tof-SANS Data Reduction – an introduction and observations regarding software provision

Richard Heenan

ISIS Facility,

Rutherford Appleton Laboratory

richard.heenan@stfc.ac.uk

Need to produce absolute scattering probabilities, allowing for all appropriate neutron wavelength and geometry dependent corrections. Every step below requires software.

"Routine reduction" for users should hide all the horrible steps and be FAST.
Calibration software for instrument scientists can be less tidy, even scripted, but still requires significant effort to write.

- What to measure and why – SANS of sample, can & possibly background; transmissions.
- Link data collection & reduction scripts for "standard experiments", batch processing.
- Normalisation to incident flux, per neutron pulse?
- Calibration & survey measurements for detector efficiency and position encoding.
- Scattering geometry effects & gravity.
- Q resolution optimisation & estimation.
- Poor λ overlap, multiple scatter – warn / correct ?
- Detector dead time – warn / correct ?
- Approximate real time I(Q) on the fly.

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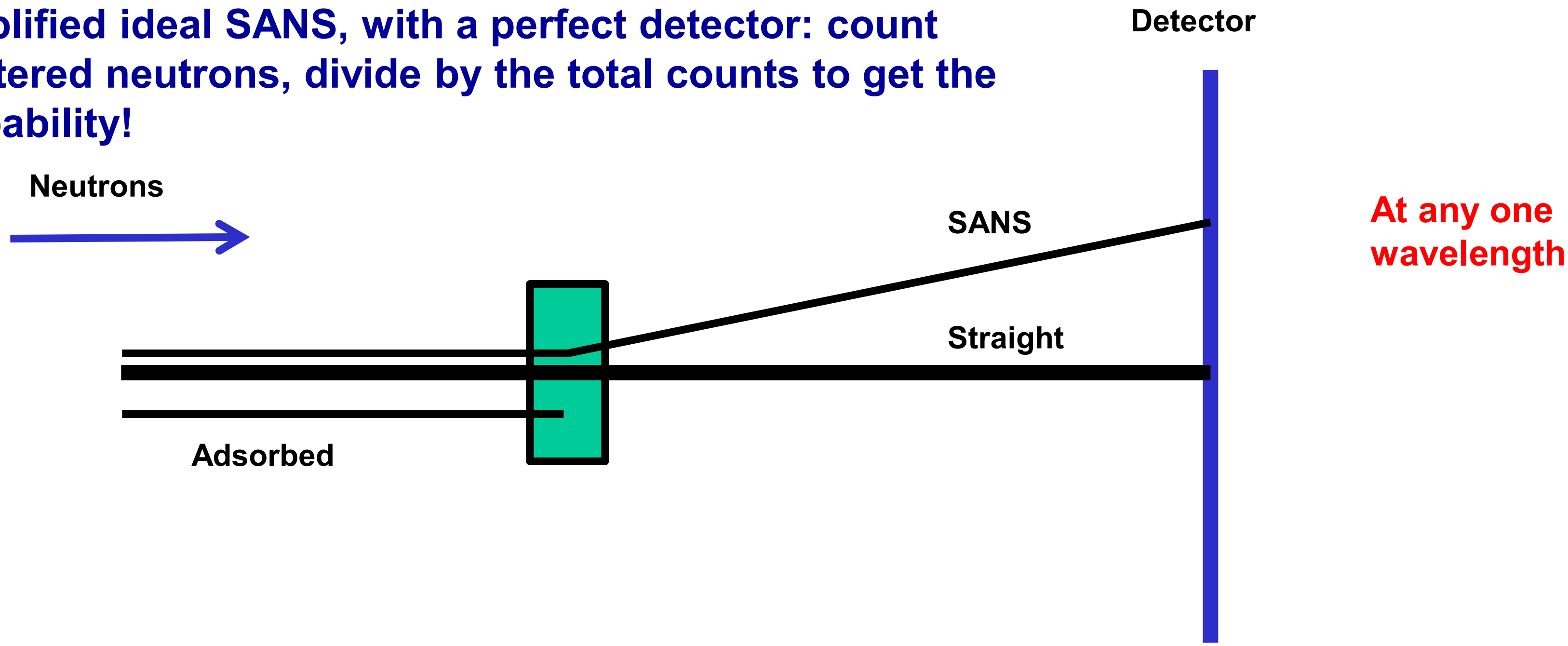
Simplified ideal SANS, with a perfect detector: count scattered neutrons, divide by the total counts to get the probability!



$$\frac{\partial\Sigma_{SANS}(Q)}{\partial\Omega}=\frac{SANS(Q)}{\int Straight+\int SANS}$$

= Probability in the absence of any other processes, we know how to calculate this from theory, so can fit a model.

$$=\frac{Counts(Q)-Straight(Q)}{\int Straight+\int SANS}$$

No transmission measurement or detector efficiency is needed
(until we get to larger scattering angles!)
Alas real detectors do not have the dynamic range and spatial uniformity required!
Alas there are also some other scattering processes to consider!

We know how to calculate the neutron scattering probabilty.
e.g. for dilute particles in solution, N particles per unit volume,

$$\frac{\partial \Sigma_{SANS}(Q)}{\partial \Omega} = I(Q) = NV^2(\rho_{particle} - \rho_{solvent})^2 F^2(Q,R)$$

Concentration Φ =NV,
volume V

Composition

size & shape

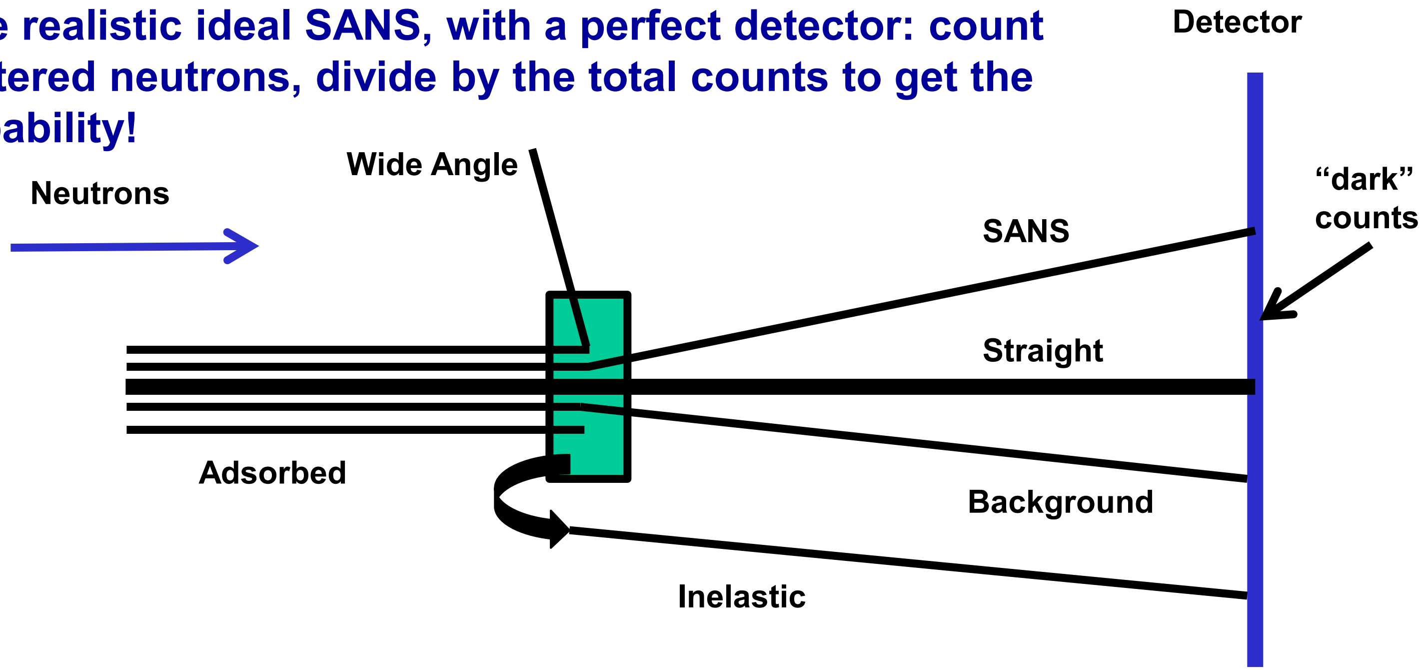
= Probability in the absence of any other processes

We would like to get identical results from a “mirror image” experiment.
e.g.
Hydrogenated particle in D_2O – high transmission, low background

Mirrored by:
Deuterated particle in H_2O – “low” transmission, high incoherent & inelastic background

In all cases we *ignore* any neutrons that are adsorbed, or contribute to wide angle diffraction, incoherent or inelastic scattering, as we do not care about, nor can we easily calculate, the probability of those events.

More realistic ideal SANS, with a perfect detector: count scattered neutrons, divide by the total counts to get the probability!



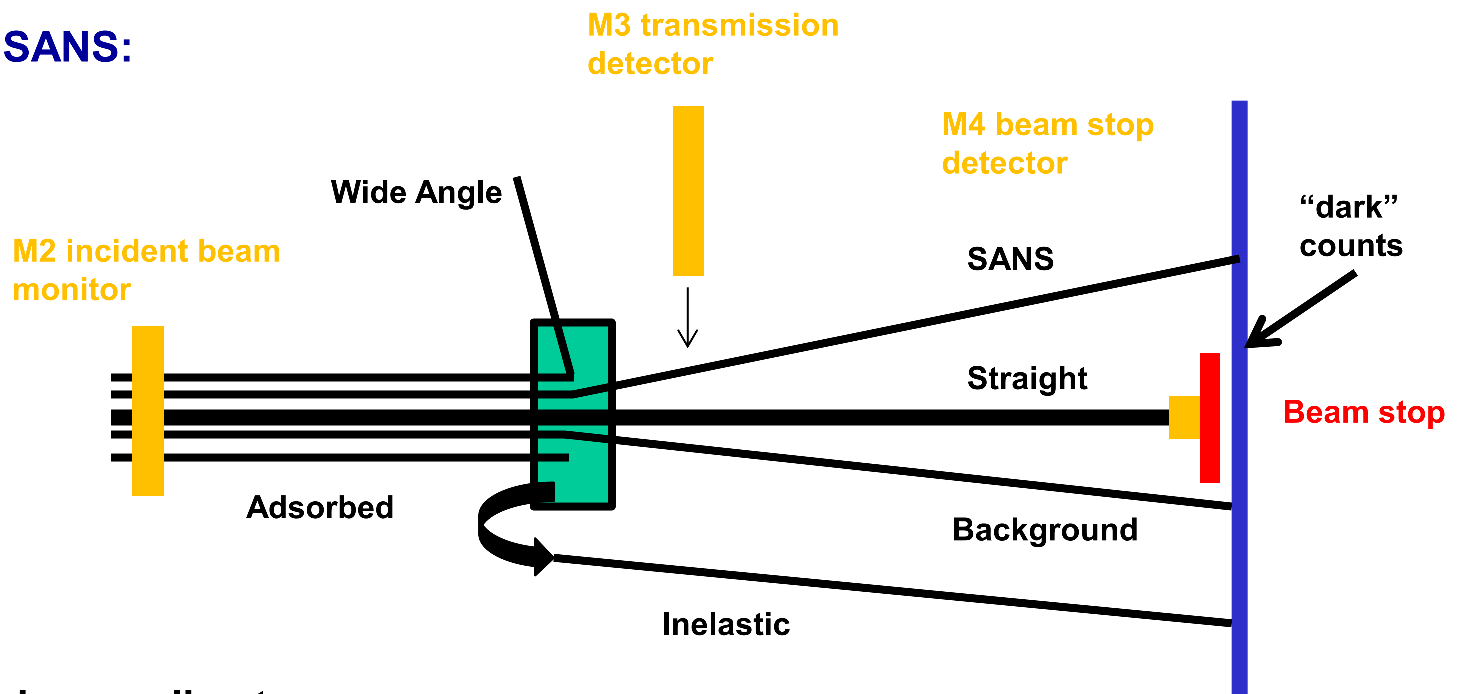
$$\frac{\partial \Sigma_{SANS}(Q)}{\partial \Omega} = \frac{SANS(Q)}{\int Straight + \int SANS}$$

= Probability in the absence of any other processes

$$= \frac{Counts(Q) - Background(Q) - Inelastic(Q) - Dark(Q) - Straight(Q)}{\int Straight + \int SANS}$$

No transmission measurement or detector efficiency is needed
(until we get to larger scattering angles!)
Alas real detectors do not have the dynamic range and spatial uniformity required!
The above slightly glosses over the Can or Cell or Background subtraction …

Real SANS:



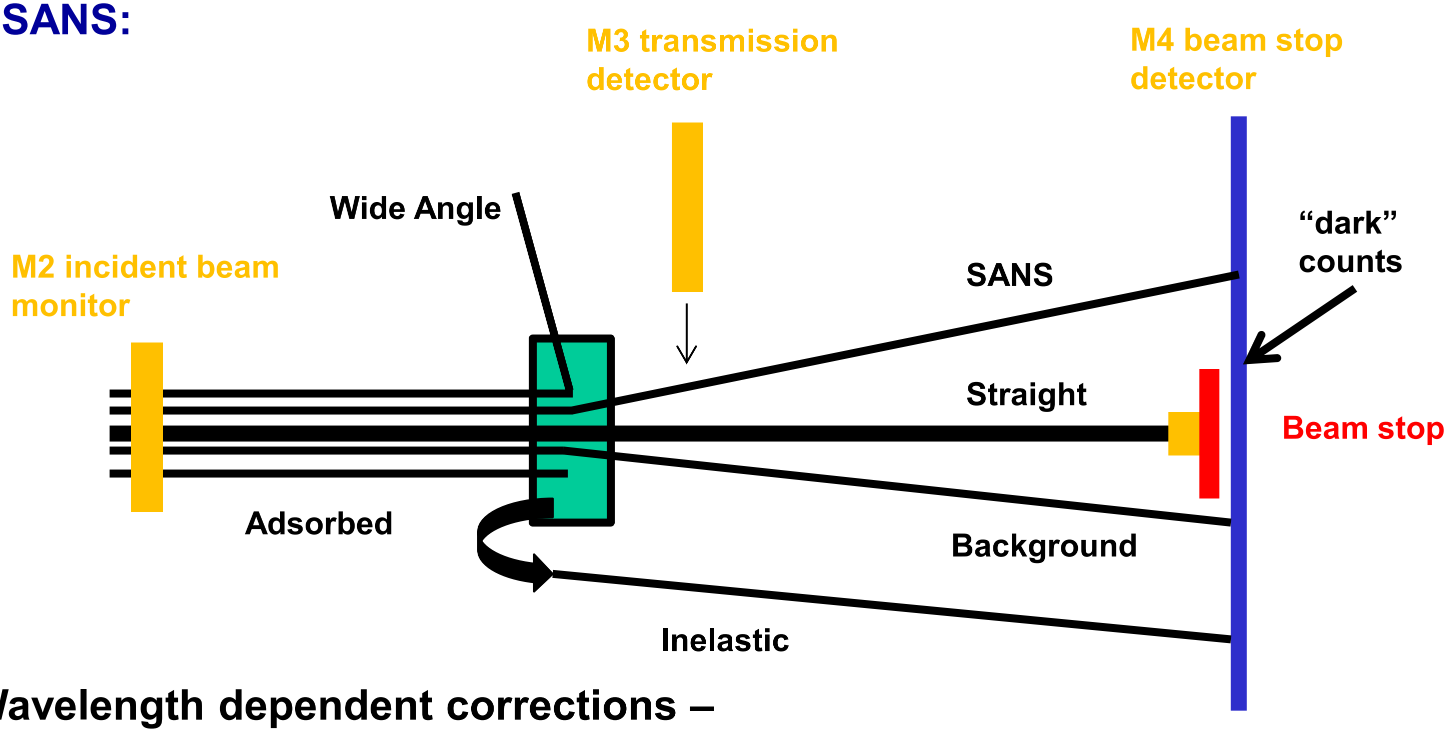
Could normalise to:

beam stop detector (M4) or a hole in beam stop – gravity issue at long L2
[monitor after sample – oops it scatters]
monitor before sample (M1 or M2) – good, but then explicitly need transmission (using M3 or M4)

Each needs relative efficiency D(λ) of monitor to main detector.
E.g. attenuate beam, remove the beam stop.
Accelerator based tof we must allow for variations in moderator temperature or proton current.

Monitors M2, M3 and M4 may not sample uniformly, nor may "beam stop out" transmissions.

Real SANS:



Wavelength dependent corrections –

- **Incident spectrum (possibly per pulse?)**
- **Transmission**
- **Relative efficiency of detectors.**
- **"wide angle" geometry**

At fixed wavelength – only a scalar error,
in tof – the shape of the merged I(Q) could be wrong.

Software – Mantid – deals separately with corrections depending on:

- **geometry**
- **wavelength**
- **geometry & wavelength**

So now we have to jump through some hoops to get the cross normalisation to work.
Start with the definition of elastic scattering cross section (probability):

$$Counts(R,\lambda) = I_0(\lambda)\frac{\partial\Sigma(Q)}{\partial\omega}\Omega(R)t.T(\lambda)\eta(\lambda)$$

R – radius on detector
t – sample thickness
T – transmission
η – detector efficiency

Incident flux:

$$I_0(\lambda) = \frac{M(\lambda)}{\eta_M(\lambda)}$$

M – incident beam monitor
C – neutron counts
Ω – solid angle
A – beam area
$V_{sam} = A_s t$ – sample volume

Wavelength λ is proportional to arrival time at detector.
Need ratio of main detector efficiency compared to monitor. e.g.
Remove beam stop and put a small hole A_H at the sample to record:

$$D(\lambda) = \frac{C_H(\lambda)}{M_H(\lambda)} = \frac{\eta(\lambda)}{\eta_M(\lambda)}\frac{A_H}{A_S}$$

D(λ) "direct beam" allows us to cross-normalise the incident spectrum to that empty beam seen on the main detector.

Rearranging:

$$I(Q) = \frac{\partial\Sigma(Q)}{\partial\Omega} = \frac{A_H \quad \sum_{R,\lambda\subset Q} C(R,\lambda)}{A_S t \sum_{R,\lambda\subset Q} M(\lambda)T(\lambda)D(\lambda)\Omega(R)}$$

Numerator sums counts in a time and space "Q bin".

Proper statistics are *not* obtained by "averaging the reduced data from a series of wavelengths" .

P.A.Seeger & R.P.Hjelm J.Appl.Cryst. 24(1991)467-478

In reality we find the absolute *Scale* factor from a standard sample (a coherent scatterer, not H_2O).

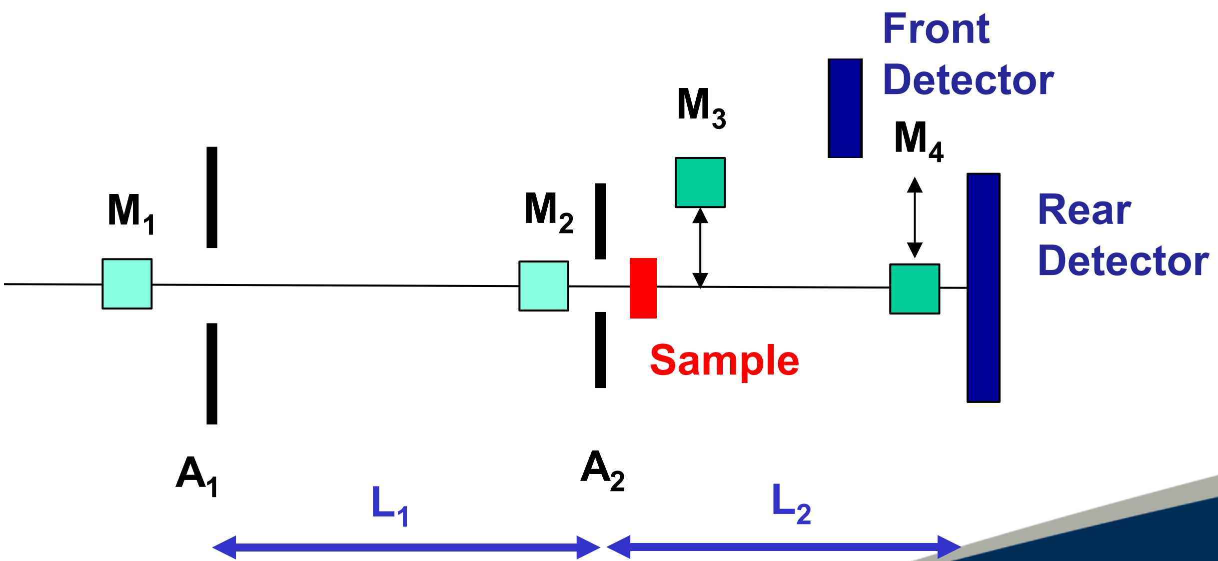
$$I(Q) = \frac{Scale}{V_{SAM}} \frac{\sum_{R,\lambda \subset Q} C(R,\lambda)}{\sum_{R,\lambda \subset Q} M(\lambda) T(\lambda) D(\lambda) \Omega(R)}$$

Have to know spatial coordinates for every detector pixel! Needs software.

ASSUME η(λ) is the same over the whole detector, and incorporate a flood source measured scalar efficiency (or area) correction for each pixel into Ω(R). Needs software.

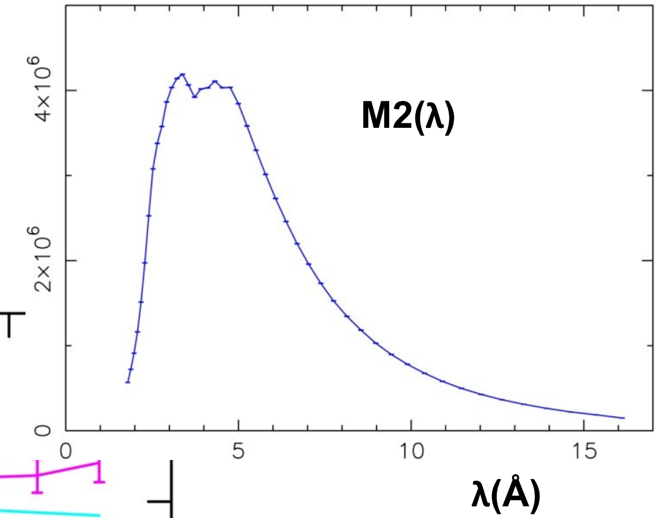
If is difficult to experimentally measure a good D(λ), so we may adjust it empirically with iterative reductions at different λ for a standard sample.

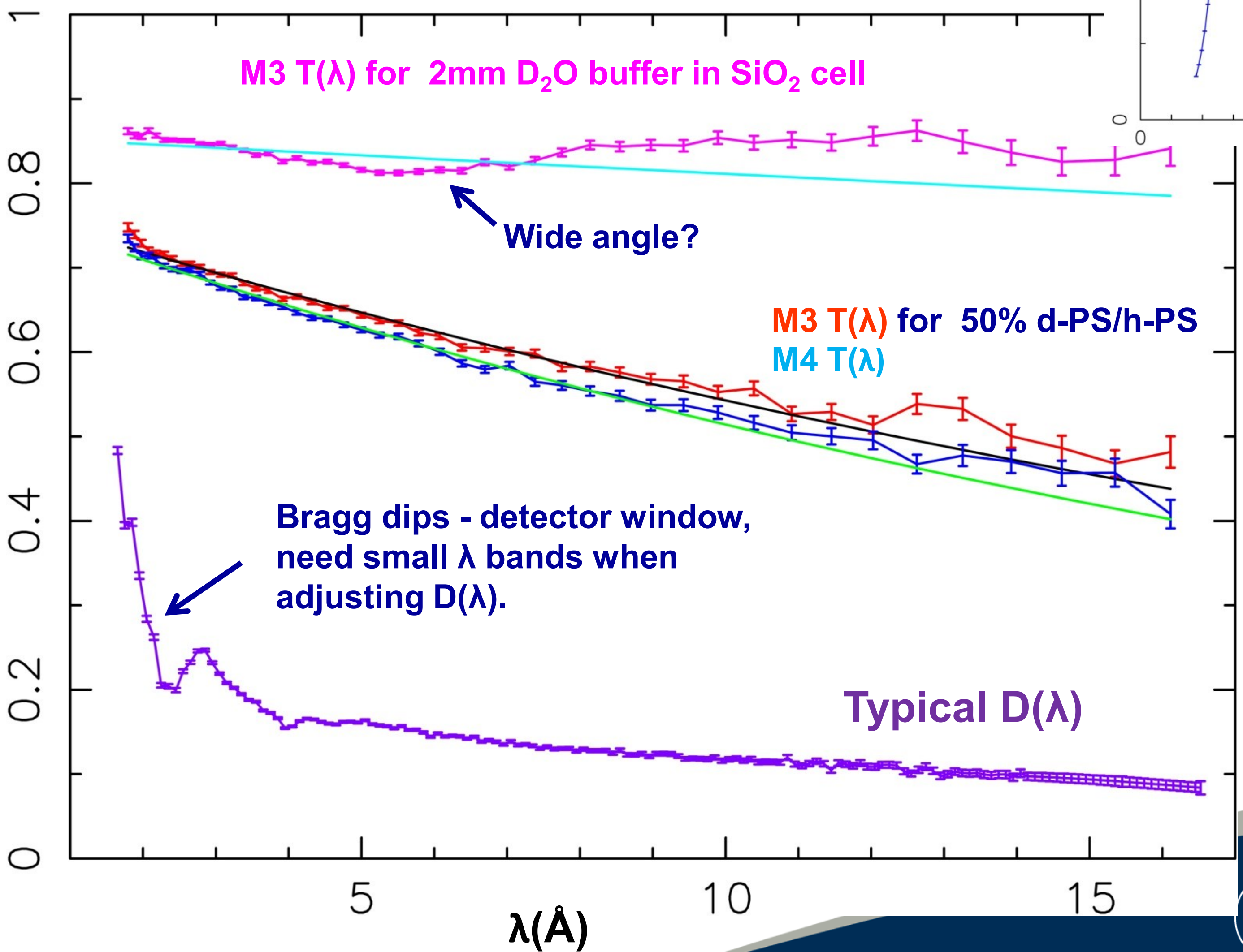
D(λ) varies slightly with L_1 and possibly A_1 and A_2 collimation as the monitor M(λ) does not see exactly the same spectrum as the sample.



On SANS2d we take out M_2 at long $L_1 = L_2$ as it scatters, and then use upstream M_1 for normalisation. LoKI needs a "halo" monitor, just outside the A_2 aperture

As expected, transmission with beam stop M4 is lower than with M3 inserted after sample, since less SANS & incoherent background signals are included.





How do we measure Transmission T(λ) ?

All methods compare transmission through a sample with an "empty beam", though we like to call this transmission a "direct beam" as the beam goes directly onto the detector. [There is some muddle here as at ISIS we also call the related empty beam into the main SANS detector the "direct beam"!!!]

Methods:

1) **Separate detector M_3 dropped in after sample, normalised to M_2 or M_1. M_3 needs to be large enough to intercept majority of SANS signal.**

2) **Use beam stop monitor M_4, normalised to M_2 or M_1 - only for weak scatter, and shorter L_2.**

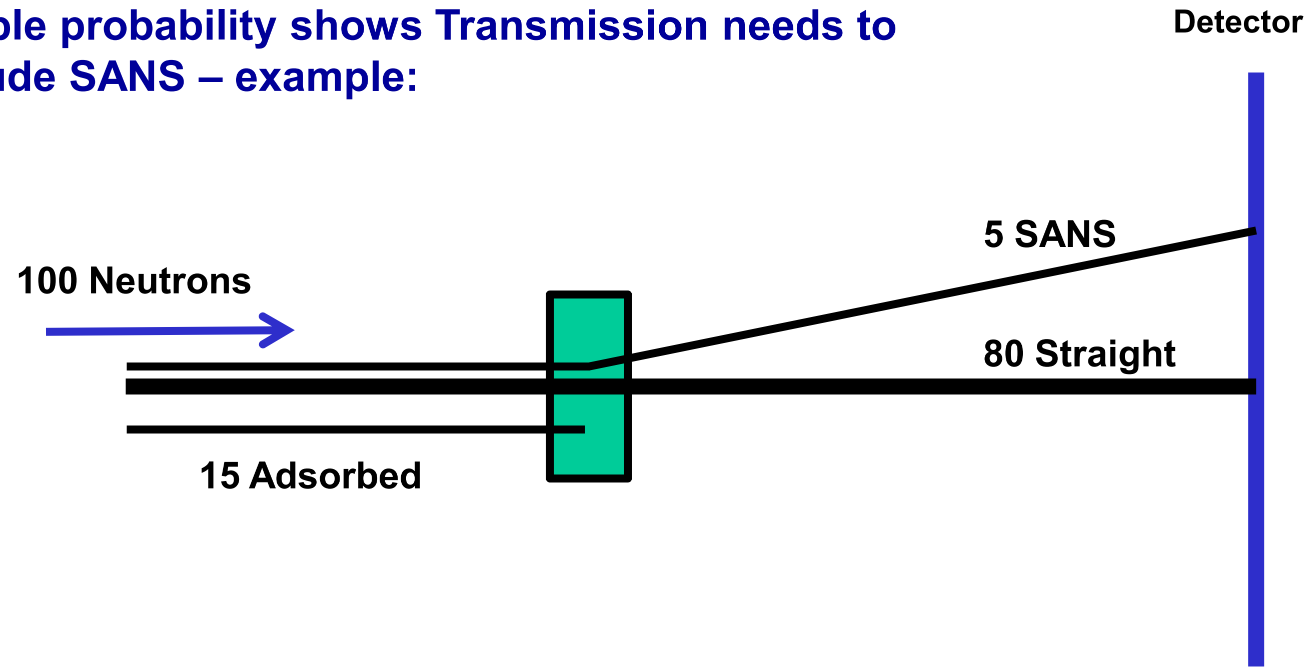
3) **Attenuate beam. Remove beam stop and use appropriate region of SANS detector. (Expensively dangerous if the detector can be degraded or damaged by high count rates. Not good e.g. with small beam that passes between tube detectors with gravity droop. Can try to diffuse with a strong scatterer in beam.)**

D33 and others like to show sample transmission as T_{SAM}/T_{CAN}, though they multiply back by T_{CAN} internally in the reduction.

NOTE, when the SANS signal is not vanishingly small the transmission should *include* the SANS signal with the direct beam but *not* any incoherent or inelastic. Fortunately the contribution from H_2O at typical detector distances is actually a fairly small fraction of 4π steradians. Thus beam stop monitor M4, is only good for simultaneous transmissions when scatter is weak, and due to gravity, when L_2 is short.

Software – will need options for several different methods

Simple probability shows Transmission needs to include SANS – example:



Classical "SANS is negligibly small" approach says T = 80/100 = 0.8, scattering probability I(Q) = counts(Q)/T = 5 / 0.8 = 6.25% which is WRONG (especially in tof).

Correct T = (80+5)/100 = 0.85, gives scattering probability I(Q) = 5 / 0.85 = 5.88%
Which is what we said in a previous slide for the "perfect detector" case:

$$\frac{\partial \Sigma_{SANS}(Q)}{\partial \Omega} = \frac{Counts(Q)}{\int Straight + \int SANS}$$

= Probability in the absence of any other processes, we know how to calculate this from theory, so can fit a model.

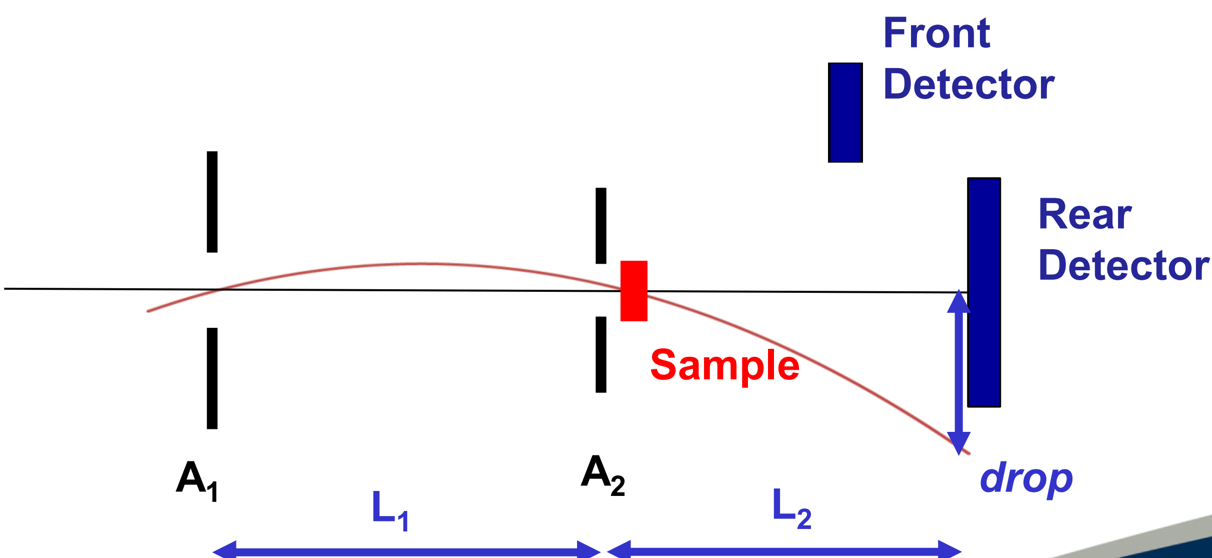
Effects of Gravity

Vertical distance H (mm) fallen by a neutron of wavelength λ (Å) in horizontal distance L (m) is

$$H = \frac{gm^2}{2h^2}\lambda^2 L^2 \approx 3.13\times10^{-4}\lambda^2 L^2$$

	Neutron fall H (mm)			
	L=5m	10m	20m	30m
λ (Å)				
2	0.03	0.13	0.5	1.1
4	0.13	0.50	2.0	4.5
6	0.28	1.13	4.5	10.1
8	0.50	2.00	8.0	18.0
10	0.78	3.13	12.5	28.2

Note – the beam centre is usually not horizontal at sample, more likely level at $L_1/2$, so *drop* may need *H* for $L^2 = (L_2 + L_1/2)^2 - (L_1/2)^2$, depending on beam divergence, baffles etc. and hence possibly on λ.



Software needs to be able to find the beam centre (at λ = 0) from the SANS pattern and allow for the drop. Best to do iterative reduction on 4 quadrants of SANS pattern.

If beam stop is centred on highest flux, them lowest Q's appear just underneath it.

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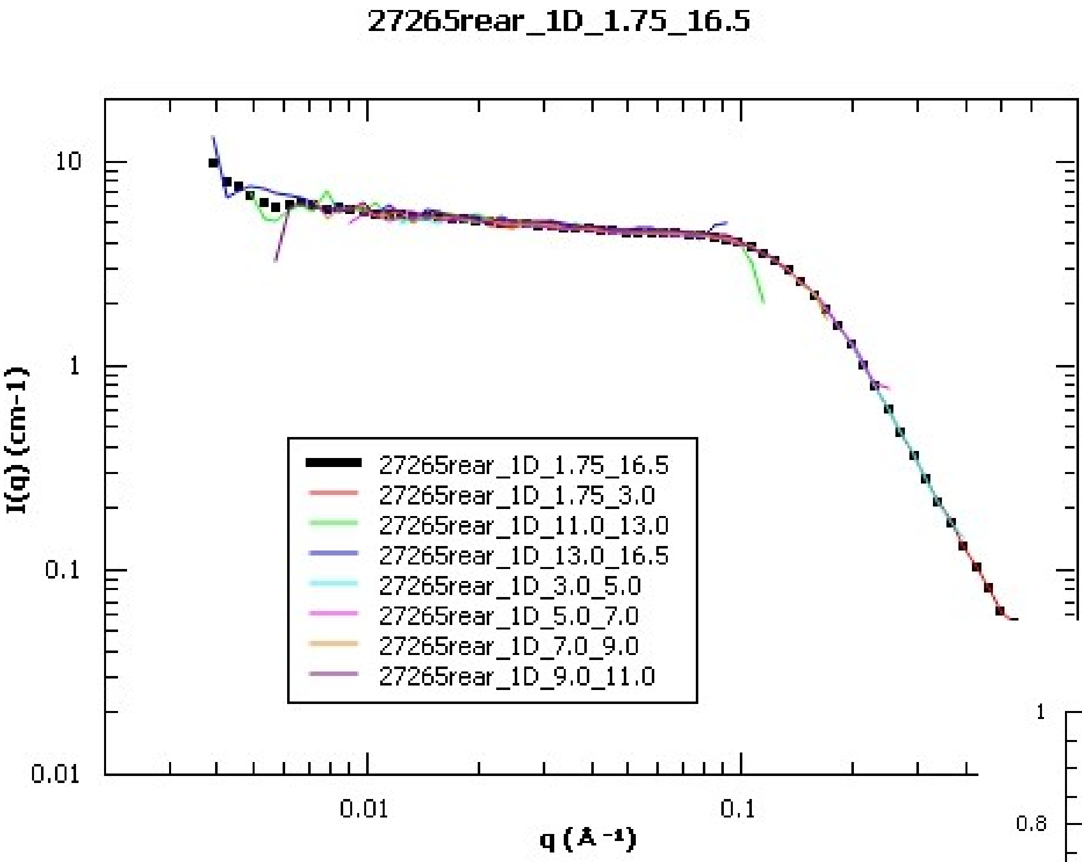
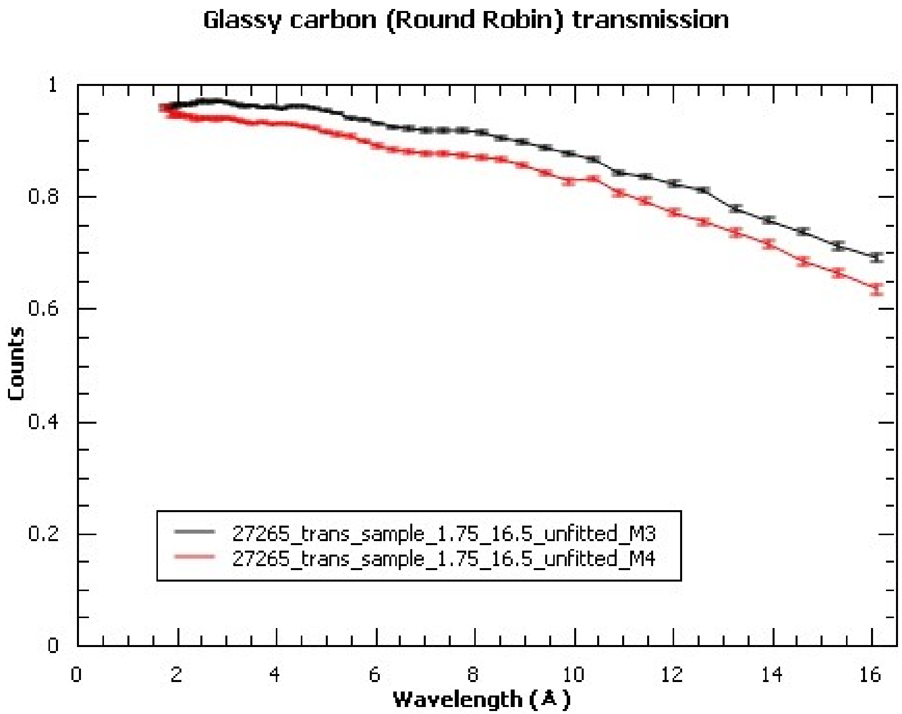
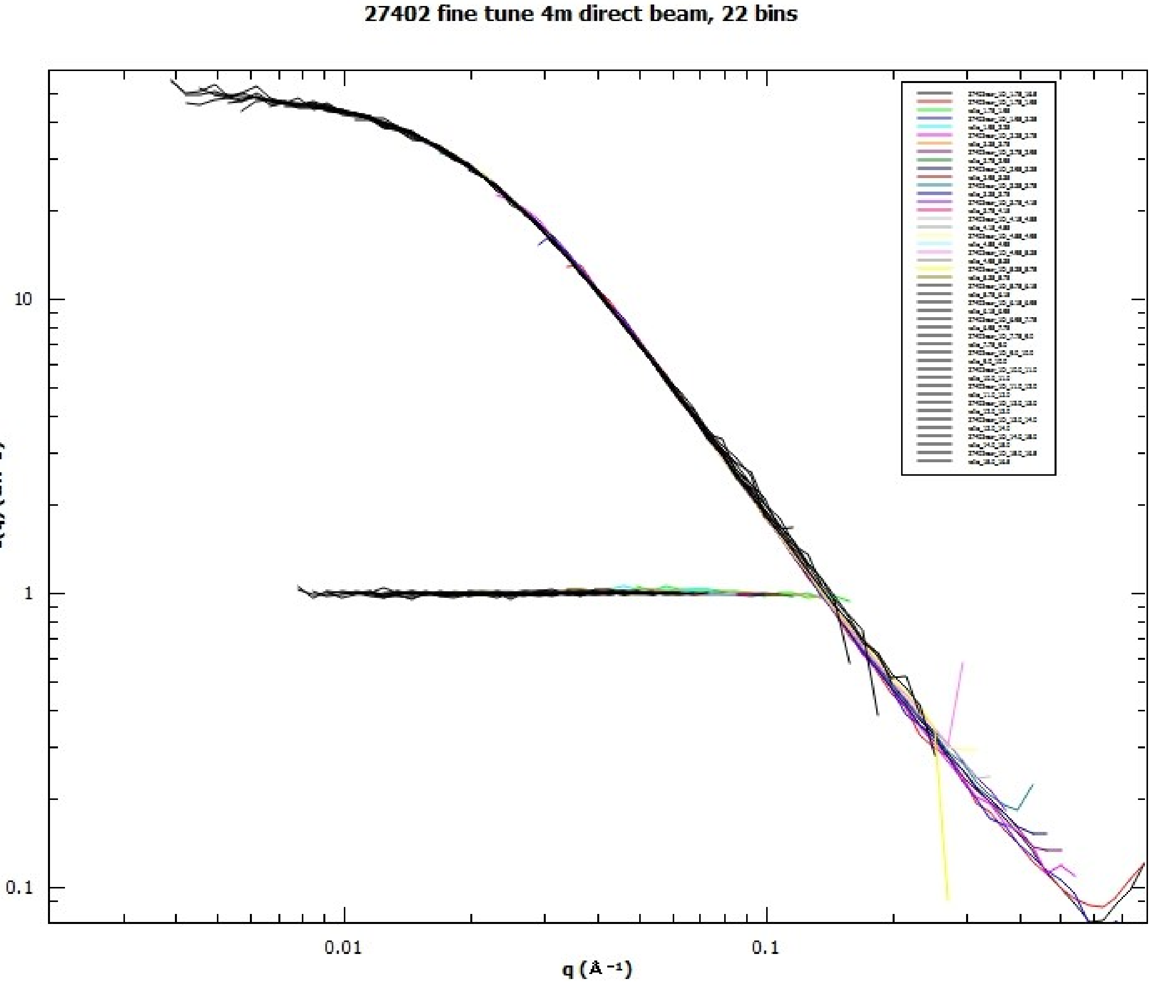
The goal – good wavelength overlap

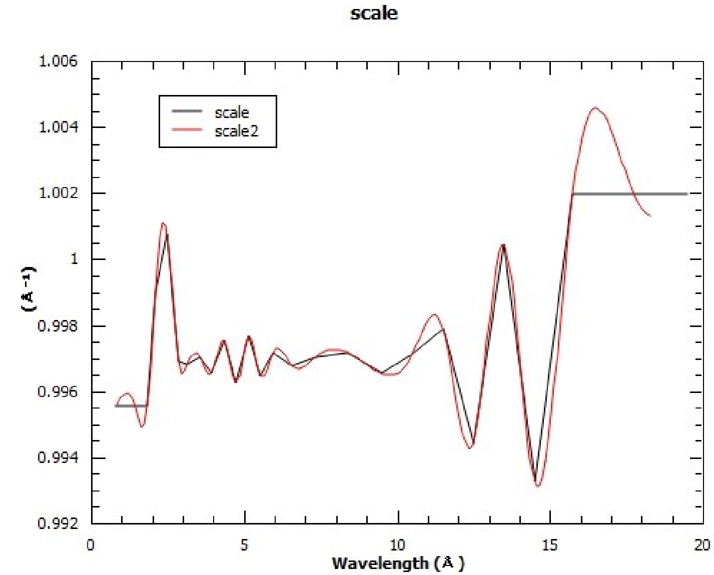


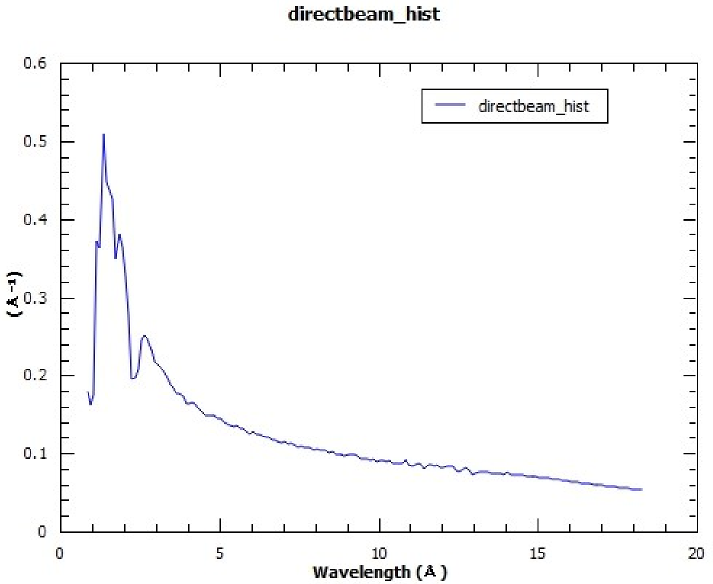
USA Glassy Carbon



Iteratively adjust direct beam shape, with standard polymer.



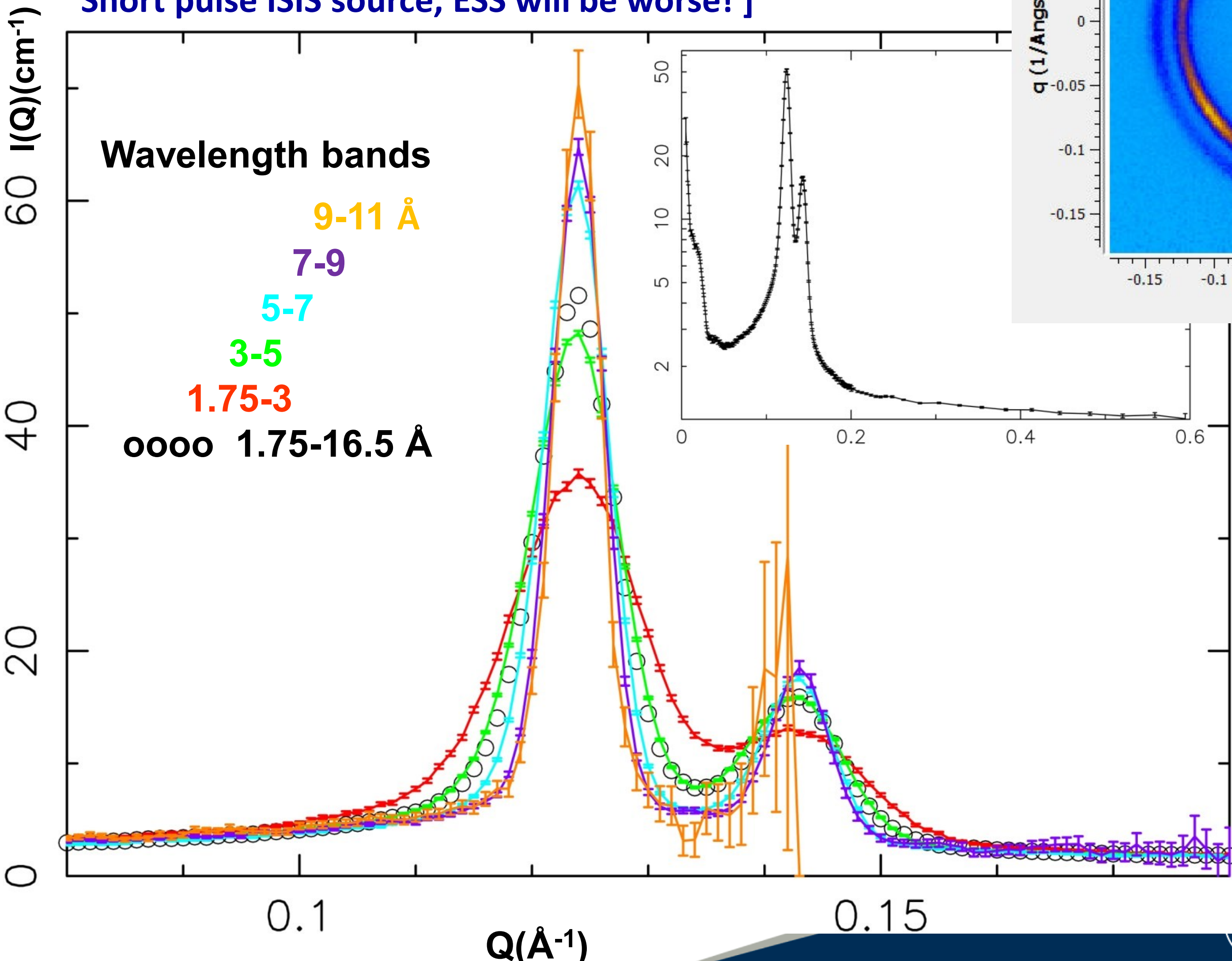


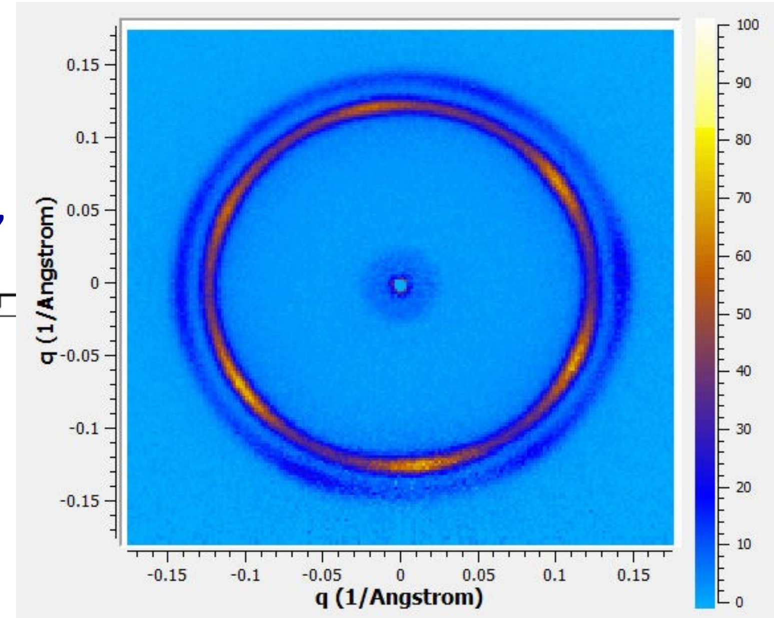


Q resolution varies inversely with λ (can estimate in software)

- can remove shorter wavelengths to sharpen peaks.

[Cubic phase silica particles on SANS2d, W.Briscoe (Bristol), Short pulse ISIS source, ESS will be worse!]





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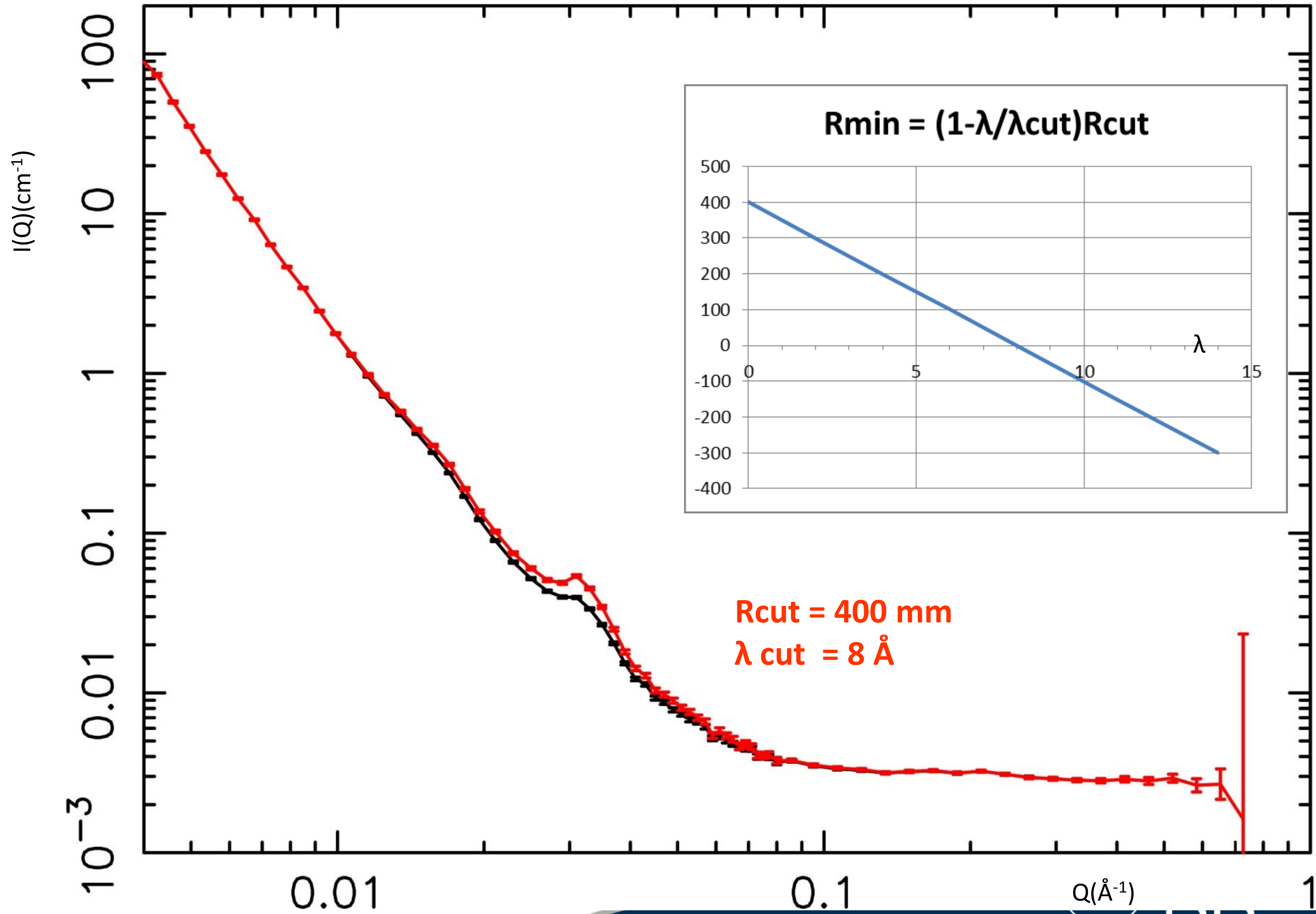
Remove short λ near beam stop (SANS2d, SDS foam, 1.75 – 16.5 Å)

Could set criteria on resolution or use a more generic method as here:



Q resolution

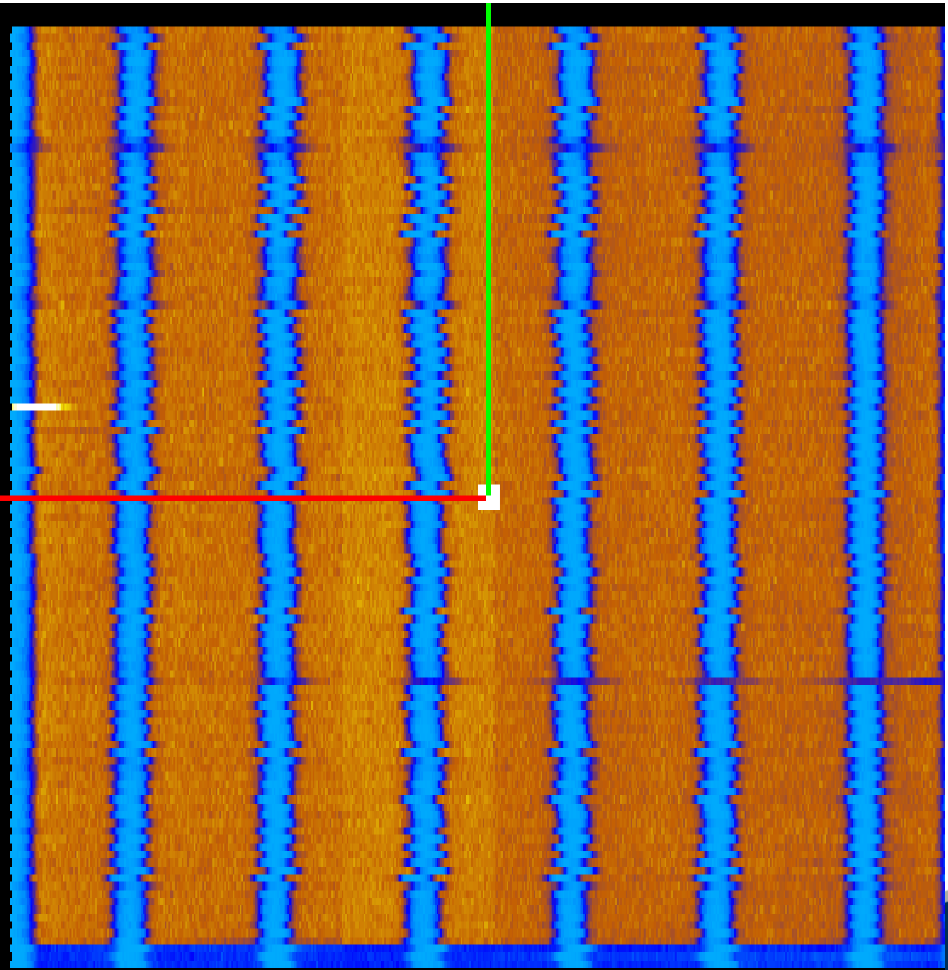
- The full resolution curve is generic for a given beam line set up and wavelength range.
- A single parameter "average" is sample dependent (resolution is better at a Q peak than in the tails).
- Thus ideally we estimate the full resolution at each, or selected, Q, but we need a format to store this and fitting software to actually use it!

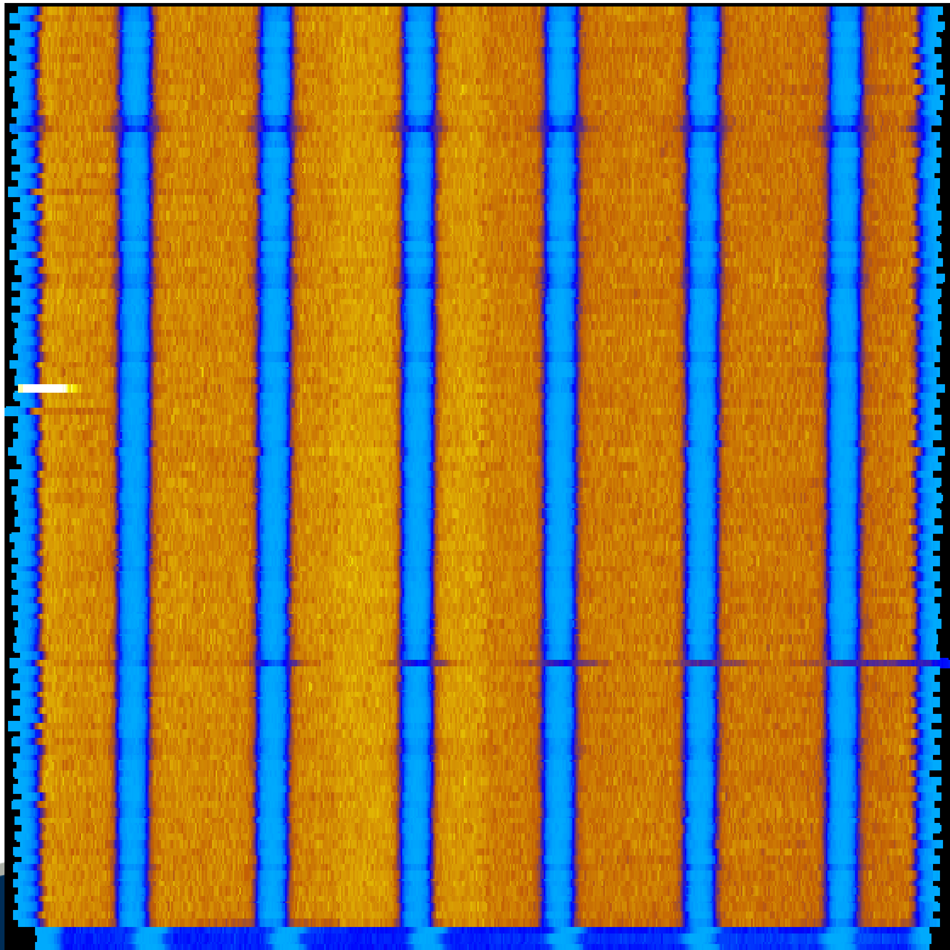
Detector calibration – example - SANS2d ^{3}He tubes

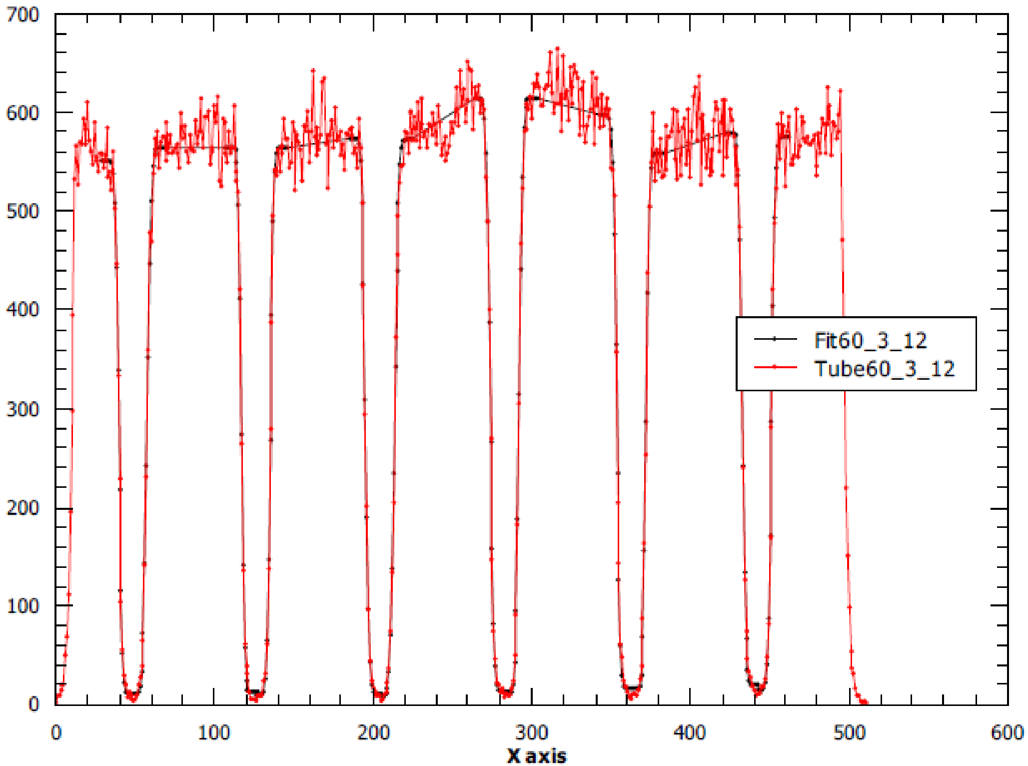
Laser theodolite survey data checks motion control & overall geometry (measure the mask position also). Large beam, 2mm H_2O sample. Scan Cd strip, or place mask with holes/stripes in Cd or ^{10}B paint. On SANS2d the "masks" below are combined data from 6 runs, moving the detector sideways part way through to remove the beam stop shadow. Check reductions using Bragg peaks – Ag behenate etc. Scan method is slow but automated, installing & removing masks in vacuum tank is labour intensive.

- this needs bespoke software

BEFORE **(6 Cd shadows stitched)** **AFTER**



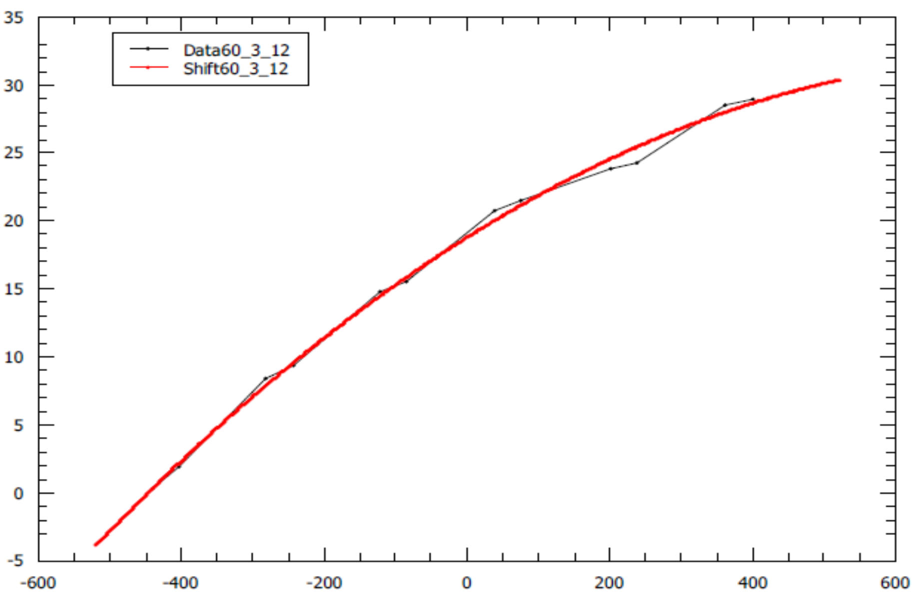




27345 rear Data60_3_12

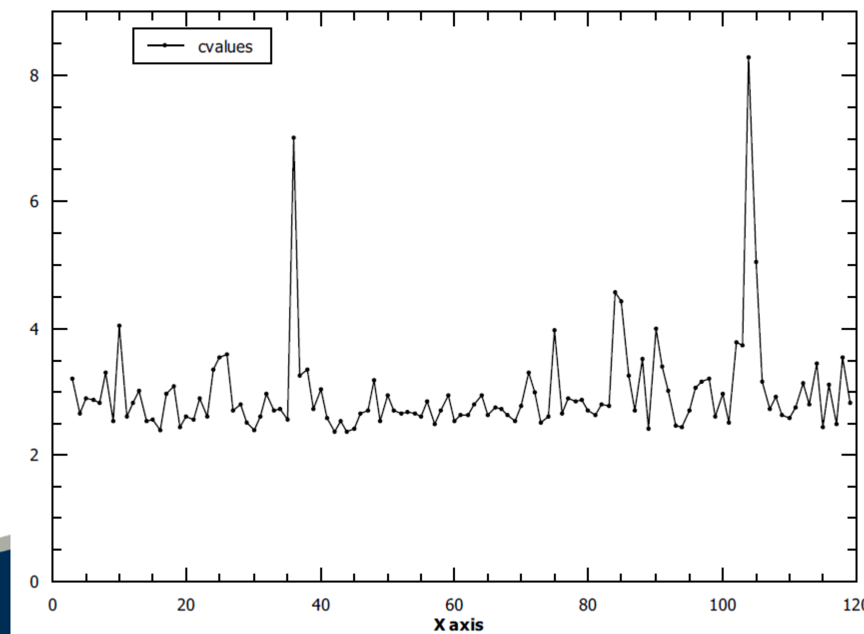
Single Tube #60

6 runs stitched. Counts vs pixel.
Fit to 12 edges, then fit quadratic.
(Cd strip ought to be wider!)
- this needs bespoke software



Mean resolution (mm) per tube

27345 rear avg cvalues



Tof SANS reduction software - Mantid status

- Reduce data, find beam centre – SANS of sample & can; dark count options; transmissions. - OK, flexible software, and pretty fast.
- Link data collection & reduction scripts for "standard experiments". – batch reductions, but not linked to collection. Robots in future ?
- Diagnose & mask "bad parts" in space and/or time – mostly OK but relies on user/scientist; "user file" stores many details for each experiment.
- Normalisation to incident flux, per neutron pulse ??? – will need work
- Calibration & survey measurements for detector efficiency and position encoding. – likely significant new effort
- Scattering geometry effects & gravity – better wider angle & more detailed gravity corrections needed
- Q resolution optimisation & estimation. – simple implementation (inputs moderator tof characteristics), room to improve
- Poor λ overlap, multiple scatter – warn / correct ? – to do
- Detector dead time warn/correct ? – on wish list, don't know how to correct in tof
- Approximate real time I(Q) on the fly - on wish list

Tof SANS reduction software - conclusions

- Tof SANS reduction is more complex than many expect and needs multiple options for how to do things.
- Ability in Mantid for instrument scientists to test new ideas or perform diagnostics & calibrations from python code is very important.
- Since most Q values come from many combinations of λ and R some effects "average out", so may be less noticeable than you expect (e.g. the flood source, or some dead pixels)
- If I(Q) falls logarithmically some users may not notice the details.
- Generally need to do a ~ ±1% reduction to combine I(Q) from different wavelengths (thus finally fit the correct R_g). This requires more care for tof than at fixed wavelength, as the corrections are usually wavelength dependent rather than a scalar.
- Mantid has a great deal of what is needed, but strongly resist the temptation to think the "its all done", as it certainly is not!

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No beam stop method

As usual

$$T(\lambda) = \frac{\sum_{All\_R} C_{RUN}(\lambda)}{M_{RUN}(\lambda)} \frac{M_{DB}(\lambda)}{C_{DB}(\lambda)}$$

Here it is emphasised $C_{RUN}(\lambda)$ are the counts summed over the whole detector, as is $C_{DB}(\lambda)$.

Imagine that we use the same A_H hole at the sample to attenuate the beam for both TRANS and DB runs, then note that in the denominator of our usual I(Q) equation.

$$M(\lambda)T(\lambda)D(\lambda) = M_{RUN}(\lambda) \frac{\sum_{All\_R} C_{RUN}(\lambda)}{M_{RUN}(\lambda)} \frac{M_{DB}(\lambda)}{C_{DB}(\lambda)} \frac{C_H(\lambda)}{M_H(\lambda)}$$

If SANS and TRANS are recorded in the same, "beam stop out", run then all the monitor spectra cancel so [need check the 1/V part]:

$$I(Q) = \frac{1}{V_{SAM}} \frac{\sum_{R,\lambda \subset Q} C(R,\lambda)}{\sum_{All\_R,\lambda \subset Q} C(R,\lambda) \sum_{R \subset Q} \Omega(R)}$$

No beam stop method – caveats!

$$I(Q) = \frac{1}{V_{SAM}} \frac{\sum_{R,\lambda \subset Q} C(R,\lambda)}{\sum_{All\_R,\lambda \subset Q} C(R,\lambda) \sum_{R \subset Q} \Omega(R)}$$

This is much easier to derive from first principles, as it gives the scattering probability per unit solid angle. Note the both the total scatter and scattered beam are attenuated by any adsorption in the sample, so we do not need a "transmission run". The sum of counts in the denominator is over all radii at all wavelengths contributing to a particular Q.

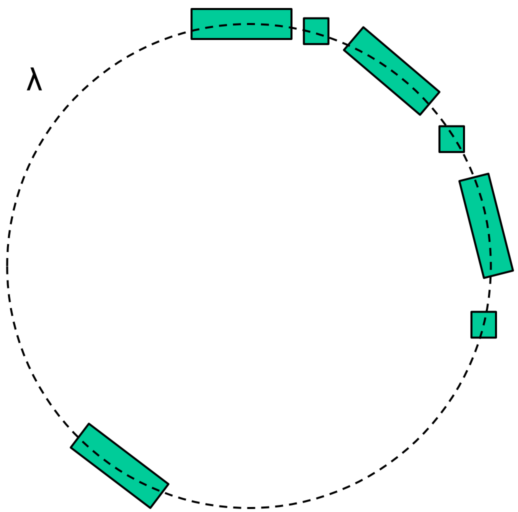
Strictly T(λ) should include the SANS but not any background such as "incoherent" from H_2O, as we are only interested in the probability of scattering by SANS and not by any other process. (Else the patterns from different wavelengths will not overlap well, and we will not get the same result for a D/H to H/D contrast reversal.)

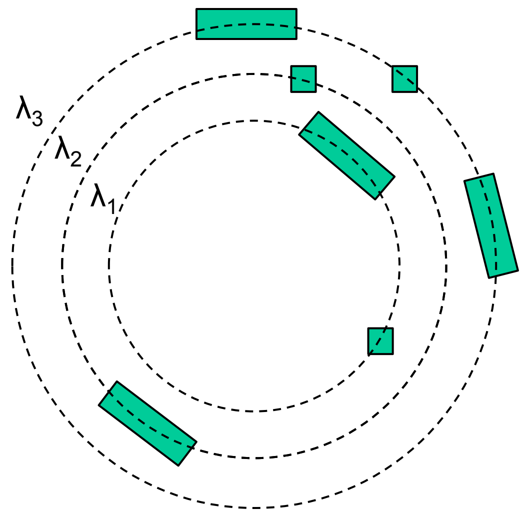
Thus "incoherent" scatter should if possible be removed. We may be able to do this by a prior subtraction of a rescaled background solvent (e.g. H2O) data set. [Needs investigation.]

Note that detector efficiency and/or dead time need to be included, especially at the beam centre, where a small beam may land on or between gas tube detectors. (Or a damaged detector may underestimate the total flux.) This will likely be a major practical limitation to the accuracy of this method.

A note on the “averaging”

$$I(Q) = \frac{Scale}{V_{SAM}} \frac{\sum_{R,\lambda \subset Q} C(R,\lambda)}{\sum_{R,\lambda \subset Q} M(\lambda)T(\lambda)D(\lambda)\Omega(R)}$$





$$I(Q) \propto \frac{\sum_{R,\lambda \subset Q} C(R)}{\sum_{R,\lambda \subset Q} \Omega(R)}$$

- **At fixed wavelength we happily merge pixels at the same radius, summing the counts, summing the solid angles.**

$$I(Q) \propto \frac{\sum_{R,\lambda \subset Q} C(R,\lambda)}{\sum_{R,\lambda \subset Q} M(\lambda)T(\lambda)D(\lambda)\Omega(R)}$$

- **In tof we equivalently merge data “pixels” from radius & wavelength combinations at same Q.**
- **(Can imagine moving pixels during tof pulse if you like!)**

“Overlap” plots generally good – but can we do better?

- Need all the effects at “few %” level,
  e.g. angle dependent T(λ), detector path length & parallax, detector efficiency.

- Transmission T(λ) is key. Actually to some extent we don’t actually need to calculate T(λ), but it is an extremely useful diagnostic.

- T(λ) should include the SANS and straight through beam, but not the Background & Inelastic. Fortunately H_2O scatter is not usually a significant effect on the transmission. Though H2O scatter is “large” the solid angle of the transmissions detector is actually small.

- M4 beam stop detector transmissions are good (possibly better?) for weak scatter.

- M3 transmissions are better for strong scatter, as they include most of the SANS.

- Beam stop out transmissions as a function of detector radius are a useful diagnostic in conjunction with “overlap checks”.

DETOUR – the traditional reactor method is the same …

I_{SAM} = raw counts of sample in can (includes $T_S T_{CAN}$)

I'_S = raw counts due to sample alone (includes T_S)

I_{CAN} = raw counts can (includes T_{CAN})

T_S = sample transmission, by measuring (sample in can)/(empty can)

$T_{SAM} = T_S . T_{CAN}$ = sample in can transmission relative to empty beam

"CAN" here may also be solvent in a cell.

Assume

$$I_{SAM} = I'_S T_{CAN} + I_{CAN} T_S$$

then

$$I'_S = \frac{1}{T_{CAN}} \left(I_{SAM} - T_S I_{CAN} \right)$$

Some reactor sources first do this, or something similar.

$$\frac{\partial \Sigma(Q)}{\partial \Omega} \propto \frac{I'_S}{T_S} = \frac{I_{SAM}}{T_S T_{CAN}} - \frac{I_{CAN}}{T_{CAN}} = \frac{I_{SAM}}{T_{SAM}} - \frac{I_{CAN}}{T_{CAN}}$$

ISIS does this, with symmetrical reduction for Sample & Can, and all transmissions relative to empty beam.

The “optimal” SANS instrument

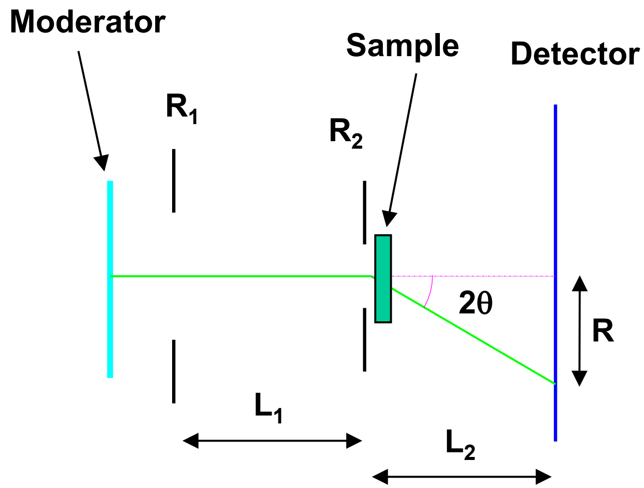
e.g. D.F.R.Mildner & J.M.Carpenter, J.Appl.Cryst. 17(1984)249-256.

For detector resolution of width ΔR at radius R

$$(\sigma_Q)^2 = \frac{1}{12}\left(\frac{2\pi}{\lambda}\right)^2\left[3\frac{R_1^2}{L_1^2} + 3\frac{R_2^2}{L'^2} + \frac{(\Delta R)^2}{L_2^2} + \frac{R^2}{L_2^2}\left(\frac{\Delta\lambda}{\lambda}\right)^2\right]$$

$$\frac{1}{L'} = \frac{1}{L_1} + \frac{1}{L_2} \qquad\qquad Q \approx \frac{2\pi\theta}{\lambda} \approx 2\pi\frac{R}{\lambda L_2}$$

$$\left(\frac{\sigma_Q}{Q}\right)^2 = \left(\frac{R_1 L_2}{2RL_1}\right)^2 + \left(\frac{R_2(L_1+L_2)}{2RL_1}\right)^2 + \frac{1}{12}\left(\frac{\Delta R}{R}\right)^2 + \frac{1}{12}\left(\frac{\Delta\lambda}{\lambda}\right)^2$$



“Optimal” SANS - best flux for best Q resolution: $L_1 = L_2$, $R_1 = 2R_2$

Note ΔR and Δλ are *rectangular* bin widths, standard deviation σ is Δ/√12 = Δ/3.464.

A Gaussian has FWHM = $(8\log_e(2))^{1/2}\sigma = 2.35482\sigma$

Note Q has 1/λ so σ_Q varies inversely with λ due to geometry

Detailed account is more complex! For resolution perpendicular to Q, see:
J.S.Pedersen, D.Posselt & K.Mortensen, J.Appl.Cryst.23(1990)321-333.