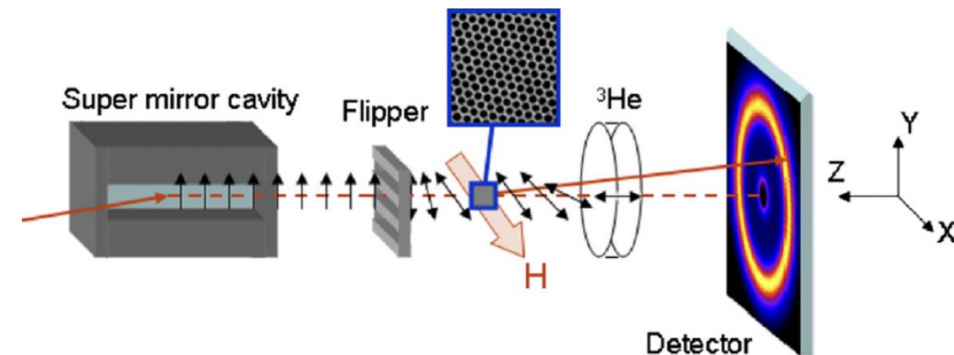
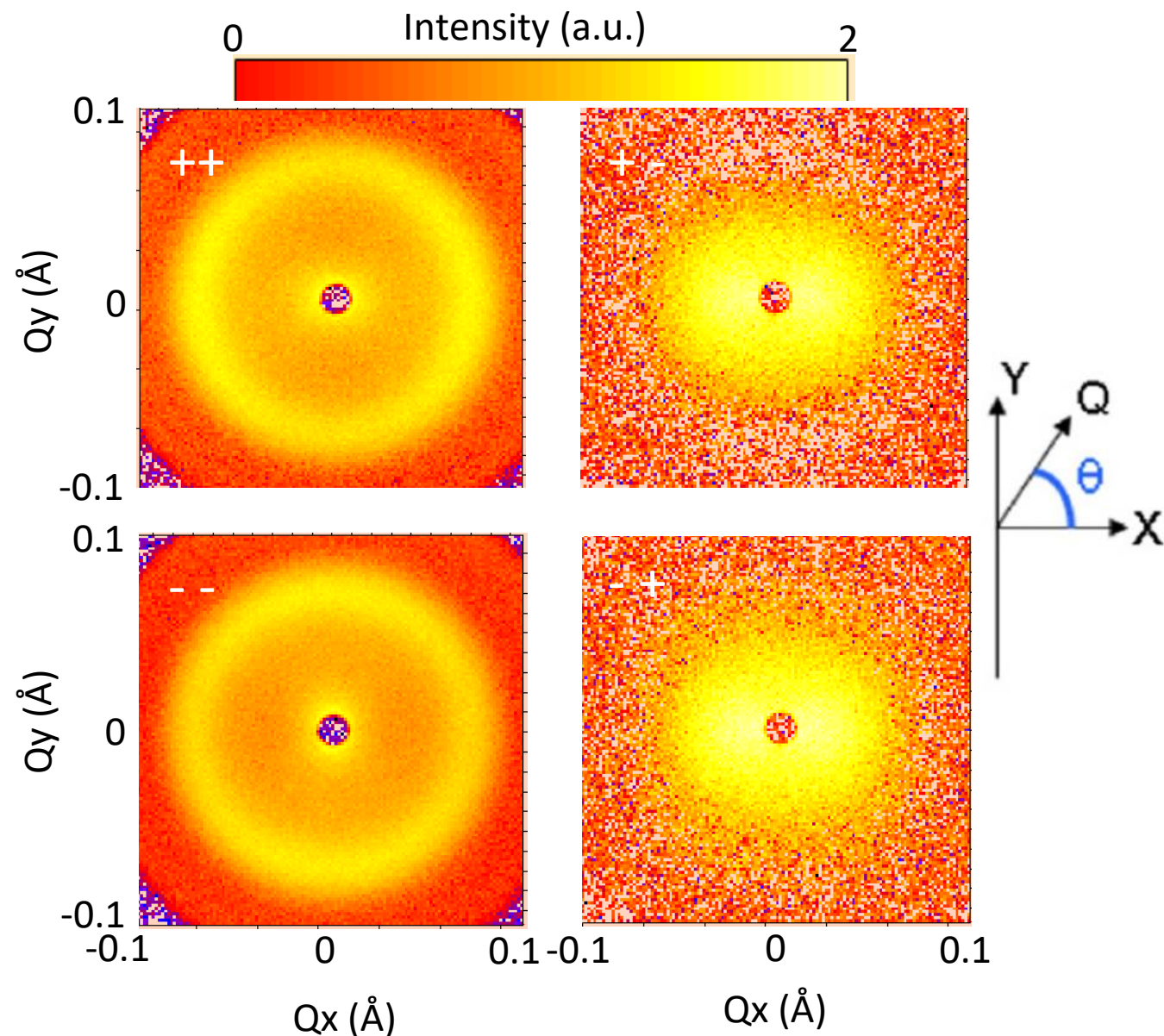


## Guide to SasView fitting for the 10 K, 1.2 T $\text{CoFe}_2\text{O}_4$ Nanoparticles

# Full Pol. data of $\text{CoFe}_2\text{O}_4$ collected at 10 K and 1.2 T

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



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

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

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

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

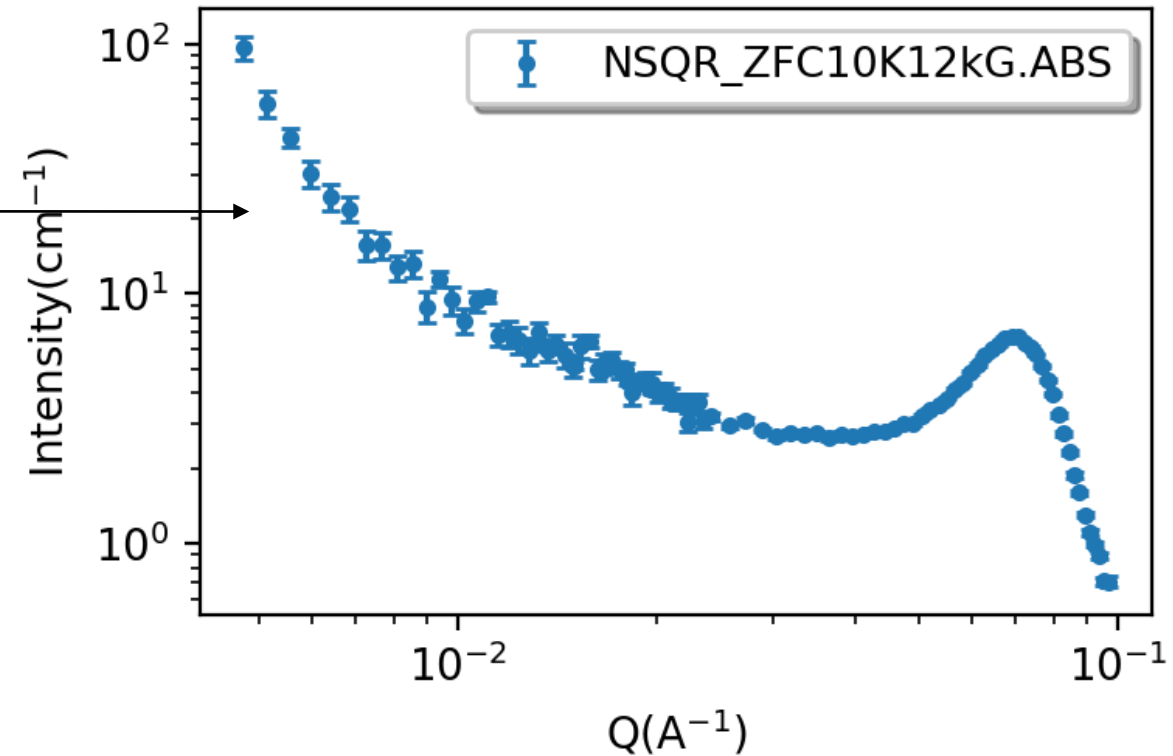
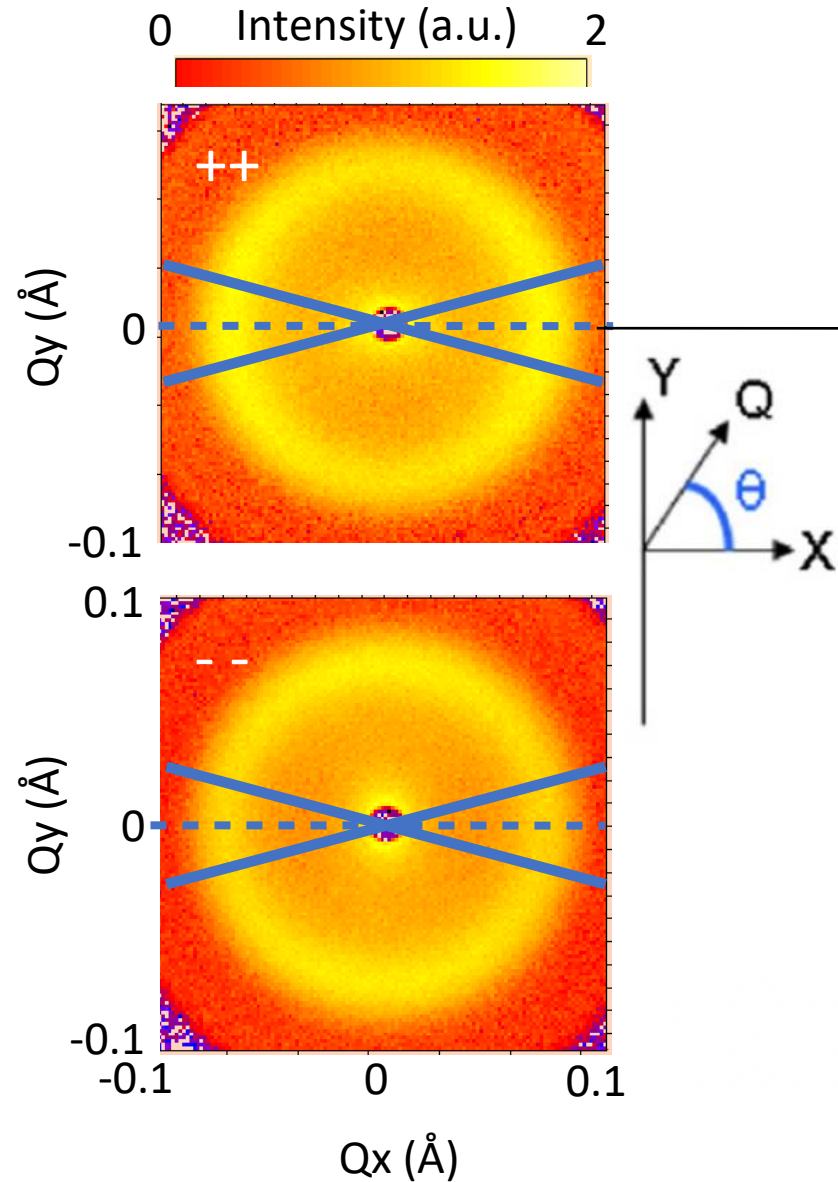
\*Assuming isotropic system with only the X (field) direction being unique

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

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

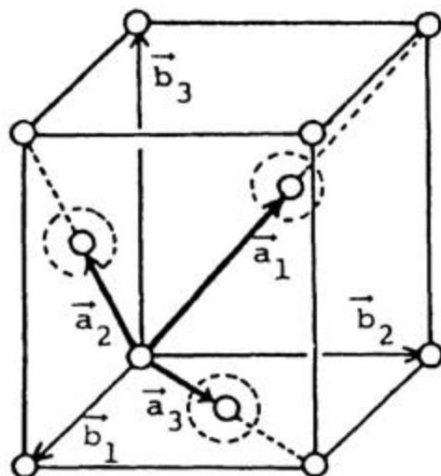
\*note that the data have been corrected for spin leakage, background/empty cell contribution, detector efficiency, etc...

# Extracting the pure nuclear scattering of $\text{CoFe}_2\text{O}_4$ 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,  $V_{\text{lattice}}$  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.

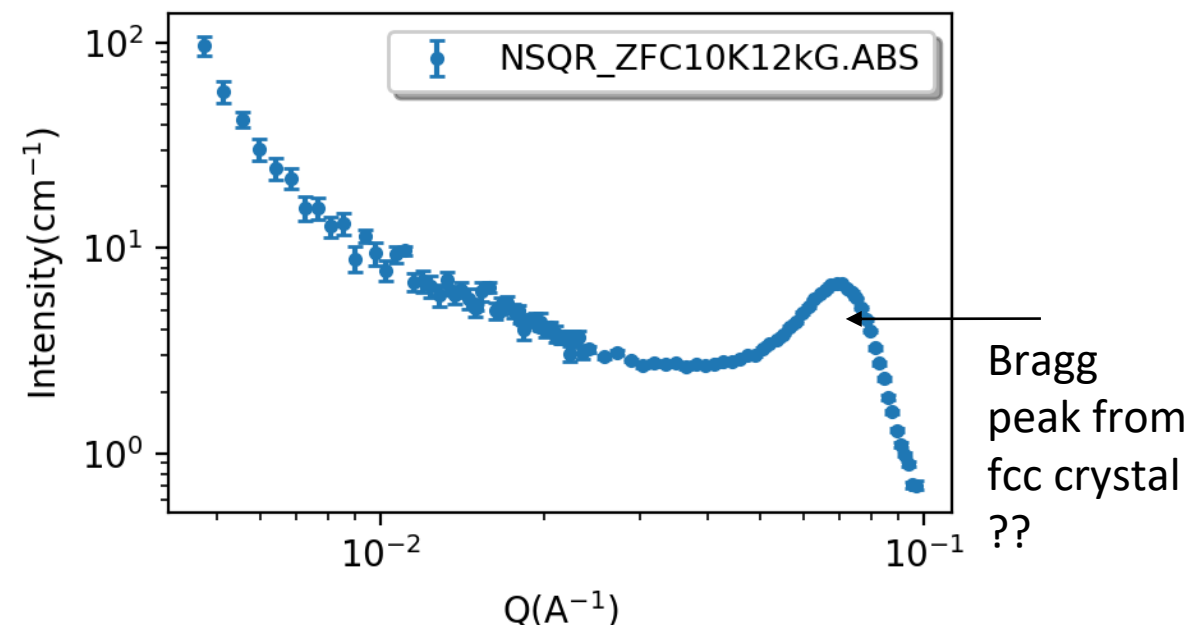
Parameter	Description	Units	Default value
scale	Scale factor or Volume fraction	None	1
background	Source background	cm <sup>-1</sup>	0.001
dnn	Nearest neighbour distance	Å	220
d_factor	Paracrystal distortion factor	None	0.06
radius	Particle radius	Å	40
sld	Particle scattering length density	10 <sup>-6</sup> Å <sup>-2</sup>	4
sld_solvent	Solvent scattering length density	10 <sup>-6</sup> Å <sup>-2</sup>	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 CoFe<sub>2</sub>O<sub>4</sub> 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,  $\Delta$ Intensity, Mean\_Q,  $\Delta$ Q, Shadow

Open SasView

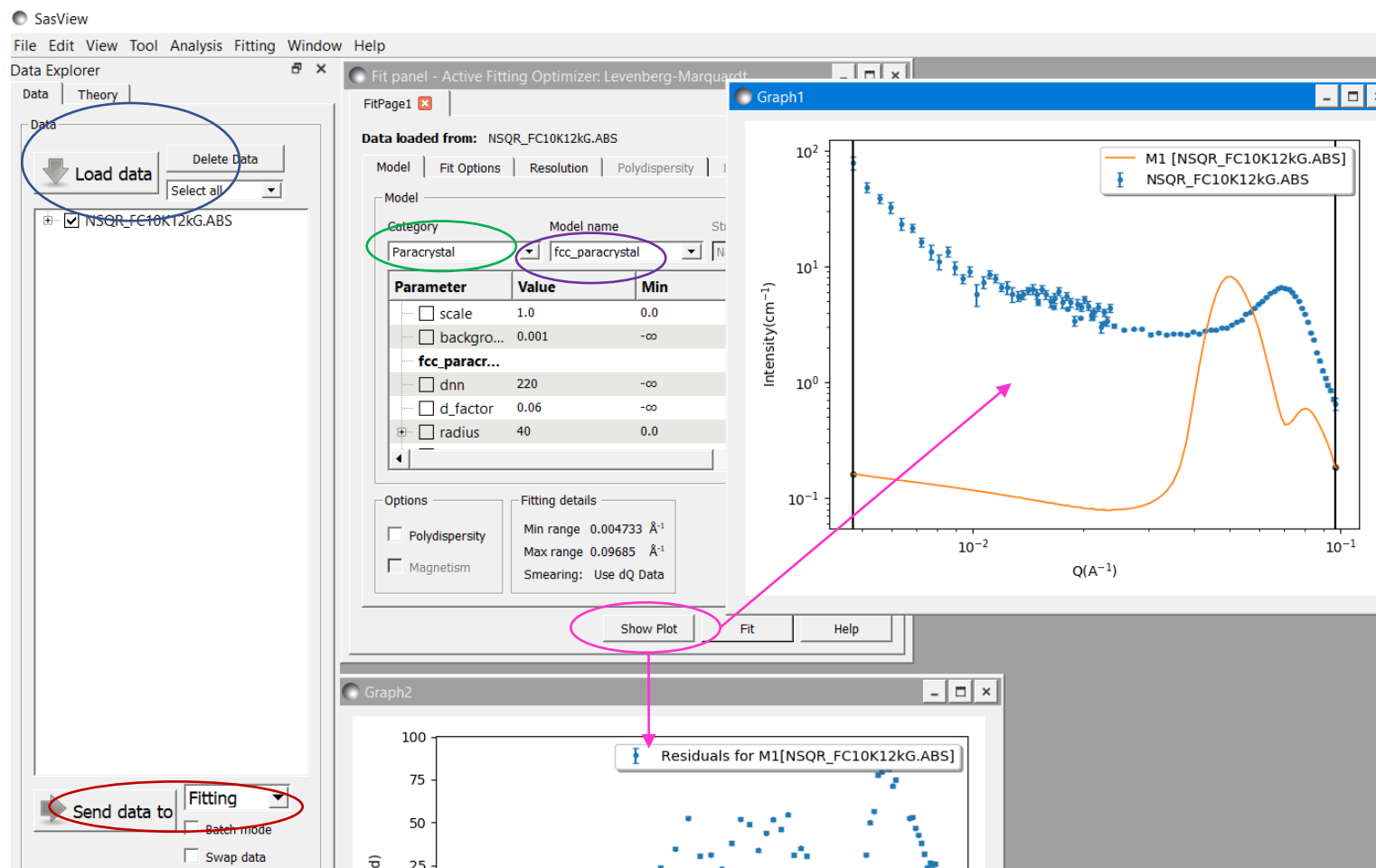
Load Data -> Choose Files ->  
NSQR\_FC10K12kG.ABS → Open

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 CoFe<sub>2</sub>O<sub>4</sub> = 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)

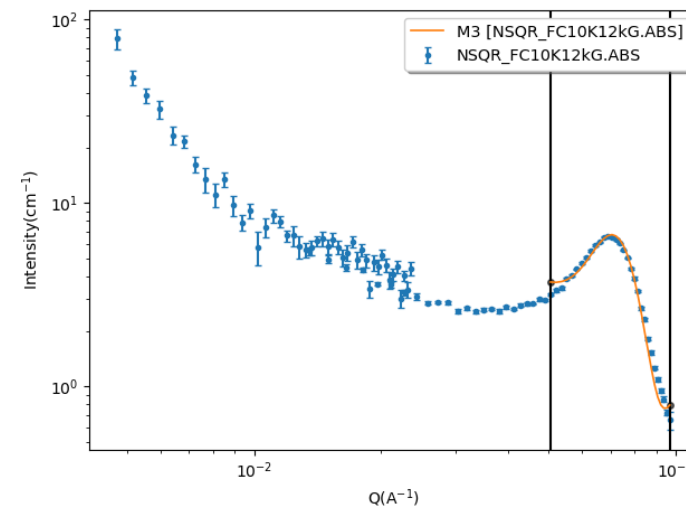
Dnn = 100 Å (constraint to 95 Å to 110 Å)

D\_factor = 0.15 (constraint from 0 to 0.4)

Fitting range taken from 0.05 Å<sup>-1</sup> to 0.09685 Å<sup>-1</sup>

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

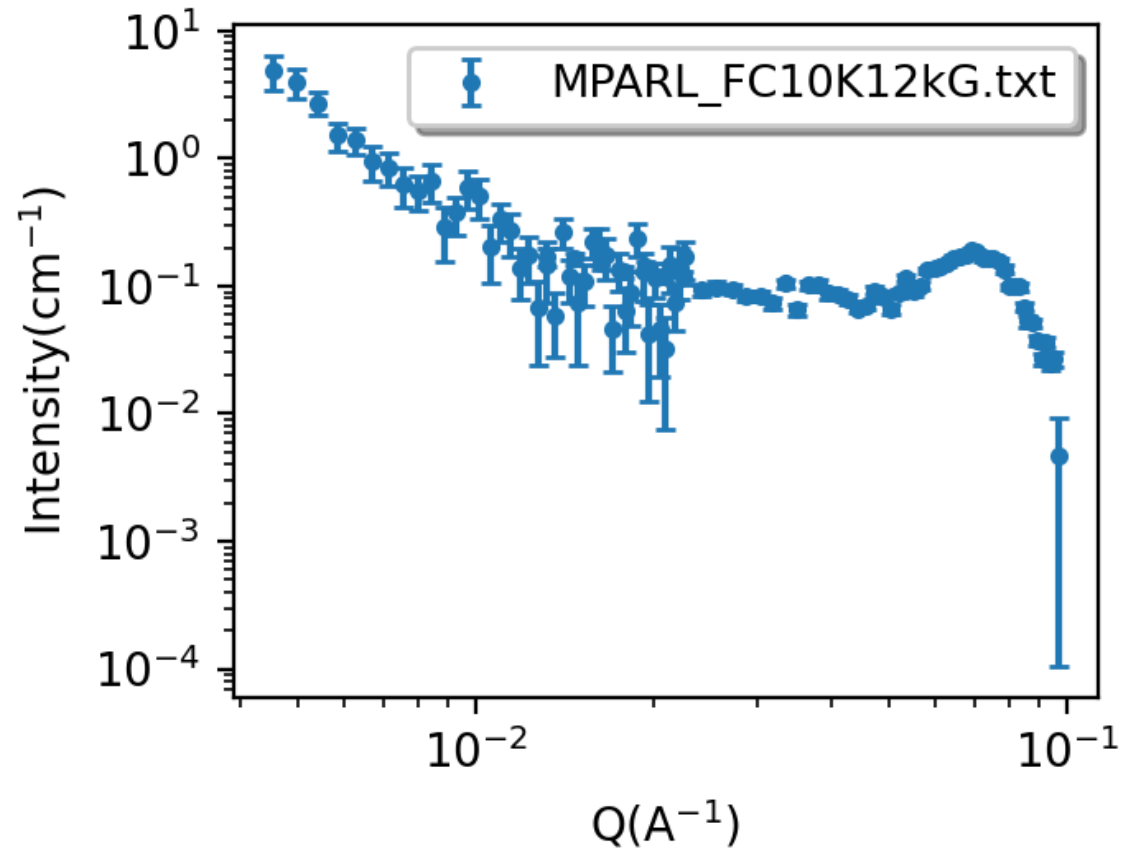
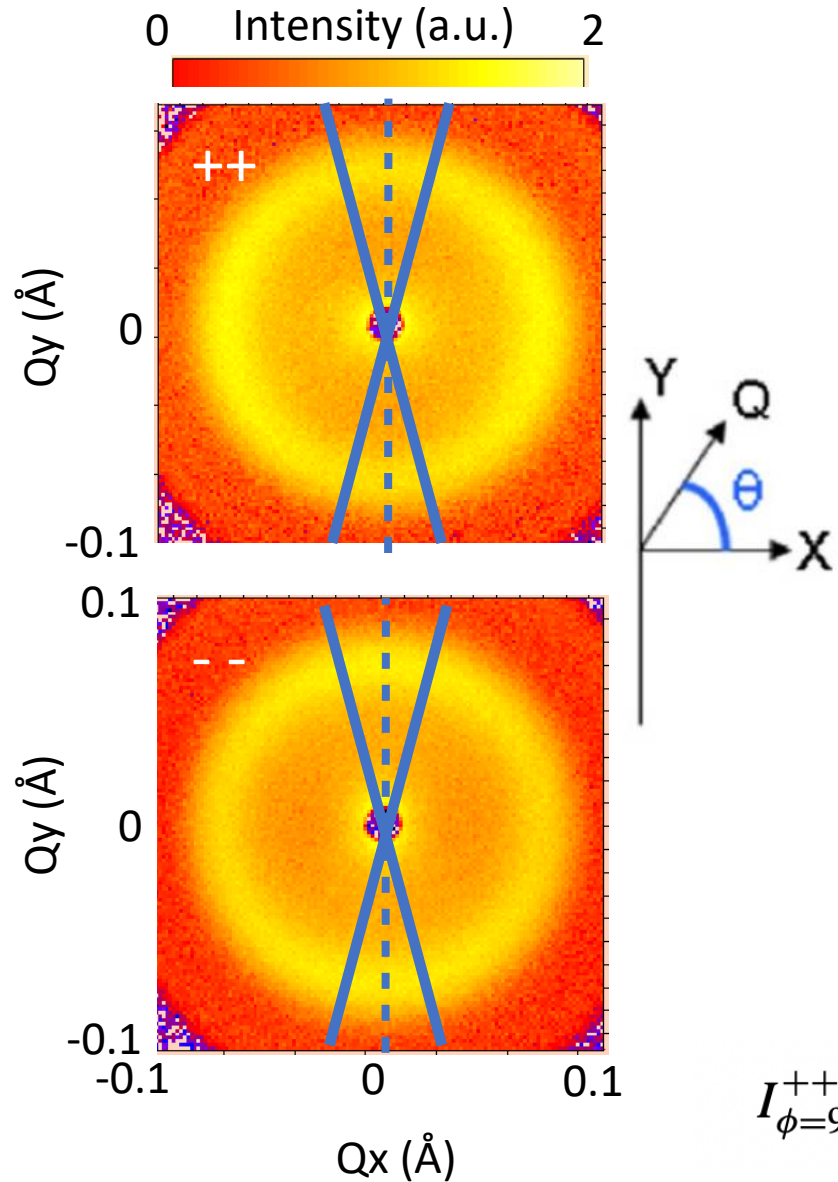
Scale ~ 0.10528

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.

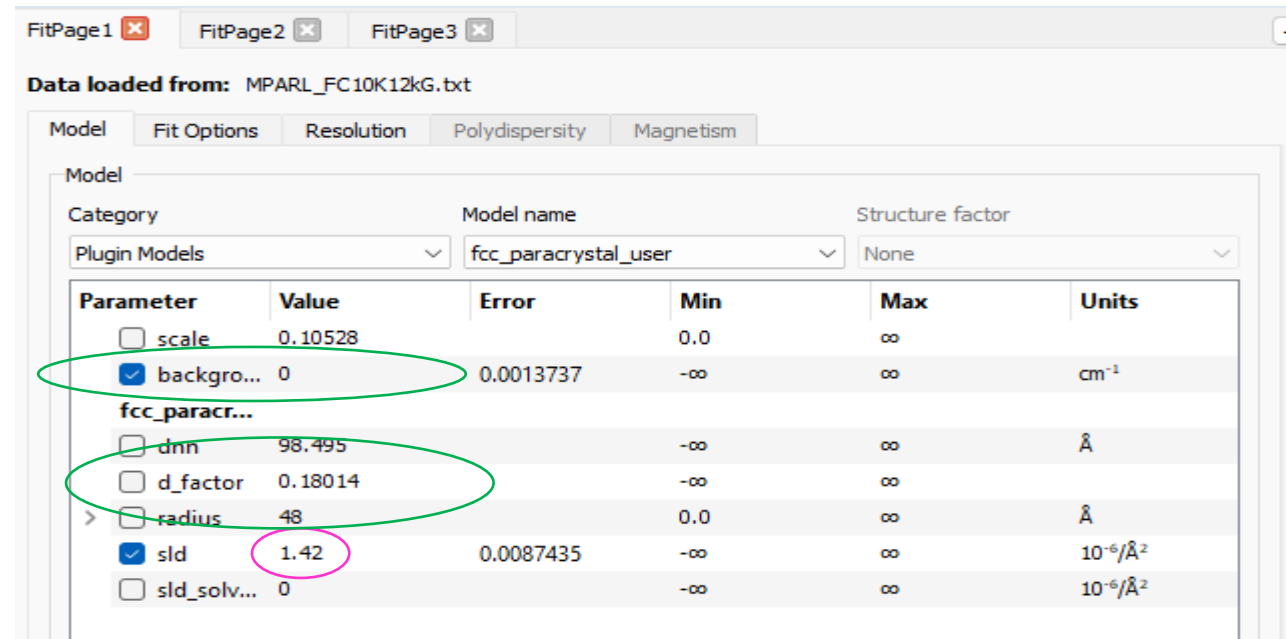
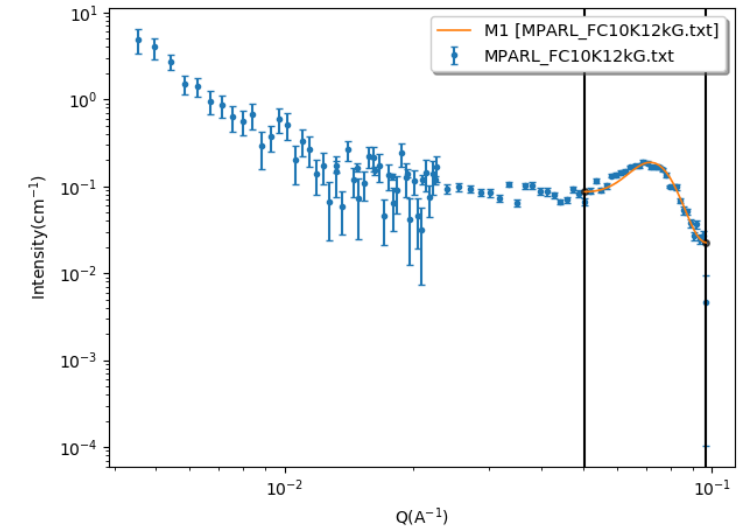
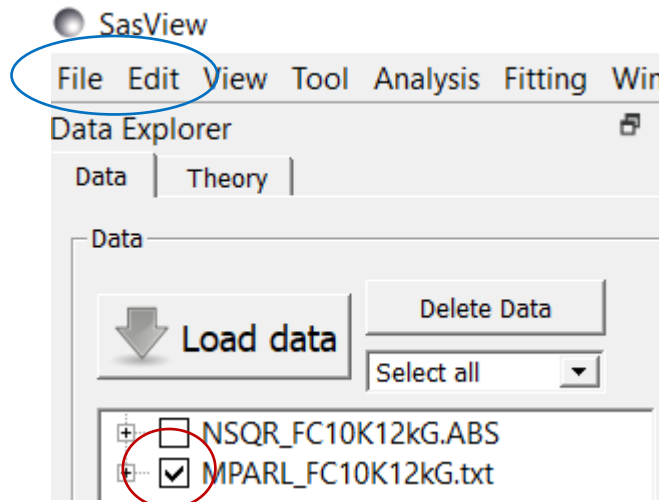
Background should be close to zero due to  
difference taken (DD – UU). SLD 6.07 ->  
1.42 (for max magnetism)

Now let the fit decide SLD

Fit result for SLD is ~ 0.94

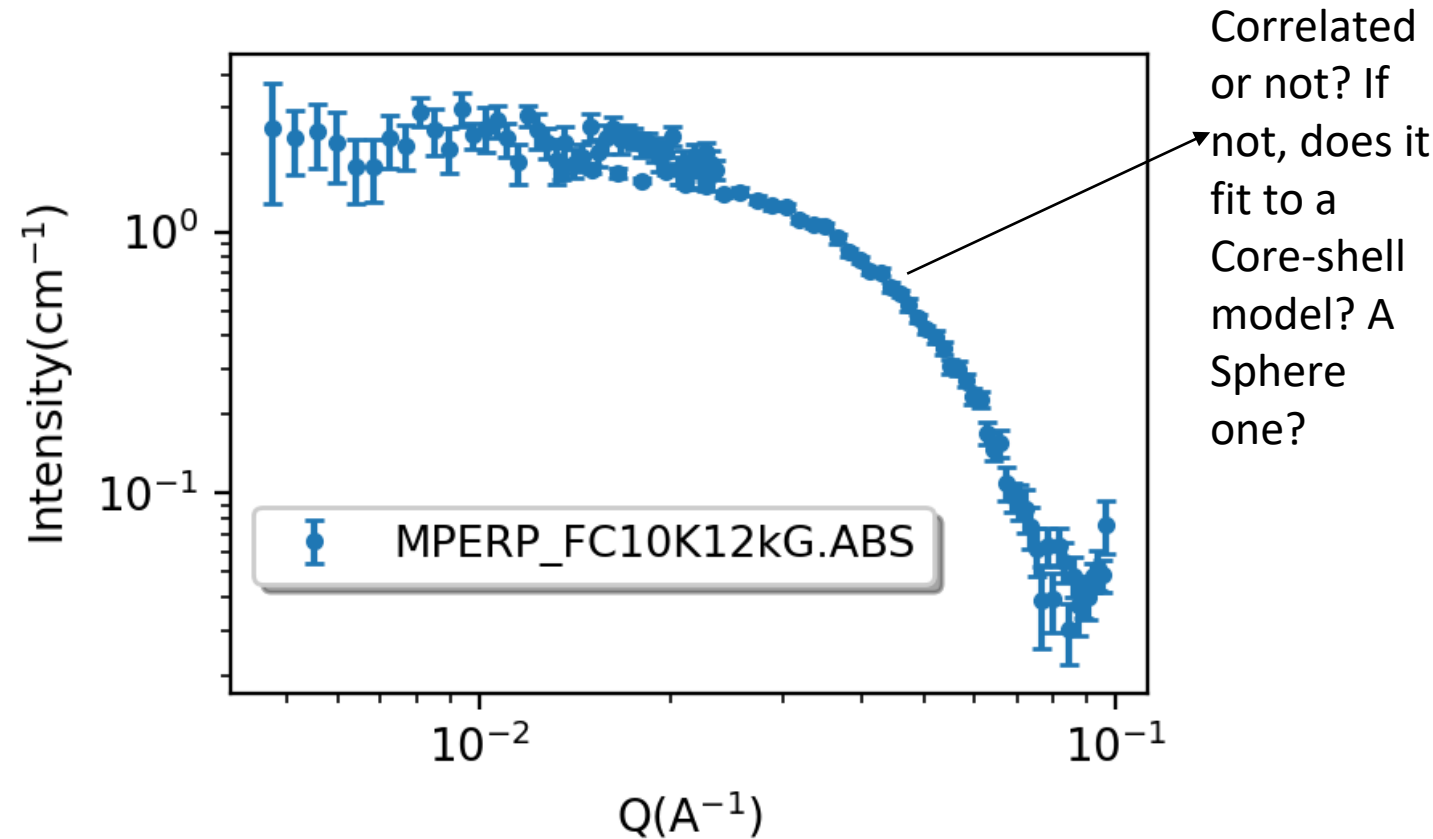
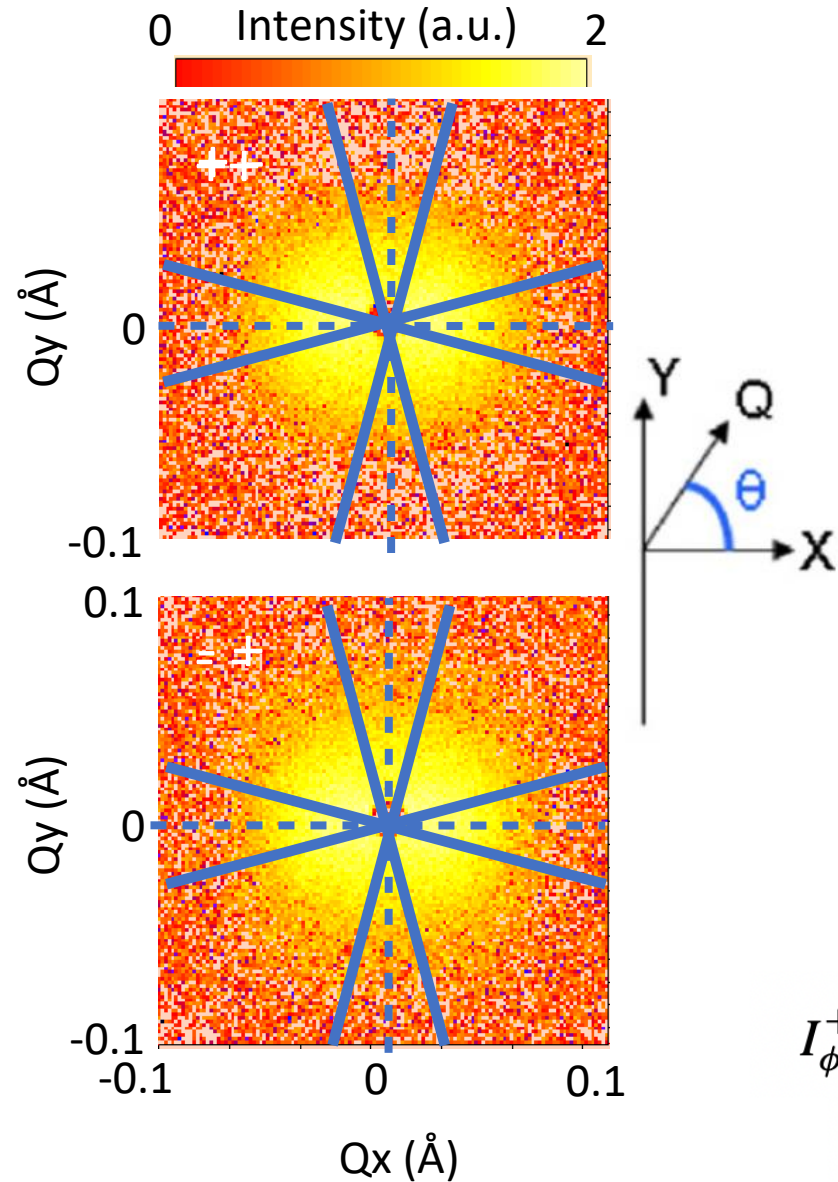
$$b_{\text{mag}} = 2.655 \times 10^{-5} \text{ Å}/\mu_B * M(\mu_B) / V_u.c(\text{Å}^3)$$

$M_{\text{CoFe2O4}}$  per f.u.= 3.94  $\mu_B$





# Extracting the scattering from magnetization perpendicular to the field ( $M_{\text{perp}}$ )



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

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

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

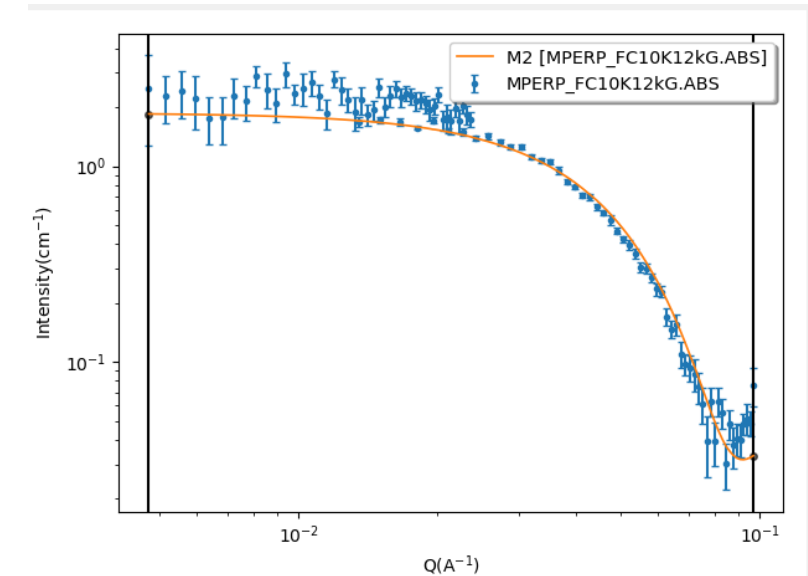
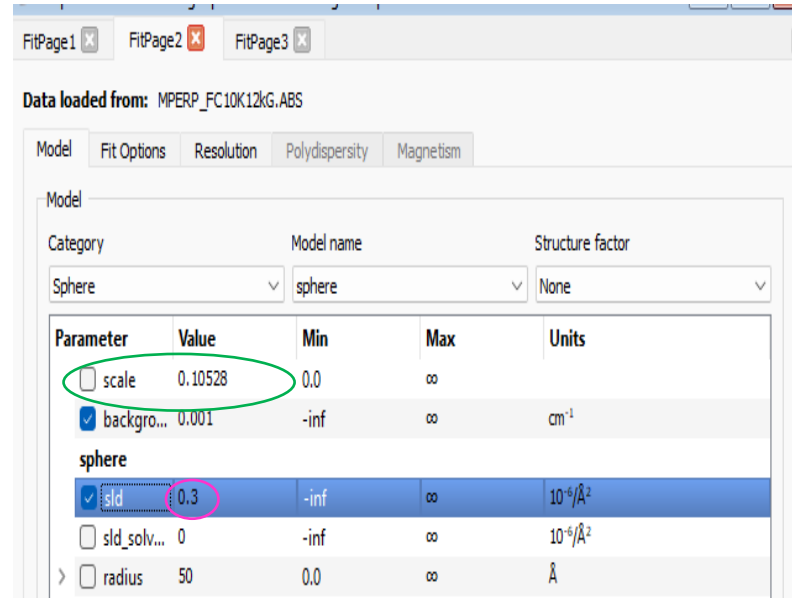
Load Data -> Choose Files ->  
MPERP\_FC10K12kG.ABS -> Open.

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.

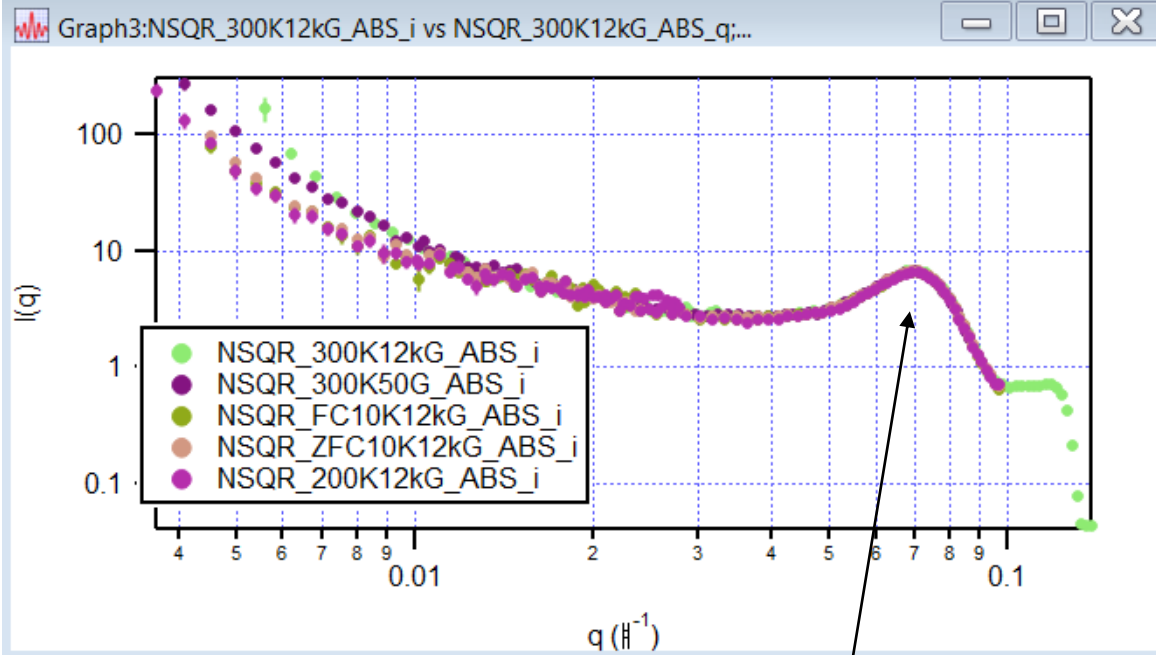
Fit result for SLD is ~ 0.577



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

$$\theta = \tan^{-1} \left( \sqrt{\frac{2M_{\text{PERP}}^2}{M_{\text{PARL}}^2}} \right) \Rightarrow 48^\circ \text{ canting angle}$$

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

