21.12.2015

ISIS run December 2015 – Data analysis

D_2O density:

I have two values: $0,935 \, g/cm^3$ ← from various papers

$0,094 \, \frac{atoms}{Å^3}$ (i.e. $\frac{molecules}{Å^3}$ i assume)

→ entered in Gudrun at ISIS by someone else ↑ No

conversion: molar weight of D_2O: $20,0276 \, \frac{g}{mol}$

Avogadro constant: $1 \, mol = 6,0221 \cdot 10^{23}$ molecules

lengths: $1 \, cm = 10^8 \, Å$

$$1 \, g/cm^3 \approx \frac{6 \cdot 10^{23} \, molecules}{mol} \cdot \frac{1 \, mol}{20 \, g} \cdot \left(\frac{1 \, cm}{10^8 \, Å}\right)^3 \cdot \frac{g}{cm^3}$$

$$= \frac{6 \cdot 10^{23}}{20} \cdot \frac{1}{10^{24}} \, \frac{molecules}{Å^3}$$

$$= \frac{3}{100} \, \frac{molecules}{Å^3} = 0,03 \, \frac{molecules}{Å^3}$$

22.12.2015

Maybe atoms means atoms. 1 molecule = 3 atoms

$$\rightarrow 1 \, g/cm^3 = \frac{6,0221}{2,00276} \cdot 10^{-2} \cdot 3 \, \frac{atoms}{Å^3} = 9,02288 \cdot 10^{-2} \, \frac{atoms}{Å^3}$$

$$\rightarrow 0,935 \, g/cm^3 = 0,0844 \, \frac{atoms}{Å^3}$$

$$\rightarrow \boxed{0,094 \, \frac{atoms}{Å^3} = 1,042 \, \frac{g}{cm^3}}$$ → Makes sense?

must be pure bulk density ignoring any possible empty spaces.

I just realised that Catherine was doing this conversion in reverse direction in her python script → take both conversion out.

✓ → check units! (m → cm)

Porod analysis:

NIST summerschool SANS : (internet)

$$\frac{\pi}{Q^*} \cdot \lim_{q \to \infty} \left(I(q) \cdot q^4 \right) = \frac{S}{V}$$

Q^* = scattering invariant $= 2\pi^2 \phi_1 (1-\phi_1)(\rho_2 - \rho_1)^2$
for an incompressible two-phase system with scattering length densities ρ_1, ρ_2

$\frac{S}{V}$ = specific surface area

what is ϕ_1?

Catherine's PhD thesis:

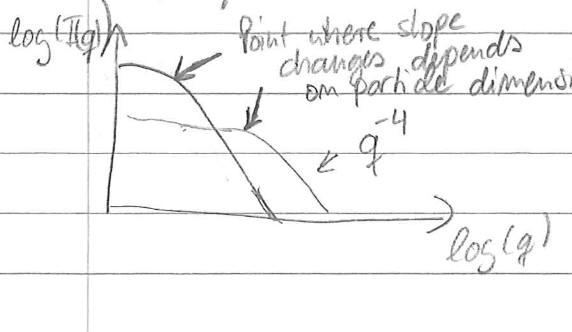
$$\Sigma_s = \frac{\lim_{Q \to \infty} (I(Q) \cdot Q^4)}{2\pi \Delta\rho^2} = \frac{K}{2\pi \Delta\rho^2} \qquad Q \hat{=} q$$

Σ_s = specific surface area $\left(= \frac{S}{V}\right)$
$\Delta\rho$ = scattering length density difference between solid and gap ("pore") $(= \rho_2 - \rho_1)$
K = Porod constant

Question: This time we had the sample under low pressure He, not vacuum. → How does that influence the scattering length density? → not all background scans

---

D size of scattering object. $q \gg 1/D$: $I(q) \sim q^{-4}$
(for any system with sharp, smooth surfaces.)



(Glatter & Kratky pp. 30-1)

from OSNS 2013, Edler

fractal systems:

for a "mass fractal" the number of particles within a sphere of radius R ist proportional to R^D, D = fractal dimension

this changes the slope in the Porod region

form factor: $F(Q) = \frac{const}{Q^{6-D}}$

(form factor for spheres: $\frac{const}{Q^4}$)

slope for spheres: Q^{-4}, rods: Q^{-2}, discs: Q^{-1}

my plots:

for $Q < 0{,}08$ Å, $I(Q) \sim Q^{-4}$

$\rightarrow$ $I(Q) \cdot Q^4 = const$

Problem: Catherine's fitroutine does not work for my data.

fix 1: change selected of range to [0,012, 0,1]

$\rightarrow$ now I get a linear region (which is not found by python)

$\rightarrow$ how to find it?

Idea: try to fit constant to data and look for the region (10 to 15 datapoints) with the least deviation from that curve. Fit range: best between 0,013 and 0,05 $Å^{-1}$

$\rightarrow$ 20 datapoints

$\rightarrow$ works!

04.03.2016: I(q) as defined here is wrong!
for corrected version see 4.3.16

unit conversion:

DCS (from Gudrun): differential cross section:

$$\frac{d\sigma}{d\Omega}(\lambda, 2\theta) \qquad \text{unit}: \frac{\text{barns}}{\text{sr}\cdot\text{atom}}$$

$$1\,\text{barn} = 10^{-28}\,m^2$$

Intensity: $I(q) = DCS(q) \cdot \rho_m \cdot \text{tweak} \cdot 3 \cdot 6{,}0221 \cdot 10^{23}$ atoms/molecule, molecules/mol

ρ_m: (g/cm^3)

$$\cdot \cancel{10^{-24}} \cdot \frac{1\,\text{mol}}{20\,g}$$

$cm^3 \to Å^3$

$$\approx DCS(q) \cdot 1\frac{g}{cm^3} \cdot \text{tweak} \cdot \frac{3.6}{20} \frac{\text{atoms}}{g} \cdot 10^{23}$$

$$= DCS(q) \cdot \text{tweak} \cdot 9 \cdot 10^{22} \frac{\text{atoms}}{cm^3}$$

@ $Q = 2 \cdot 10^{-2}\,Å^{-1}$

$$DCS \approx 100 \frac{\text{barn}}{\text{sr atom}}, \quad \text{tweak} \approx 9$$

$$\to I(q) \approx 10^2 \cdot 80 \cdot 10^{22-28} \frac{m^2}{cm^3}$$

$$= 80 \cdot 10^{-4} \frac{m^2}{cm^3} = 8 \cdot 10^{-3} \frac{m^2}{cm^3}$$

Specific Surface Area: $\dfrac{\lim_{Q\to\infty} I(Q) \cdot Q^4}{2\pi\,\Delta\rho^2} = \Sigma_S$

scattering length density: $\Delta\rho = 5{,}4 \cdot 10^{-6}\,Å^{-2}$

$$\to \Sigma_S = \frac{80 \cdot 10^{-4} \frac{m^2}{cm^3} \cdot (2 \cdot 10^{-2}\,Å^{-1})^4}{2\pi \cdot (5{,}4 \cdot 10^{-6}\,Å^{-2})^2}$$

$$\approx \frac{80 \cdot 10^{-4} \cdot 16 \cdot 10^{-8}}{2 \cdot 3{,}1 \cdot 30 \cdot 10^{-12}} \frac{m^2}{cm^3} \approx 7 \cdot 10^{-4-8+12} \frac{m^2}{cm^3}$$

$$= 7 \cdot 10^{0} \frac{m^2}{cm^3} = 7 \frac{m^2}{cm^3}$$

When density is given in $\frac{\text{atoms}}{Å^3}$:

$$I(q) = DCS \cdot \rho_a \cdot \text{tweak}$$

$$\approx 100 \cdot 0{,}094 \cdot 9 \cdot 10^{-28} \frac{m^2}{Å^3}$$

$$\approx 85 \cdot 10^{-28} \frac{m^2}{Å^3} = 85 \cdot 10^{-28} \cdot 10^{24} \frac{m^2}{cm^3}$$

$$= 85 \cdot 10^{-4} \frac{m^2}{cm^3}$$

23.12.2015

$$K = 85 \cdot 10^{-4} \frac{m^2}{cm^3} \cdot (2 \cdot 10^{-2} Å^{-1})^4$$

$$\doteq 85 \cdot 16 \cdot 10^{-4-8} Å^{-4} \frac{m^2}{cm^3} = 1{,}4 \cdot 10^{3-12} \frac{m^2}{cm^3 Å^4}$$

$$= 1{,}4 \cdot 10^{-9} \frac{m^2}{cm^3 Å^4}$$

$$\Sigma_s = \frac{1{,}4 \cdot 10^{-9} \, m^2}{2\pi \cdot (5{,}4 \cdot 10^{-6} Å^{-2})^2 \, cm^3 Å^4} = \frac{1{,}4 \cdot 10^{-9}}{1{,}8 \cdot 10^{2-12}} \frac{m^2}{cm^3}$$

$$= 8 \frac{m^2}{cm^3}$$

Surface Roughness

07.01.2015

From: Surface Roughness by X-Ray and Neutron Scattering Methods
S.K. Sinha, Acta Physica Polonica A (1996)

Roughness exponent: h (values between 0 and 1)
$h = 1/2$: corresponds to the case of random-walk fluctuations
small h: sharp and jagged surface
h approaching 1: surface becomes more gently rounded

asymptotic power law: $I(q) \approx q^{-(2+\frac{1}{h})}$

Extreme cases: $h \to 0 \Rightarrow 2+\frac{1}{h} \to \infty \to$ super steep slope

$h = 1 \Rightarrow 2+\frac{1}{h} = 3 \to I(q) \sim q^{-3}$

random walk: $h = \frac{1}{2} \Rightarrow 2+\frac{1}{h} = 4 \to I(q) \sim q^{-4}$ (Porod law)

↳ what does that mean?

↳ randomly oriented vapour deposition

Roughness exponent:

$\beta = 2 + \frac{1}{h} \Rightarrow h = \frac{1}{\beta - 2}$

$\Delta h = \frac{\Delta\beta}{(\beta-2)^2}$

values of $\beta < 3$ don't make sense as h would then be out of the range $[0,1]$.

→ Force fit to stop at $\beta = 3$

$\boxed{11.01.2016}$

Tweak factor variations:

The tweak factors that lead to the best DCS matches in budrun seem to change with temperature (and possibly time), but fluctuate a lot.

First attempt to solve this: assume tweak changes linearly with temp and time and fit this.

Fit results for 1h-averages and for individual files match within the errorbars, but have seem to be more reliable for individual files.

Extreme cases: $h \to 0 \Rightarrow 2 + \frac{1}{h} \to \infty \to$ super steep slope

$h = 1 \Rightarrow 2 + \frac{1}{h} = 3 \Rightarrow I(q) \sim q^{-3}$

random walk: $h = \frac{1}{2} \Rightarrow 2 + \frac{1}{h} = 4 \Rightarrow I(q) \sim q^{-4}$ (Porod law)

↳ what does that mean?

↳ randomly oriented vapour deposition

Roughness exponent:

$\beta = 2 + \frac{1}{h} \Rightarrow h = \frac{1}{\beta - 2}$

$\Delta h = \frac{\Delta \beta}{(\beta - 2)^2}$

values of $\beta < 3$ don't ma

then be out of the range

→ Force fit to stop at β

intensity fit going crazy for some files
→ check that.

happens only for the high-T crap data but can't prevent it there.

11.01.2016

Tweak factor variations:

The tweak factors that lead to the best DCS matches in budrun seem to change with temperature (and possibly time), but fluctuate a lot.

First attempt to solve this: assume tweak changes linearly with temp and time and fit this.

Fit results for 1h-averages and for individual files match within the errorbars, but have seem to be more reliable for individual files.

11.01.2016

Continue with ISIS (Dec) data analysis

Tweak Factors:

Use time and temperature dependence fit results from individual scan analysis and force Python / Gudrun to run with those only.
(Save in the same folder structure and move the previous results to archive folders.)

Uncertainties:

$$tweak = a + b \cdot T + c \cdot t$$

$$\Delta tweak = \sqrt{(\Delta a)^2 + (\Delta b \cdot T)^2 + (b \cdot \Delta T)^2 + (\Delta c \cdot t)^2 + (c \cdot \Delta t)^2}$$

problem, when I include this uncertainty in the uncertainties for the specific surface area, those grow huge → I'll leave it out at the moment.

The new Gudrun results look good (DCS is in range 97 % – 103 % for most scans (sample 4, 1 h averages: 2 x DCS of 104.5 %)

12.01.2016

DCS deviations are stronger for individual scans, some scans don't process at all (Gudrun)
→ analyse them manually later

Scans with very little (less than 3 min) data:

sample 2: 39876, 39880 – 39885
sample 3: 39886, 39888, 39909 – 39912, 39967 – 39969
sample 4: 40007 – 40009, 40030 – 40032, 40036 – 40039, 40050, 40058 – 40059, 40068, 40148

Scans that didn't process:

sample 2: 39829, 39876, 39881-39885
sample 3: 39911, 39967-39968, 39978
sample 4: 40007-40009, 40031, 40037-40038, 40068

Scans that didn't process, but have > 3 min data:

sample 2: 39829 } at first look: nothing special, both on heat ramp
sample 3: 39978
sample 4: /

15.01.2016

Fit tweak factor as function of temperature only:

The tweak fit functions make more sense now.
SSA and roughness results don't change much
files that didn't process: the same as before

First summary:

Files to be excluded from final plots:
→ see Excel file in summary folder.

20.01.2016

Estimate expected SSA (see Excel sheet):

tweak uncertainty: $\Delta \text{Tweak} = \sqrt{(\Delta a)^2 + (\Delta b \cdot T)^2 + (b \cdot \Delta T)^2}$

particle volume: $V = \frac{4}{3}\pi \cdot \left(\frac{d}{2}\right)^3 \Rightarrow \Delta V = 4\pi \left(\frac{d}{2}\right)^2 \cdot \frac{\Delta d}{2}$

$\Rightarrow \frac{\Delta V}{V} = 3 \cdot \frac{\Delta d}{d}$

particle surface area: $A = 4\pi \left(\frac{d}{2}\right)^2 \Rightarrow \Delta A = 8\pi \frac{d}{2} \cdot \frac{\Delta d}{2} \Rightarrow \frac{\Delta A}{A} = 2 \cdot \frac{\Delta d}{d}$

20.01.2016

Continue with ISIS (Dec) data analysis

Particles per m^3: $m = \frac{1}{V \cdot Tweak}$

$$\Rightarrow \Delta m = \sqrt{\left(\frac{\Delta V}{V^2 \cdot Tweak}\right)^2 + \left(\frac{\Delta Tweak}{V \cdot Tweak^2}\right)^2}$$

$$= m \cdot \sqrt{\left(\frac{\Delta V}{V}\right)^2 + \left(\frac{\Delta Tweak}{Tweak}\right)^2}$$

$$SSA = A \cdot m \quad \Rightarrow \quad \Delta SSA = \sqrt{(\Delta A \cdot m)^2 + (\Delta m \cdot A)^2}$$

Sample	Specific Surface Area (m^2/cm^3) (at 105 K) Theoretical	Observed	
	+/-	+/-	
1	0.52 +/- 0.84	4.65 +/- 0.14	
2	1.02 +/- 1.60	7.13 +/- 0.13	
3	1.22 +/- 1.92	5.37 +/- 0.32	
4			
	Specific Surface Area (m^2/cm^3) (at 225 K) Theoretical	**Observed**	
	+/-	+/-	
1	0.45 +/- 0.73	0.78 +/- 0.15	
2	0.97 +/- 1.53	0.78 +/- 0.12	
3	1.15 +/- 1.81	1.16 +/- 0.12	
4		0.30 +/- 0.11	after 40 h
4			

21.01.2016

$$SSA = A \cdot m = 4\pi \left(\frac{d}{2}\right)^2 \cdot \frac{1}{\frac{4}{3}\pi\left(\frac{d}{2}\right)^3} \cdot \frac{1}{Tweak}$$

$$\boxed{SSA = 3 \cdot \frac{2}{d} \cdot \frac{1}{Tweak}}$$ ← wrong. no need to include tweak factor ⇒ see 16.03.2016

Unit Conversion: $1 \frac{m^2}{cm^3} = 1 \frac{m^2}{10^{-6} m^3} = 10^6 \frac{1}{m} = 10^6 \cdot \frac{1}{10^6 \mu m} = \frac{1}{\mu m}$

27.01.2016

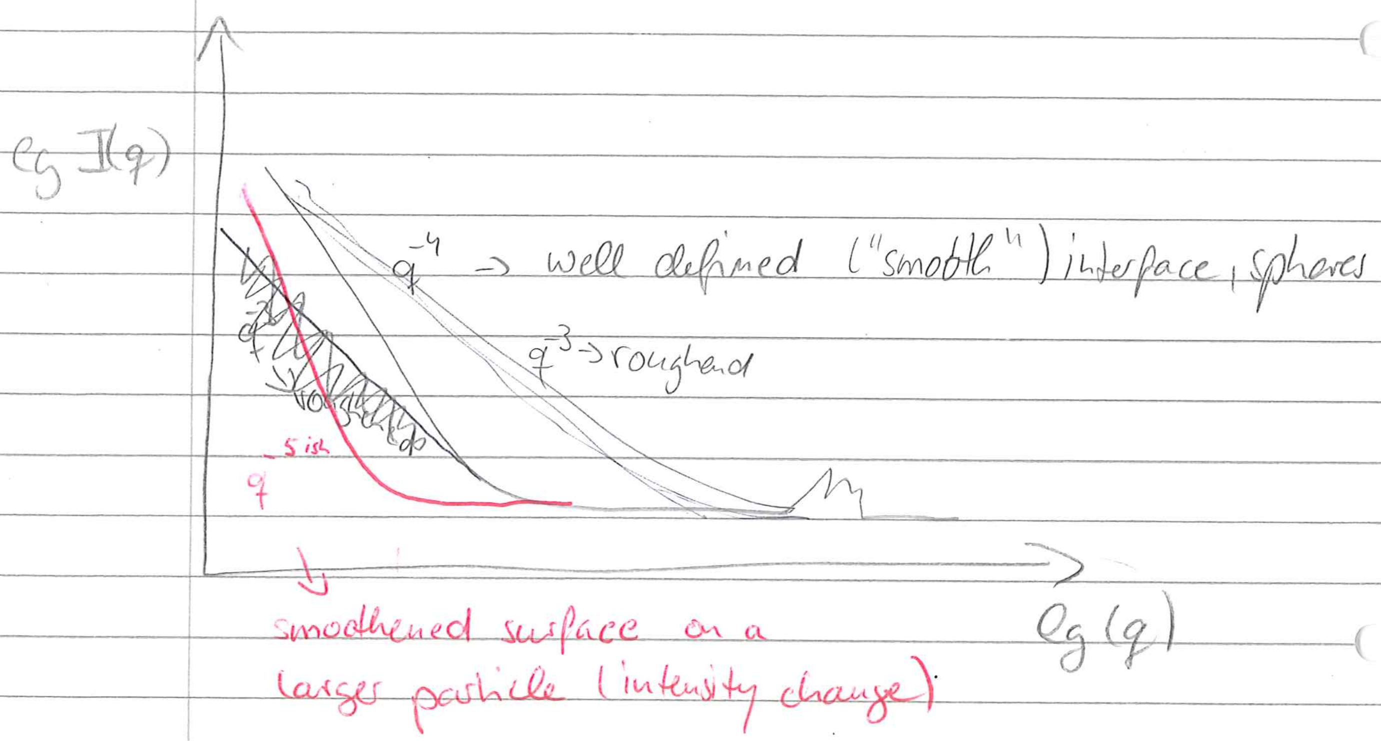
Discussion at ISIS:

Helens email from 20th

$\beta = 6 - D$ D: fractal dimension

$\Rightarrow \beta = 4 \hat{=} D = 2$ = smooth balls

D going up > surface getting rougher
(β goes down)



including size distribution could make up for missing SSA

$I = dcs \cdot \rho \cdot tweak \cdot 10^{-4}$

density = $0.094 \ \frac{atoms}{Å^3}$

$K = F \cdot q^4$

$SSA = K \cdot \left(2 \cdot \pi \cdot (\Delta\rho)^2\right)^{-1}$

proposal in April, or even a rapid access proposal to complete the data for a good publication. We should also discuss SANS2d and other options (of sclaes up to the micron size, to try and disentangle surface roughening versus sintering

j. though we never see water liquid, a question is would we be senstive or have enoughliquid water at a dynamic wetted interface to see it in this situation?

I think that is everything we disucssed. See you all next week:)

Helen

On 18/01/2016 11:32, Sabrina.Gaertner wrote:

> Hi everybody,
>
> Please find attached a (very rough) summary of the latest results from the data analysis. There are still some things on my to do list for the analysis (like looking at the other output files from Gudrun – d-spacings, g(r)), but I'm curious to hear your input.
> What do you think, we still need to do to get this data to publication stage (provided the run in Feb works and we do the same to all that data as well)?
>
> Looking forward to our discussion round next week at ISIS.
>
> Cheers,
> Sabrina

--

Dr Helen Jane Fraser
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Walton Hall,
Milton Keynes,
MK7 6AA

01908 332921

Re: ISIS run Dec (icy particles) - summary

Helen.Fraser [helen.fraser@open.ac.uk]

Sent: 20 January 2016 08:15

To: Sabrina.Gaertner [sabrina.gaertner@open.ac.uk]; Jürgen Blum [j.blum@tu-bs.de]; Bastian Gundlach [b.gundlach@tu-bs.de]; ratte.judy@googlemail.com; Bowron, Daniel (STFC,RAL,ISIS); Headen, Tom (STFC,RAL,ISIS); Youngs, Tristan (STFC,RAL,ISIS)

Sabrina - Jurgen and I chatted:- I tired to sumarise below - do interveen if I forgot anything...

a. it would be good to just compare "red and "blue" data (original) - done in last nights email I think (smile)

b. I am not sure if the "h" is the best beta model... can you plot beta as is and just remind me why you chose the"h" model - I recall we discussed it - my feeling is the roughness has no trend at all with T or with t? Why is 'h' the best fit - I have some recollections of h being a special fractal dimension, I wonder if we get the same trend in "roughness" If we choose a Beta model - we should chat about this with Daniel Tristan and Tom

c. it looks like there is no change in roughness of the surface at all - this seems strange! I'd like to look at the value you took from catherine for the surface volume density - is that associated with D2O, D or ASW? if so we might need to change this value for crytsaliine ice. Do you have an origin for her number - that would be good to know, and then we should do a literature search to check, and probably discuss again with D T & T

d. since there seems to be no directional dependence of the high Q data (Tristan and Tom did this in circular and longitudianal directions but I don't have copies of the original data so we should get those graphs to see the data in case we need it for the paper) so we need to (a) addign all the high Q peaks, (b) work out the iC and IH ratios and exactly where the ic is converted to IH - and check which hlk miller indicies are leading to the data - I still don't understand why our intensities are reproducable but not "standard" Ih or Ic. We should perhaps look at the g(r) and d-spacings data - probably for the 1 hr isothermal scans as the 3 min scans are too noisy

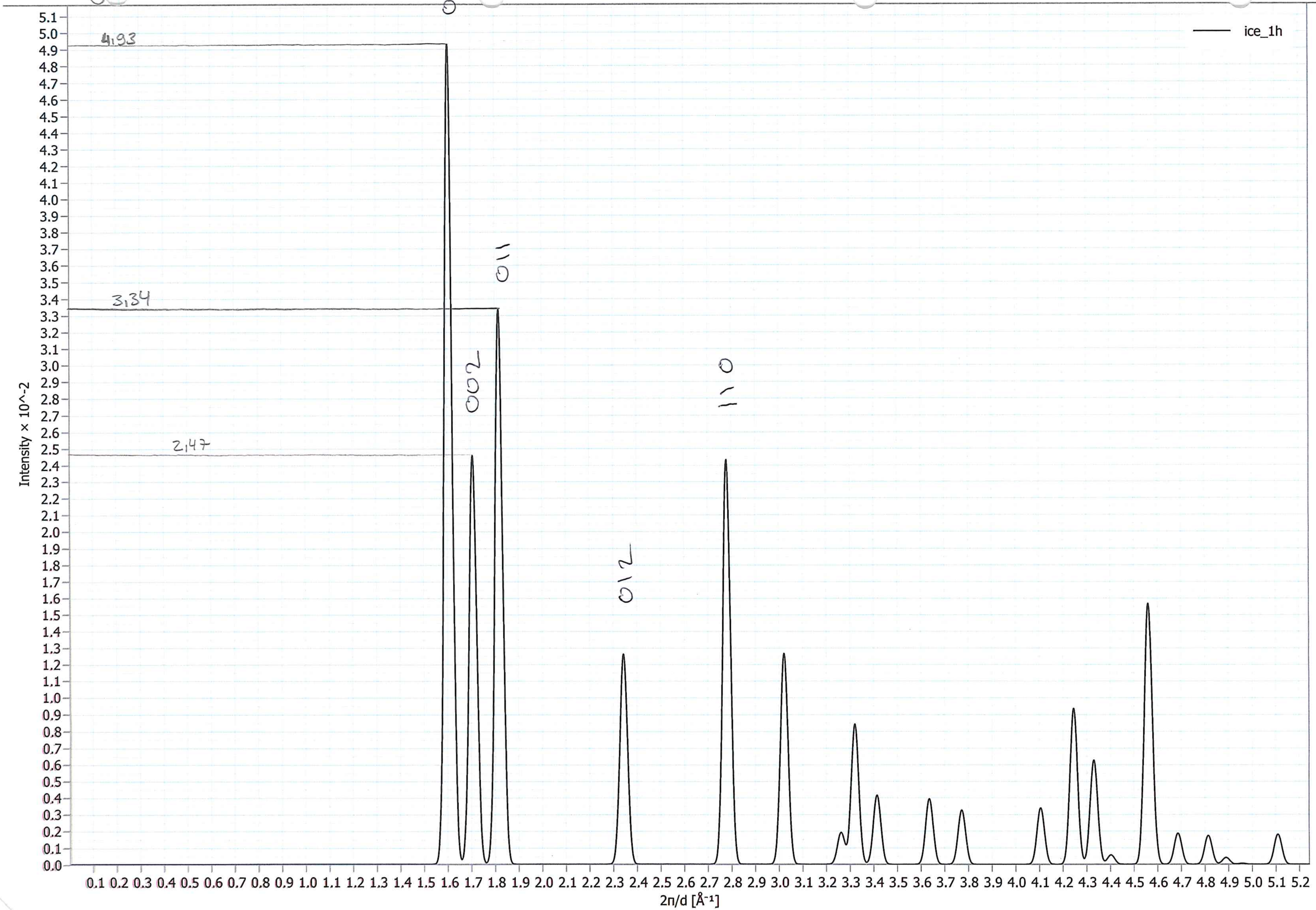
e. we should discuss if the tweek factor rise with T is real or not and why so much scatter is required to fit the data.

f. the question of sintering versus surface strcutre is still key. Why should the SSA be a factor 10 at least > than anticipated with the packing fraction - is the heating to 100 K from the liquid N2 environment leading to potting of the surface - Jurgen says the "roughness' seen in the image sof the ice balls is insufficient to explain this

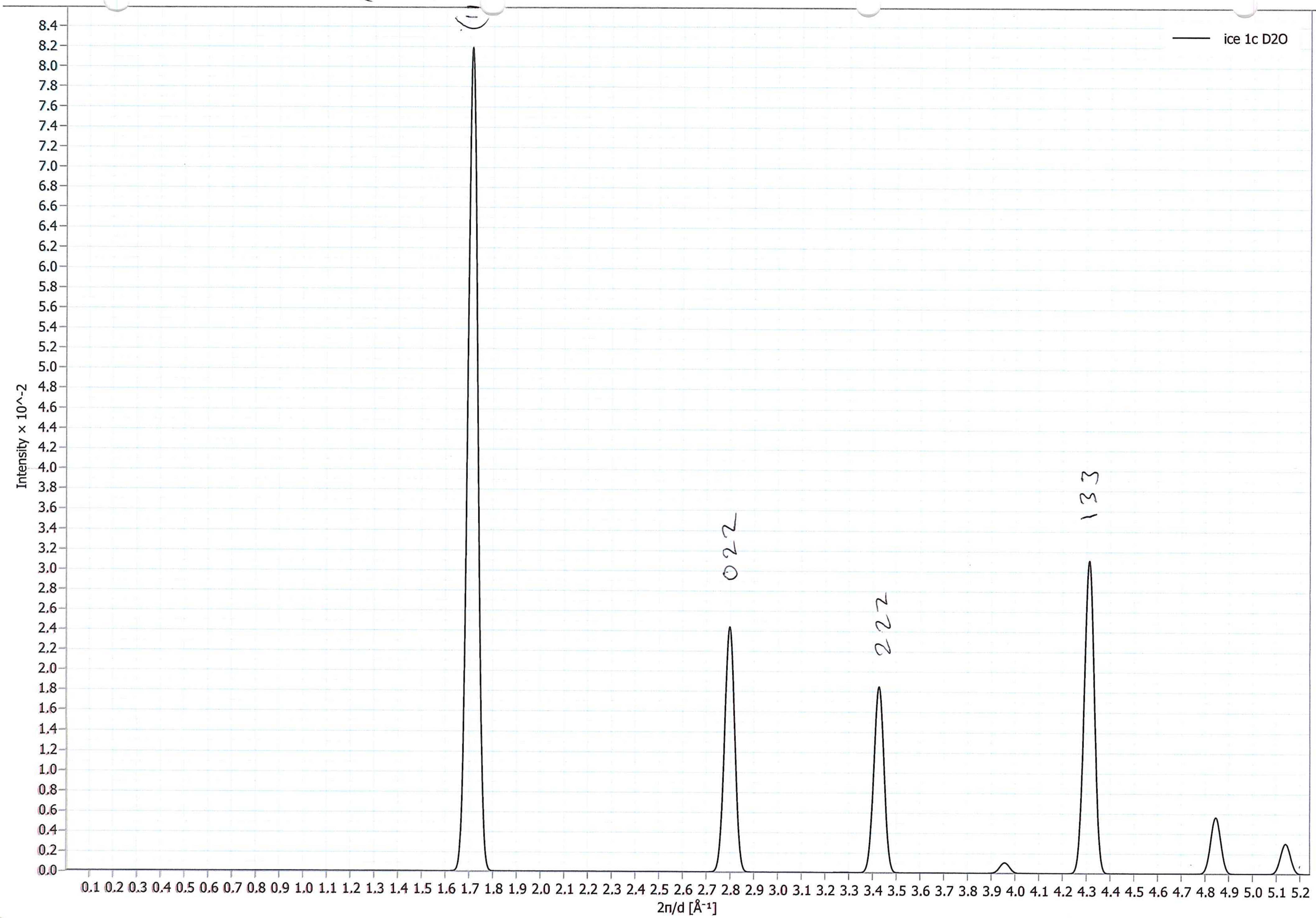
g. we should try using tirstans kinet programme to analyse the isothermal data at particular length scales and to analyse the onet point of the isothermal blue and red data to see if the porod slope onset is shifting with T or only the angle is changing.

h. we need to think how to display the data for pulications. I don't like the yellow as it is hard to see - a red to blue or light to dark grey scale may work better.

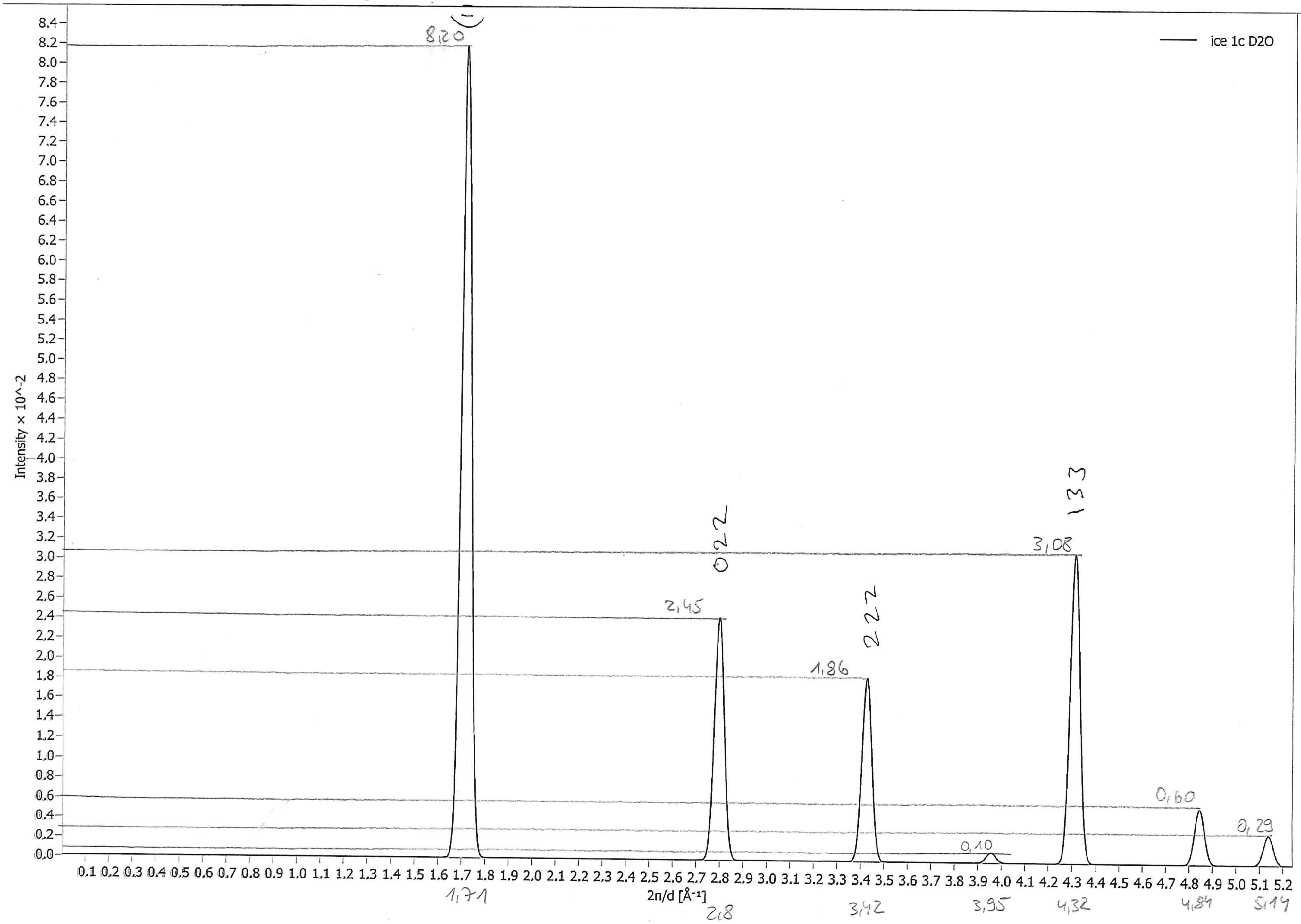
i. looking at the data and the questions we should plan a simple repeat for Feb - one blue and one red sample to reprodcue the results. If there are other questions or ideas we can either make a regular hexagonal ice

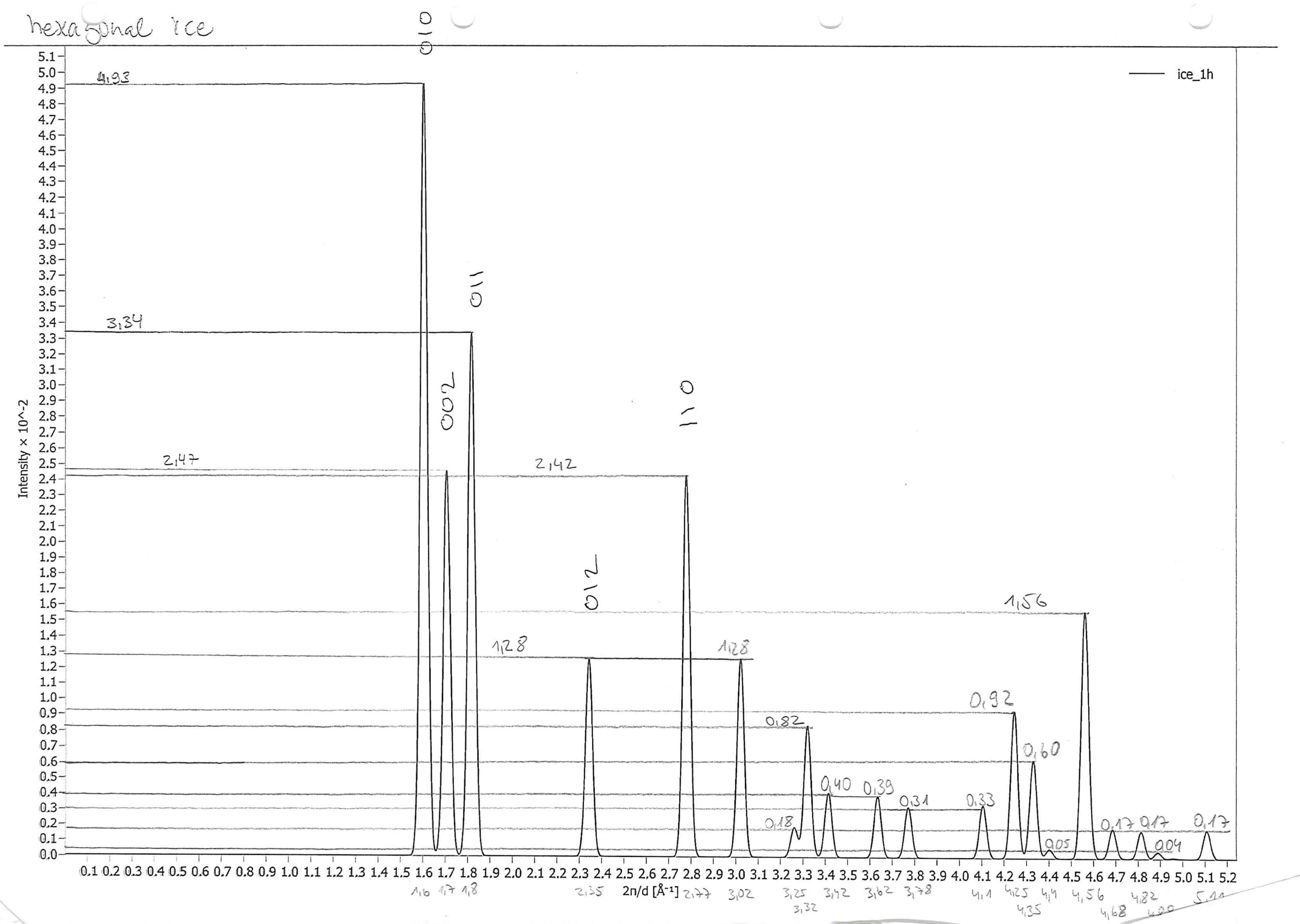


cubic Ice (D_2O)



cubic Ice (D_2O)




hexagonal ice
ice_1h
Intensity × 10^-2
2π/d [Å⁻¹]
010
002
011
012
110
4,93
3,34
2,47
2,42
1,28
1,28
1,56
0,92
0,82
0,60
0,40
0,39
0,31
0,33
0,18
0,05
0,17
0,17
0,04
0,17
1,6
1,7
1,8
2,35
2,77
3,02
3,25
3,32
3,42
3,62
3,78
4,1
4,25
4,35
4,4
4,56
4,68
4,82

27.01.2016

Continue with data analysis ISIS (dec)

tweak factor change:
average one hour of heating ramps as well
and fit temperature dependence again
(run Gudrun again for averages)

background change 0,1 – 1 Å^{-1}: plot .mint
files and see if it's still there

28.01.2016

In original processing, I did not
set a top hat width for Gudrun
(sample tab) → no mint files were produced
→ run Gudrun again with −10 top hat width

11.02.2016

tweak factor averaging:
In today's meeting with Helen, we agreed to
use average values for the final analysis.

Results:

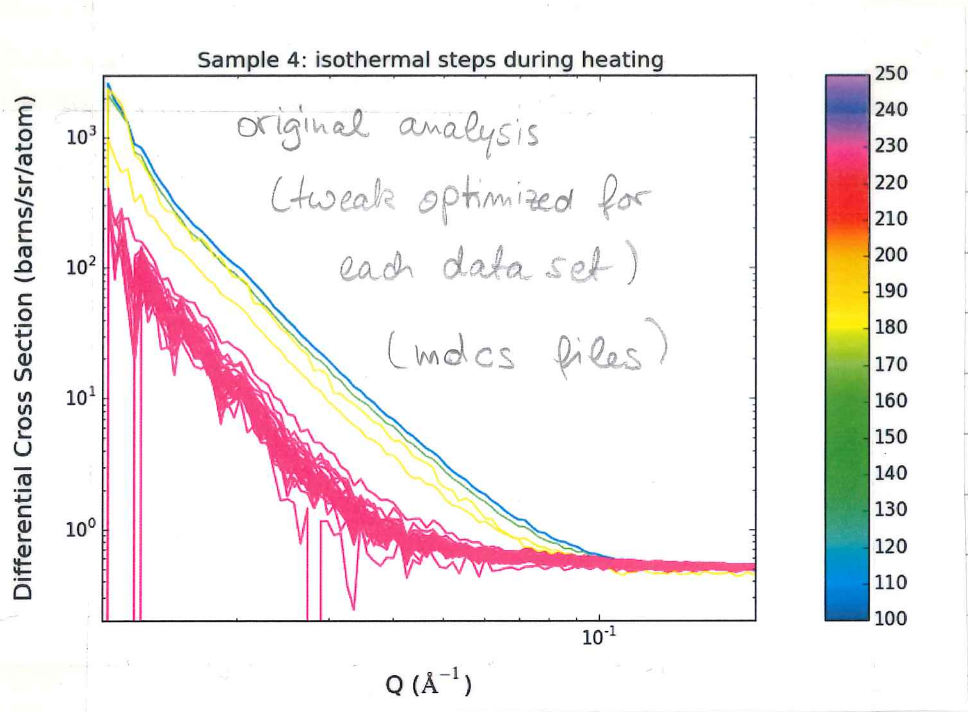
Sample	isothermal steps	individual scans
2	9,0300 ± 0,1529	8,8705 ± 0,0805
3	8,7210 ± 0,0889	8,5747 ± 0,0723
4	7,3620 ± 0,0247	7,3146 ± 0,0238

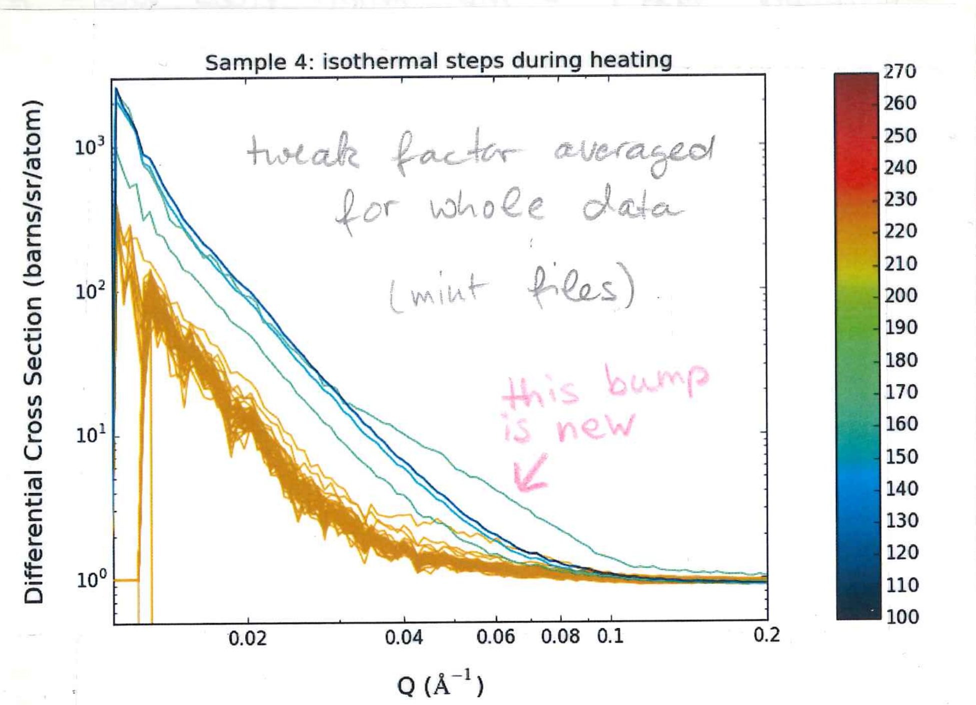
Note: * the isothermal average is always higher
than the individual average
* uncertainties depend on number of datapoints
and scatter

For final analysis, I will use the isothermal
averages and their uncertainties.

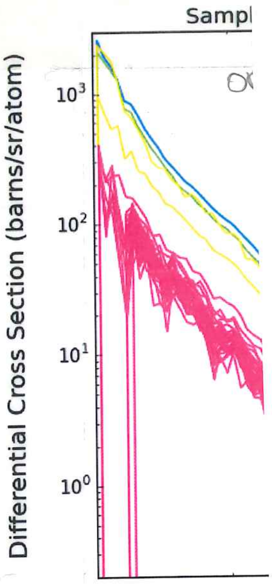
⇒ New plots of 1h isothermal steps.
Working mostly fine, but sample 4 last six
minutes before beam off show funny bump now
that wasn't there before.

12.02.2016: bumps only in mint files, not in mdcs
17.02.2016: Tristan says: due to bad statistics → use mdcs



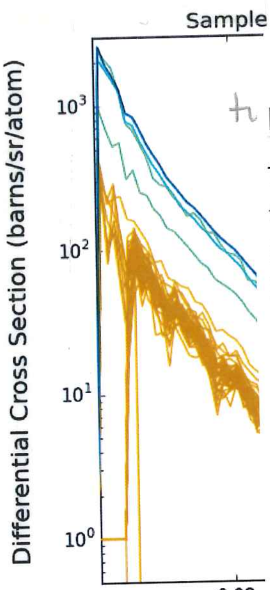


12.02.2016: bumps only in unit files, not in modes
17.02.2016: Tristan says: due to bad statistics → use modes



Returns:	**popt : array** Optimal values for the parameters so that the sum of the squared error of `f(xdata, *popt) - ydata` is minimized **pcov : 2d array** The estimated covariance of popt. The diagonals provide the variance of the parameter estimate. To compute one standard deviation errors on the parameters use `perr = np.sqrt(np.diag(pcov))`. How the sigma parameter affects the estimated covariance depends on absolute_sigma argument, as described above.
Raises:	**OptimizeWarning** if covariance of the parameters can not be estimated. **ValueError** if ydata and xdata contain NaNs.

See also:
leastsq (scipy.optimize.leastsq.html#scipy.optimize.leastsq)



Notes

The algorithm uses the Levenberg-Marquardt algorithm through leastsq (scipy.optimize.leastsq.html#scipy.optimize.leastsq). Additional keyword arguments are passed directly to that algorithm.

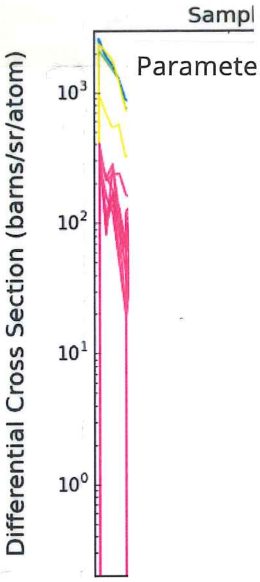
Examples

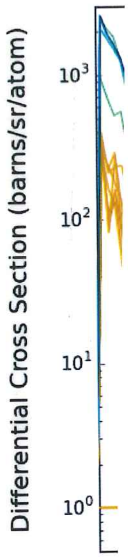
```
>>> import numpy as np
>>> from scipy.optimize import curve_fit
>>> def func(x, a, b, c):
...     return a * np.exp(-b * x) + c
```

```
>>> xdata = np.linspace(0, 4, 50)
>>> y = func(xdata, 2.5, 1.3, 0.5)
>>> ydata = y + 0.2 * np.random.normal(size=len(xdata))
```

```
>>> popt, pcov = curve_fit(func, xdata, ydata)
```

12.02.2016: bumps only in mult files, not in mdes
17.02.2016: Tristan says: due to bad statistics → use mdes





Returns: **popt** : array

Optimal values for the parameters so that the sum of the squared error of `f(xdata, *popt) - ydata` is minimized

Parameters: **f** : callable

The model function, f(x, ...). It must take the independent variable as the first argument and the parameters to fit as separate remaining arguments.

xdata : An M-length sequence or an (k,M)-shaped array

for functions with k predictors. The independent variable where the data is measured.

ydata : M-length sequence

The dependent data — nominally f(xdata, ...)

p0 : None, scalar, or N-length sequence, optional

Initial guess for the parameters. If None, then the initial values will all be 1 (if the number of parameters for the function can be determined using introspection, otherwise a ValueError is raised).

sigma : None or M-length sequence, optional

If not None, the uncertainties in the ydata array. These are used as weights in the least-squares problem i.e. minimising `np.sum( ((f(xdata, *popt) - ydata) / sigma)**2 )` If None, the uncertainties are assumed to be 1.

absolute_sigma : bool, optional

If False, sigma denotes relative weights of the data points. The returned covariance matrix pcov is based on estimated errors in the data, and is not affected by the overall magnitude of the values in sigma. Only the relative magnitudes of the sigma values matter. If True, sigma describes one standard deviation errors of the input data points. The estimated covariance in pcov is based on these values.

check_finite : bool, optional

If True, check that the input arrays do not contain nans of infs, and raise a ValueError if they do. Setting this parameter to False may silently produce nonsensical results if the input arrays do contain nans. Default is True.

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ize=len(xda

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ydata)

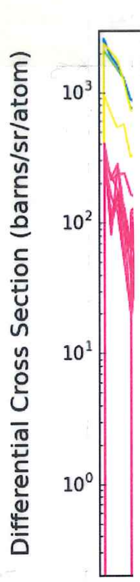
12.02.2016: bumps only in multi fits, not in modes

2016: Tistrup says: due to bad statistics → use modes

Returns: popt : array

Optimal values for the parameters so that the sum of the squared error of `f(xdata, *popt) - ydata` is minimized

Sampl



Parameters: f : callable

The model function, f(x, ...). It must take the independent variable as the first argument and the parameters to fit as separate remaining arguments.

xdata : An M-length sequence or an (k,M)-shaped array

for functions with k predictors. The independent variable where the data is measured.

ydata : M-length sequence

The dependent data — nominally f(xdata, ...)

p0 : None, scalar, or N-length sequence, optional

Initial guess for the parameters. If None, then the initial values will all be 1 (if the number of parameters for the function can be determined using introspection, otherwise a ValueError is raised).

sigma : None or M-length sequence, optional

If not None, the uncertainties in the ydata array. These are used as weights in the least-squares problem i.e. minimising `np.sum( ((f(xdata, *popt) - ydata) / sigma)**2 )` If

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SciPy.org Sponsored By ENTHOUGHT (../index.html)

curve fit

weighted by Yerr

Scipy.org (http://scipy.org/) Docs (http://docs.scipy.org/)

SciPy v0.16.1 Reference Guide (../index.html)

Optimization and root finding (**scipy.optimize**) (../optimize.html)

index (../genindex.html) modules (../py-modindex.html)

modules (../scipy-optimize-modindex.html) next (scipy.optimize.brentq.html)

previous (scipy.optimize.rosen_hess_prod.html)

>>>

>>>

(xda

scipy.optimize.curve_fit

scipy.optimize.**curve_fit**(f, xdata, ydata, p0=None, sigma=None, absolute_sigma=False, check_finite=True, **kw) [source] (http://github.com/scipy/scipy/blob/v0.16.1/scipy/optimize/minpack.py#L454)

Use non-linear least squares to fit a function, f, to data.

Assumes `ydata = f(xdata, *params) + eps`

>>>

Previous topic

scipy.optimize.rose
(scipy.optimize.rose

Next topic

scipy.optimize.bren
(scipy.optimize.bren

12.02.2016

Continue with ISIS Dec data analysis

Surface Roughness:

- Modified Python script to do weighted fit (curve_fit instead of leastsq) and to allow manual fit for predefined exeptions.
- New fit function: $I(q) = \text{offset} + \text{const} \cdot q^{-\text{beta}}$ that works ok (took out moving fit window)
- problem: on linear scale those values with low Intensity have less absolute deviation
  ⇝ curve_fit gives them preference over high Intensity data points.
  To improve this, I would like to go to log space with fit routine (⚠ log means ln in numpy)
  $(\lg I(q) = \lg(\text{offset} + \text{const} \cdot q^{-\text{beta}}))$
  in my first attempts the fit always crashed.
- → problem solved, but fit is still off in low q region, also uncertainties are super small
- Solution: artificially scale up uncertainties of log data (Scaling factor going from 2 to 3 (low q to mid q))
  → now the fit looks good in low q region and uncertainties of fit parameters look reasonable
- New fit has no constraints on β values!
- Include $\beta = 6 - D$ model:
  fractal dimension: $D = 6 - \beta$

Timeconstant for SSA change

16.02.2016

Sample 4, 220 K: 5,21 h

SSA loss for sitting still at 180 K can not be fitted with same function (is stronger than at 220K)

29.02.2016

Surface Roughness:

Sinha 88: surface in xy plane with height distribution in z direction:

$$\langle [z(x',y') - z(x,y)]^2 \rangle = g(X,Y)$$

$$g(X,Y) = g(R) = A R^{2h} \qquad (0 < h < 1)$$

$$R = (X^2 + Y^2)^{1/2}$$

$$\vec{A} = \vec{e_z} \qquad \text{question: is } A = |\vec{A}| \text{ ?}$$

$$\Rightarrow A = 1 \text{ ???}$$

fractal dimension: $D = 3 - h \quad \Rightarrow h = 3 - D$

$\Rightarrow \quad 2 < D < 3$

Smooth surface: Porod case : $\beta = 4 = 6 - D \Rightarrow D = 2$

$(g(R) = 0) \qquad S(q) \sim q^{-4}$

Rough Surface (no cutoff) : $S(\vec{q})\big|_{q_r = 0} \sim q_z^{-(2+\frac{2}{h})}$

Randomly oriented rough surfaces:

$$S(q) = \frac{A_1}{q^4} + \frac{A_2}{q^{3+h}}$$

$$= \frac{A_1}{q^4} + \frac{A_2}{q^{6-D}}$$

oriental averaging yields $\boxed{S(q) \sim \frac{1}{q^{3+h}}}$

single surfaces yield $\frac{1}{q^{2+\frac{2}{h}}}$

$\beta = 6 - D = 6 - 3 + h = 3 + h$

$\beta = 2 + \frac{2}{h}$

very different for single surfaces and oriental averaged surfaces

sinha 96 : $g(R) \sim R^{2h}$

rough surface: $S(q) \propto q^{-(2+2/h)}$

depending on instrument I might be $\sim q_z^{-(2+1/h)}$ (eq. 5.3 & 5.4)

$\hookrightarrow$ ask Tom + Tristan

04.03.2016

Specific Surface Area:

In the previous calculations, I most likely did a mistake on the tweak factor.
→ Go through Gudrun manual to get it right

Differential cross section (DCS)

$$\left(\frac{d\sigma}{d\Omega}\right)(\lambda, 2\theta)$$

σ: cross-section, Ω: solid angle
λ: neutron wavelength, 2θ: scattering angle

Scattered radiation amplitude:

Array of N point atoms at positions $\vec{R}_j$:

$$A(Q) = \sum_j b_j e^{i\vec{Q}\vec{R}_j}$$

b_j: scattering length (form factor) for atom j

Structure factor (scattered intensity per unit atom):

$$F(\vec{Q}) = \frac{1}{N} |A(\vec{Q})|^2$$ (dimensionless)

$$= \frac{1}{N} \sum_{jk} b_j b_k e^{i\vec{Q}(\vec{R}_j - \vec{R}_k)}$$

$\vec{Q} = \vec{k}_i - \vec{k}_f$, change in wavevector by scattering

$$|\vec{Q}| = Q = \frac{4\pi}{\lambda} \sin\theta$$

Gudrun N analysis:

average number of particles (neutrons) per unit area per unit time per unit wavelength, impinging on an assembly of N atoms:

$$\langle \Phi(\lambda,t)\rangle_t = \frac{\int_0^t \Phi(\lambda,t)\,dt}{\Delta t}$$

number of neutrons scattered by that assembly in the same time interval Δt in the wavelength range $\lambda, \lambda+\Delta\lambda$ into a solid angle $\Delta\Omega(2\theta)$ in the direction 2θ relative to incident beam:

$$CNT(\lambda, 2\theta)$$

Differential Cross Section:

$$\frac{d\sigma}{d\Omega}(\lambda, 2\theta) = \frac{CNT(\lambda,2\theta)}{N\langle\Phi(\lambda,t)\rangle_t\,\Delta\lambda\,\Delta\Omega(2\theta)\,\Delta t}$$

(assuming N is small enough to ignore multiple scattering etc.)

Problems: - neutron sources not overly stable
- beams not precisely defined
- detectors do not count every particle entering them (neutron-energy dependent efficiency)
- in reality samples cannot be as small as assumed above (to low signal)

Cross Sections:

$$\sigma^{(t)}(\lambda) = \sigma^{(s)}(\lambda) + \sigma^{(a)}(\lambda)$$

total cross section scattering cross section absorption cr. sec.

$$\sigma^{(s)}(\lambda) = \int_\Omega \left(\frac{d\sigma}{d\Omega}\right)(\lambda, 2\theta)\,d\Omega$$

(in reality measurement never covers all 4π sr of Ω
→ use tabulated single atom values for $\sigma^{(s)}$
$\sigma^{(s)}$ more or less independent of λ (exceptions e.g. materials containing H/D)

[table of neutron scattering lengths :
www.ncnr.nist.gov/resources/n-lengths]

for problematic materials use neutron source with fixed wavelength (tuned to avoid resonances etc.)

for H/D solution can be to use transmission crosssection measured from special detector at instrument (forward direction) / or use tables for the right wavelength range

$\sigma^{(a)}$ depends linearly on λ (is usually quoted for $\lambda = 1{,}7982\,\text{Å}$

$$\Rightarrow \quad \sigma^{(a)}(\lambda) = \sigma^{(a)}(\lambda_0) \cdot \frac{\lambda}{\lambda_0}$$

<u>neutron transmission cross section:</u>

$$\mathrm{TRANS}(\lambda) = e^{-\mathrm{MUT}(\lambda)\cdot \underset{\substack{\uparrow\\ \text{sample}\\ \text{thickness}}}{L}} = e^{-\underset{\substack{\uparrow\\ \text{average atomic}\\ \text{number density}}}{\rho}\,\sigma^{(t)}(\lambda)\cdot L}$$

(calculations get a bit more complicated if sample is not a flat slab)

include detector efficiencies $E(\lambda)$ ($_t$: transmission monitor, $_m$: incident monitor)

$$\frac{\overset{\text{transmission}}{\mathrm{RAWTRANS}(\lambda)}}{\underset{\text{incident}}{\mathrm{RAWMON}(\lambda)}} = \mathrm{TRANS}(\lambda)\,\frac{E_t(\lambda)}{E_m(\lambda)}$$

monitor

Signal must be <u>normalised</u> to <u>incident beam strength</u> (integrated monitor count $\langle \bar{\Phi}(\lambda, t)\rangle_t\,\Delta t$) (monitor placed in incident beam)

$$\mathrm{RAWMON}(\lambda) = \langle \Phi(\lambda, t)\rangle_t\, E_m(\lambda)\,\Delta\lambda\,\Delta t$$

$\rightarrow$ normalise all measured intensities (sample + background) to this $\Rightarrow$ $\mathrm{NORMON}(\lambda, 2\theta) = \dfrac{\mathrm{CNT}(\lambda, 2\theta)}{\mathrm{RAWMON}(\lambda)}$

Subtract the background(s):

$$SUBBAK(\lambda, 2\theta) = NORMON_s(\lambda, 2\theta) - NORMON_b(\lambda, 2\theta)$$

Calibration:

So far calibration factor (detector efficiency terms) is unknown
→ vanadium reference (calib) scan with same settings as for exp.
⇒ $VANCOR(\lambda, 2\theta)$ → smoothing ⇒ $SMOVAN(\lambda, 2\theta)$
(overdoing the smoothing can lead to artifacts at the ends of the spectra, no effect for fixed wavelength instrument)
→ normalisation of data: $NORMVAN(\lambda, 2\theta) = \dfrac{SUBBAK_s(\lambda, 2\theta)}{SMOVAN(\lambda, 2\theta)}$

Attenuation and multiple scattering corrections: ⇒ $MULCOR(\lambda, 2\theta) - M_s$
multiple scattering

⇒ $ABSCOR(\lambda, 2\theta) = \dfrac{MULCOR(\lambda, 2\theta)}{NA}$

number of atoms in the beam
self attenuation factor < 1

Tweak factor: accounting for less than fully packing in container
inverse of packin fraction

$$\text{sample atomic density} = \frac{\text{specified atomic density}}{\text{tweak factor}}$$

« sample with 100% packing

only applied to sample (not containers / backgrounds)

⇒ ABSCOR is in $\frac{\text{barns}}{\text{atom} \cdot \text{sr}}$, but just for one of many detectors
Combine detectors: statistically weighted average
(how many counts do detectors show)
before that re-bin data for each detector on common x-scale
⇒ MDCS (merged all detectors)
↳ Differential Cross Section per atom

Gudrun manual page 135: List of Gudrun output files

Specific Surface Area:

$$\lim_{Q\to\infty} \frac{I(Q)\cdot Q^4}{2\pi \Delta\rho^2}$$

We want to have the surface area per unit volume of a sample with our atomic density for a sample with atomic density ρ_A the intensity per unit volume would be

$$I(Q) = DCS(Q) \cdot \rho_A$$

units: $[I] = \frac{\text{barn}}{\text{atom sr}} \cdot \frac{\text{atom}}{Å^3} = \frac{?? \ cm^2}{Å^3}$

for our sample the atomic density is

$$\rho_A = \frac{\rho_a}{\text{tweak}}$$

ρ_a = atomic density of material at 100% packing

$\frac{1}{\text{tweak}}$ = volume filling factor

$$\Rightarrow I(Q) = DCS(Q) \cdot \frac{\rho_a}{\text{tweak}}$$

still wrong

see 24.03.16

⚠

Scattering length density:

We do not have a solution (mix of materials) but one material in a Helium atmosphere (which should have been subtracted with the background scans)

$$\Rightarrow \Delta\rho = \rho_{D_2O}$$

Estimate SSA for same example values as on 21.12.2015

$$SSA \approx \frac{DCS \cdot \rho_a \cdot Q^4}{tweak \cdot 2\pi \cdot (\Delta\rho)^2}$$

$DCS(@\ Q = 2 \cdot 10^{-2} \frac{1}{Å}) \approx 100 \frac{barn}{sr\ atom}$

tweak ≈ 9 $\qquad \rho_a \approx 0{,}1 \frac{atom}{Å^3}$

$\Delta\rho = 5{,}4 \cdot 10^{-6}\ Å^{-2}$ $\qquad 1\,barn = 10^{-28}\ m^2$

$$SSA \approx \frac{100 \cdot 10^{-28}\ m^2 \cdot 10^{-1}\ atom \cdot (2 \cdot 10^{-2} \frac{1}{Å})^4}{atom\ Å^3 \cdot 9 \cdot 2 \cdot \pi \cdot (5{,}4 \cdot 10^{-6}\ Å^{-2})^2}$$

$$\approx \frac{1 \cdot 10^{2-28-1-8} \cdot 16\ m^2}{2 \cdot 9 \cdot 3 \cdot 30 \cdot 10^{-12}\ Å^3} \approx \frac{16 \cdot 10^{-35}\ m^2}{4 \cdot 9 \cdot 45 \cdot 10^{-12} \cdot 10^{-30}\ m^3}$$

$$\approx \frac{4}{4 \cdot 100} \cdot 10^{-5+12} \frac{m^2}{m^3} \approx 10^{7-2} \frac{m^2}{m^3}$$

$$= 10^5 \frac{m^2}{m^3} = \boxed{10^{-1} \frac{m^2}{cm^3}}$$

estimates for SSA (smooth particles, no size distribution) are between 0,5 and 12 $\frac{m^2}{cm^3}$
these get a bit lower when size distribution is included (more big particles than small ones)

16.03.2016

Continue with ISIS Dec 15 + Feb 16 Data Analysis

Estimate expected Specific Surface Area:

For spheres with diameter d and no size distribution:

$$\boxed{SSA = \frac{6}{d}} \qquad \left(SSA = \frac{\text{surface}}{\text{Volume}} = \frac{A}{V} = \frac{4\pi \left(\frac{d}{2}\right)^2}{\frac{4}{3}\pi \left(\frac{d}{2}\right)^3} \right)$$

Now consider sample with a size distribution:

Probability to have size d: $p(d) = \frac{1}{\sqrt{2\pi}\,\sigma_d} e^{-\frac{(d-d_0)^2}{2\sigma_d^2}}$

Average SSA:

$$SSA = \int_0^\infty p(d) \frac{6}{d} \, dd$$
$$= \frac{6}{\sqrt{2\pi}\,\sigma_d} \int_0^\infty \frac{1}{d} e^{-\frac{(d-d_0)^2}{2\sigma_d^2}} \, dd$$

$\Rightarrow$ does not work because $\lim_{d \to 0} \left(\frac{1}{d} e^{-\frac{(d-d_0)^2}{2\sigma_d^2}} \right) = \infty$

17.03.2016

Average surface:

$$A = \int_0^\infty \frac{4\pi}{\sqrt{2\pi}\,\sigma_d} \left(\frac{d}{2}\right)^2 e^{-\frac{(d-d_0)^2}{2\sigma_d^2}} \, dd$$
$$= \sqrt{\frac{\pi}{2}}\frac{1}{\sigma_d} \int_0^\infty d^2 e^{-\frac{(d-d_0)^2}{2\sigma_d^2}} \, dd$$

$d_0 = 1{,}45\ \mu m$, $\sigma_d = 0{,}65\ \mu m$ $\Rightarrow A = 6{,}39234\ \mu m^2$

$d_0 = 0{,}71\ \mu m$, $\sigma_d = 0{,}31\ \mu m$ $\Rightarrow A = 1{,}04945\ \mu m^2$

(Integrals calculated with wolfram alpha)

Average Volume :

$$V = \int_0^\infty \frac{4}{3}\pi \left(\frac{d}{2}\right)^3 \cdot \frac{1}{\sqrt{2\pi}\,\sigma_d} \cdot e^{-\frac{(d-d_0)^2}{2\sigma_d^2}} \, dd$$

$$= \int_0^\infty \frac{1}{6}\sqrt{\frac{\pi}{2\sigma_d}} \cdot e^{-\frac{(d-d_0)^2}{2\sigma_d^2}} d^3 \, dd$$

$d_0 = 1{,}45\ \mu m$, $\sigma_d = 0{,}65\ \mu m$ $\Rightarrow$ $V = 2{,}06308\ \mu m^3$

$d_0 = 0{,}71\ \mu m$, $\sigma_d = 0{,}31\ \mu m$ $\Rightarrow$ $V = 0{,}164033\ \mu m^3$

Average SSA :

19.07.16

factor of 2 missing

$d = 2 r$

$= 2 \cdot 1{,}45\ \mu m$

$d_0 = 1{,}45\ \mu m$, $\sigma_d = 0{,}65\ \mu m$ $\Rightarrow$ $SSA = \frac{A}{V} = 3{,}09845\ \frac{\mu m^2}{\mu m^3}$

(blue aspirator inset)

$\Rightarrow$ $\boxed{SSA = 3{,}09845\ \frac{m^2}{cm^3}}$

$d_0 = 0{,}71\ \mu m$, $\sigma_d = 0{,}31\ \mu m$ $\Rightarrow$ $\boxed{SSA = 6{,}39780\ \frac{m^2}{cm^3}}$

(red aspirator inset)

wrong by factor 2

see 02.11.2016 for correct values

18.03.2016

Crystalline Phases :

in my attempts to determine the contributions to our diffraction peaks from different ice phases, I used one of Catherine's amorphous ice data sets to compare it to our data and two simulations (I_h , I_c).
Each of these fitted peak sets comes with a scaling factor compared to our data (s_i) and a fitted amount (a_i)

$$\Rightarrow DCS_{sample} = \sum_i a_i s_i DCS_i \quad , \text{where } i = h, c, HGW$$

The absolute amounts of each phase thus are :

$$\boxed{A_i = \frac{a_i s_i}{\sum_j a_j s_j}}$$ (so they sum up to 1)

Determine the scaling factors s_i:

I_a, I_c: they come from the same simulation
$\Rightarrow$ should have the same scaling factors

$$\boxed{s_h = s_c}$$

s_h can be determined from a scan that shows only hexagonal contribution

$$\Rightarrow \quad a_h \cdot s_h = 1$$

$$\Rightarrow \quad \boxed{s_h = \frac{1}{a_h}} \qquad \Delta s_h = \frac{\Delta a_h}{a_h^2}$$

I_{HGW}: Catherine used different settings for the Gudrun processing $\rightsquigarrow$ find those and work back from them

$\rightarrow$ She had Gudrun set to give output in the same units as I do

$$\Rightarrow \quad \boxed{s_{HGW} = 1} \qquad \Delta s_{HGW} = 0$$

Amount uncertainties:

$$\Delta A_i = \left[\left(\frac{\Delta a_i \cdot (s_i \cdot (\sum_j a_j s_j) - a_i s_i s_i)}{(\sum_j a_j s_j)^2} \right)^2 + \left(\frac{\Delta s_i \, (a_i \cdot (\sum_j a_j s_j) - a_i s_i a_i)}{(\sum_j a_j s_j)^2} \right)^2 \right.$$
$$\left. + \sum_{k \neq i} \left\{ \left(\frac{a_i s_i \, \Delta a_k s_k}{(\sum_j a_j s_j)^2} \right)^2 + \left(\frac{a_i s_i \cdot \Delta s_k \, a_k}{(\sum_j a_j s_j)^2} \right)^2 \right\} \right]^{1/2}$$

$$= \frac{1}{(\sum_j a_j s_j)^2} \cdot \left[\left(\Delta a_i \, s_i \cdot \sum_{k \neq i} a_k s_k \right)^2 + \left(\Delta s_i \, a_i \sum_{k \neq i} a_k s_k \right)^2 \right.$$
$$\left. + \sum_{k \neq i} \left\{ \left(a_i s_i \cdot \Delta a_k s_k \right)^2 + \left(a_i s_i \, \Delta s_k \, a_k \right)^2 \right\} \right]^{1/2}$$

$$= \frac{1}{\sum_j a_j s_j} \cdot \left[\left(\sum_{k \neq i} a_k s_k\right)^2 \cdot \left\{ (\Delta a_i s_i)^2 + (\Delta s_i a_i)^2 \right\} + (a_i s_i)^2 \sum_{k \neq i} \left\{ (\Delta a_k s_k)^2 + (\Delta s_k a_k)^2 \right\} \right]^{1/2}$$

Calculate s_h in case a_{hgw} is never 0:

$$DCS_{sample} = (a_h DCS_h + a_c DCS_c) \cdot s_h + a_{hgw} DCS_{hgw}$$

$$\Rightarrow \quad s_h = \frac{DCS_{sample} - a_{hgw} DCS_{hgw}}{a_h DCS_h + a_c DCS_c}$$

all DCS equal

$$\rightsquigarrow \quad \boxed{s_h = \frac{1 - a_{hgw}}{a_h + a_c}}$$

This should work in all cases and leads to the previous result in the special case $a_{hgw} = a_c = 0$.

$$\Delta s_h = \sqrt{\left(\frac{\Delta a_{hgw}}{a_h + a_c}\right)^2 + \left(\frac{1 - a_{hgw}}{(a_h + a_c)^2} \Delta a_h\right)^2 + \left(\frac{1 - a_{hgw}}{(a_h + a_c)^2} \Delta a_c\right)^2}$$

24.03.2016

Specific Surface Area:

Units: $[I] = \frac{10^{-24} cm^2}{Å^3} = 10^{-24} \frac{(10^{-2} m)^2}{(10^{-10} m)^3} = 10^{-24-4+30} \frac{1}{m} = 10^2 \cdot \frac{1}{10^2 cm} = \frac{1}{cm}$

$\rightsquigarrow$ as Tom said in his email.

$\Rightarrow$ If i set Gudrun to output in $\frac{1}{cm}$, I should not have to do the DCS $\to$ I conversion

$$\Rightarrow \quad SSA = \frac{K}{2\pi \Delta\rho^2} \qquad \left(K = \lim_{Q \to \infty} (I(Q) \cdot Q^4) \right)$$

$\rightsquigarrow$ try that

$$= \frac{1}{\sum_j a_j s_j} \cdot \left[\left(\sum_{k\neq i} a_k s_k\right)^2 \cdot \left\{ (\Delta a_i s_i)^2 + (\Delta s_i a_i)^2 \right\} + (a_i s_i)^2 \sum_{k\neq i} \left\{ (\Delta a_k s_k)^2 + (\Delta s_k a_k)^2 \right\} \right]^{1/2}$$

Calculate s_h in case a_{hgw} is never 0:

$$DCS_{sample} = (a_h DCS_h + a_c DCS_c) \cdot s_h + a_{hgw} DCS_{hgw}$$

$$\Rightarrow s_h = \frac{DCS_{sample} - a_{hgw} DCS_{hgw}}{a_h DCS_h + a_c DCS_c}$$

all DCS equal $\approx>$

$$\boxed{s_h = \frac{1 - a_{hgw}}{a_h + a_c}}$$

This should work in all cases and leads to the previous result in the special case $a_{hgw} = a_c = 0$.

$$\Delta s_h = \sqrt{\left(\frac{\Delta a_{hgw}}{a_h + a_c}\right)^2 + \left(\frac{1 - a_{hgw}}{(a_h + a_c)^2} \Delta a_h\right)^2 + \left(\frac{1 - a_{hgw}}{(a_h + a_c)^2} \Delta a_c\right)^2}$$

SSA

Hi Sabrina,

Having looked at this I think you may be calculating the surface area per unit of ***illuminated volume*** rather than solid sample volume, if I'm right this should bump up your volumetric surface area by the tweak factor used. I'll explain my reasoning below (Daniel, Tris if you spot an error please let me know)

- Your equation for I(Q) at the top of page 3 should give I(Q) in SANS units of cm^-1: [barns/atom]*[atoms/Ang^3]=barns/Ang^3= [10^-28 m^2]/[10^-30 m^3]= 10^2 m^-1 = 1 cm^-1. Note that as you have divided by the tweak factor this is the scattering cross section per illuminated volume. Double check this is corrected by asking Gudrun to calculate in cm^-1 as previously described
- The surface area then calculated is per unit of illuminated volume, therefore to get it per volume of solid you need to multiply by the tweak factor. For a tweak factor of 10 this would push your SSA up to 1-2 m^2/cm3, much closer, but still a bit lower than the expected value, perhaps more easily explain by a bit of particle sintering?

I realise that I may have said the opposite to this during our meeting in the NIMROD cabin – so apologies for that!

Any questions/thoughts/flaws in my argument please let me know,

All the best,

Tom

Dr Thomas Headen
Mob: +44 (0) 7584 142603
Short Code: 1156

23.03.2016

PDRA Meeting

Peak fits: show combination of $I_c + I_h$ vs $I_c + I_h + I_{HGA}$ to show which one fits better

Surface Roughness: Length scales of Porod region:

$Q = \frac{2\pi}{d} \quad \Rightarrow d = \frac{2\pi}{Q}$

$Q = 0{,}01 - 0{,}05$

$\Rightarrow d \approx 6 \cdot \left(\frac{1}{100} Å^{-1}\right)^{-1} - 6 \cdot \left(\frac{5}{100} Å^{-1}\right)^{-1}$

$= 600 \text{ Å} - 100 \text{ Å}$

$= (6-1) \cdot 10^{2} \cdot 10^{-10} \text{ m}$

$= (6-1) \cdot 10^{-8} \text{ m}$

$= (6-1) \cdot 10^{-2} \cdot 10^{-6} \text{ m}$

$\boxed{= (0{,}01 - 0{,}06)\ \mu m}$

01.04.2016

Size of H_2O molecule:

$3 Å = 3 \cdot 10^{-10}$ m

$\Rightarrow 6 \cdot 10^{-8}$ m $= 200$ molecules

22.06.2016

$Q = 0.15 Å^{-1} \Rightarrow d = 42 Å$

$\Rightarrow d = 4.2 \cdot 10^{1-10}$ m $\approx 4 \cdot 10^{-9}$ m

$= 4$ nm

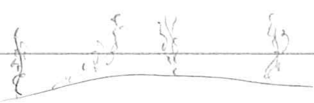
$Q = 1 Å^{-1} \Rightarrow d = 0.6$ nm

$Q = 6 Å^{-1} \Rightarrow d = 1.04 Å$

$Q = 1.2 Å^{-1} \Rightarrow d = 0.52$ nm

diffuse interfaces

1-D (line fractal)



DLCCA diffusion limited cluster growth

high SSA lower SSA

$D = 1.8$

$D = 2$

look for liquid like surface (beta → 6)

05.05.2016

Porod analysis (ASW):

✓ plot position where data best fits Q^{-4} vs T

✓ SSA: how much ice would we expect to have upon deposition ~> what SSA would that be for homogeneous + compact sample (-> 22.07.16)

compare SSA: * Bossa papers (Leiden): He-Ne laser to determine height of ice

* Bruce Kay (B.D. Kay) mid-late 90's -> SSA vs. temp by TPD + volumetric analys

* Raut 1970 or 2007 co-author baragola: SSA from TPD

* Martin Manca + Rubin 90's 2000's

heating ramp data analysis

Gunier-Porod analysis: split in two sets (two bumps) for low T

✓ try to treat higher T ones like icy particles ($Q^{-\beta}$ fit only)

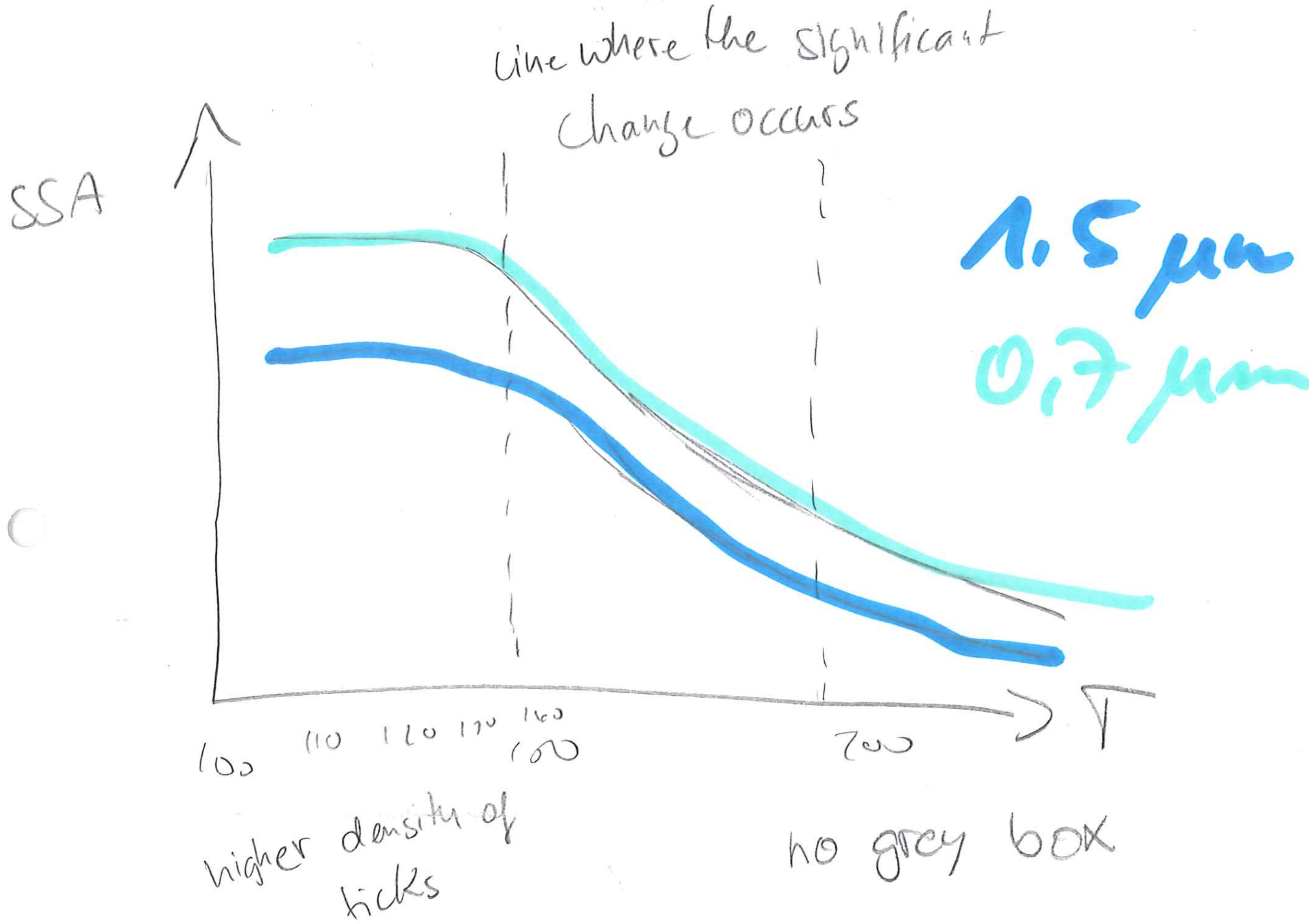
keep calling d-parameter "d" not β (that's in the literature)

Icy particles paper: do not put details of analysis but refer to other papers which give them.

27th May: give PRL (icy particles) draft to Helen (printed paper copy!)

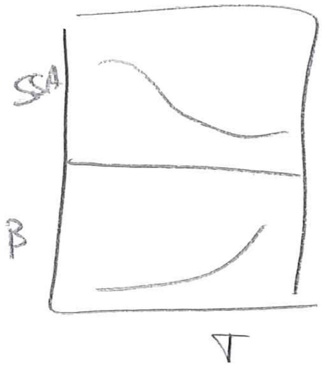
20th May: send 60s final report to Helen she'll send it back until 27th May

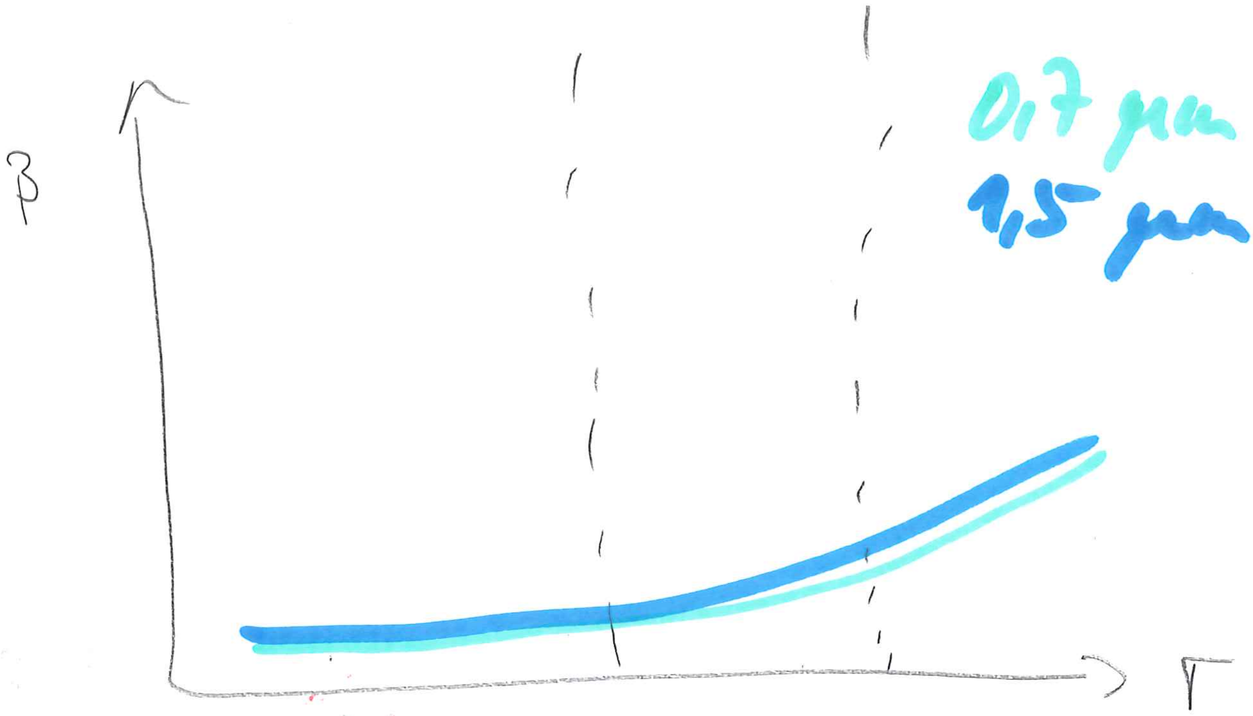
SSA


line where the significant change occurs
SSA
1.5 μm
0,7 μm
T
100
110
120
200
higher density of ticks
no grey box

Surface Roughness

together with SSA



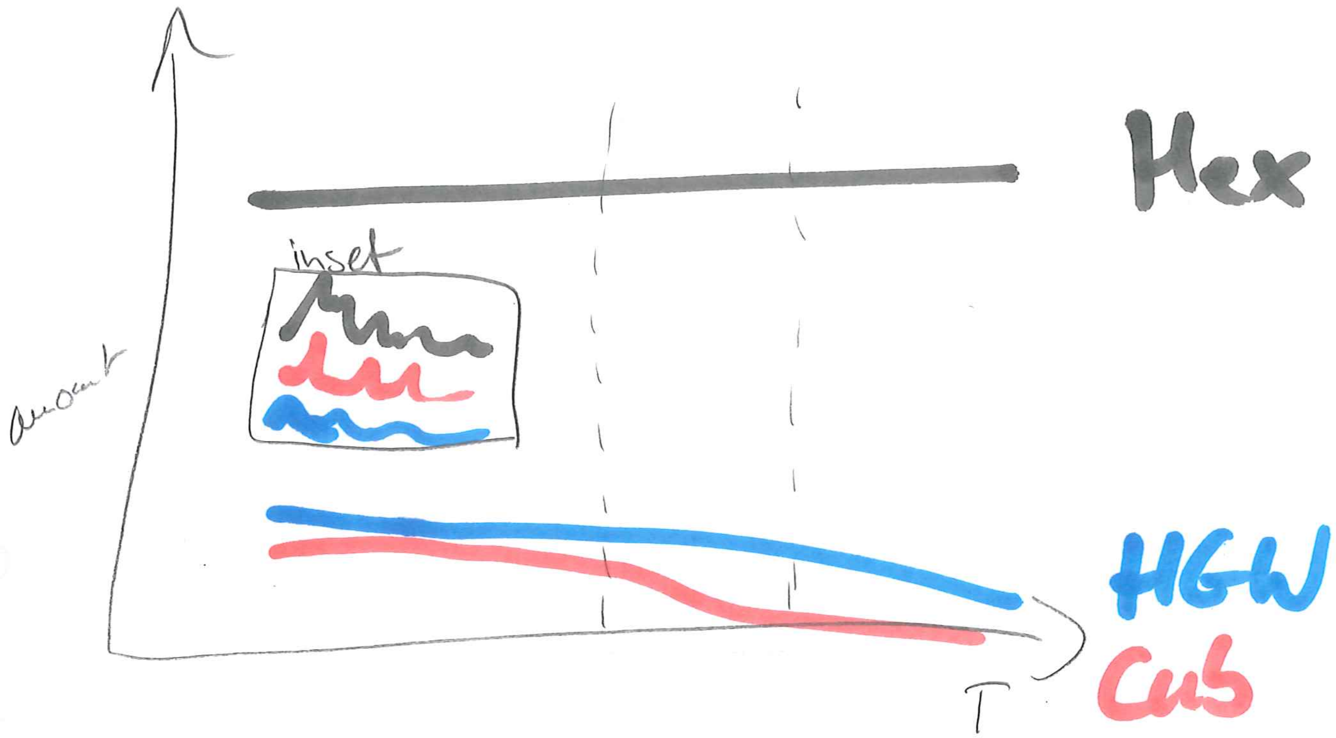


particle size doesn't influence surface roughness

interpolation for 250 K datapoint

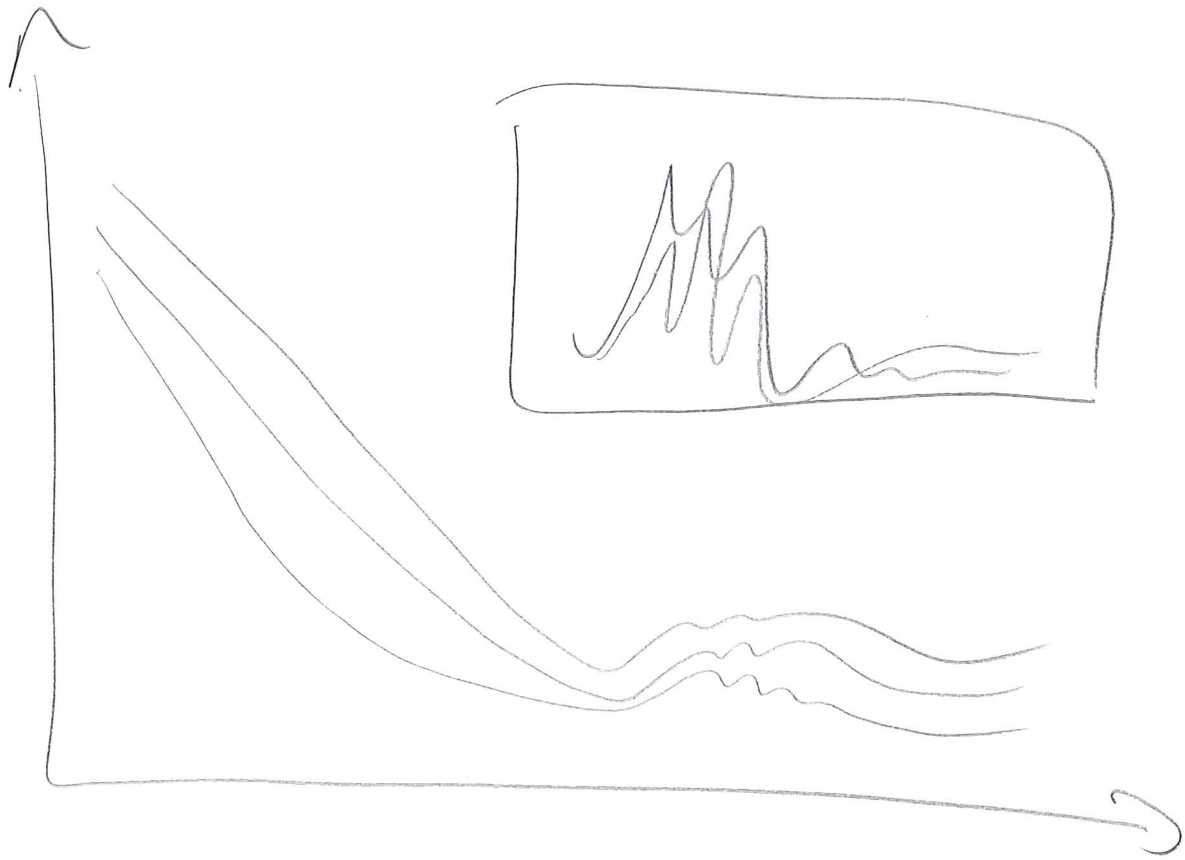
caption: smooth spheres / liquid like layer
but not on the figure

$\beta = 6 - D$

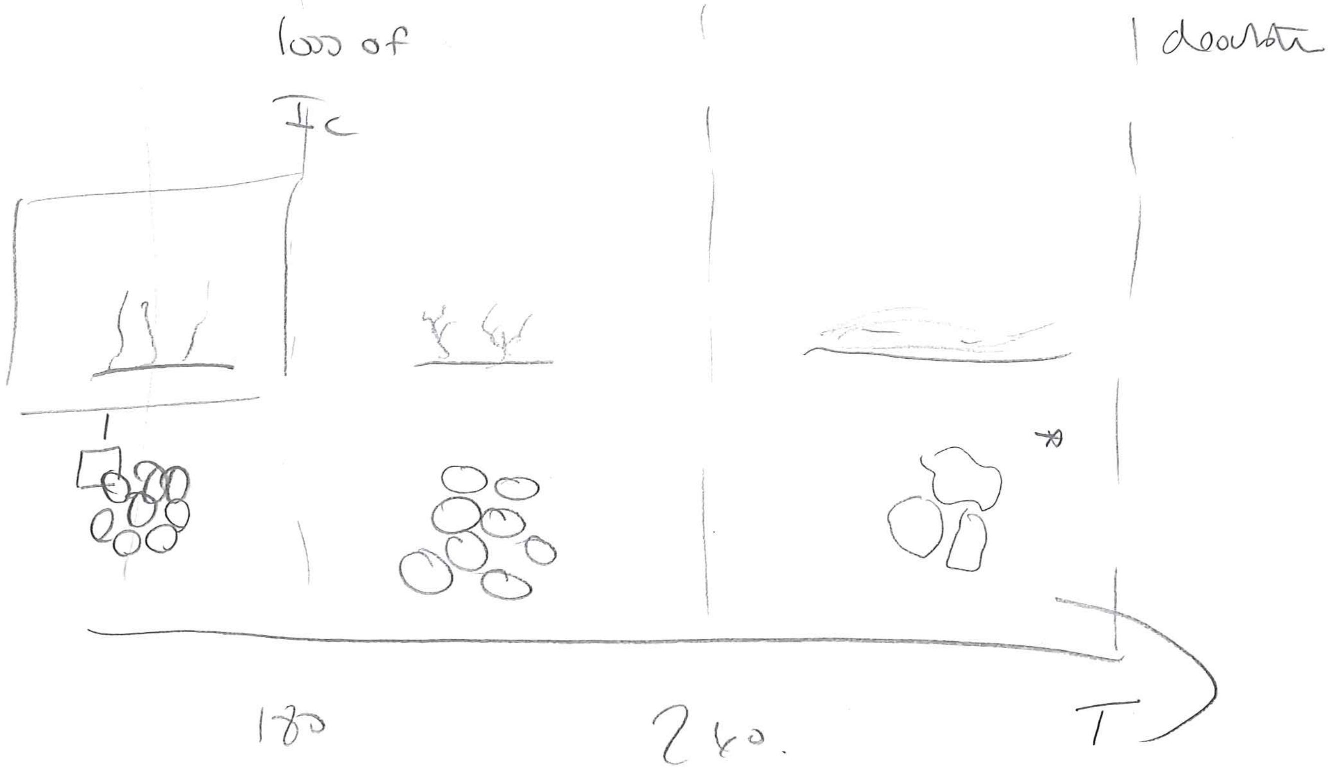


normalise in a way that hex stays constant to show material loss.

amorphicity that arises from the stacking disorder of the ice. disordered layers. huge defect density raw data



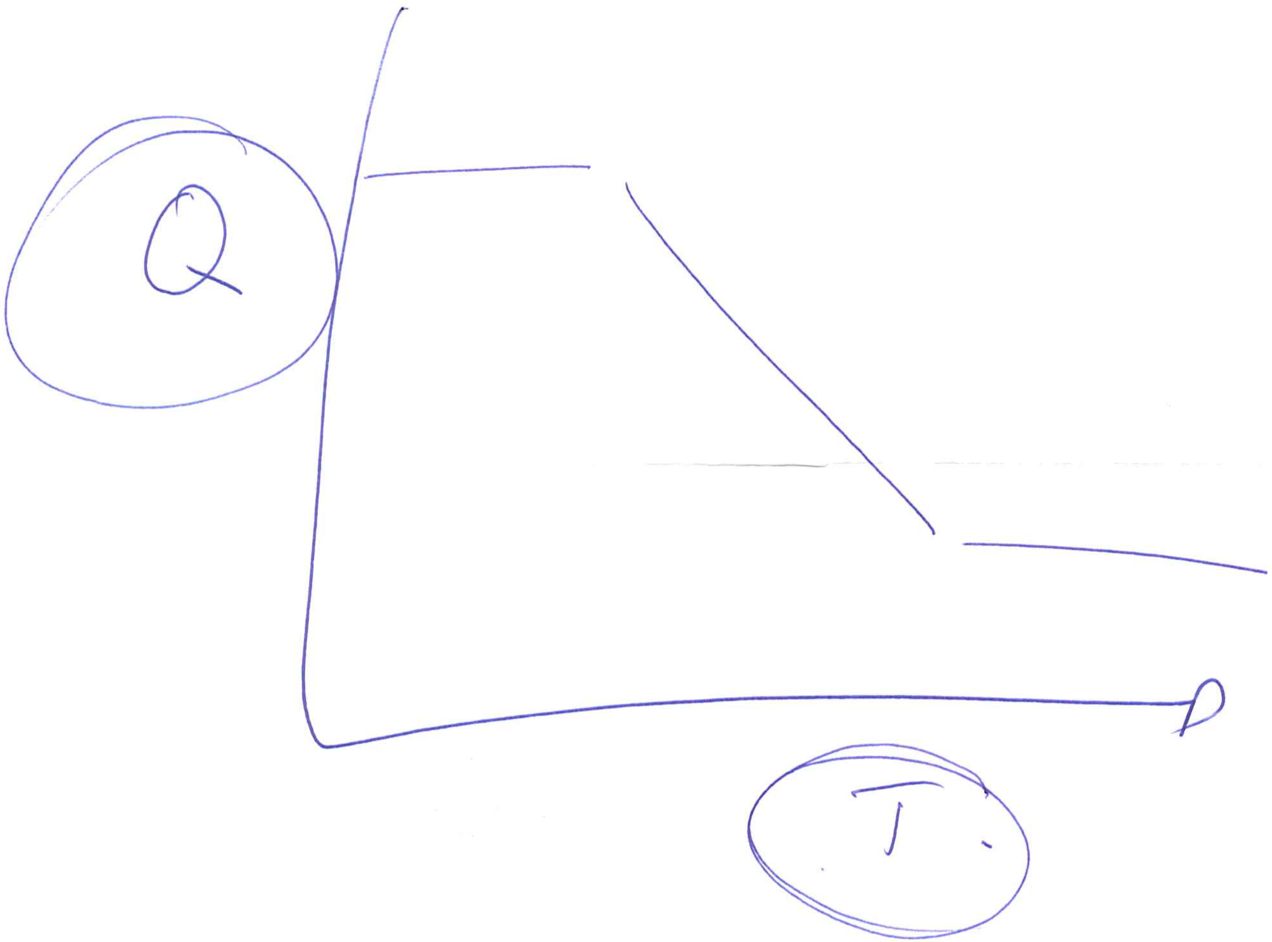
Sample change with temp.



Collin effect
collision

* low T Sem
low T SEM

Plot for ASW Porod analysis



24.03.2016

Continue with data analysis on Icy Particles (Dec 2015 + Feb 2016)

SSA (from SANS introduction, Jackson (NIST) 2008):

$$SSA = \frac{\pi}{Q^*} K$$

scattering invariant: $Q^* = 2\pi^2 \phi_1 \cdot (1-\phi_1)(\rho_2-\rho_1)^2$

for an incompressible two-phase system

They don't say what ϕ_1 is. I assume the relative amount of phase 1.

Problem: in our case, we have only one phase

But: if I leave the ϕ_1-factors away, I get exactly the formula I was using so far.

~> I guess that's ok then.

Except: This is where the tweak factor would be needed, because we have our one phase not everywhere ???

$$SSA = \frac{K}{2\pi \Delta\rho^2} \Rightarrow \text{units}:$$

$$[SSA] = \frac{[I][Q]^4}{[\Delta\rho]^2} = \frac{\AA^4}{cm} \cdot \AA^4 = \frac{1}{cm} = \frac{1}{10^{-2}} \cdot \frac{1}{m} = 10^2 \frac{m^2}{m^3}$$

$$= 10^2 \frac{m^2}{(10^2 cm)^3} = 10^{2-6} \frac{m^2}{cm^3} = 10^{-4} \frac{m^2}{cm^3}$$

Skype with Tom:

04.04.2016

compare peak heights (mint file) - work out proper tweak factor -> add H_2O in Gudrun to get DCS levels right (assume that highest tweak factor contains no H_2O)

mgor 01 -> bond distances (peaks) change for other isotopes beta > 4 : diffuse interface → density gradient

09.05.2016

Plots for paper: average data sets for each particle size

⚠ sample 2 Dec: highest T was 250 K instead of 240 K
→ interpolate results R to get 240 K point

$$R(240K) = \frac{R(250K) - R(220K)}{T(250K) - T(220K)} \cdot (T(240K) - T(220K)) + R(220K)$$

ice phases: normalize to first data point to show loss of material (→ hex amount = const)

original formula: $A_i = \frac{a_i s_i}{\sum_j a_j s_j} \Rightarrow \sum_i A_i = 1$

now: $\cancel{A_i = \frac{a_i s_i}{\sum_j a_j[0]\, s_j[0]} \Rightarrow \sum_i A_i[t] = \frac{\sum_i a_i[t]\, s_i[t]}{\sum_j a_j[0]\, s_j[0]}}$

~~→ graphs do not change~~

try: $\boxed{A_i[t] = \frac{a_i[t] \cdot s_i[0]}{\sum_j a_j[0] \cdot s_j[0]}}$

→ graphs change ~ looks like slight gain and then loss in hex component with T
→ checked fit results: they show this trend

10.05.2016

SSA: compare to Tom's results (Porod constant)
my fit: $\approx 0{,}25 \cdot 10^{-9} \frac{m^2}{cm^3 \cdot A^4}$ Tom's fit: $\approx 2{,}5 \cdot 10^{-6} \frac{1}{cm\, A^4}$

$$1 \frac{m^2}{cm^2} = \frac{1 m^2}{(10^{-2} m)^2} = 10^4 \frac{m^2}{m^2} = 10^4 \rightsquigarrow 0{,}25 \cdot 10^{-9} \frac{m^2}{cm^3 A^4} = 2{,}5 \cdot 10^{-10+4} \frac{1}{cm\, A^4}$$

$$= 2{,}5 \cdot 10^{-6} \frac{1}{cm\, A^4} \checkmark$$

10^{-3} mbar

Cryo-SEM notes

T unknown

very few particles (less than 1 Layer on cooling plate) to better controll temperature

Bashi tries thermodynamic calculations

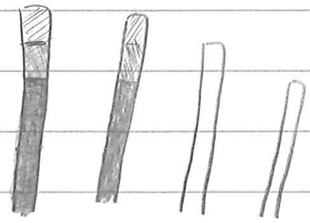
pics $-120°C$ to $-50°C$

$\rightsquigarrow$ 150 K to 180 K

10.05.2016

Continue with data analysis of Icy Particles (ISIS)
(Dec 15 + Feb 16)

Ice phase plots: not line, but bar:



Length scales with no scattering signal
(-> no structure):

01.06.2016

Q: 0,1 to 1,2 $\mathring{A}^{-1}$ $\rightsquigarrow$ $d = \frac{1}{2\pi Q}$: 1 to 0,1 $\mathring{A}$

$\Rightarrow$ 1 to 10 mm

Literature research for references in paper:

[Hill 15] ice:

Hatzes '88

Higa '96

Higa '98

Heißelmann '10

Hill '15

general:

21.07.2016

SSA compare Tom's and my analysis of 2nd Feb 100k data (41158 - 41161)

	Sabrina	Tom
Tweak factor:	11.038	≈ 11
Porod constant K:	$2.54 \cdot 10^{-10} \frac{m^2}{cm^3} \cdot Å^{-4}$	$3.29 \cdot 10^{-6}\, cm^{-1} Å^{-4} = 3.29 \cdot 10^{-10} \frac{m^2}{cm^3} Å^{-4}$
Density:	$0.094 \frac{atom}{Å^3}$	← that was entered in Gudrun at ISIS by Daniel + Helen. No clue, where it comes from
SSA:	$0.99 \frac{m^2}{cm^3}$	$1.45 \frac{m^2}{cm^3}$

Question: How did Tom determine K? Why is it so high?
(I am looking for the 20 datapoints (interval) between $0.013 Å^{-1}$ and $0.05 Å^{-1}$ that are most constant in $I(Q) \cdot Q^4$

25.07.2016

Tom did a rough estimate by eye. He says my method is probably fine, but will check with experts

Tom found different values for D_2O's atomic density and scattering length density:
$\Delta\rho = 5.995 \cdot 10^{-6} Å^{-2}$ ← https://www.ncnr.nist.gov/resources/activation

$\rho_A = 1.041\, g/cm^3 = 1.041 \cdot 0.092288 \frac{atoms}{Å^3} = 0.096 \frac{atoms}{Å^3}$

↑ see 22.12.2015

12.07.2016

Agenda:

Work planning → next meeting

Lab Move ✓

60s final report → return to Helen by Wednesday 13.07.

PRL Paper

16.06.2016

Icy Particles

Diffuse interface fit: compare intensity / slope changes in lab data (Feb vs Dec)

thickness: average

SSA expected value: —*— line with star at low T

1 bilayer ≈ 4 Å (Thürmer + Nie pnas 110 11757), 2013)

17.08.2016

table summarising collision experiments in Catherine's thesis

also starting point for atmospheric literature research

thickness errorbars: deviation of the mean

10.06.2016

PDRA Meeting

Agenda:

✓ Paper ('ay particles - PRL):
- work trough comments / pics etc.
- schedule : June 25th send paper to co-authors
  21st get comments from Helen
  17th send paper to Helen

✓ 60s :- researchfish after paper
- budget Helen deals with it

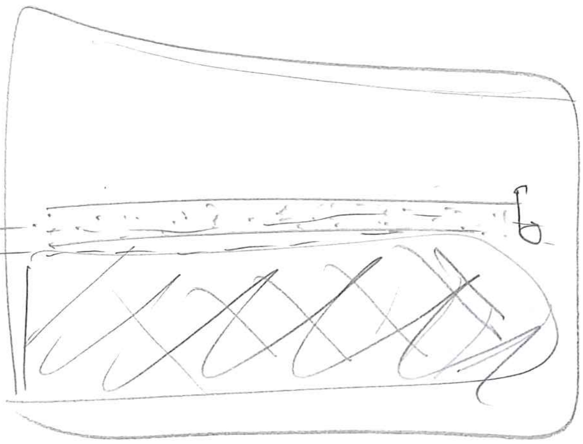
✓ Ogden :- prep for this year's workshop
- feedback on collaboration so far
word doc with instructions by 24th give to Helen

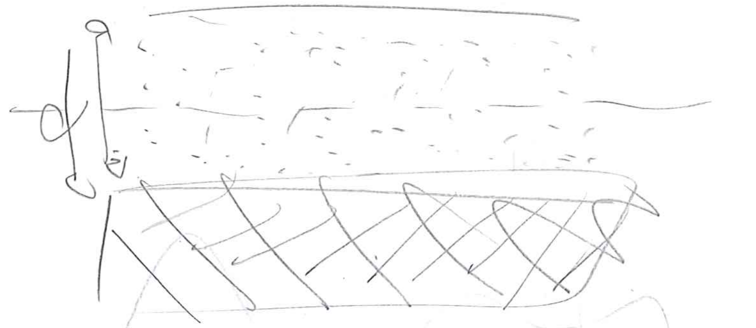
PF proposal : 17.06.15

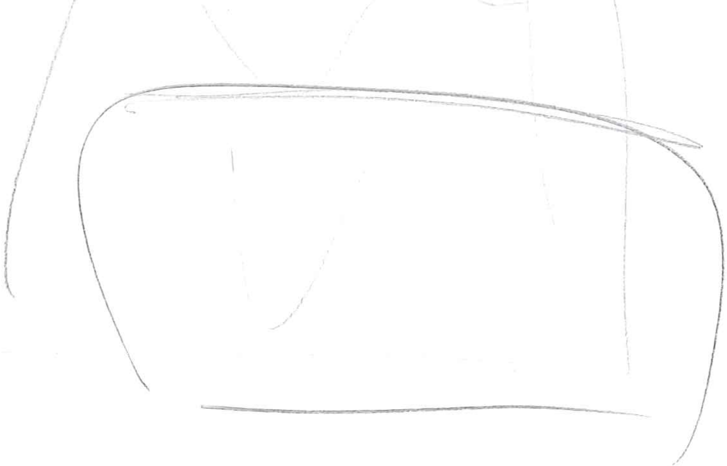
✓ Lab clean-up : When do we meet with M.S.
after June holiday

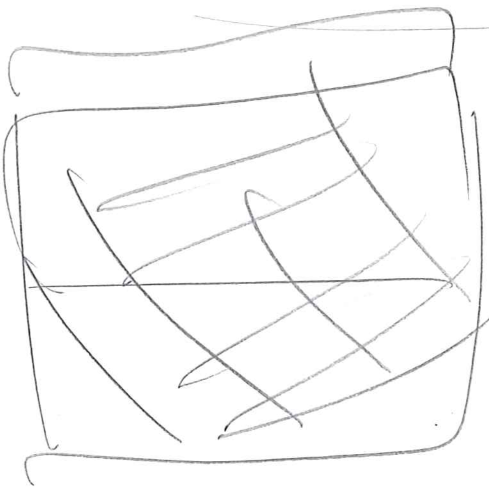
✓ October : 30.09. to 10.10. 4 days working from Germany
3 days holiday

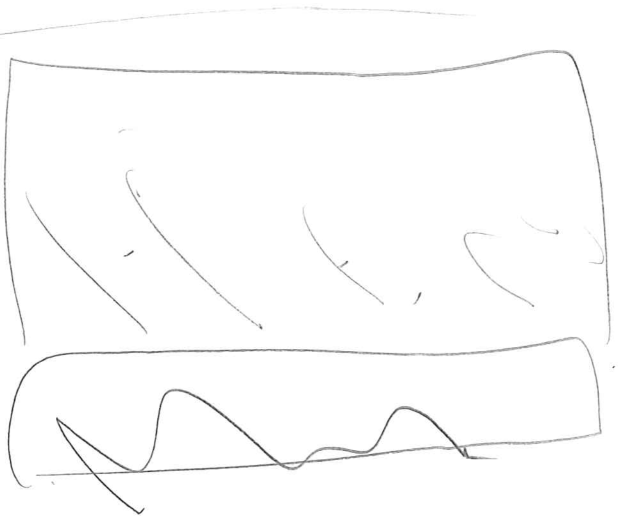
✓ CDSA . draft by 20th evening
make summary of 3+2 form



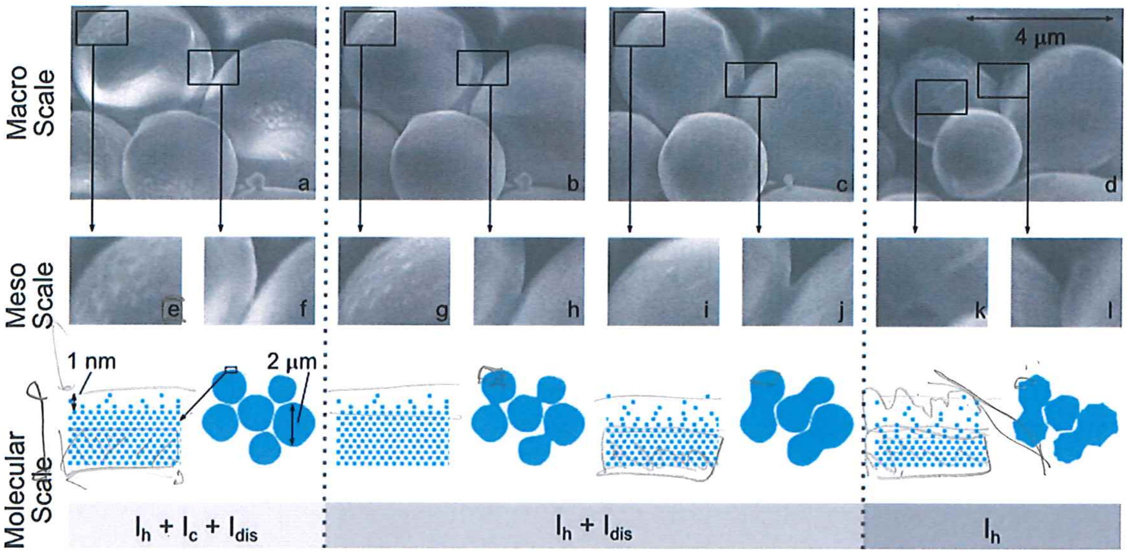








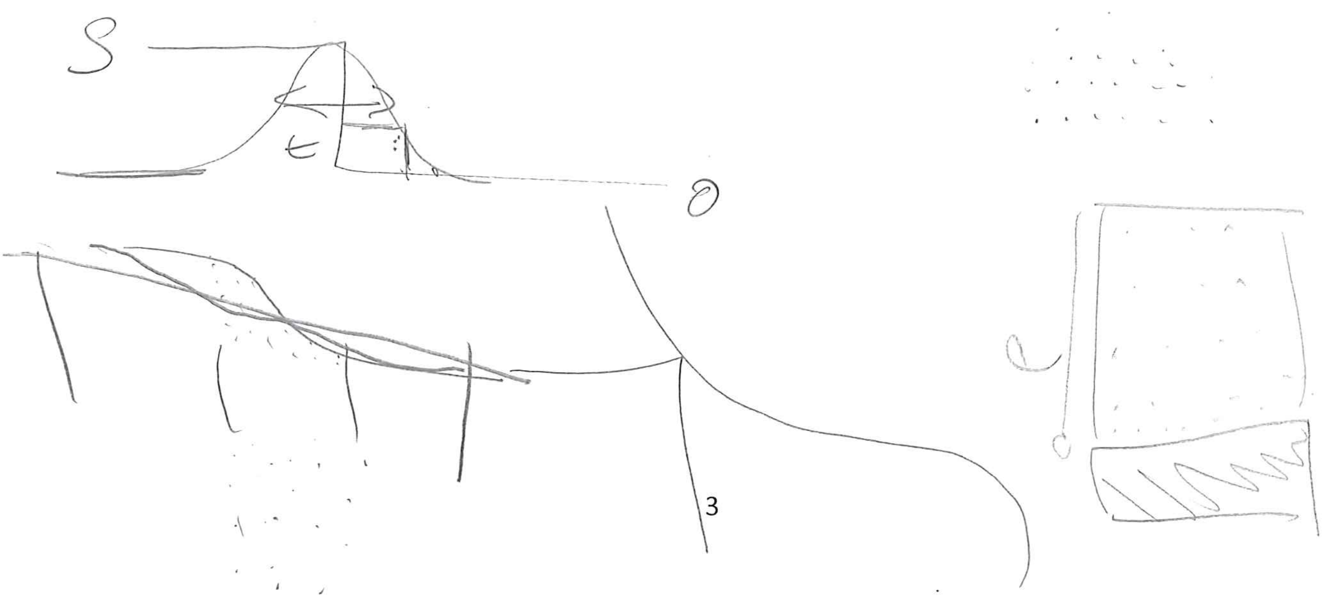
Summary



- Low T: particles almost spherical with bumps and diffuse interface (≈ 2.5 bilayers).
- T-increase: number of bumps gets less, only minor changes in diffuse interface, slow onset of sintering, disappearance of I_c.
- Further T-increase: bumps gone, onset of rapid sintering, increase of diffuse interface thickness, outer molecule become more mobile -> liquid like layer, disappearance of I_{dis}, larger scale surface features appear, particles less spherical.

Key Conclusions

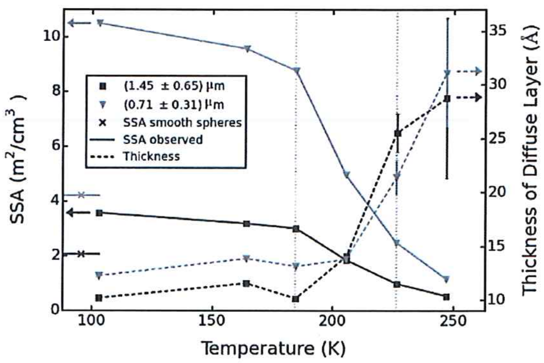
- Particles always crystalline -> all collision experiments so far with crystalline particles.
- Drastic changes in particle surface and composition in temperature range around 200 K -> probably explains previously observed changes in physical properties (stickiness etc.).
- Sintering happens at low temperatures as well.
- conjecture to what would happen to "T points" @ lower P
- Increase in stickiness explainable by surface wetting -> in experiments take care to have high vacuum to avoid surface wetting.
- How are the particles similar/different to those used in other collision experiments?
- Surface wetting helps stickiness more than "frosty" bumps.



estimate amount of sample that is surface ~> how much can it contribute to scattering

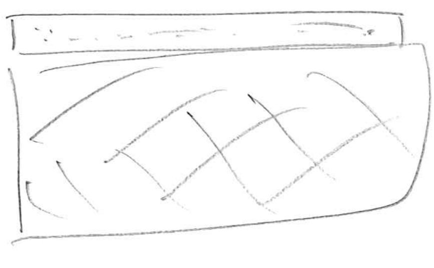
Surface

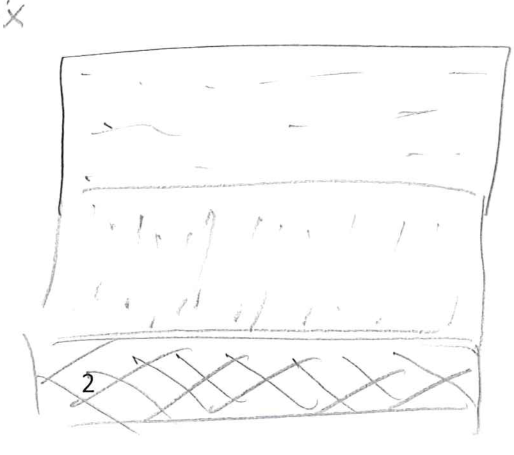
- Analysis of low-Q data
- Standard method would be two analyses (fit SSA to region that best follows Porod law, determine Porod exponent by fitting slope)
- Porod (beta) indicated diffuse interface
- Chose different fit function (to background corrected data):
  $I(Q) = 2\pi * (\Delta\rho)^2 * SSA * Q^{-4} * \exp(-Q^2 t^2)$
  $\Delta\rho$ = scattering length density difference (footnote to say that $\Delta\rho = \rho(D_2O) - 0$)
  SSA = specific surface area
  t = thickness of diffuse interface
- One analysis over whole low Q range instead of two analyses over parts of the range Fits shown in enlarged view of low-Q in fig 1 (top left graphs, dashed lines).
- SSA plot shows average for each particle size, uncertainties propagated from fit uncertainties (state typical relative uncertainty in %)
- SSA for smaller particles bigger (expected).
- Calculated SSA values indicated on graph
- SSA starts above smooth sphere value decreases below smooth sphere value
- Absolute values of SSA with potential systematic error (H_2O issue), trend not affected by that.
- SSA trend with T very similar for different particle sizes.
- At this point can't tell whether it's surface changes or sintering
- We'll come back to this later (fig 4)



- Thickness of interface starts off at ≈ 2.5 bilayers, increases to ≈ 7.5 bilayers at high T.
- Some careful sentence saying that we averaged Dec and Feb runs, but thickness data showed similar trends between the two runs, but more pronounced in one case than the other, so data are average here for clarity.
- Error bars represent standard deviation of mean
- Trend matches trend in beta, not quite inverse to SSA trend
- Not surface changes alone are responsible for SSA change

show individual data in appendix





Color code

changes to the story

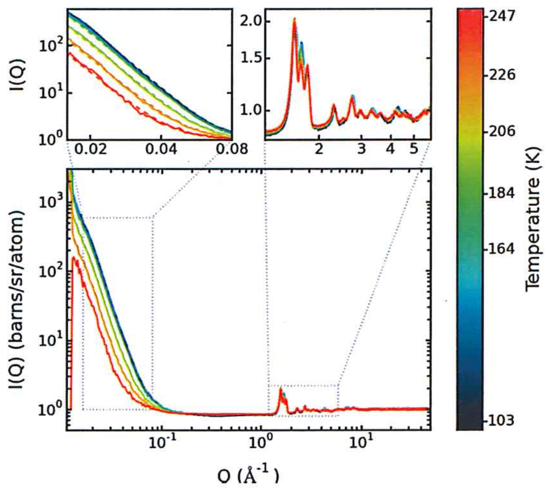
nothing new

Key Questions

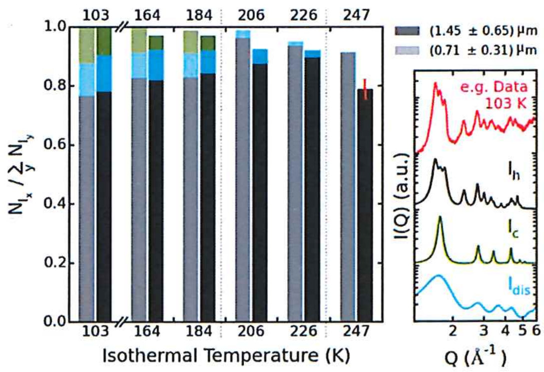
- Planet formation -> collision experiments -> hard to compare because of different p-T conditions
- What makes ice particles stick? Hypothesis, based on previous literature (Bridges / Hill / Gundlach etc): frosting, surface changes, sintering, ice phase? Influence of p-T conditions of local environment?
- Comparison to atmospheric chemistry literature: snow and hail studies (p-T range)
- What ice phase are the particles made of?
- What does their surface look like?
- How do both change with temperature? (@ fixed P)
- Can those changes explain the observed changes in stickiness?

Raw Data

- I(Q) changes with T.
- Changes look very similar for all samples. (Size & run independent)
- Slope in low-Q: Granular material
- Low-Q intensity decreasing with increasing T.
- Low-Q tells about particle surface.
- More detailed analysis later.
- High-Q peaks: tell us phase of material, probe molecular bonding: crystalline material.
- Changes in peak intensities with T.



Ice Phase



- Analysis of high Q data
- Peaks tell us about ice phases.
- Work out approx. composition from peak intensities.
- Stacking disordered ice (I_h, I_c, I_{dis}).
- Composition changes with T.
- First I_c decreases, then I_{dis}.
- Critical isothermal steps for change of ice composition: 184 K, 226 K.
- Loss of material from ice phase for high T.
- No significant difference between particle sizes within errors and accuracy of fitting

Icy Particles

I still have lots of questions about how to improve the paper, i.e. the comments I got. However, after I finally have the comments from the ISIS team, I think there will be a major change of story for the paper, so we should get this right first.

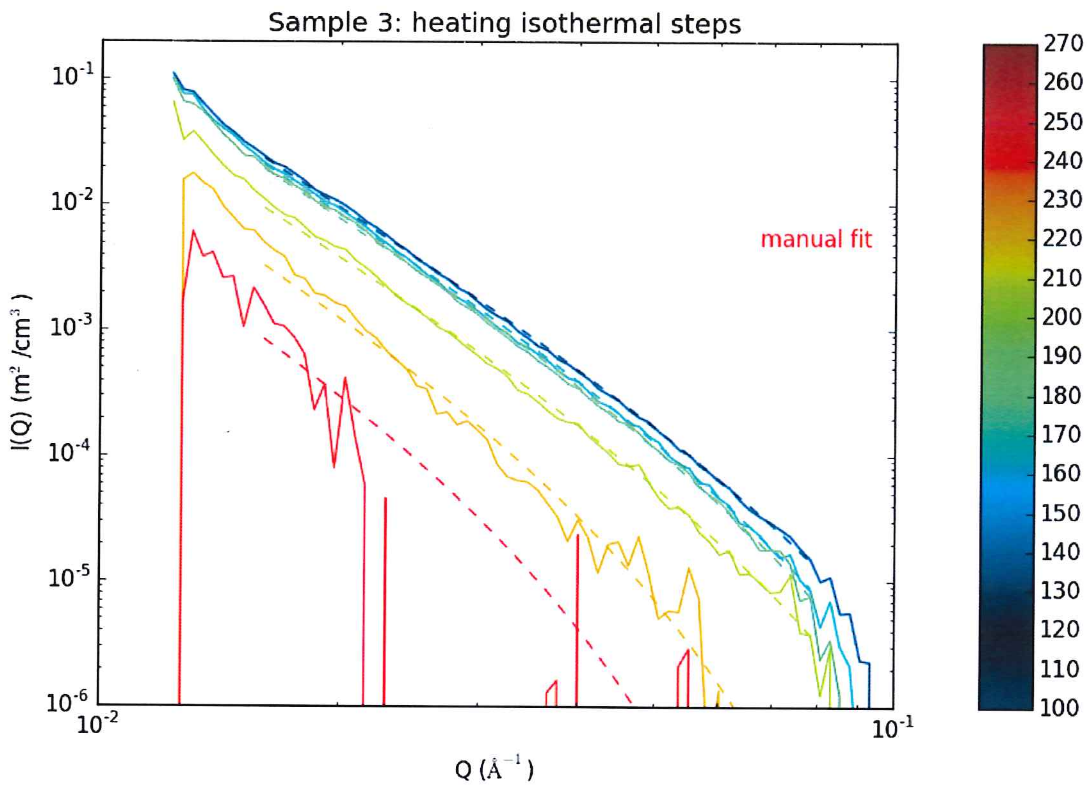
Tom said, that the tendril thing we found in the Hiemenz book, does not apply. They are talking about mass fractals and that fractal dimension must not be used in $\beta = 6 - D$. Also, the tendrils would drastically increase the observed SSA, i.e. our values would still be far lower than the expectation.

He suggested to only use the diffuse interface interpretation for the whole $\beta > 4$ range. He also found a paper (*1991 Strey: Small Angle Neutron Scattering from Diffuse Interfaces – 1. Mono- and Bilayers in the Water-Octane-$C_{12}E_5$ System* http://pubs.acs.org/doi/pdf/10.1021/j100172a070), which has a model to describe the scattering from diffuse interfaces via the specific surface area (SSA) of the sample and the effective thickness (t) of the diffuse layer.

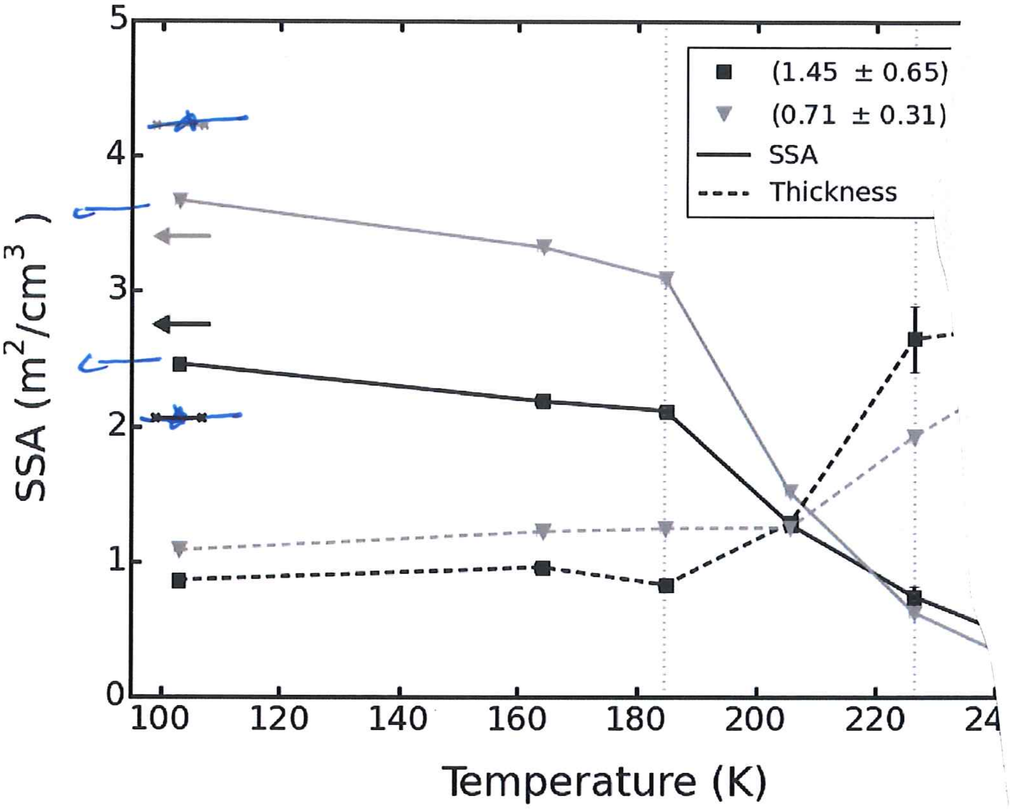
$$I(Q) = 2\pi * (\Delta\rho)^2 * SSA * Q^{-4} * \exp(-Q^2 t^2) + \text{background}$$

$\Delta\rho$ = scattering length density difference, the background term can be omitted, when using the .mint01 files.

I've tried this on our data and get very nice results - mostly. Results from a quick analysis with this method are shown below.



Example of the fits for Dec sample 3 (smaller particles, good run)



Summary of the SSA and layer thickness results for two runs on each particle size

x---x marks the expected SSA values for the individual particle sizes.

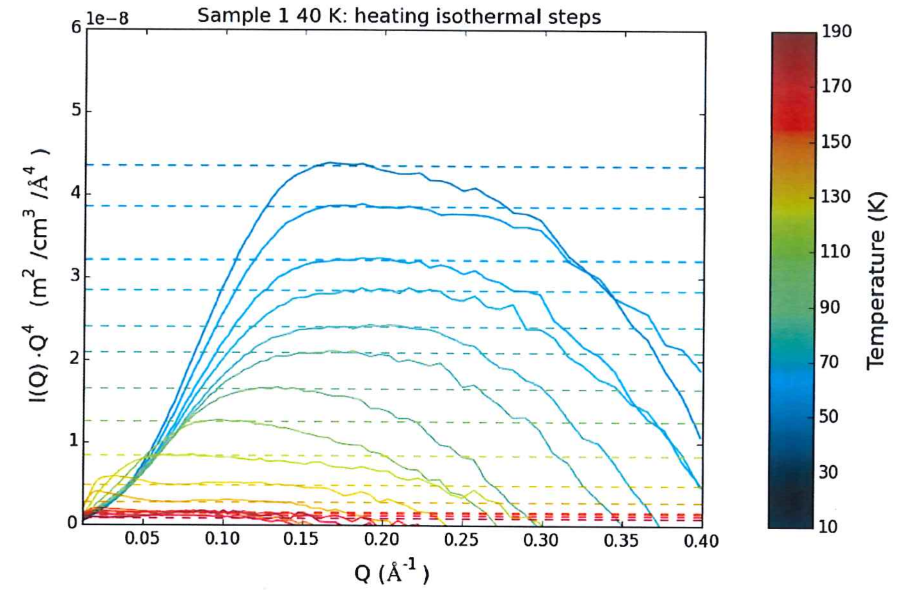
Some of the thickness results seem to jump a bit, so I will look into these fits again in m
Overall, I think this analysis makes much more sense than the two individual analyses I d

However, that means we have no clue what the bumps are, where they come from and w
disappear. Can we discuss this at some point soon?

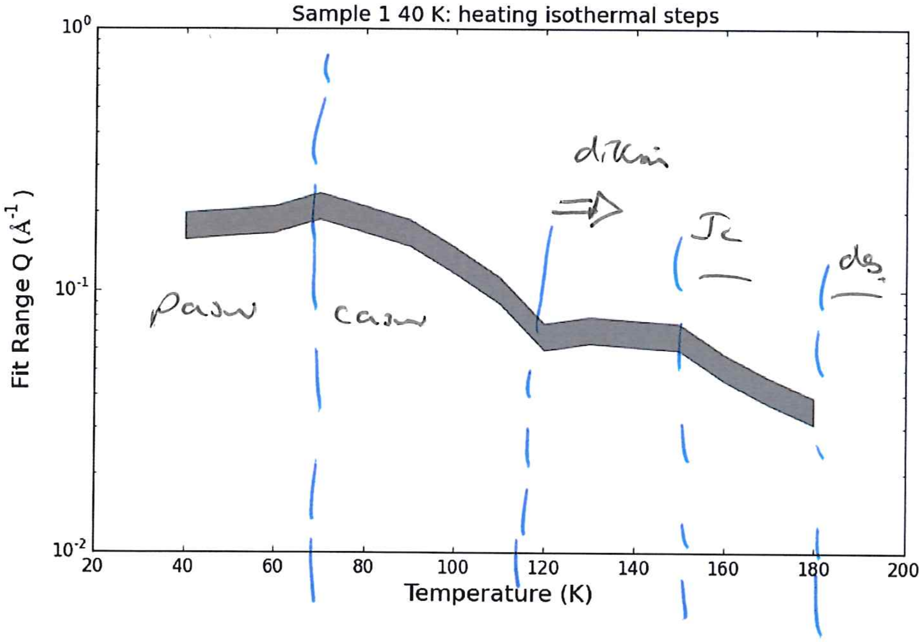
ASW data

Porod Range

I've plotted the window that my fitting routing chooses for the determination of the Porod constant (-> SSA) against temperature. With increasing temperature it is moving towards smaller Q-values.



Plot of the Porod constant fits to I(Q)Q^{-4} for sample 1.*



Plot of the range where I(Q) best follows Porod law (Q^{-4}) versus temperature.

Note: the width of the fitting window is fixed to 20 data points (width of shaded area in plot). Only the starting position of this window is varied.

vary window width and see how that changes the picture

SSA

Estimate of SSA for compact homogeneous sample

If we had deposited all gas used from the bottles (i.e. ignore the pumps), we would expect a specific surface area of

$$\text{SSA} \approx 0.2\,\frac{\text{m}^2}{\text{cm}^3}$$

Observation

For sample 1 (40 K deposition) we observe at 40 K:

$$\text{SSA} \approx 170\,\frac{\text{m}^2}{\text{cm}^3}$$

Comparison to literature values

Still needs to be done.

Analysis of heating ramp data

Still needs to be done.

Double Guinier-Porod Fits

Fit Function

I've tried several approaches. The one that worked in the end is the following:

For the low temperature data, use the sum of two Guinier-Porod functions (T ≤ .

$$GP_i\,(Q) = \begin{cases} G_i\; Q^{-s_i}\, e^{-\frac{Q^2 R_{gi}^2}{3-s_i}} & , Q \le Q_i \\ A_i\; Q^{-d} & , Q_i < Q \end{cases} \qquad Q_i = \frac{1}{R_{gi}} \sqrt{(\ldots}$$

$$I(Q) = GP_1(Q) + GP_2(Q)$$

For the medium temperature data use only one GP function (110 K < T ≤ 170 K).

For the highest temperature (T = 180 K) use only Porod power law ($A*Q^{-d}$).

This approach works nicely for temperatures between 40 and 90 K and between 130

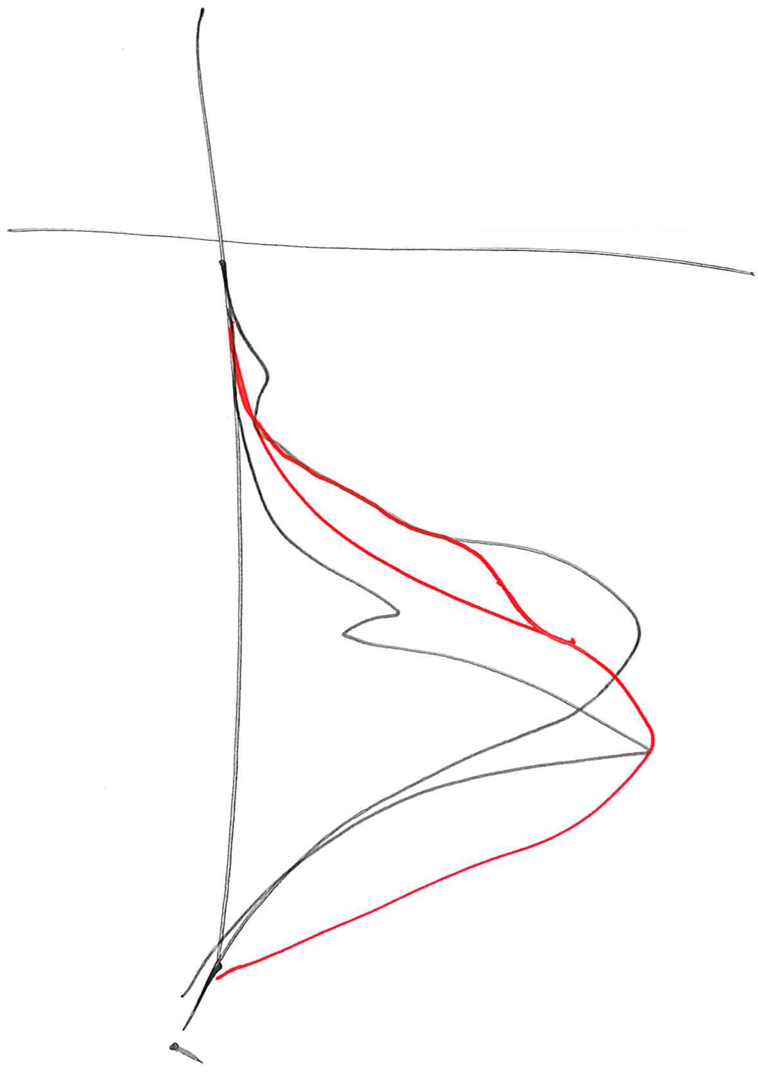
Results

For the three isothermal points 100, 110, 120 K, the fit results are a bit jumping. I prol do manual fits there.

I need only one d-parameter in all three cases of fit functions. This is scattering around Porod case.

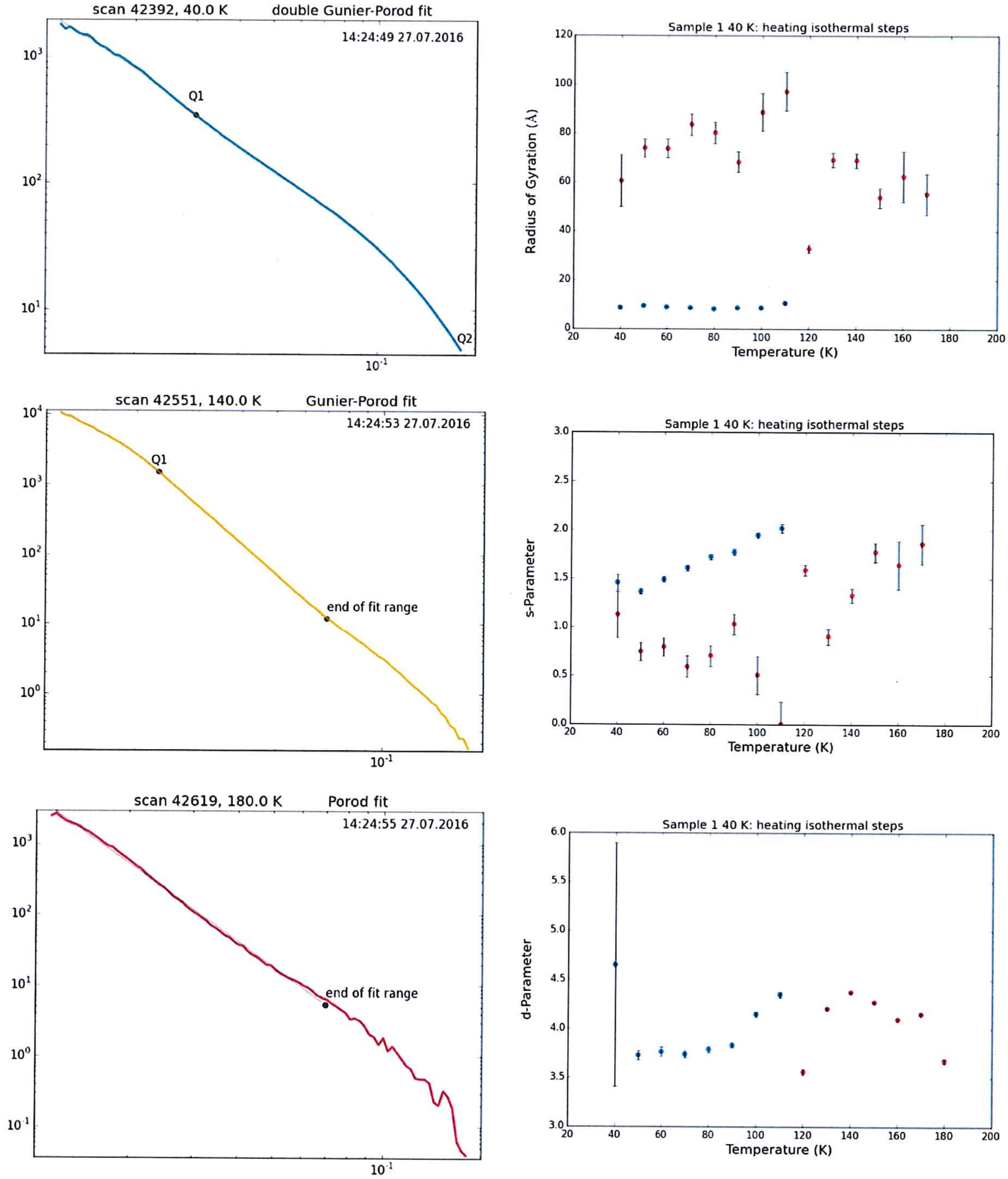
There seem to be two types of pores:

1. Small ($R_g \approx 10$ Å), start off between cylindrical and platelet shape, linearly change before disappearing, getting a bit bigger R_g just in the very last steps before disap



2. Big ($R_g \approx$ 60-70 Å), start off between cylindrical and spherical shape, change to platelet before disappearing, R_g scattering a lot, maybe getting slightly smaller before disappearing.

Note: The double-bump structure never fully disappears, but at some point the double GP fit does not work anymore. Thus I limited the fit range for the normal GP and the Porod fit to the left bump (see plots).



Left: Three examples of fits to low-Q data. Top: double Guinier-Porod, Middle: Guinier-Porod, Bottom: Porod

Right: Results for fit parameters. Top: Radius of gyration, Middle: s-parameter, Bottom: d-parameter.

28.09.2016

PDRA Meeting

Agenda:

Gant Chart + Timescales

Next steps Icy Particles paper
- 11th Oct. final draft
- how many words do we need to get rid of
- 15th Oct send to Co-authors
- 21st get back from Co-authors
- ask Jürgen for editor to send PRLs to

next : ASW paper
- very good draft before Xmas
- Jan → finish it

email dates to Helen

15.03.2017

Priorities for next months:

1. Icy Particles PRL
2. ASW data analysis & paper
3. Sedimentation chamber : quick data analysis & proposal (16.4.)
4. ASW deposition paper
5. Cold dust paper

Helen away 10.-16. April

To do: quick analysis of ASW deposition & individual scans

Icy Particles

Low-Q Analysis

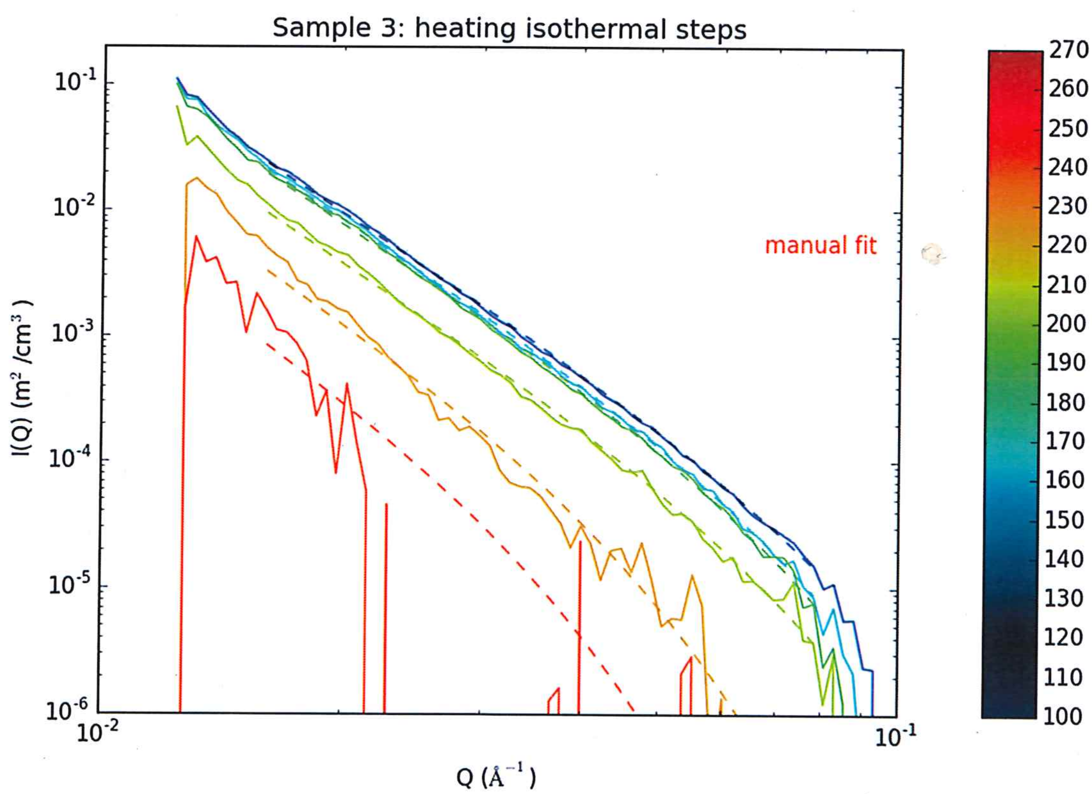
Tom said, that the tendril thing we found in the Hiemenz book, does not apply. They are talking about mass fractals and that fractal dimension must not be used in $\beta = 6 - D$. Also, the tendrils would drastically increase the observed SSA, i.e. our values would still be far lower than the expectation.

He suggested to only use the diffuse interface interpretation for the whole $\beta > 4$ range. He also found a paper (*1991 Strey: Small Angle Neutron Scattering from Diffuse Interfaces – 1. Mono- and Bilayers in the Water-Octane-$C_{12}E_5$ System* http://pubs.acs.org/doi/pdf/10.1021/j100172a070), which has a model to describe the scattering from diffuse interfaces via the specific surface area (SSA) of the sample and the effective thickness (t) of the diffuse layer.

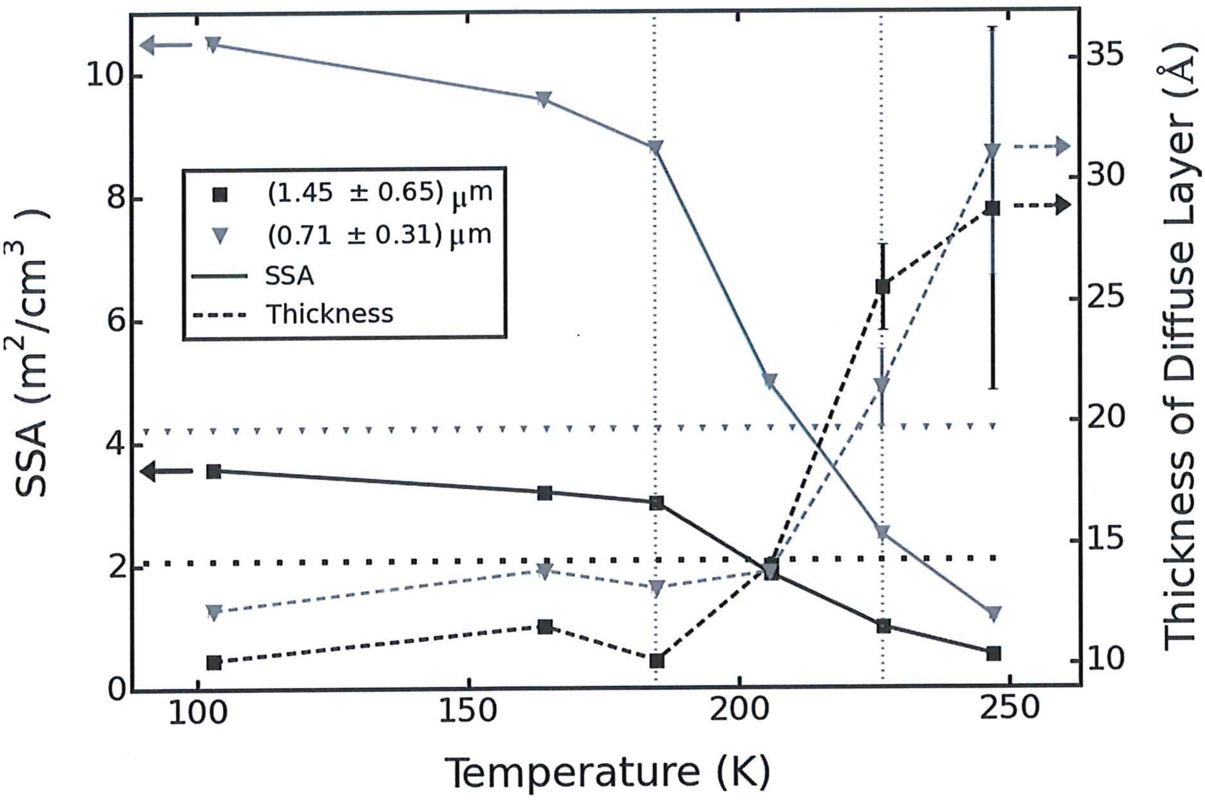
$$I(Q) = 2\pi * (\Delta\rho)^2 * SSA * Q^{-4} * \exp(-Q^2 t^2) + background$$

$\Delta\rho$ = scattering length density difference, the background term can be omitted, when using the .mint01 files.

I've tried this on our data and get very nice results - mostly. Results from a quick analysis with this method are shown below.



Example of the fits for Dec sample 3 (smaller particles, good run)



Summary of the SSA (left y-scale) and layer thickness (right y-scale) results for two runs on each particle size (average).

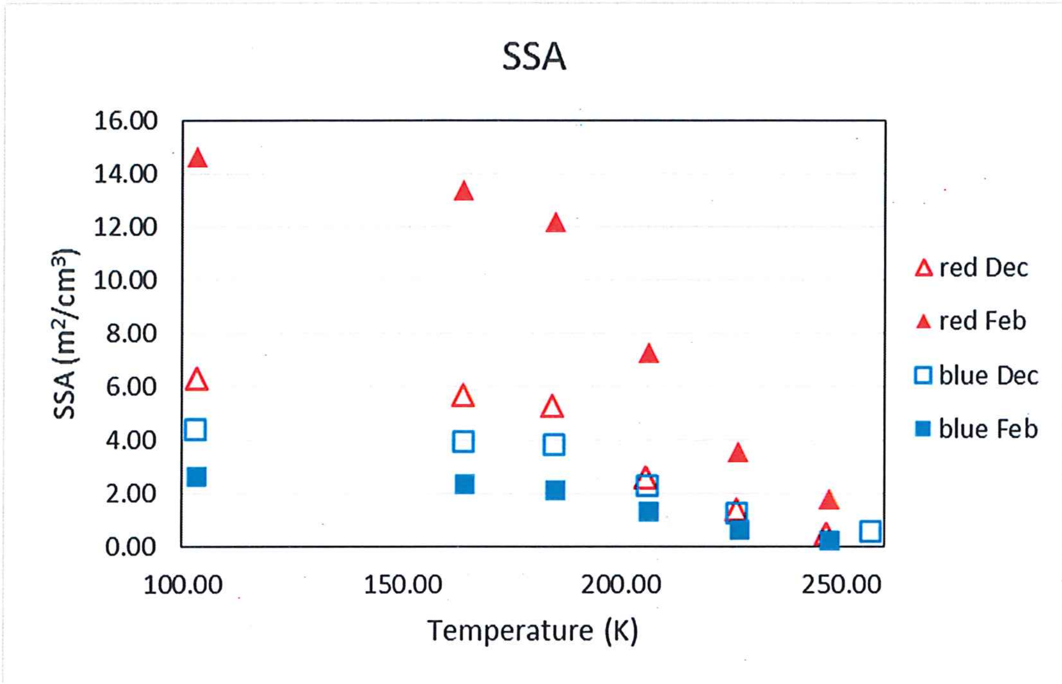
Lines of small symbols mark the expected SSA values for the individual particle sizes.

Some of the thickness results seemed to jump a bit, so I looked into these fits again in more detail (see next section). Overall, I think this analysis makes much more sense than the two individual analyses I did before.

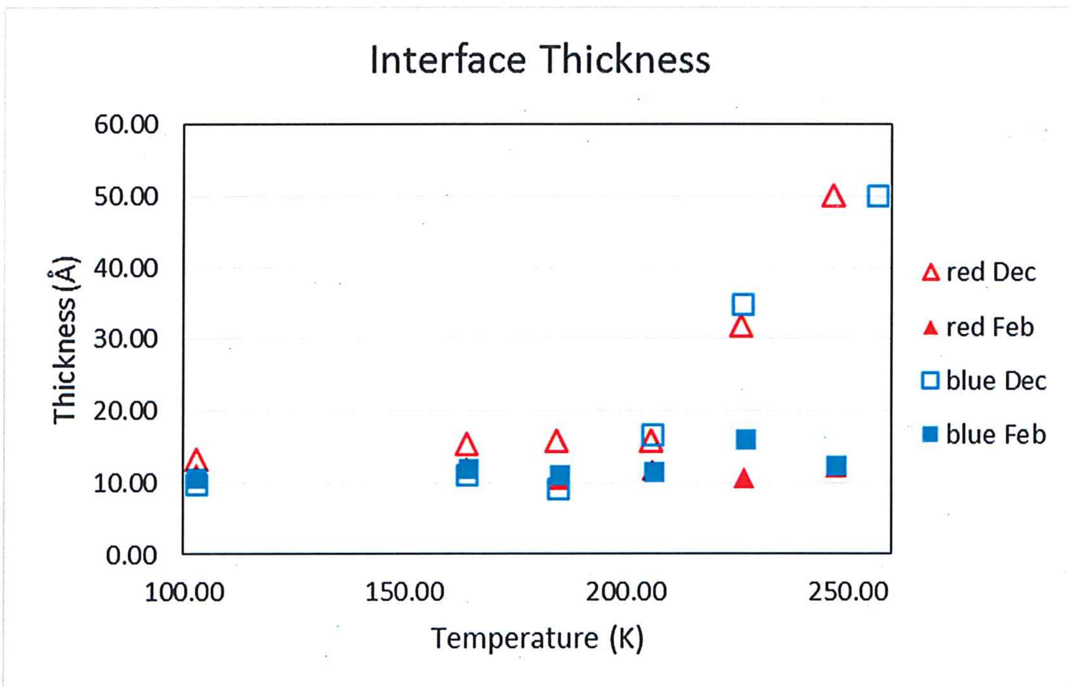
However, that means we have no clue what the bumps are, where they come from and why they disappear.

Individual Samples

On a closer look, I've noticed that there is a difference in the surface changes between the two runs. The SSA is still showing more or less random absolute values due to the H_2O issue. However, the thickness of the diffuse interface shows a drastic increase with temperature for the Dec samples and basically no change for the Feb samples.



Specific surface area versus temperature for the individual samples.



Interface thickness versus temperature for the individual samples.

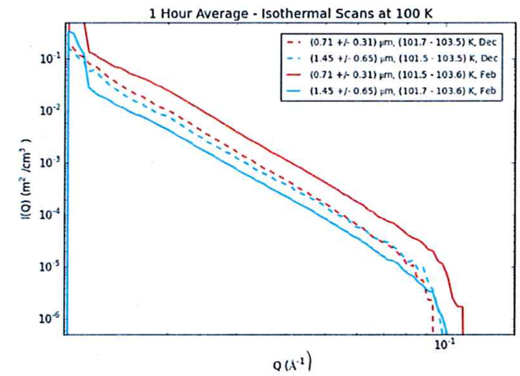
I can't think of anything that we changed in the treatment of the particles. My first assumption would be that maybe the pressure in the bin was a bit different, but that isn't logged anywhere. Also it would be really weird to see such a drastic effect of slight pressure changes.

There seems to be no correlation with the SSA differences between the two runs.

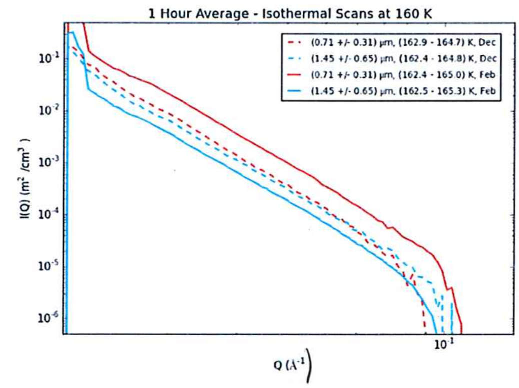
Isothermal Steps

Comparison of the four samples' I(Q) at each isothermal step.
Dashed lines: Dec run, solid lines: Feb run.

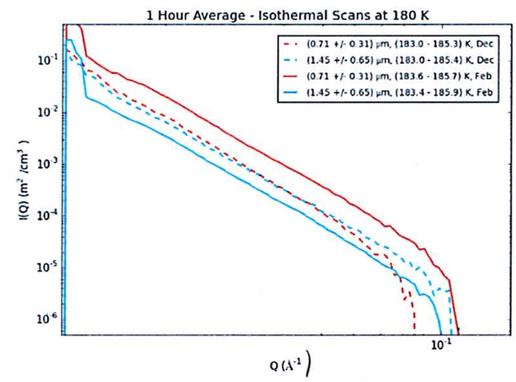
100 K



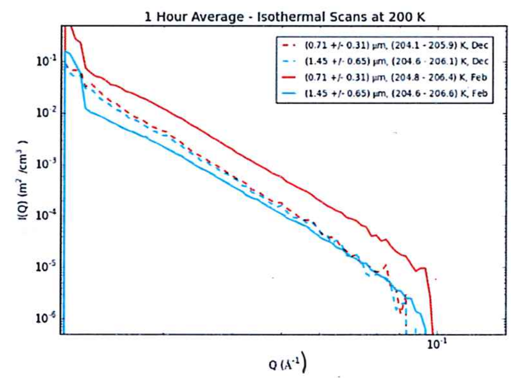
160 K



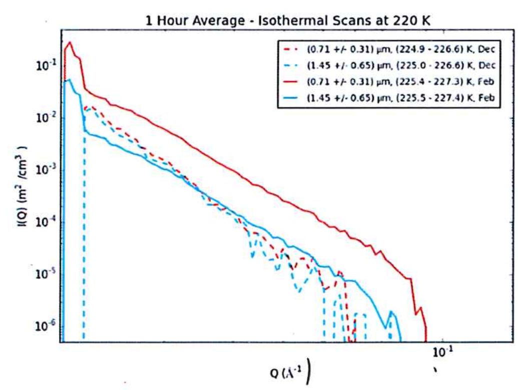
180 K



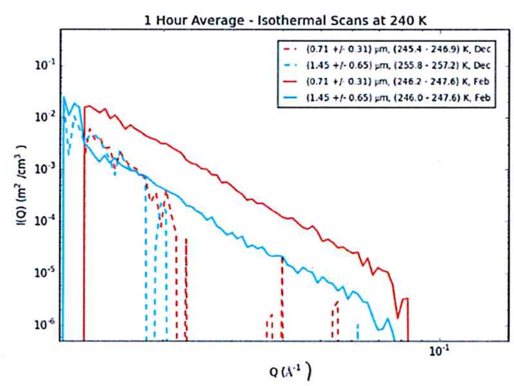
200 K



220 K



240 K



Gudrun Processing

The only difference in processing the data (apart from individual tweak factors for each sample) is:

Dec: One background scan for empty cell NIMROD00039593.raw

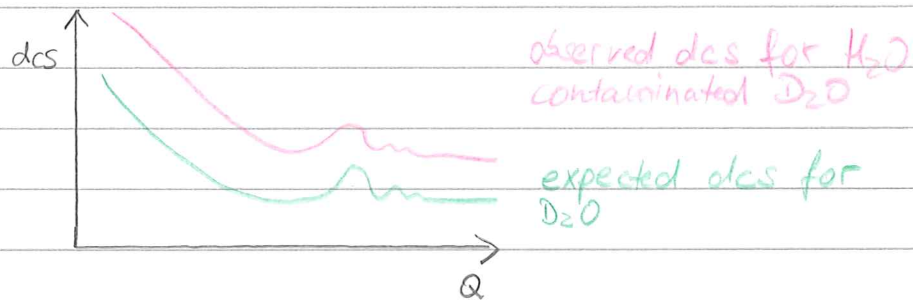
Feb: Two background scans for empty cell NIMROD00040992.raw, NIMROD00040993.raw

01.11.2016

ISIS on Icy Particles - Data Analysis - SSA

$I(Q) = 2\pi(\Delta\rho)^2 \cdot SSA \cdot Q^{-4} \cdot e^{-Q^2 t^2}$	fit function
$I(Q) = dcs \cdot \tilde{\rho} \cdot \cancel{tweak} \cdot 10^{-4}$ ↑ density ↑ 13.01.2017 ↑ unit conversion	dcs to $I(Q)$ conversion

H_2O influence:



⇒ Gudrun would suggest, the dcs level was too high, i.e. the amount of sample in the beam higher than entered in Gudrun. This would then be corrected by using a higher tweak factor, i.e. having less sample in the same volume.

⇒ In this case, the derived $I(Q)$ would be too high.
⇒ The SSA would be too high.

Basti worked out, that the maximum contamination in a non-purged atmosphere would be $\approx 15\%$ of H_2O.

This would scale up the tweak factor by $\approx$ a factor of 2.

A contamination of $\approx 4\%$ would scale up the tweak factor by ≈ 1.3.

02.11.2016

Expected SSA for sample of size distributed spheres:

Average surface:

$$A = \sqrt{\frac{\pi}{2\sigma_d}} \int_0^\infty d^2 \, e^{-\frac{(d-d_0)^2}{2\sigma_d^2}} \, dd$$

(Integrals solved by Wolfram Alpha)

$d_0 = 2 \cdot 1.45\,\mu m$, $\sigma_d = 2 \cdot 0.65\,\mu m$ $\Rightarrow A = 36.1605\,\mu m^2$

$d_0 = 2 \cdot 0.71\,\mu m$, $\sigma_d = 2 \cdot 0.31\,\mu m$ $\Rightarrow A = 5.86646\,\mu m^2$

Average volume:

$$V = \frac{1}{6}\sqrt{\frac{\pi}{2\sigma_d}} \int_0^\infty d^3 e^{-\frac{(d-d_0)^2}{2\sigma_d^2}} \, dd$$

$d_0 = 2.9\,\mu m$, $\sigma_d = 1.3\,\mu m$ $\Rightarrow V = 23.3411\,\mu m^3$

$d_0 = 1.41\,\mu m$, $\sigma_d = 0.62\,\mu m$ $\Rightarrow V = 1.82631\,\mu m$

Average SSA:

$$SSA = \frac{A}{V}$$

$d_0 = 2.9\,\mu m$, $\sigma_d = 1.3\,\mu m$ $\Rightarrow \boxed{SSA = 1.5492\,\frac{m^2}{cm^3}}$

$d_0 = 1.41\,\mu m$, $\sigma_d = 0.62\,\mu m$ $\Rightarrow \boxed{SSA = 3.2122\,\frac{m^2}{cm^3}}$

13.01.2017

ISIS data analysis SSA & tweak factor

Conversion from dcs to $I(Q)$: $\boxed{I(Q) = \text{dcs} \cdot \underbrace{\tilde{\rho}}_{\text{density}} \cdot \underbrace{10^{-4}}_{\text{unit conversion}}}$

As long as Gudrun-output is set to barn/sr/atom, the tweak factor does <u>not</u> need to be included in $I(Q)$. (Density in $\frac{\text{atoms}}{Å^3}$)

fit function: $\boxed{I(Q) = 2\pi \underbrace{(\Delta\rho)}_{\text{scattering length density difference}}{}^2 \cdot SSA \cdot Q^{-4} \cdot e^{-Q^2 t^2}}$

H_2O influence for icy particles: