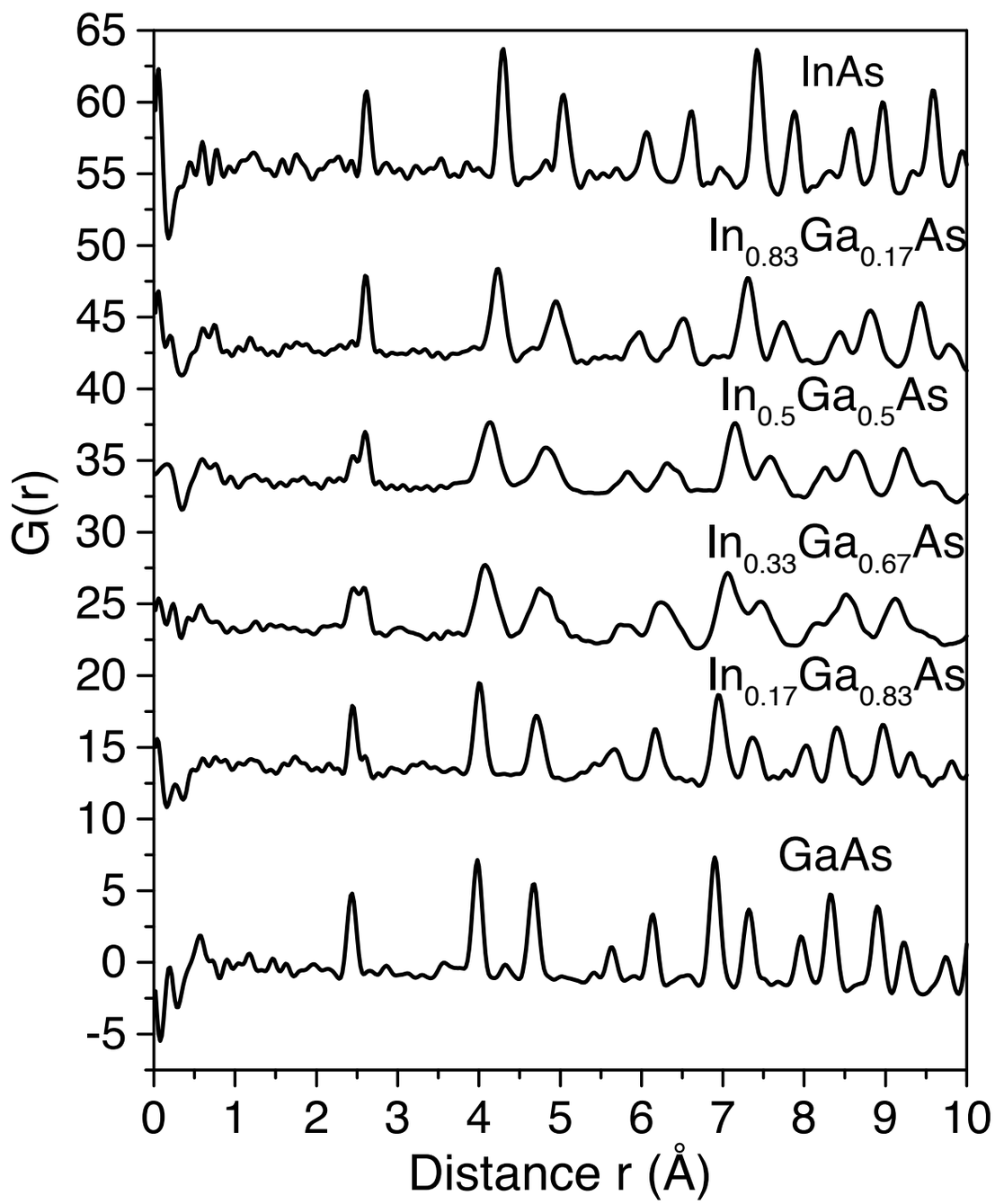


High real-space resolution measurement of the local structure of $Ga_{1-x}In_xAs$ using x-ray diffraction

V. Petkov[1], I-K. Jeong[1], J. S. Chung[1], M. F. Thorpe[1], S. Kycia[2] and S. J. L. Billinge[1]
[1] *Department of Physics and Astronomy and Center for Fundamental Materials Research, Michigan State University, East Lansing, MI 48824-1116.* [2] *Cornell High Energy Synchrotron Source, Cornell University, Ithaca, NY 14853*
(February 7, 2008)

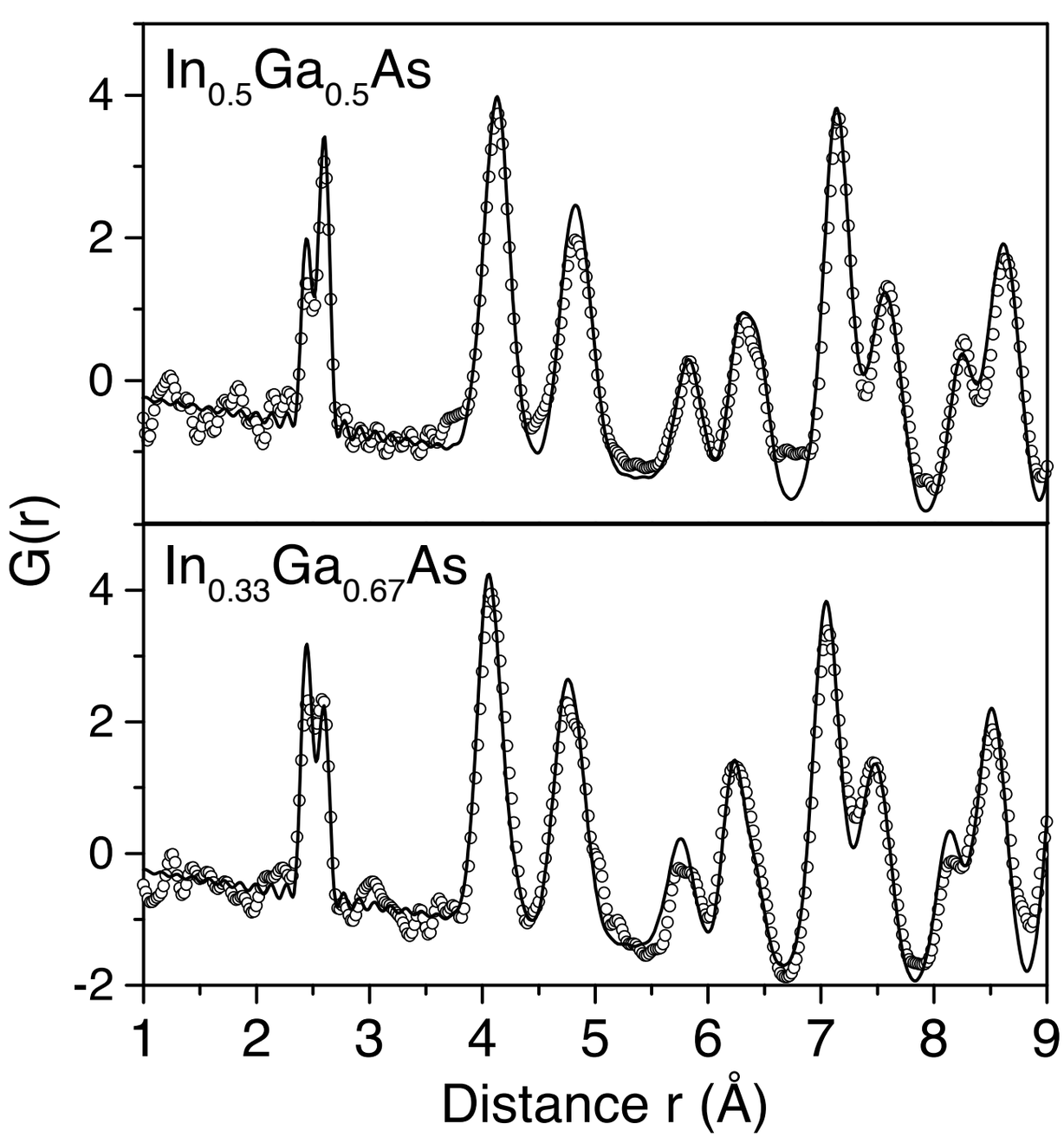
PHYSICAL REVIEW LETTERS · NOVEMBER 1999

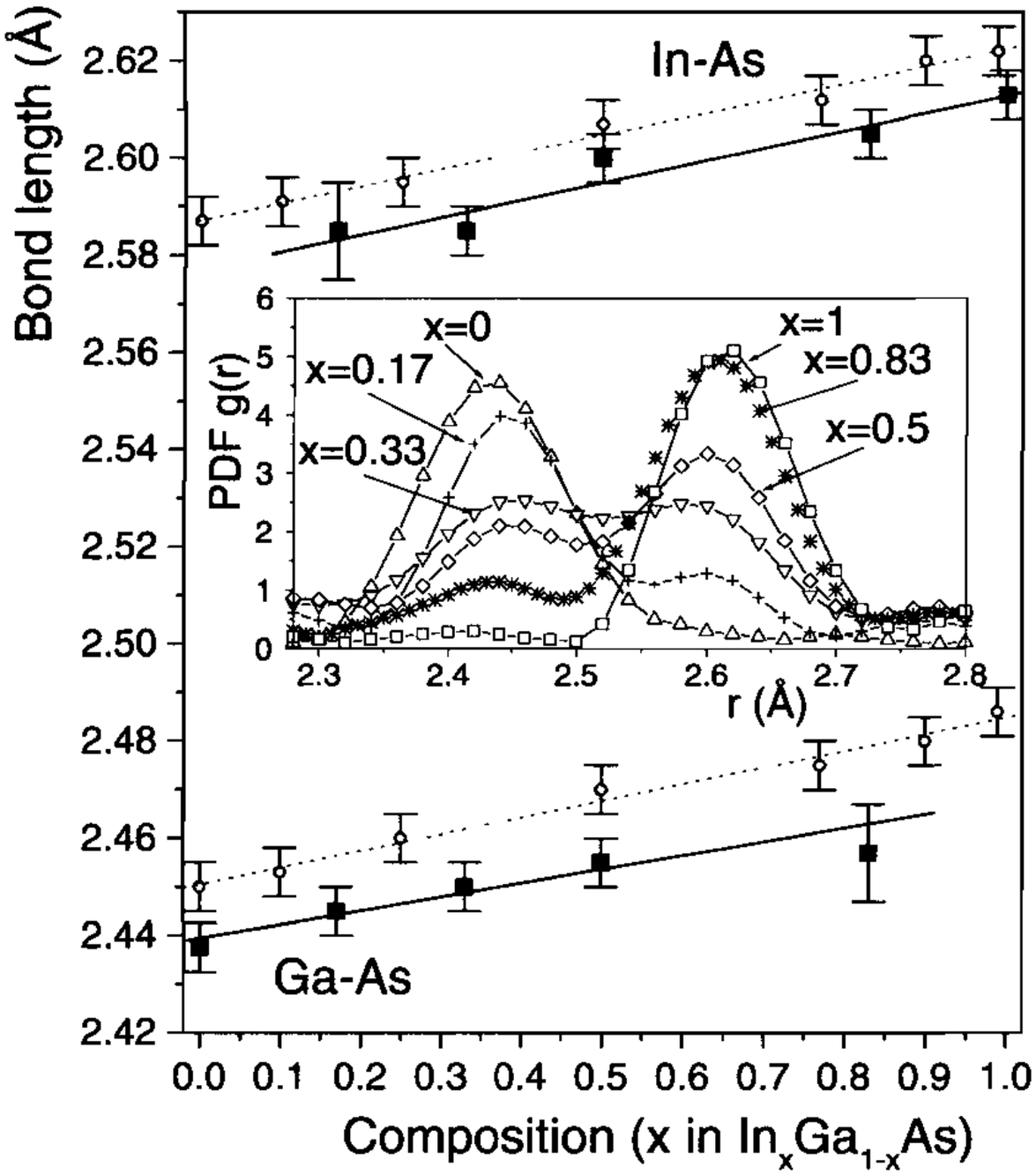




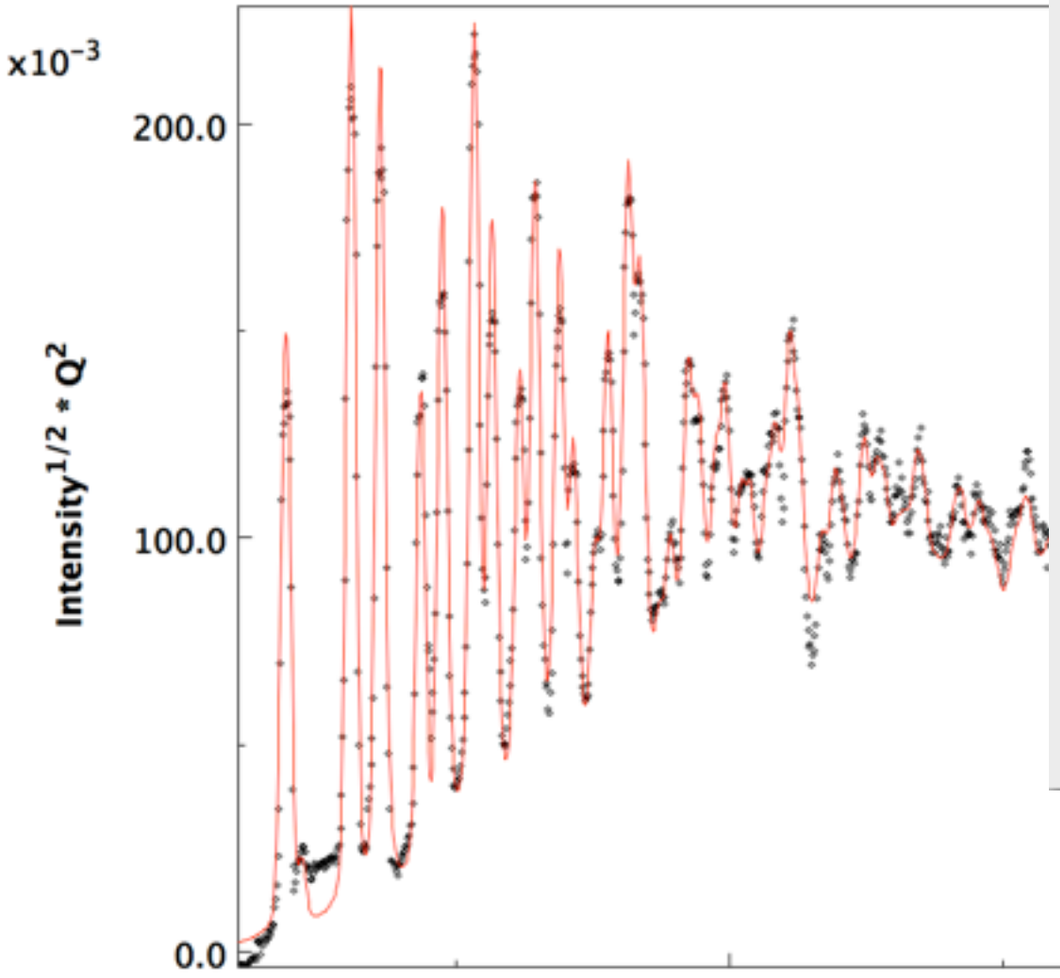
InGaAs modeling in PDF

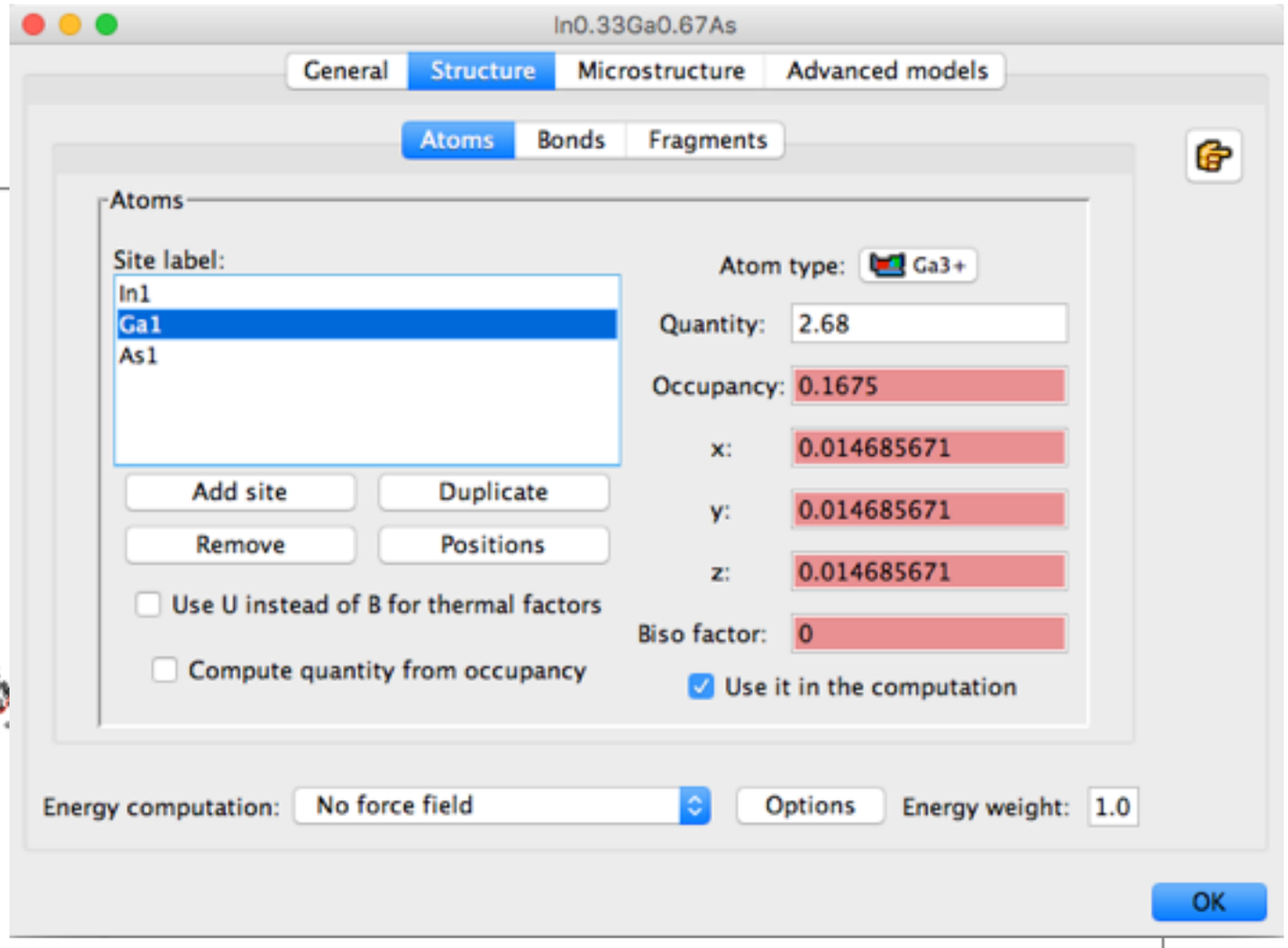
Modeling





PdfgetX3 examples: InGaAs in Maud



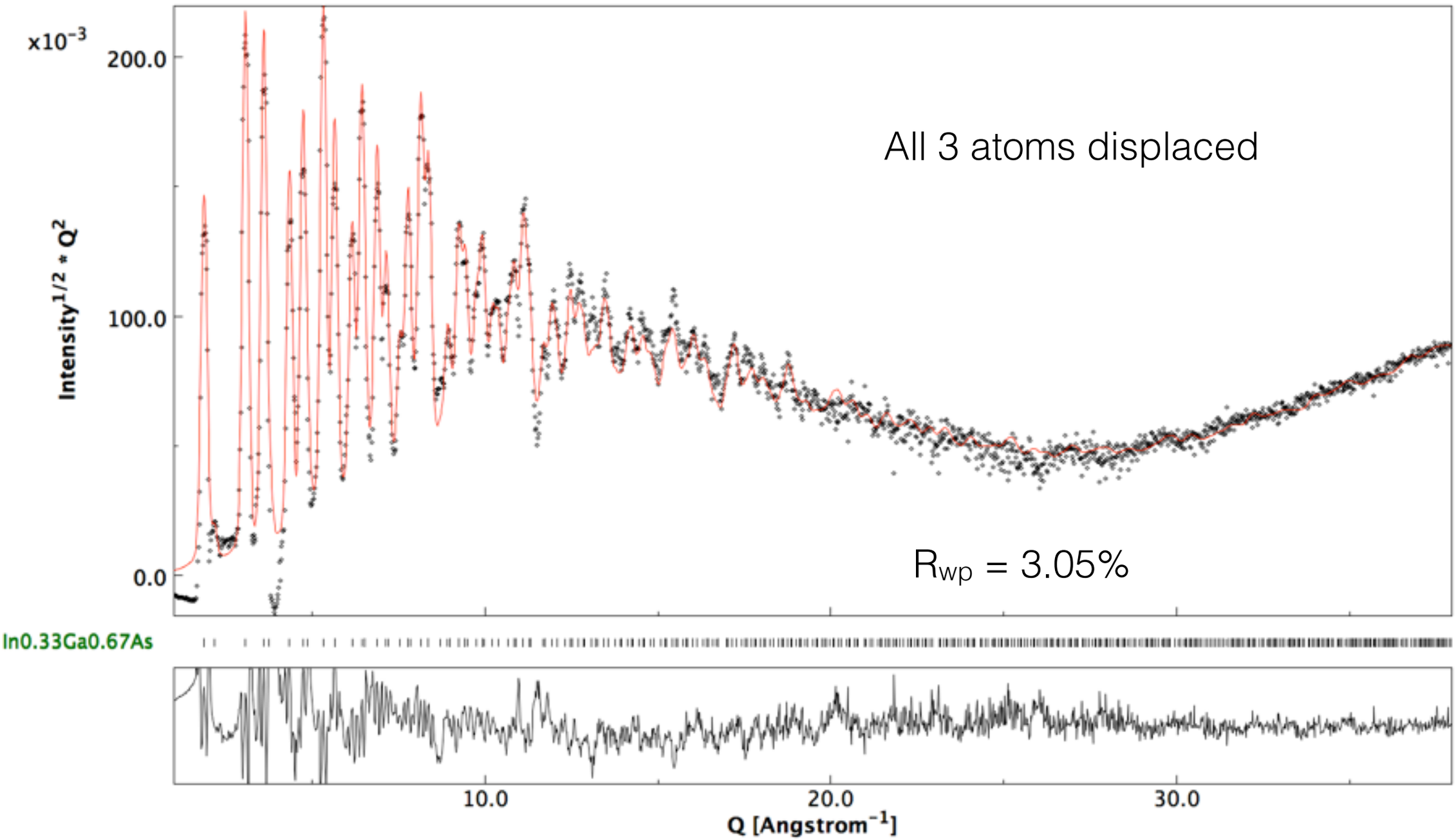


R_{wp} = 3.14%

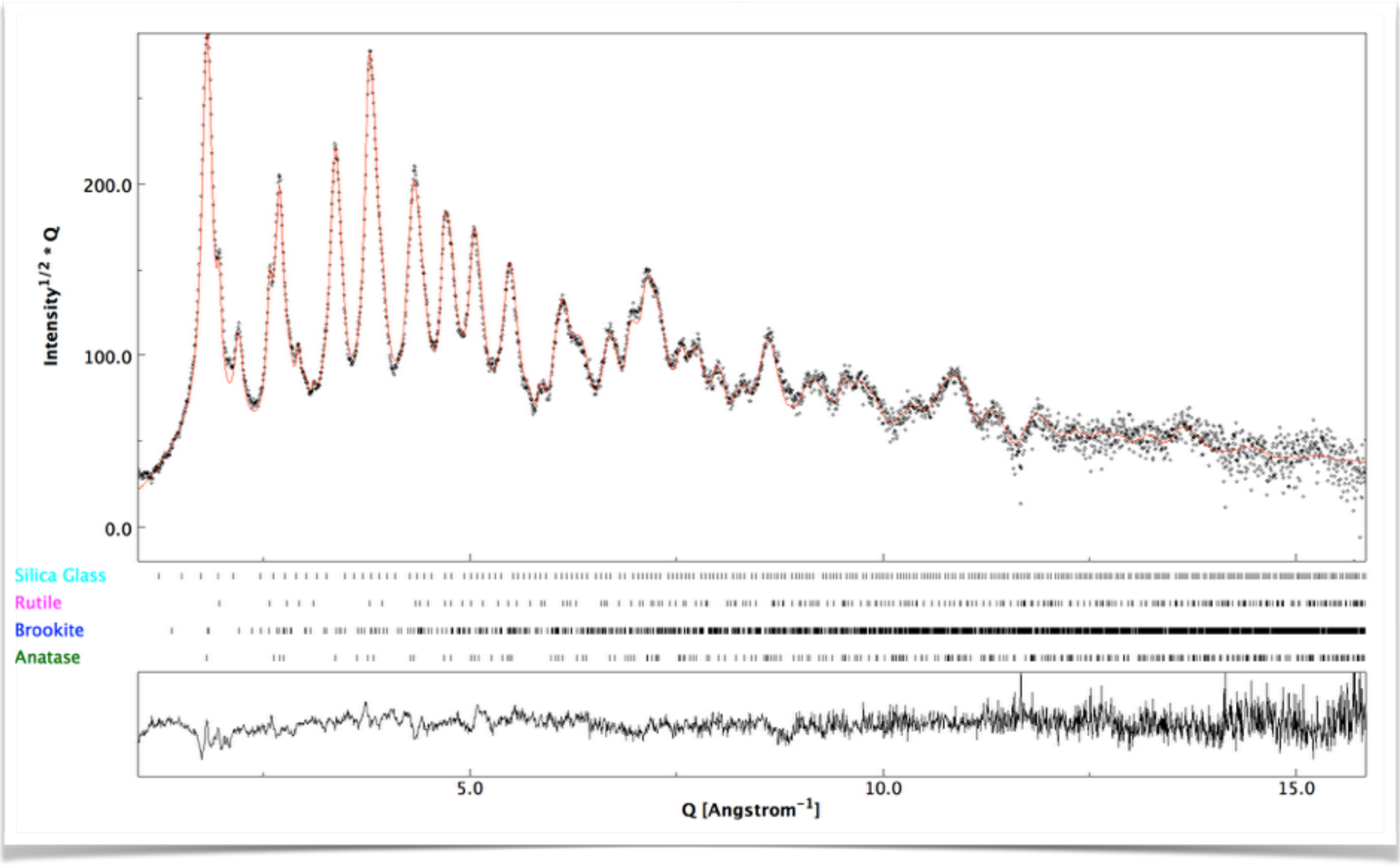
In0.33Ga0.67As

10.0
20.0
30.0
Q [Angstrom^-1]

PdfgetX3 examples: InGaAs

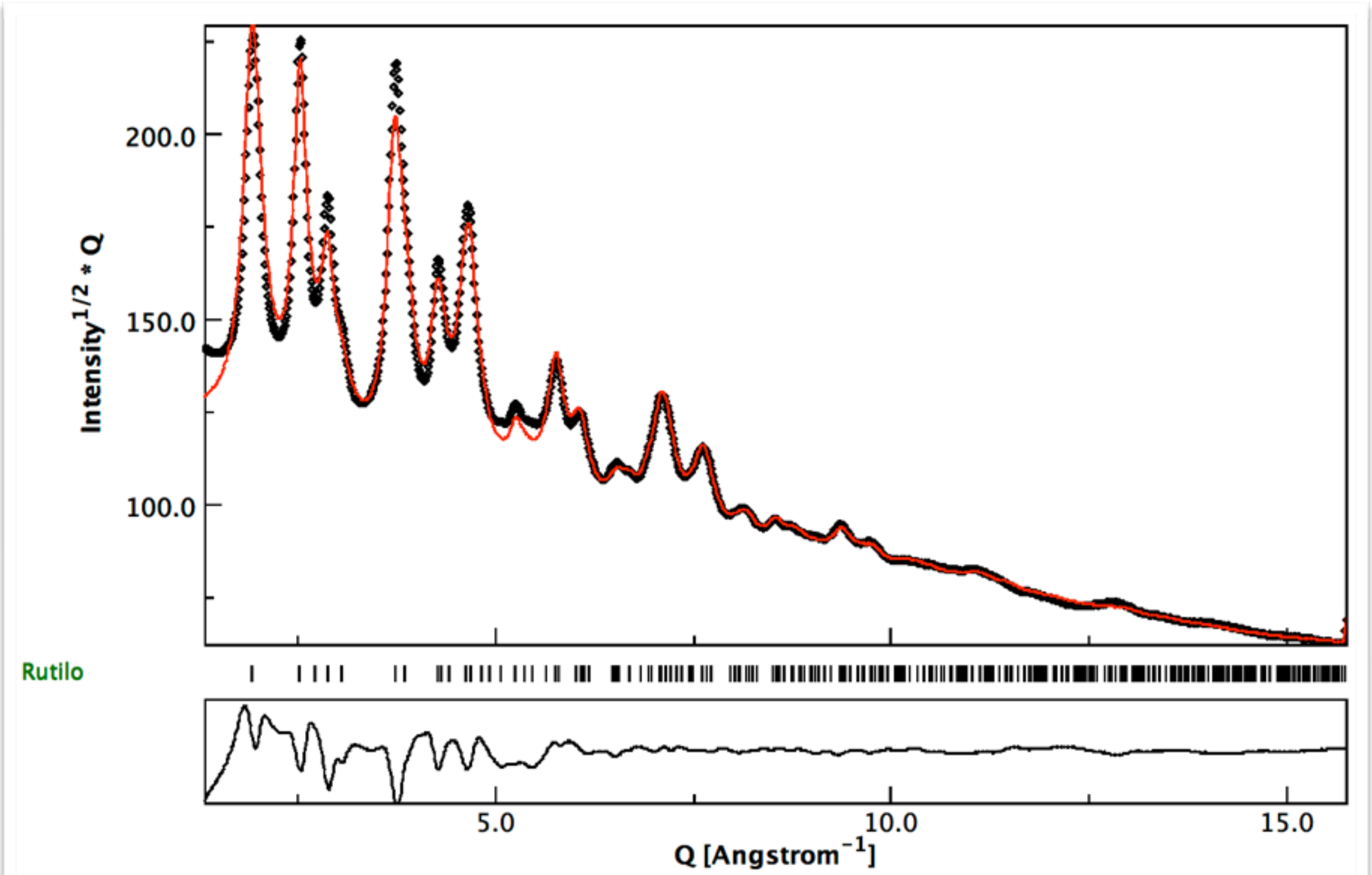


We are not limited to one phase



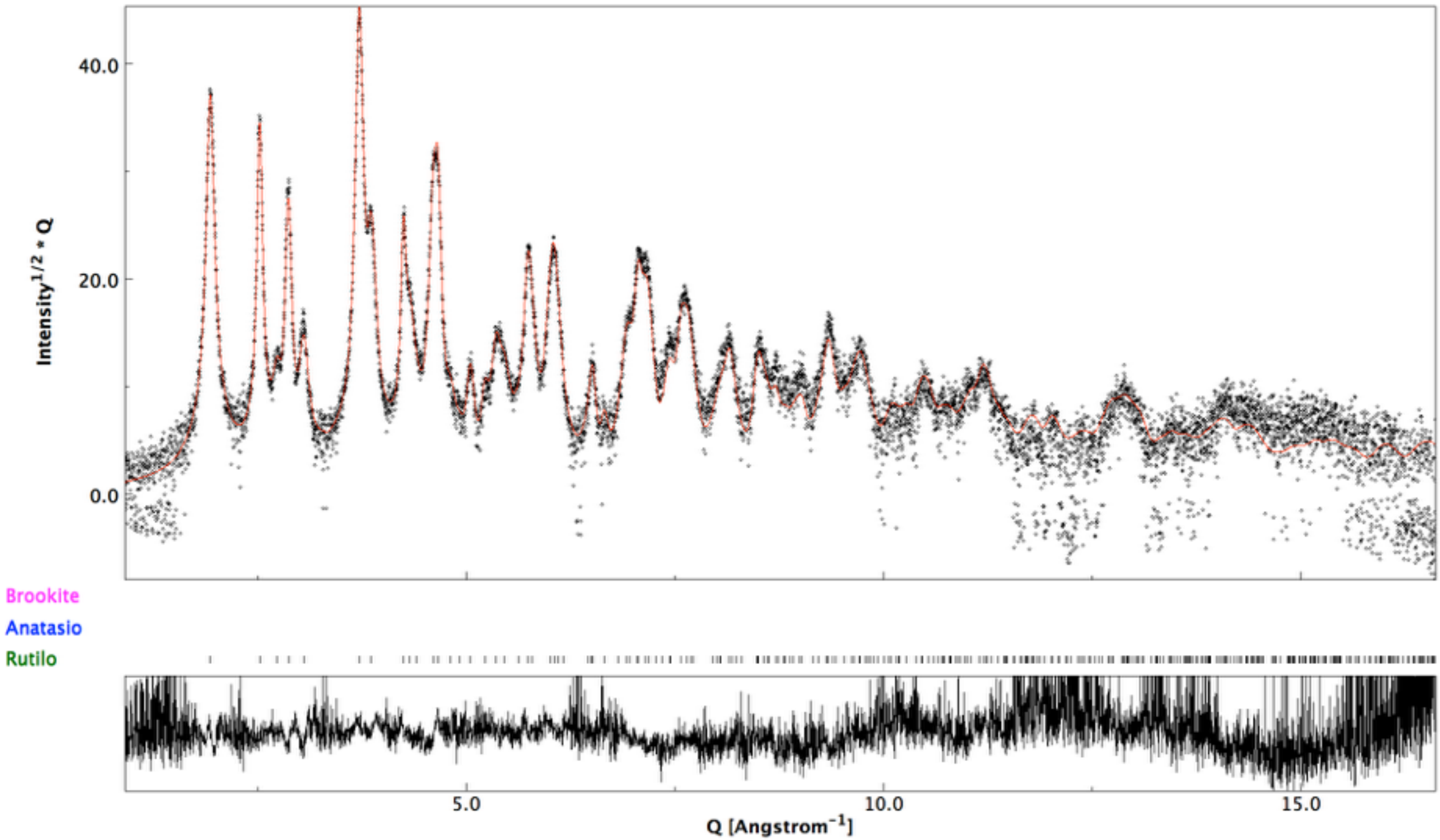
INEL CPS 120˚ Ag Kα, capillary sample holder

Works with electron diffraction



We can apply the Blackman dynamical correction

TiO_2 @ MARS-Soleil

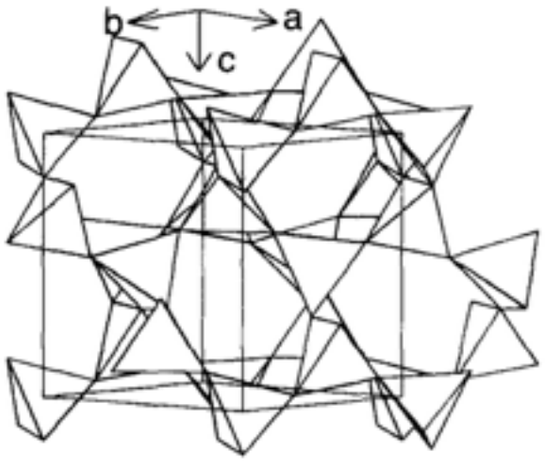


Pseudo-amorphous approximation

Journal of Non-Crystalline Solids 183 (1995) 39–42

Modelling the silica glass structure by the Rietveld method

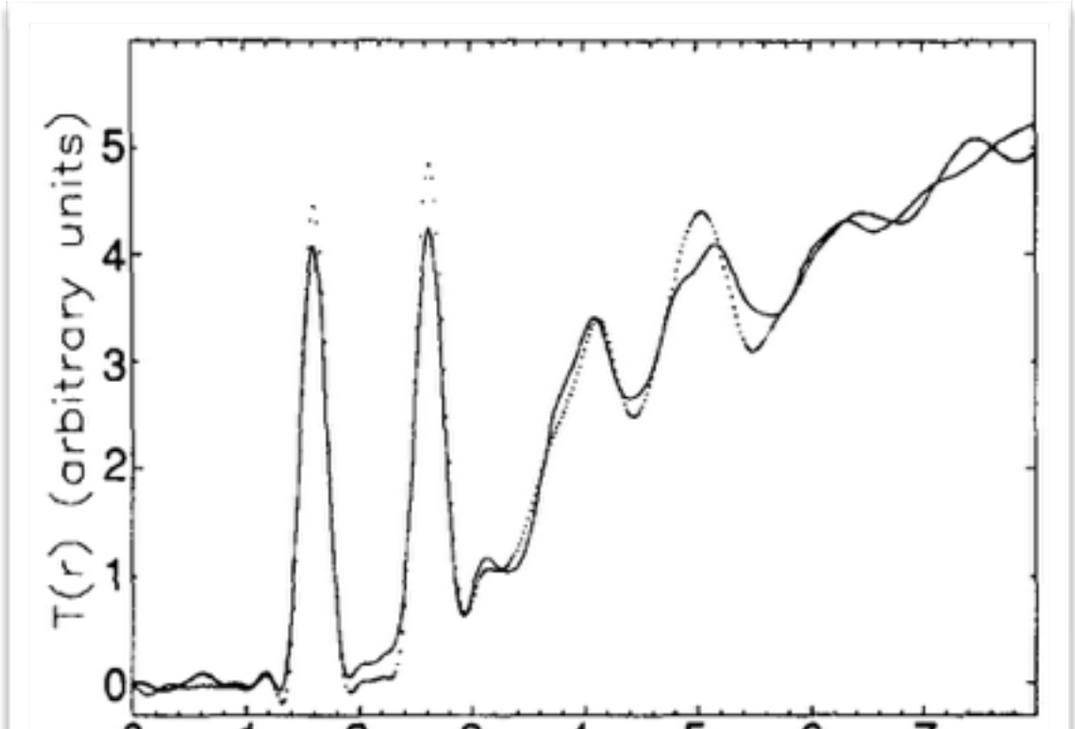
A. Le Bail *

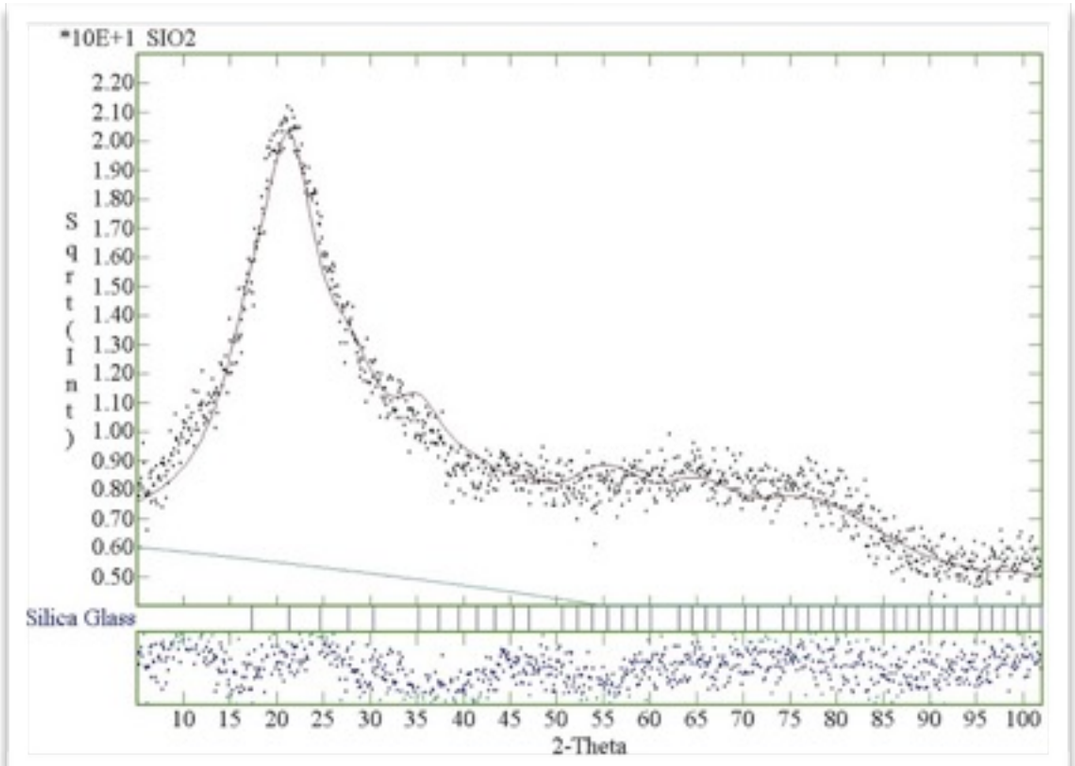


Refined parameters of the amorphous SiO_2 model in spacegroup $P2_12_12_1$

Atom	x	y	z
Si(1)	0.277(2)	0.275(2)	0.282(1)
Si(2)	0.009(1)	0.022(3)	0.016(3)
O(1)	0.125(3)	0.149(4)	0.152(3)
O(2)	0.635(4)	0.657(4)	0.061(5)
O(3)	0.065(4)	0.646(6)	0.654(7)
O(4)	0.670(3)	0.060(4)	0.621(4)

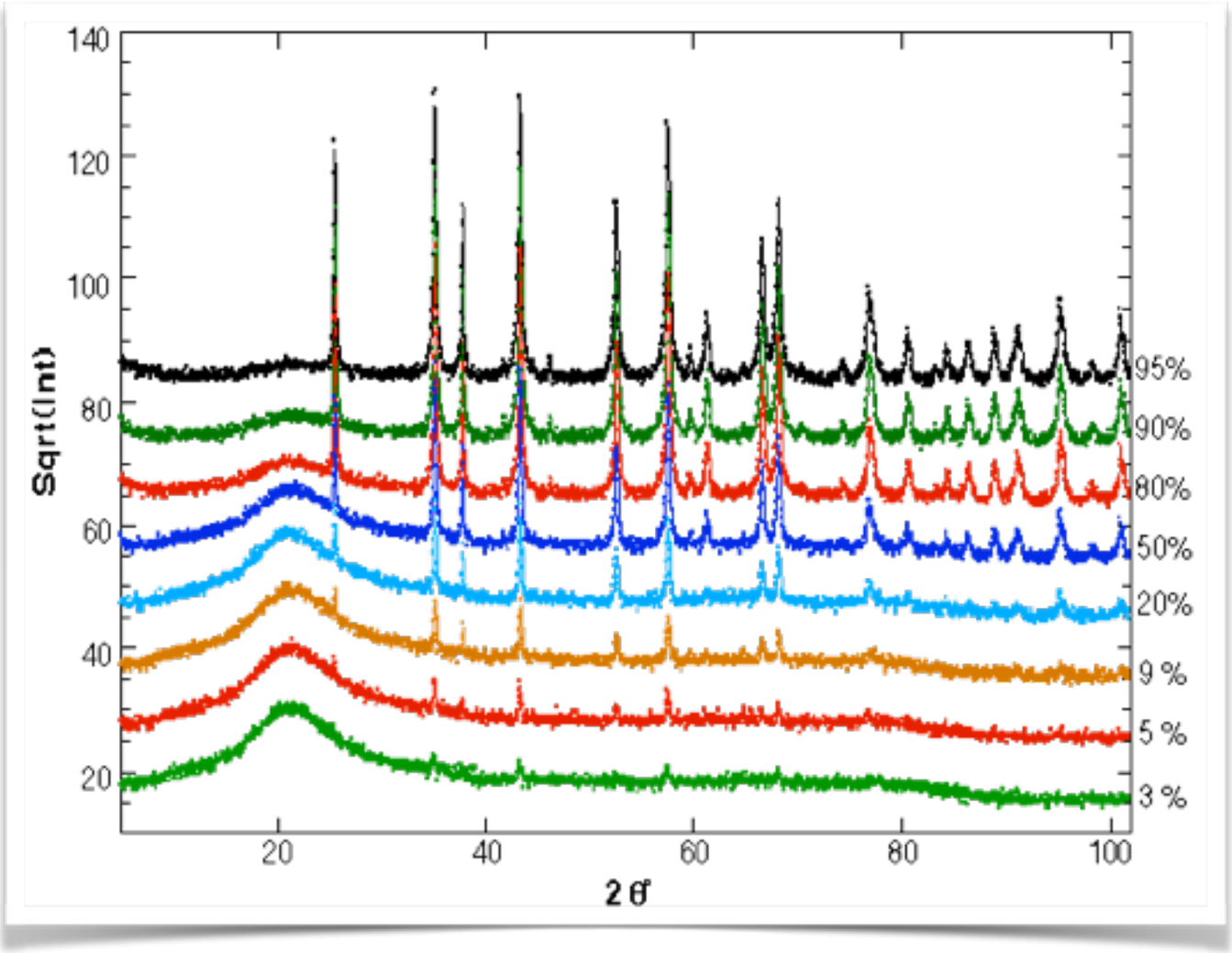
Lattice parameters: $a = 7.22(7)$ Å, $b = 7.09(6)$ Å and $c = 7.30(6)$ Å. V/molecule $= 46.7(2)$ Å^3 with $Z = 4$. Halfwidth parameters for the simultaneous fit of diffraction data rebuilt at a fictitious 2θ scale: $U = 22(6)$, $V = 0.5$, $W = 5.2(1)$ (neutron, $\lambda = 0.35$ Å) and $U = 42(11)$, $V = 1.0$, $W = 15(1)$ (X-ray, $\lambda = 0.5$ Å). The number in parentheses denotes the estimated standard deviation in the last digit.

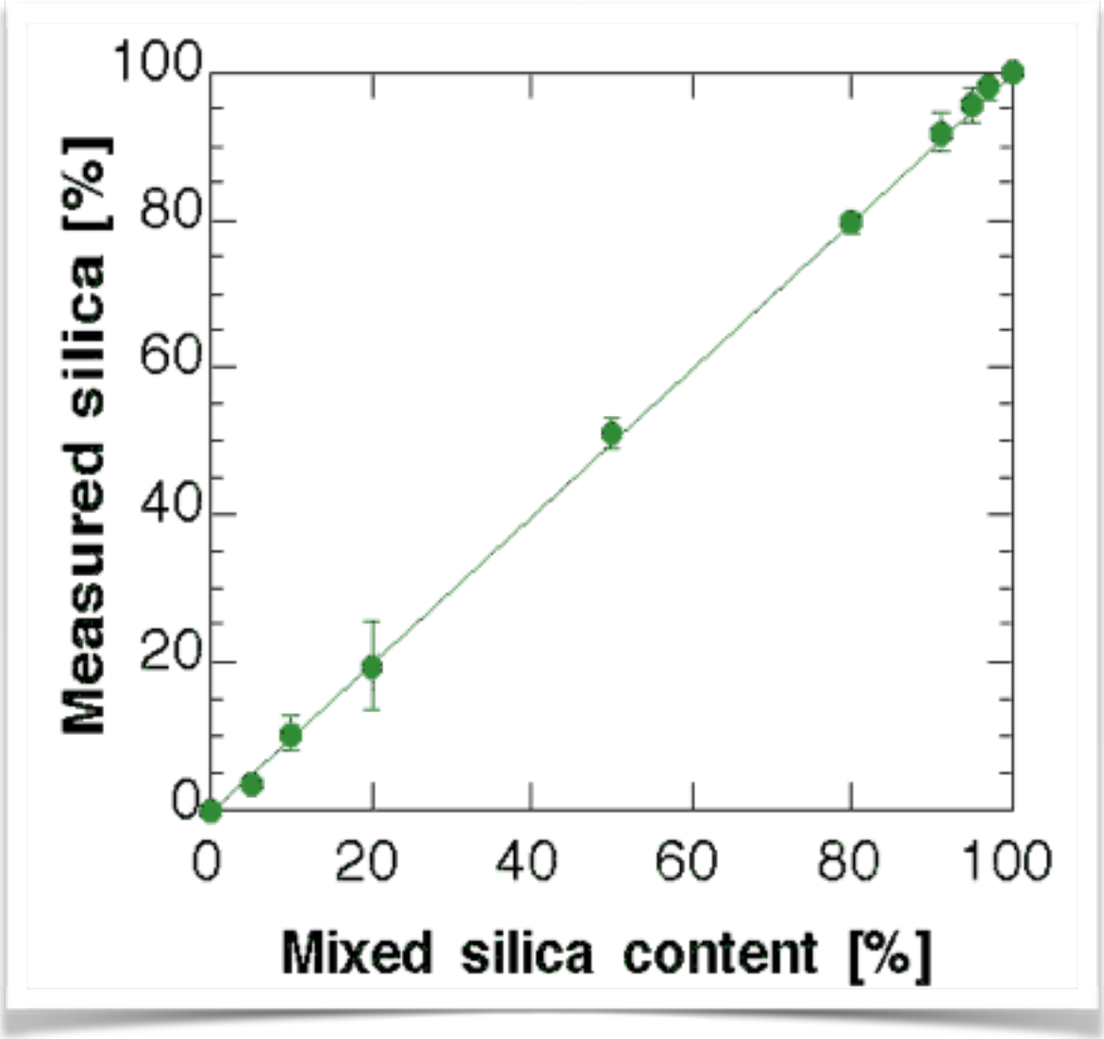




Corundum+amorphous silica mixed

Validation of the method testing with know amount of silica-alumina

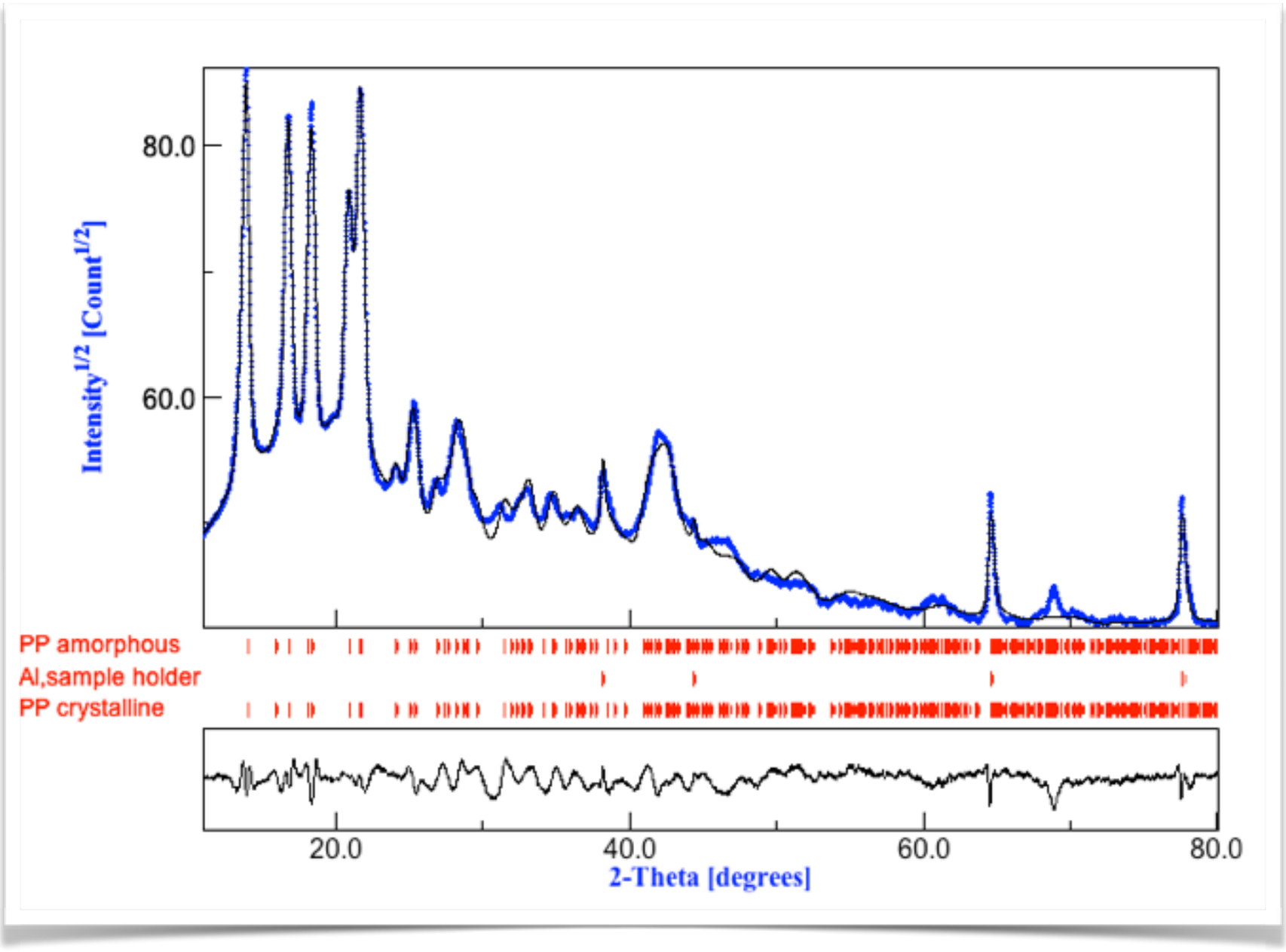




Lutterotti L., Ceccato R., Dal Maschio R., Pagani E.: Quantitative analysis of silicate glass in ceramic materials by the Rietveld method. Mater. Sci. Forum, 278-281, 87-92 (1998)

Crystalline fraction for polypropylene

- Same crystal structure for amorphous and crystalline
- Results: 43(1) % crystalline - 57(1) % amorphous



Application: Nifedipine/PVP composite

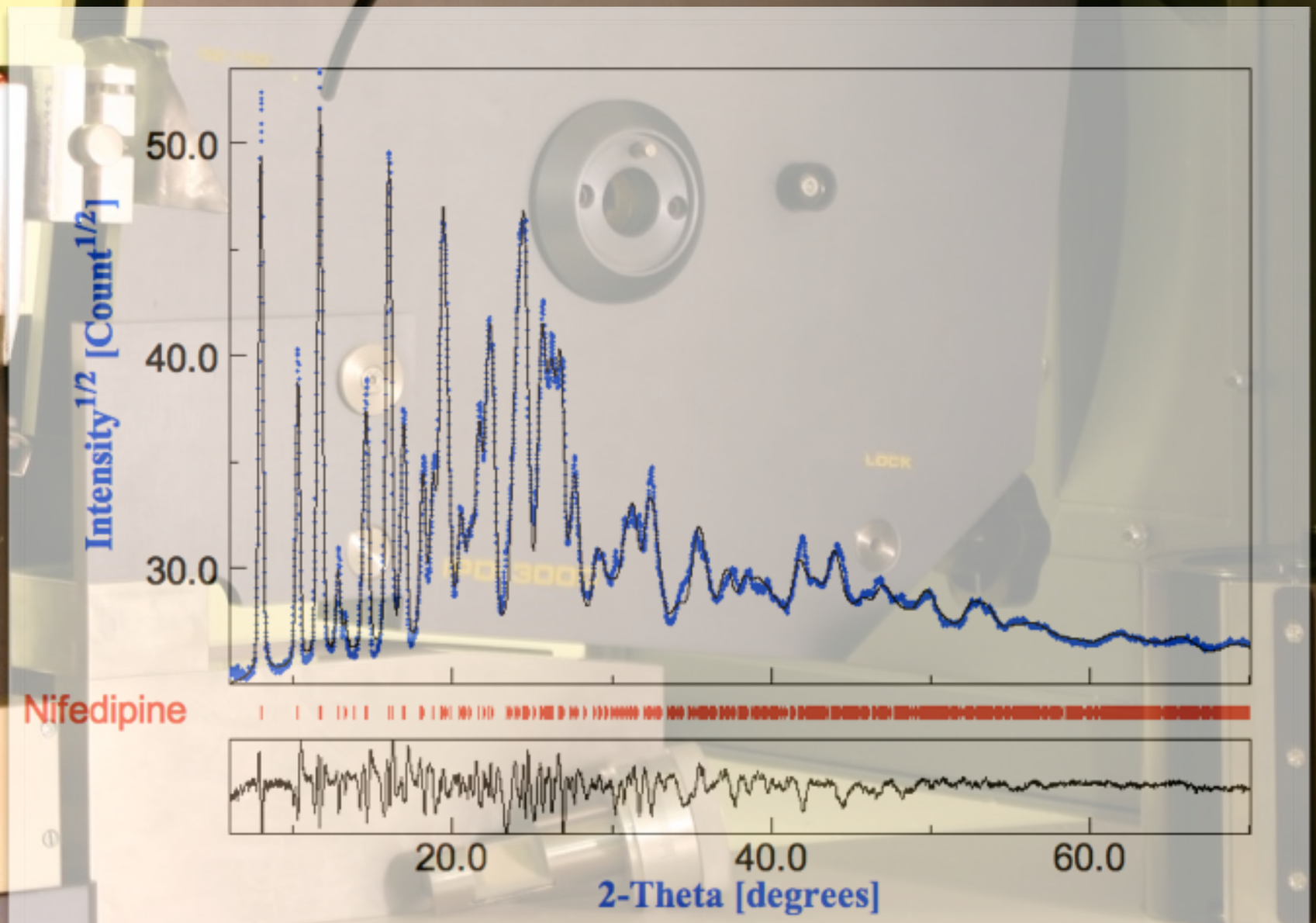
- Nifedipine is used to treat high blood pressure and to control chest pain
- To control activity and release we study the dispersion of nifedipine inside a polymer (PVP)
- By ball milling we aim at stabilize the nifedipine up to a molecular level (amorphous) inside the PVP
- We need to characterize the crystallization state of the nifedipine inside the polymer after milling at different times and energies -> PXRD

Milling of the composite

- Planetary milling system: Fritsch Pulverisette 7
- Carrier/drug weight ratio 50:50 %
- Milling times (minutes): 0, 5, 10 15, 20, 25, 30, 45, 60

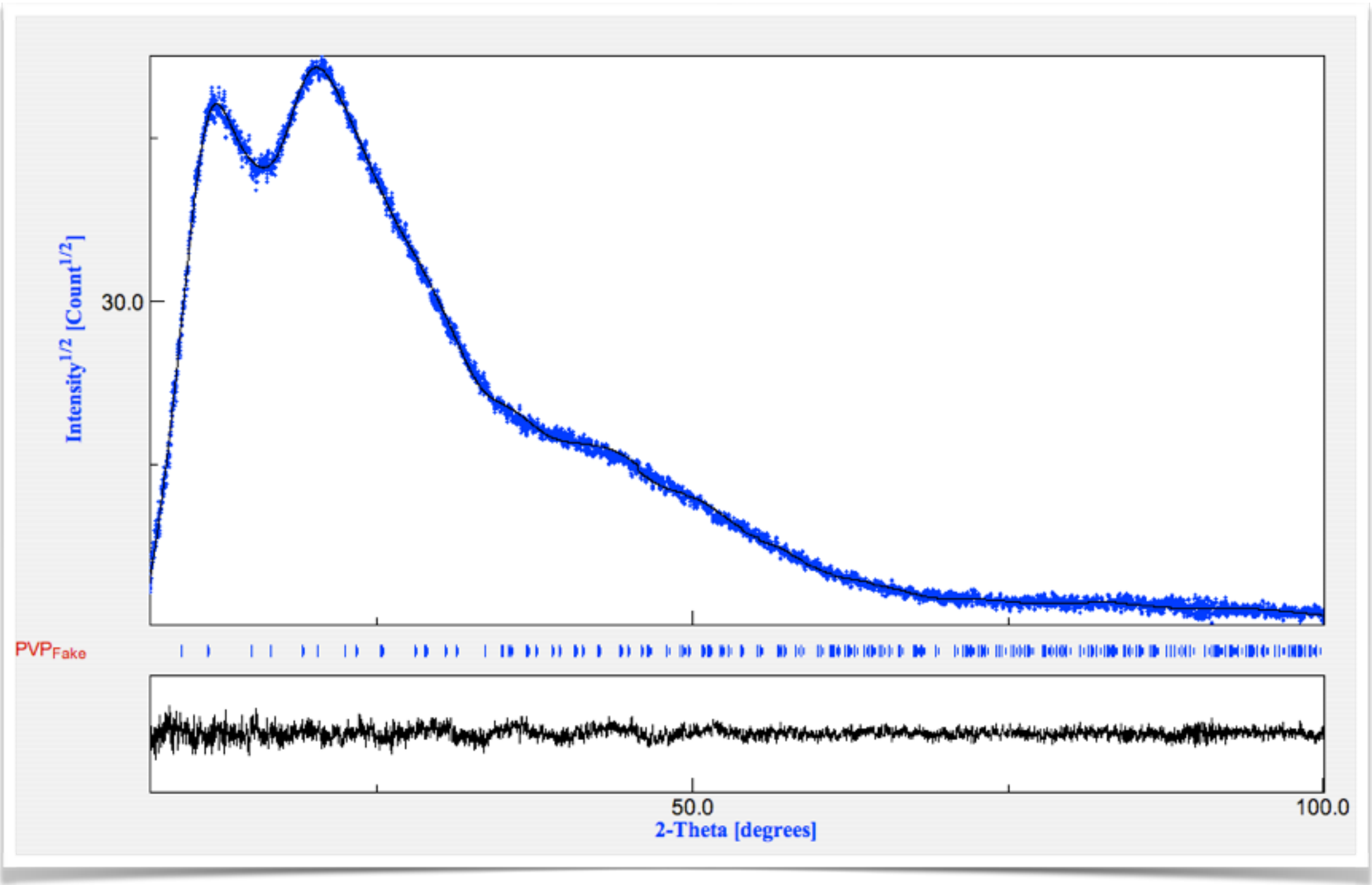
Nifedipine refinement

- Image plate (IPD 3000) data, high intensity
- The Nifedipine has a tendency to orient 100



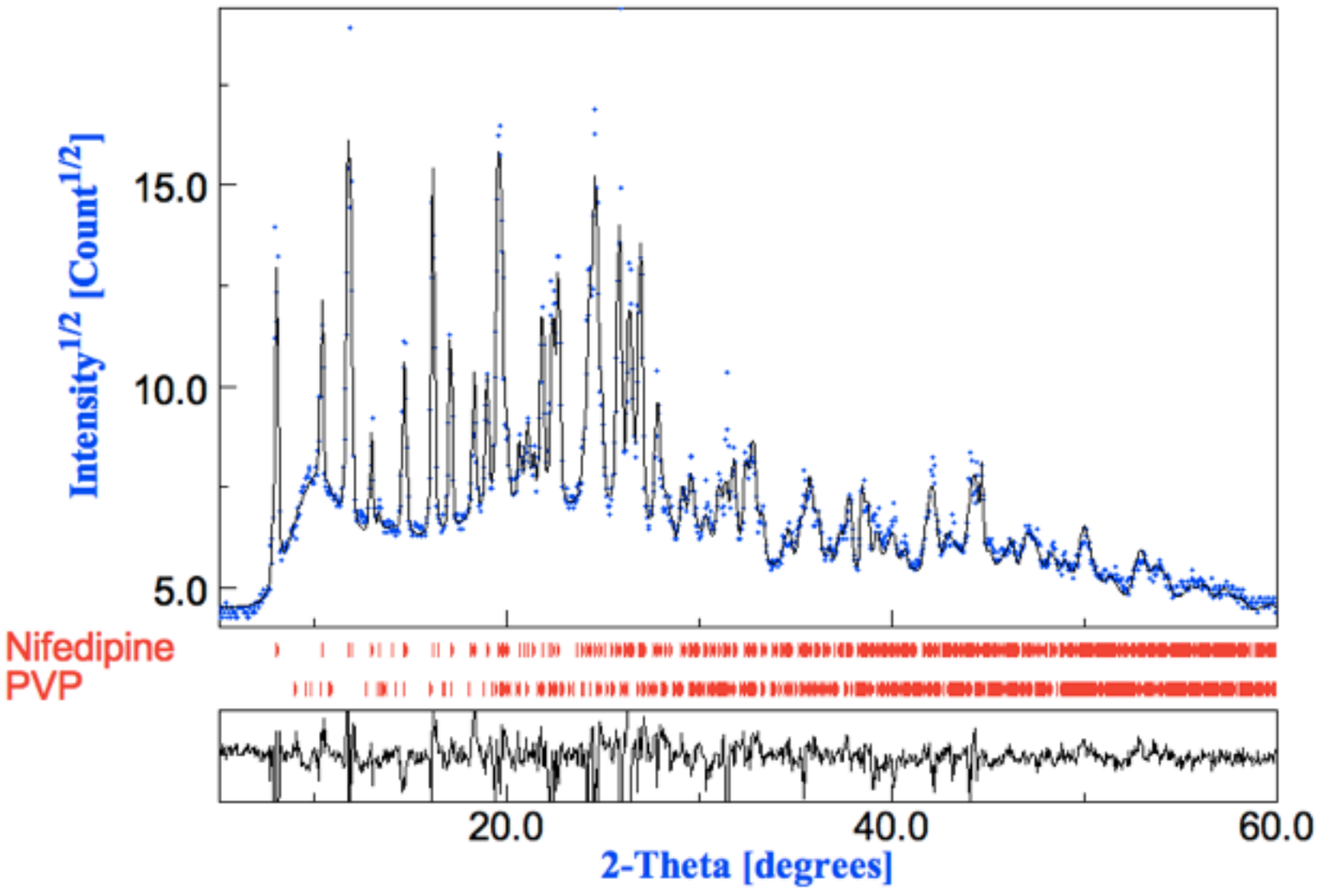
The PVP amorphous pseudo-structure by MEEM

- Rw = 0.58%, Rw(no bkg) = 0.64%, linear bkg
- Crystallites = 15 Angstrom, microstrain = 0.02



PVP MEEM fit and calibration

- The spectrum was fitted by knowing the nifedipine structure and performing a focused indexing and MEEM refinement for the PVP (phases quantities imposed for the 50-50 wt% as prepared sample)



Amorphization results