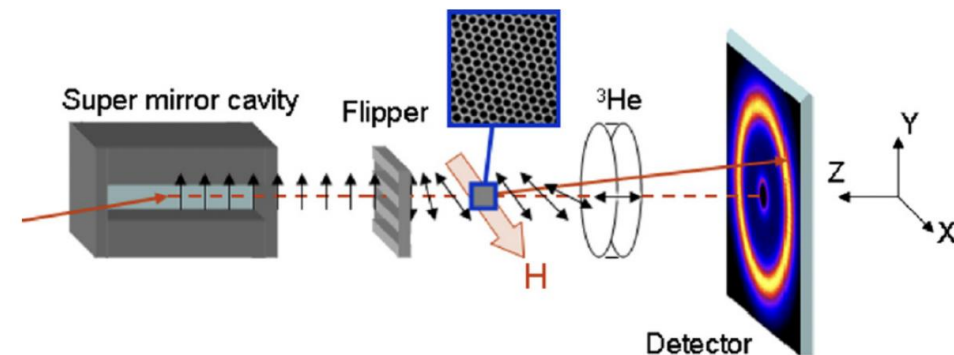
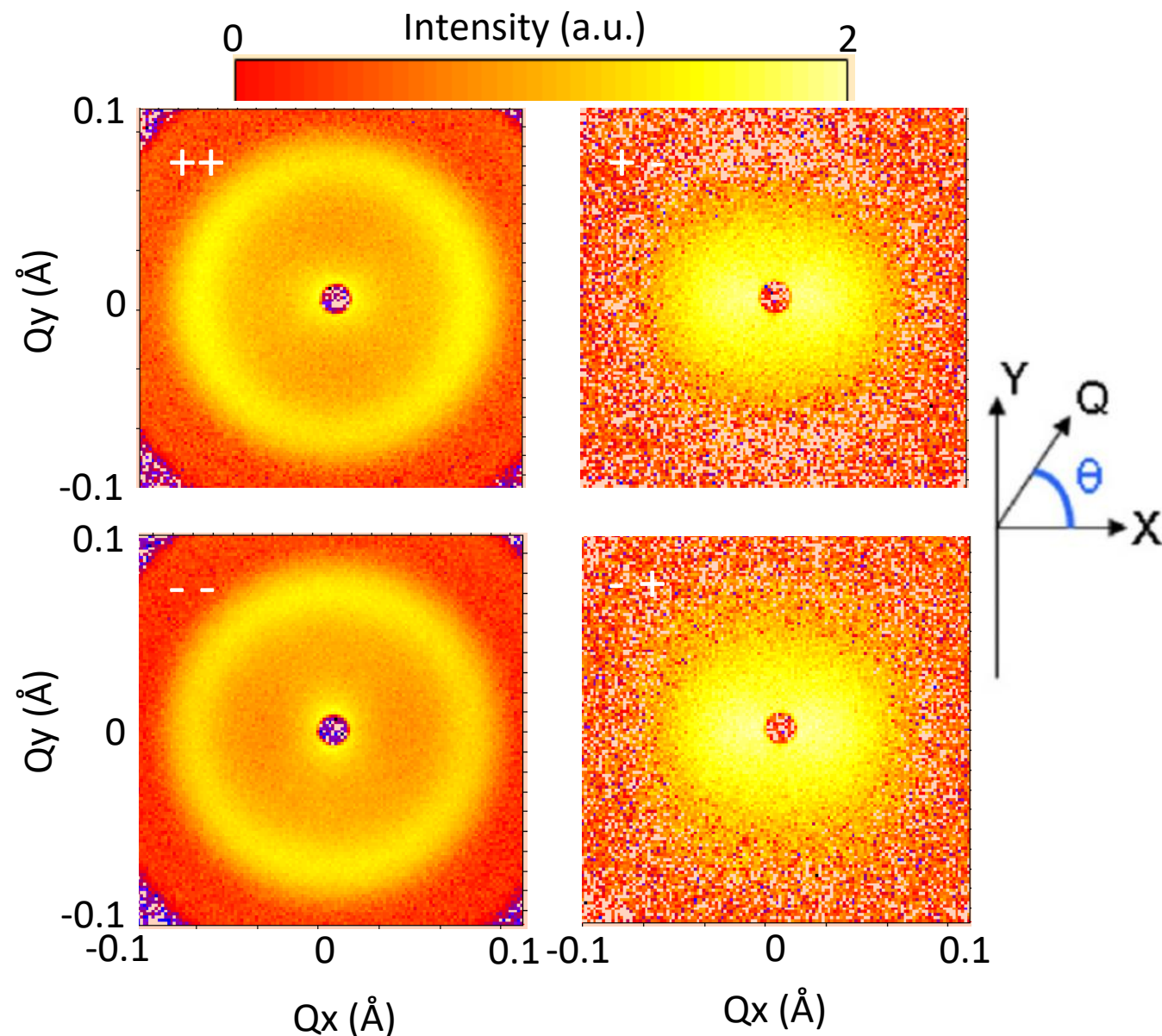


Guide to SasView fitting for the 10 K, 1.2 T CoFe_2O_4 Nanoparticles

Full Pol. data of CoFe_2O_4 collected at 10 K and 1.2 T

K. L. Krycka, et al., J Appl Cryst **45**, 554–565 (2012)



$$I_{\phi=0^\circ}^{++,--} = N^2,$$

$$I_{\phi=90^\circ}^{++,--} = N^2 + M_X^2 \mp 2NM_X,$$

$$I_{\phi=0^\circ}^{+-,-+} = M_Y^2 + M_Z^2 = 2M_{\text{PERP}}^2,$$

$$I_{\phi=90^\circ}^{+-,-+} = M_Z^2 = M_{\text{PERP}}^2,$$

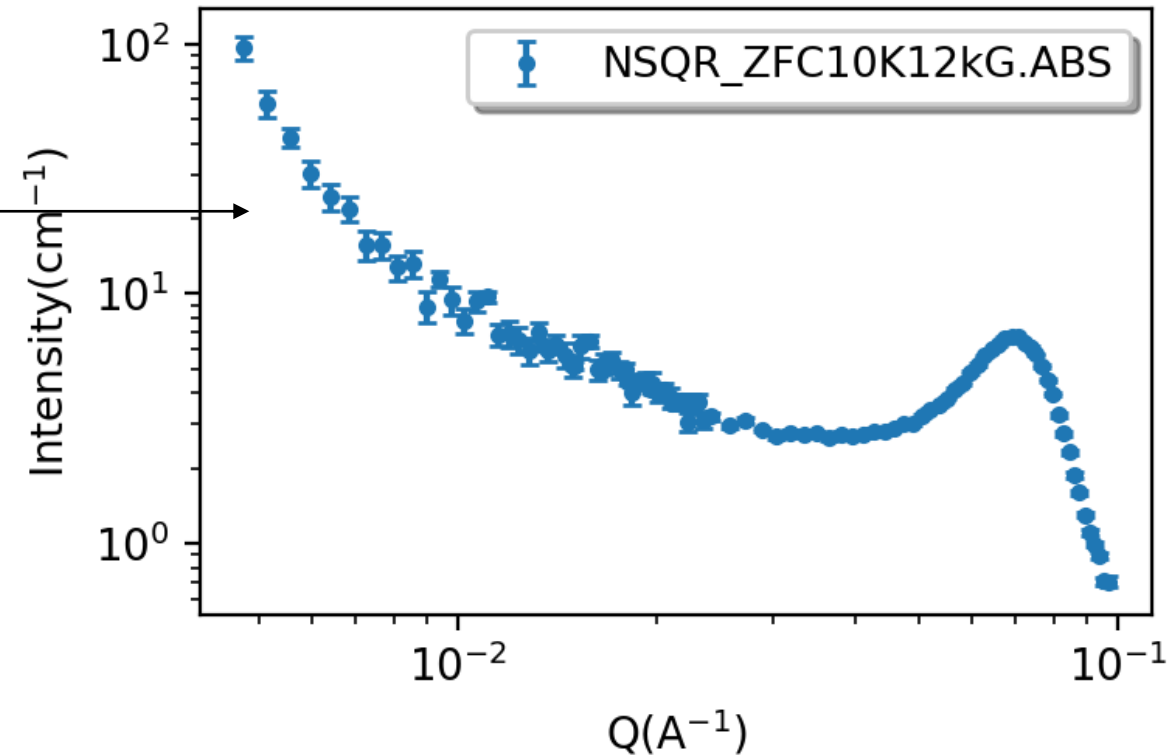
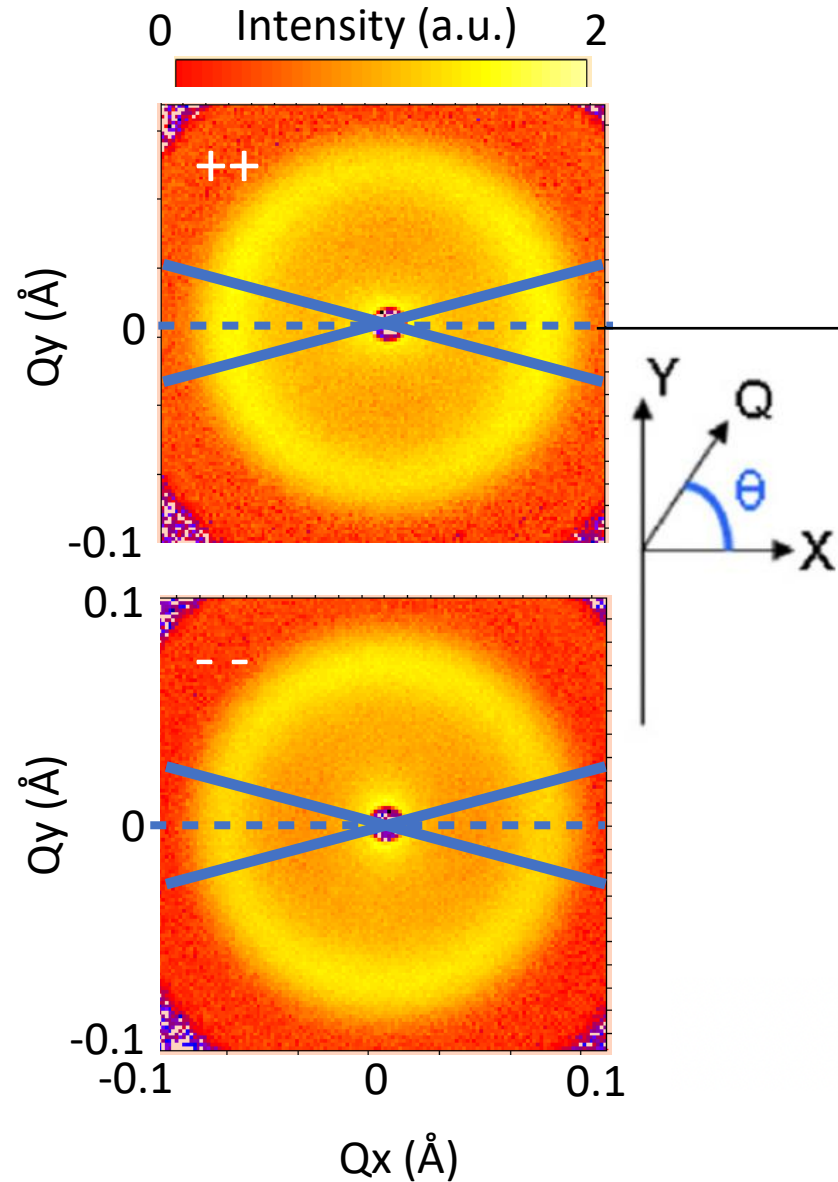
*Assuming isotropic system with only the X (field) direction being unique

$$M_X^2 = M_{\text{parl}}^2$$

$$M_Y^2 = M_Z^2 = M_{\text{perp}}^2$$

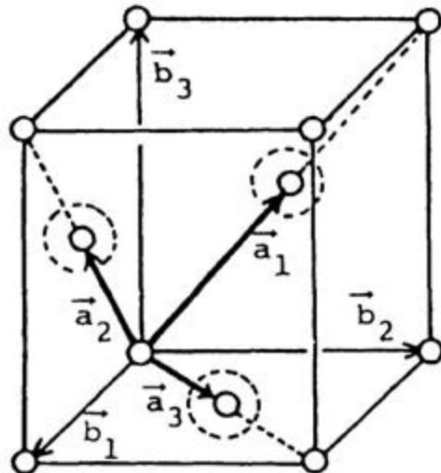
*note that the data have been corrected for spin leakage, background/empty cell contribution, detector efficiency, etc...

Extracting the pure nuclear scattering of CoFe_2O_4 using Pol. SANS



$$I_{\phi=0^\circ}^{++,--} = N^2 \longrightarrow N^2(Q) = \frac{1}{2}(I_{\theta=0^\circ}^{++} + I_{\theta=0^\circ}^{--})$$

Fit of the pure Nuclear scattering (10 K, 1.2 T)



The scattering intensity $I(q)$ is calculated as

$$I(q) = \frac{\text{scale}}{V_p} V_{\text{lattice}} P(q) Z(q) + \text{background}$$

where *scale* is the volume fraction of crystal in the sample volume, V_{lattice} is the volume fraction of spheres in the crystal, V_p is the volume of the primary particle, $P(q)$ is the form factor of the sphere (normalized), and $Z(q)$ is the paracrystalline structure factor for a face-centered cubic structure.

Finally, the position of the Bragg peaks for the fcc lattice are indexed by (reduced q-values):

$$\frac{qa}{2\pi} = \frac{qD}{\sqrt{2}\pi} = \sqrt{h^2 + k^2 + l^2}.$$

where the scattering condition imposes that h,k, and l are all odd or all even.

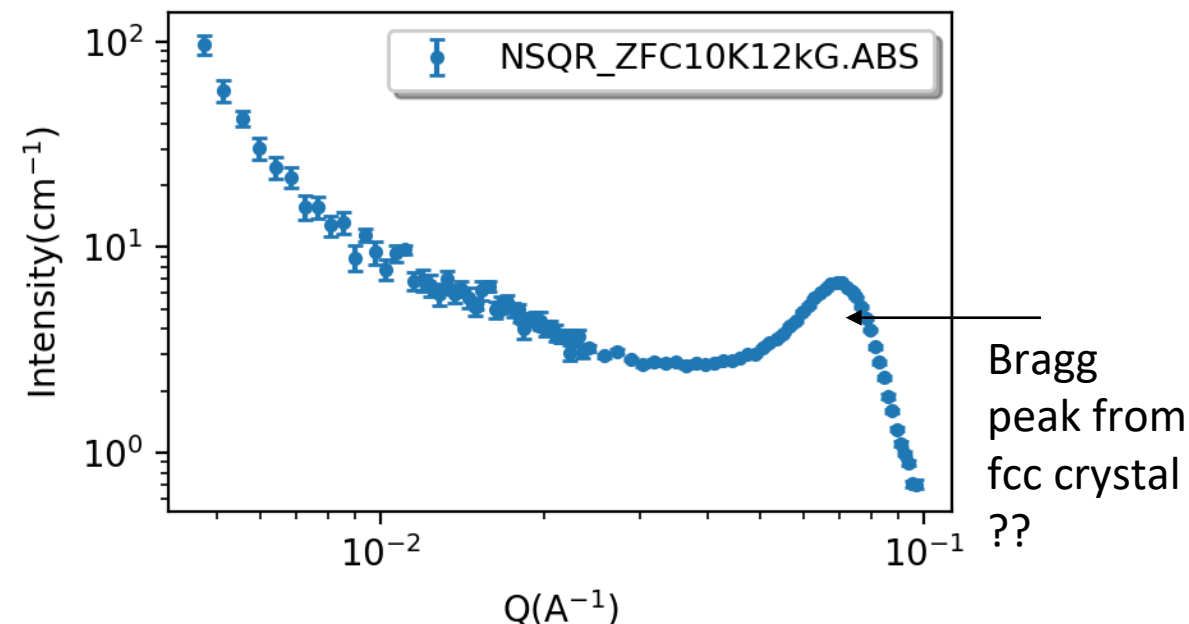
Parameter	Description	Units	Default value
scale	Scale factor or Volume fraction	None	1
background	Source background	cm ⁻¹	0.001
dnn	Nearest neighbour distance	Å	220
d_factor	Paracrystal distortion factor	None	0.06
radius	Particle radius	Å	40
sld	Particle scattering length density	10 ⁻⁶ Å ⁻²	4
sld_solvent	Solvent scattering length density	10 ⁻⁶ Å ⁻²	1
theta	c axis to beam angle	degree	60
phi	rotation about beam	degree	60
psi	rotation about c axis	degree	60

$$b_{\text{Co}} = 2.490 \times 10^{-5} \text{ Å}$$

$$b_{\text{Fe}} = 9.450 \times 10^{-5} \text{ Å}$$

$$b_{\text{O}} = 5.803 \times 10^{-5} \text{ Å}$$

Unit cell volume ($V_{\text{u.c.}}$) of CoFe₂O₄ is $\sim 73.5 \text{ Å}^3$



Fit of the pure Nuclear scattering (10 K, 1.2 T)

Loading the 6 Column Data – Q, Intensity, Δ Intensity, Mean_Q, Δ Q, Shadow

Open SasView

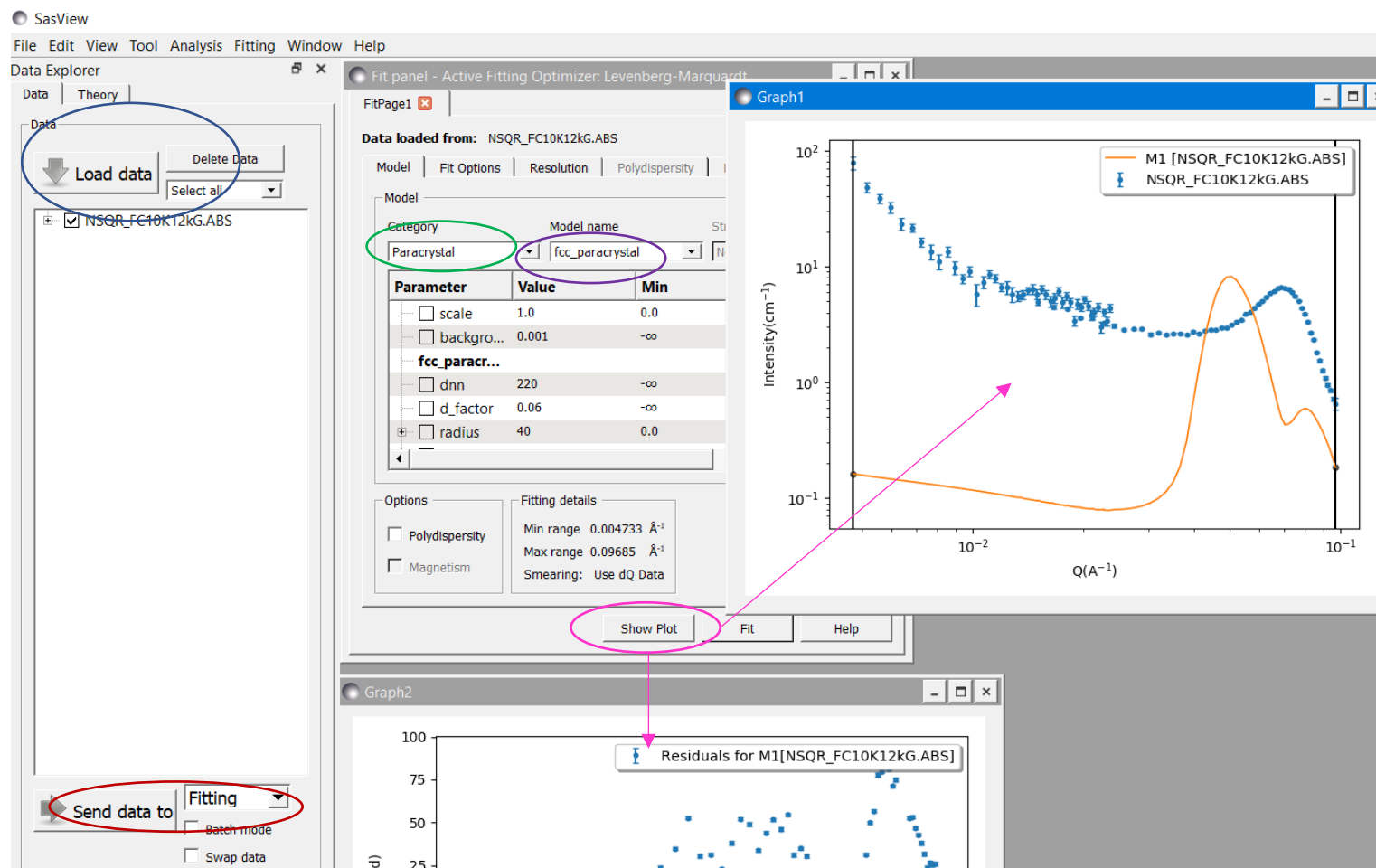
Load Data -> Choose Files ->
NSQR_FC10K12kG.ABS → Open

Send Data To → Fitting (you could also create a new plot at this point, but you don't need to)

In FitPage1 Tab, Model -> Category -> Paracrystal

Model Name → FFC Paracrystal

Show Plot. Two plots will pop up. You may want to drag the residuals away to the bottom for now.



Fit of the pure Nuclear scattering (10 K, 1.2 T)



Scale is set by volume fraction and sample thickness (which we do not know)

Background in this case is dominated by incoherent scattering from hydrogen

Dnn is the nearest-neighbor distance between nanoparticles

D_factor is the parameter associated to an isotropic distortion of the FCC lattice (larger widens Bragg peak)

Radius = 50 Å (fixed from TEM results)

SLD of CoFe₂O₄ = 6.07 (fixed)

SLD of "solvent" = 0 (air) (fixed)

Help at bottom describes the model in more detail

Before fitting the idea is to change the parameters within physically reasonable limits to get a near fit to the data (fitting before getting close often leads the model astray). Try:

Scale = 0.1 (constraint from 0 to 1)

BKGD = 0.5 (constraint from 0 to 0.5)

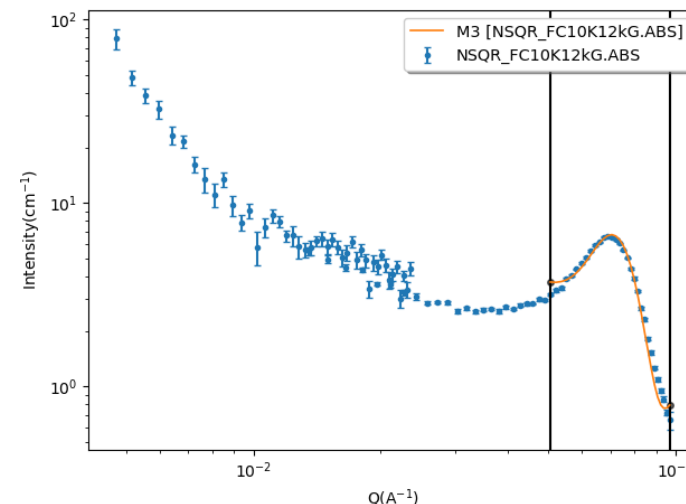
Dnn = 100 Å (constraint to 95 Å to 110 Å)

D_factor = 0.15 (constraint from 0 to 0.4)

Fitting range taken from 0.05 Å⁻¹ to 0.09685 Å⁻¹

Polydispersity can be added, but not needed (if so, please use 0.1). Note that polydispersity will slow down the fitting considerably.

The overshoot of the scattering at low-Q likely means D_factor is artificially large to cover some of the FCC stacking faults (a different form of disorder)



Results for the fitted parameters

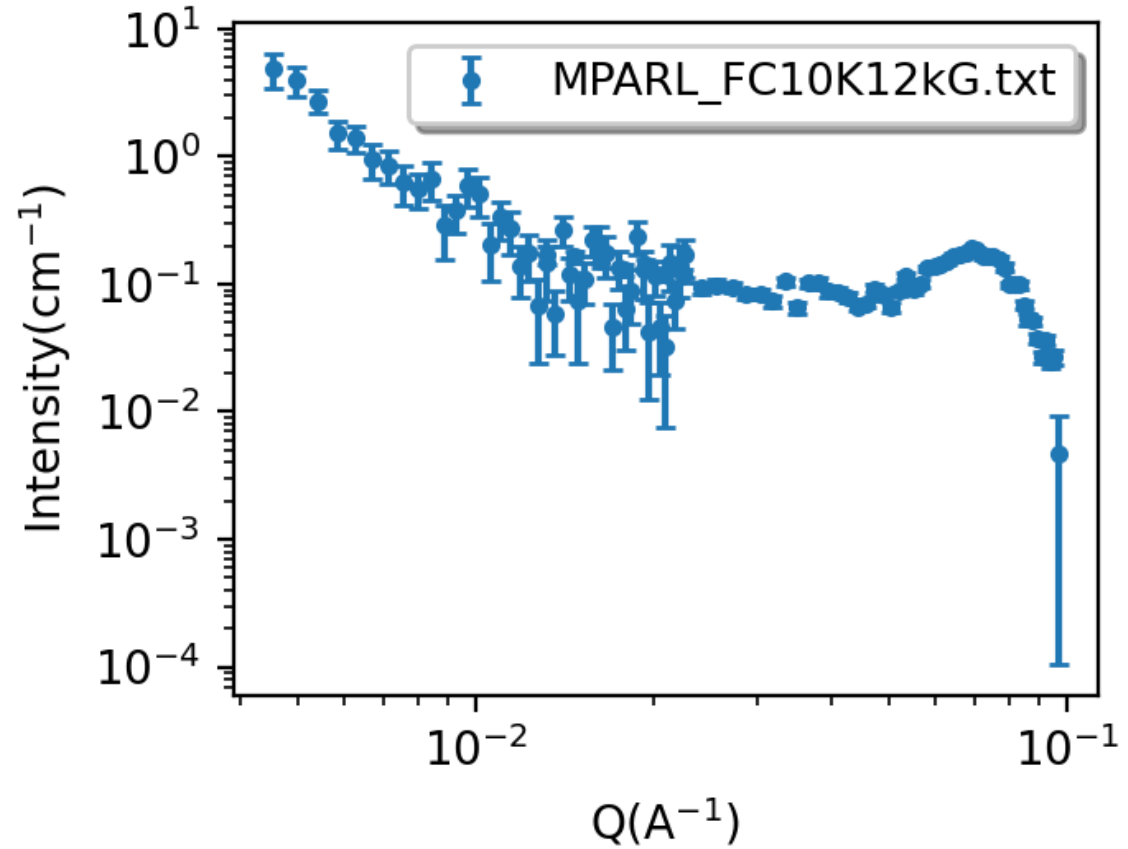
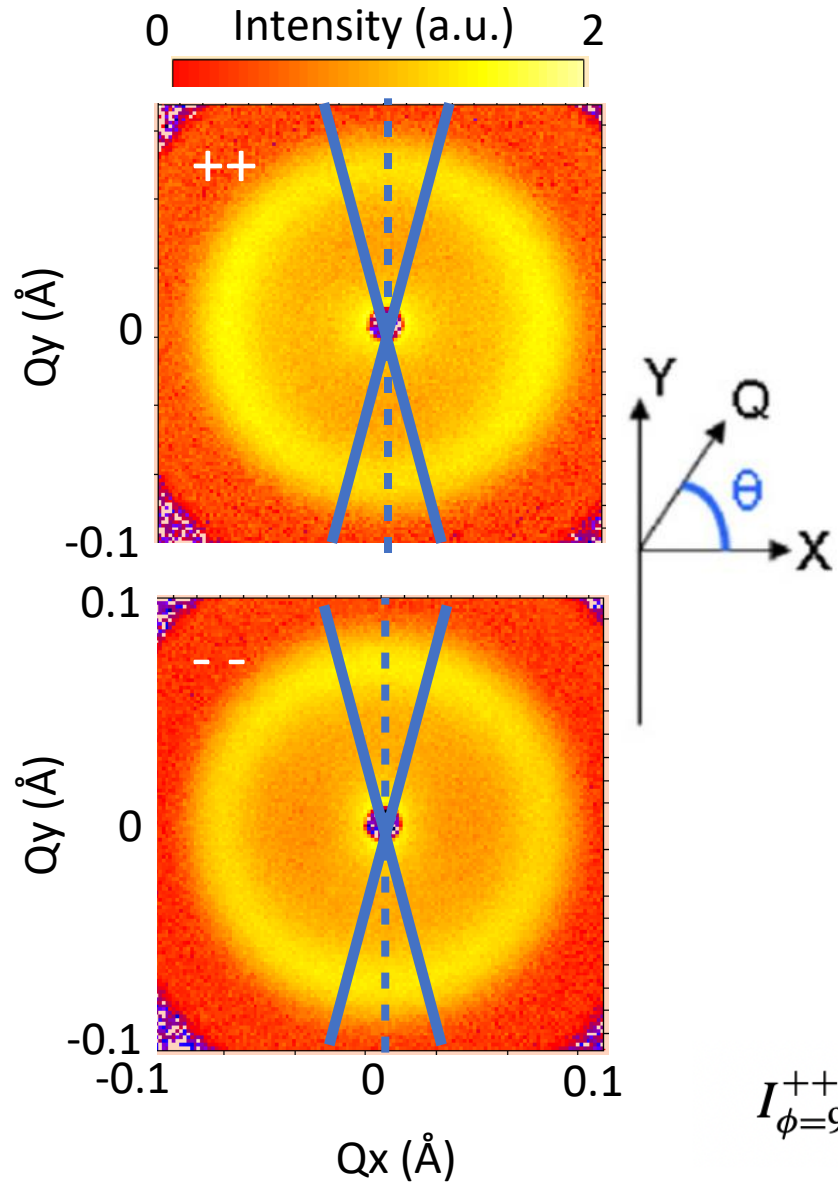
Scale ~ 0.10528

BKGD ~ 0.5

Dnn ~ 98.5

D_factor ~ 0.18

Extracting the scattering from magnetization parallel to the field (Mpar)



$$I_{\phi=90^\circ}^{++,--} = N^2 + M_X^2 \mp 2NM_X, \longrightarrow M_{\text{PARL}}^2(Q) = \frac{(I_{\theta=90^\circ}^{--} - I_{\theta=90^\circ}^{++})^2}{16N^2}$$

Load Data -> Choose Files ->
MPARL_FC10K12kG.txt. Open.

Check MPARL_FC10K12kG.txt only and
then Send Data To → Fitting. This
creates a fit page 2.

Set the parameters and Q-range to match
that of the structural scattering. Avoid
fitting the last Q-point.

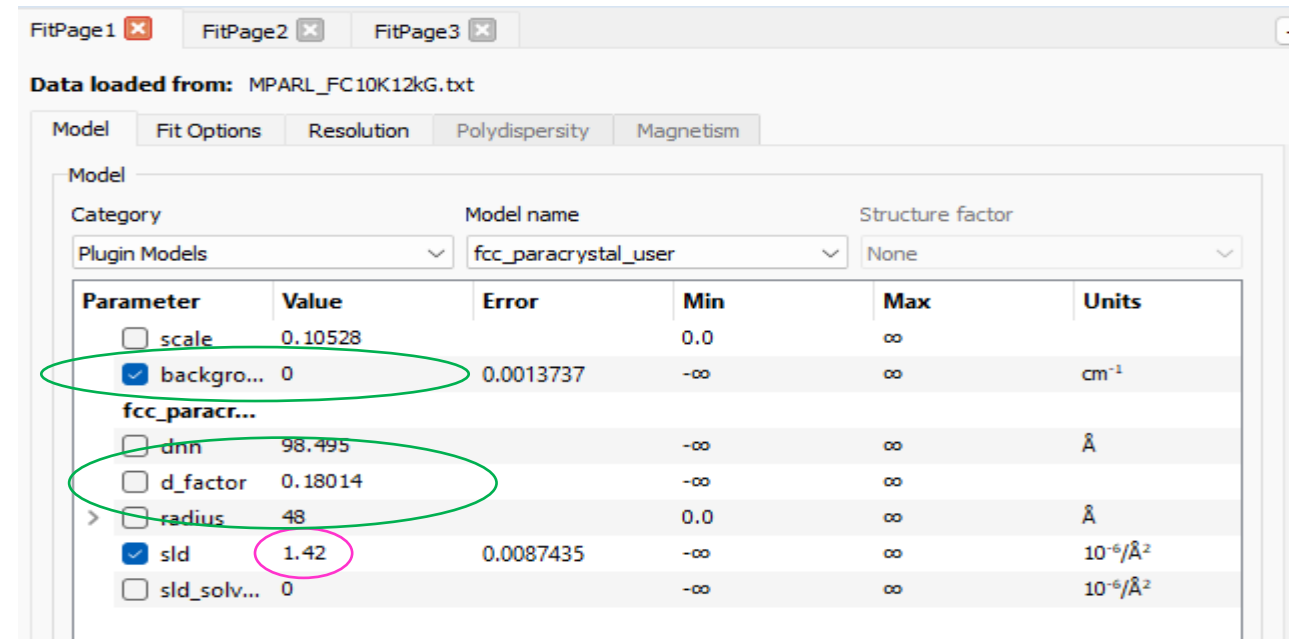
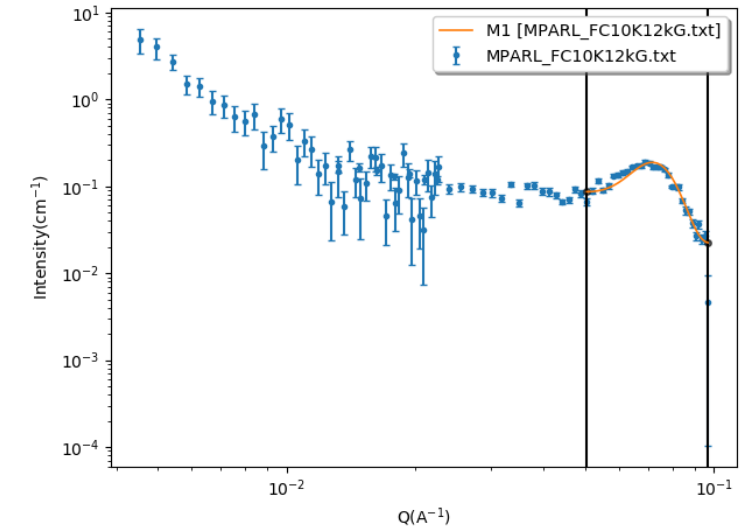
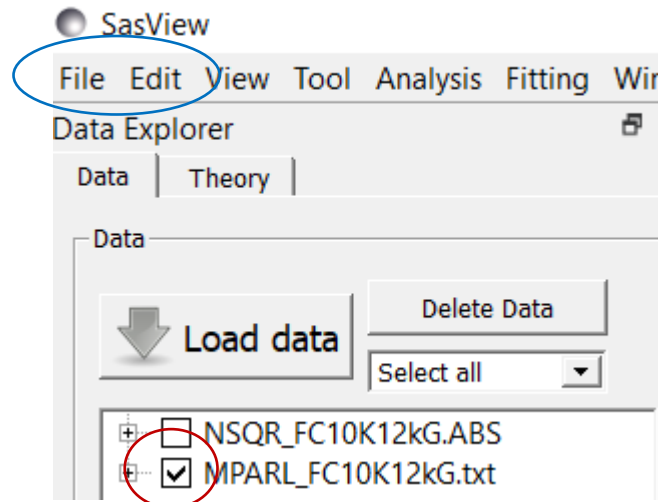
Background should be close to zero due to
difference taken (DD – UU). SLD 6.07 ->
1.42 (for max magnetism)

Now let the fit decide SLD

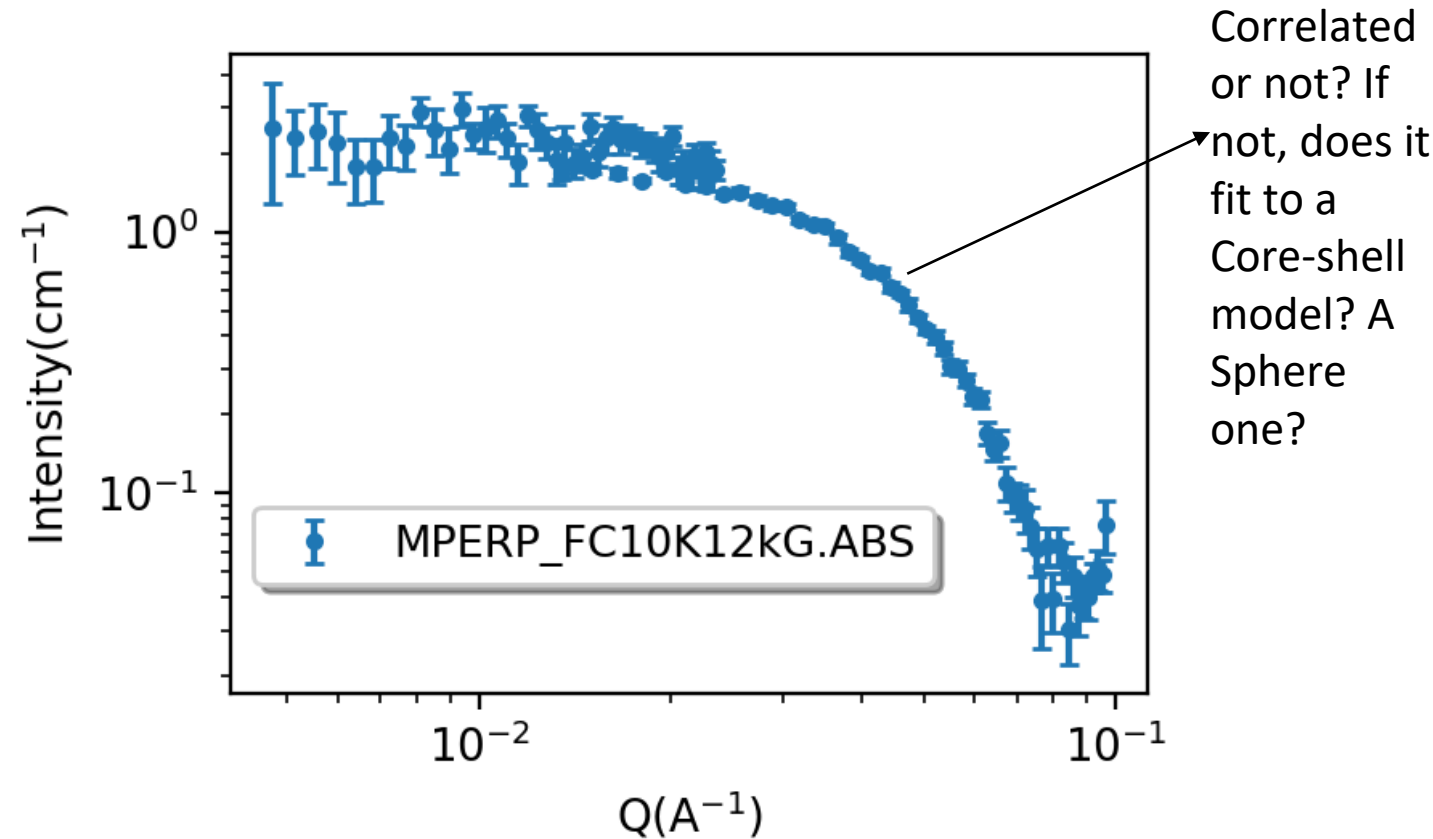
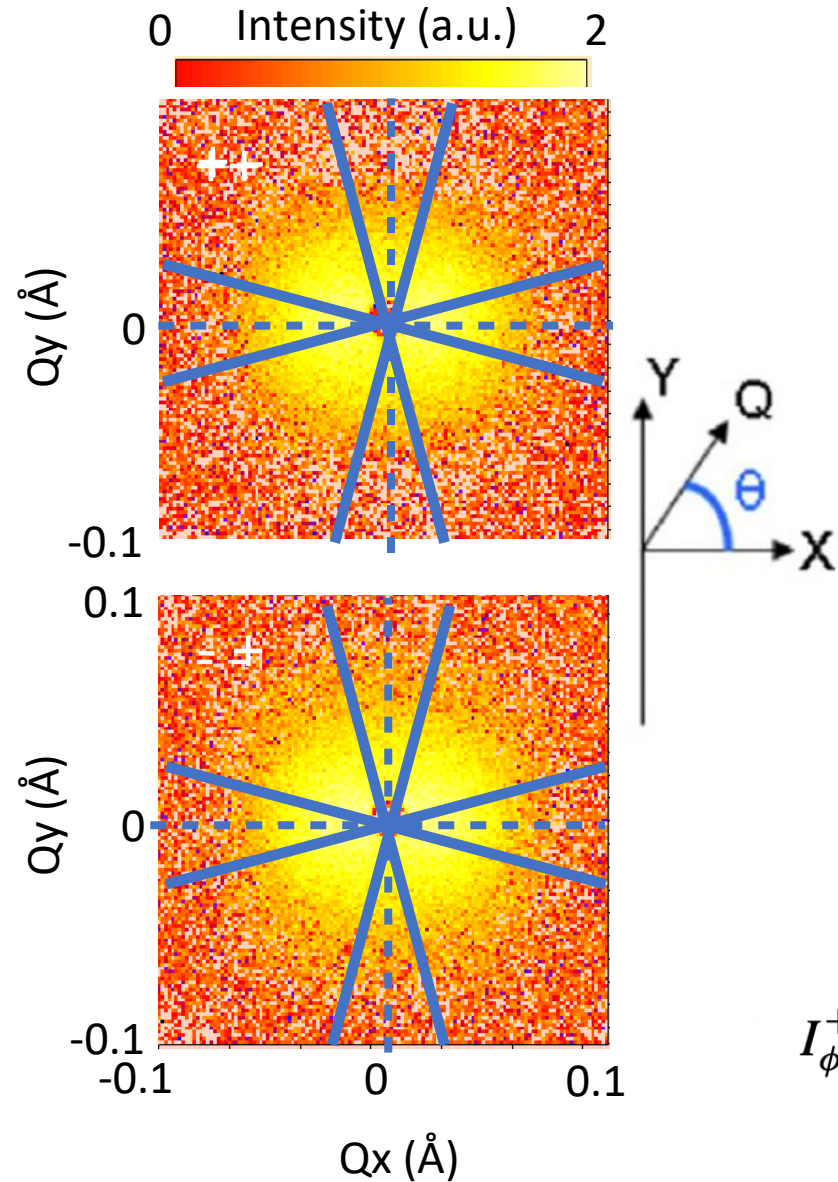
Fit result for SLD is ~ 0.94

$$b_{\text{mag}} = 2.655 \text{e-}5 \text{ \AA}/\mu_B * M(\mu_B) / V_u.c(\text{\AA}^3)$$

$M_{\text{CoFe}_2\text{O}_4}$ per f.u.= 3.94 μ_B



Extracting the scattering from magnetization perpendicular to the field (M_{perp})



$$I_{\phi=0^\circ}^{+-,-+} = M_Y^2 + M_Z^2 = 2M_{\text{PERP}}^2$$

$$I_{\phi=90^\circ}^{+-,-+} = M_Z^2 = M_{\text{PERP}}^2$$

$$M_{\text{PERP}}^2(Q) = \frac{1}{6}(I_{\theta=0^\circ, 90^\circ}^{+-} + I_{\theta=0^\circ, 90^\circ}^{-+})$$

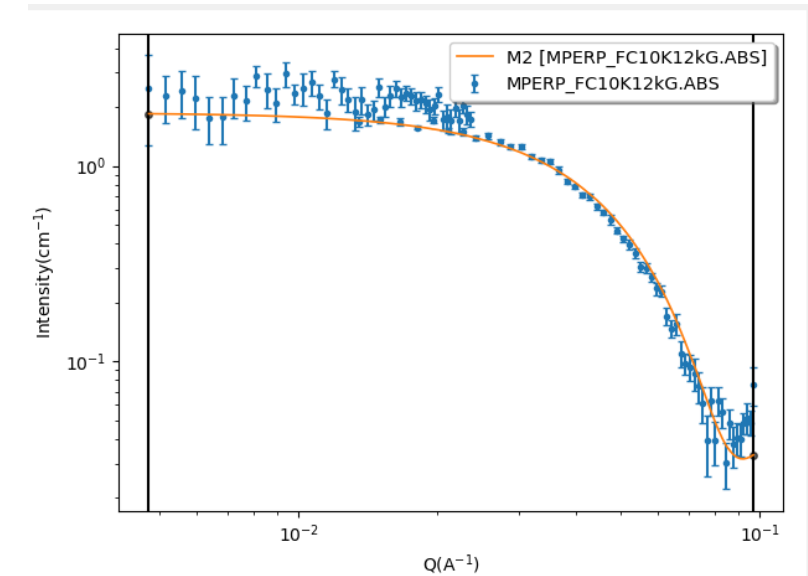
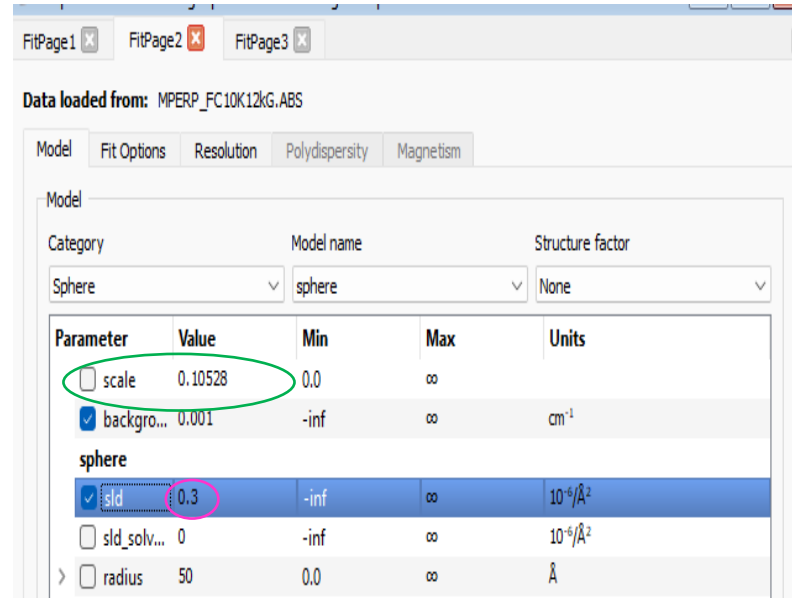
Load Data -> Choose Files ->
MPERP_FC10K12kG.ABS -> Open.

Check MPERP_FC10K12kG.ABS only
and then Send Data To → Fitting.
This creates a fit page 3.

Choose Category = Sphere, Model
Name = Sphere. Set the same scale as
obtained from the Nuclear fit.

Now fit the SLD, background and
radius.

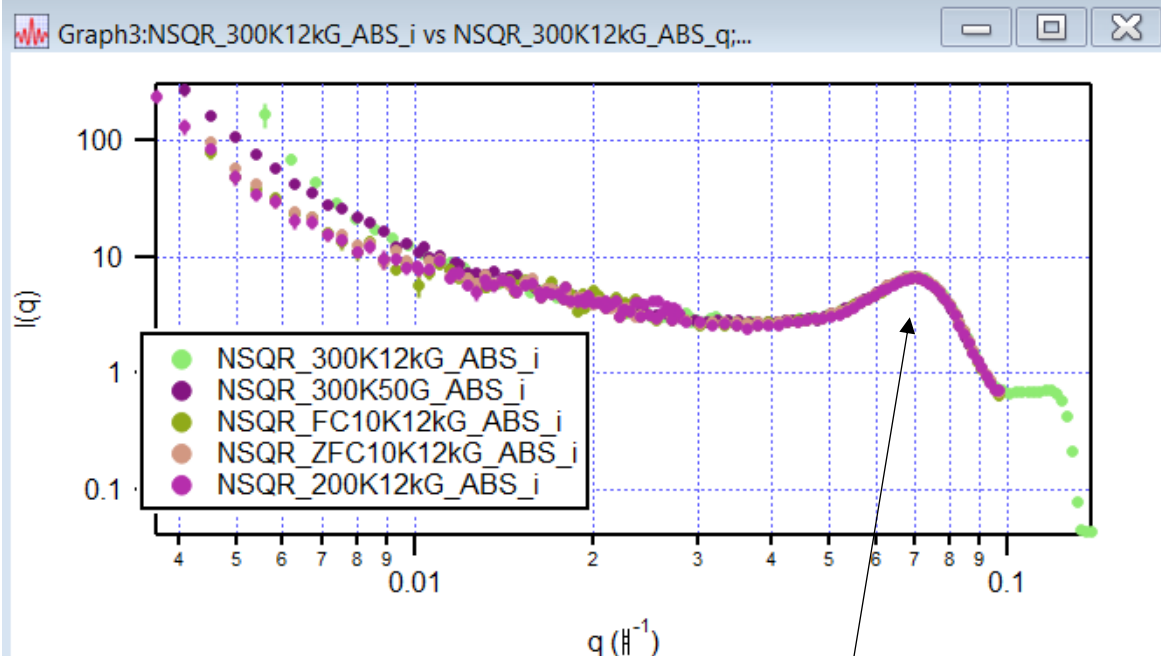
Fit result for SLD is ~ 0.577



Now $(0.577 + 0.94)/1.42 \sim 107\%$ so all the magnetization is accounted for.

$$\theta \equiv \tan^{-1} \left(\frac{m_{\text{PERP}}}{m_{\text{PARL}}} \right) \Rightarrow 31.5^\circ \text{ canting angle}$$

An overview of conditions to analyze:
 10 K, 1.2 T Field-cooled and zero field-cooled
 200 K, 1.2 T
 300 K, 1.2 T
 300 K, 0.005 T



Except at low-Q where long-range magnetism differs, the Bragg peak remains constant and need only be fit once

