SMALL-ANGLE NEUTRON SCATTERING (SANS) & TIME-OF-FLIGHT (TOF)

Endurance

Charles Dewhurst

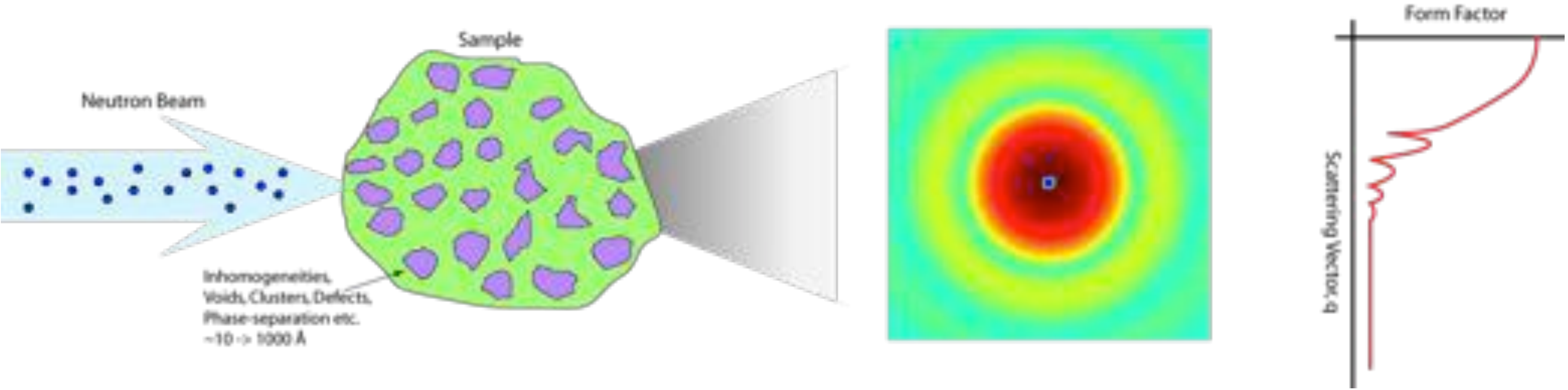
ILL - Grenoble

Scientific Assistant to DPT Director
& Endurance Coordinator

Special thanks to: Isabelle Grillo, Michel Bonnaud & D33 project team

SMALL-ANGLE NEUTRON SCATTERING (SANS):

- Scattering methods for 'dilute' samples are analogous to
  Young's single-slit diffraction experiment



- **Fourier transform of the particle shape function**

- Scattering depends on the **'scattering-length densities'** of the elementary volumes

- Neutrons have large natural differences in scattering length between elements (compared to x-rays)
  also H / D sensitivity -> **contrast variation**

- **Neutron spin** – gives a sensitivity to magnetic structures

Small-Angle Neutron Scattering (SANS): Geometry

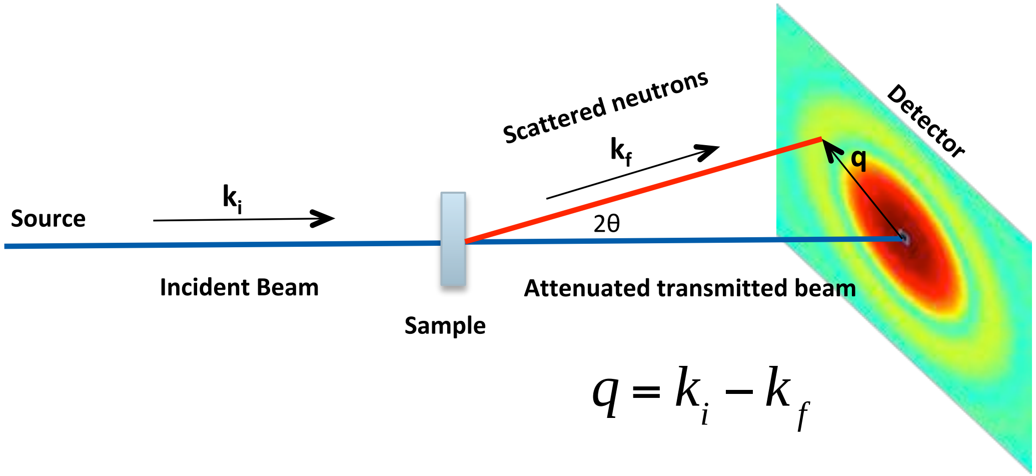
- Consider scattering geometry & Bragg law:

$$\lambda = 2d\sin(\theta)$$

- Switch to reciprocal space:

$$q = \frac{2\pi}{d}$$

$$q = \frac{4\pi}{\lambda}\sin(\theta)$$



$$q = k_i - k_f$$

Small-Angle Neutron Scattering (SANS): Resolution -> How to build a SANS instrument

- Differentiating the Bragg law tells us:
  - Resolution δq in determining q
    i.e. how accurate the measurement could be in determining the size and shape of particles
  - Gives us a clue as to how to build a SANS instrument

Bragg Law: $$q = \frac{4\pi}{\lambda}\sin(\theta)$$

Partial derivatives: $$\frac{\delta q_\lambda}{\delta\lambda} = \frac{-4\pi}{\lambda^2}\sin(\theta) \qquad \frac{\delta q_\theta}{\delta\theta} = \frac{4\pi}{\lambda}\cos(\theta)$$

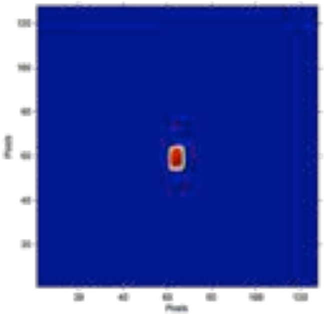
- Add resolution components in quadrature: $$\delta q^2 = \delta q_\lambda^2 + \delta q_\theta^2$$
- Substitute for q and use $\cos^2(\theta) = 1 - \sin^2(\theta)$
- Resolution, δq:

$$\delta q^2 = \left(\frac{\delta\lambda}{\lambda}\right)^2 q^2 + \delta\theta^2\left[\left(\frac{4\pi}{\lambda}\right)^2 - q^2\right]$$

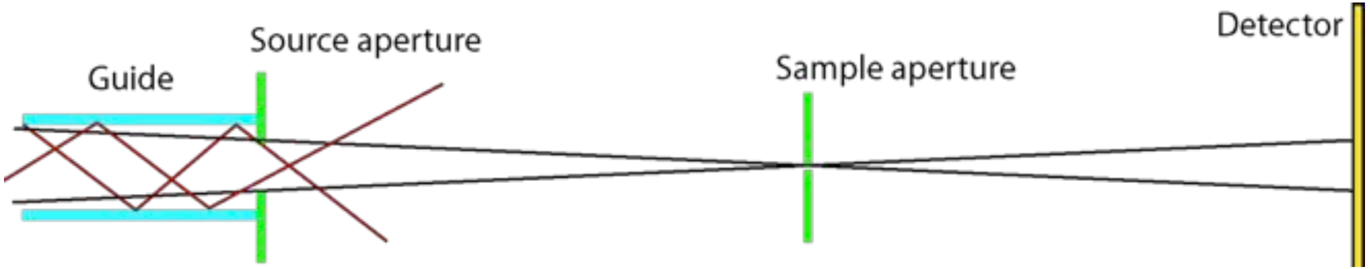
Small-angle approximation: as q -> 0

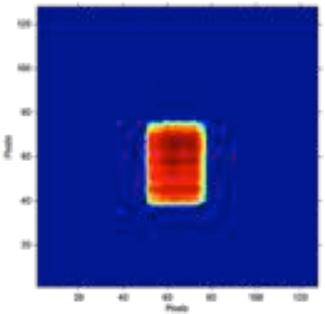
$$\delta q_{q=0} \approx \frac{4\pi}{\lambda}\delta\theta$$

Small-Angle Neutron Scattering (SANS): Resolution -> Instrument parameters



- **Beam Divergence, δq, determined by collimation distance and source aperture size**
  - Try to keep within the 'pin-hole' approximation for the sample size
  - Beam 'size' on the detector
  - Most important for detector pixels close to the beam centre





- **Wavelength spread, $\delta\lambda/\lambda$:** Typically 10% from a velocity selector
  - $\delta\lambda$ leads to a radial ($|q|$) smearing of data
  - Most important for scattering at higher angles ......

    ..... and could be relaxed further under some conditions to gain flux .....

    ..... but need also an option for higher resolution for some highly ordered systems

SMALL-ANGLE NEUTRON SCATTERING (SANS): RESOLUTION -> WHY SANS INSTRUMENTS ARE BIG

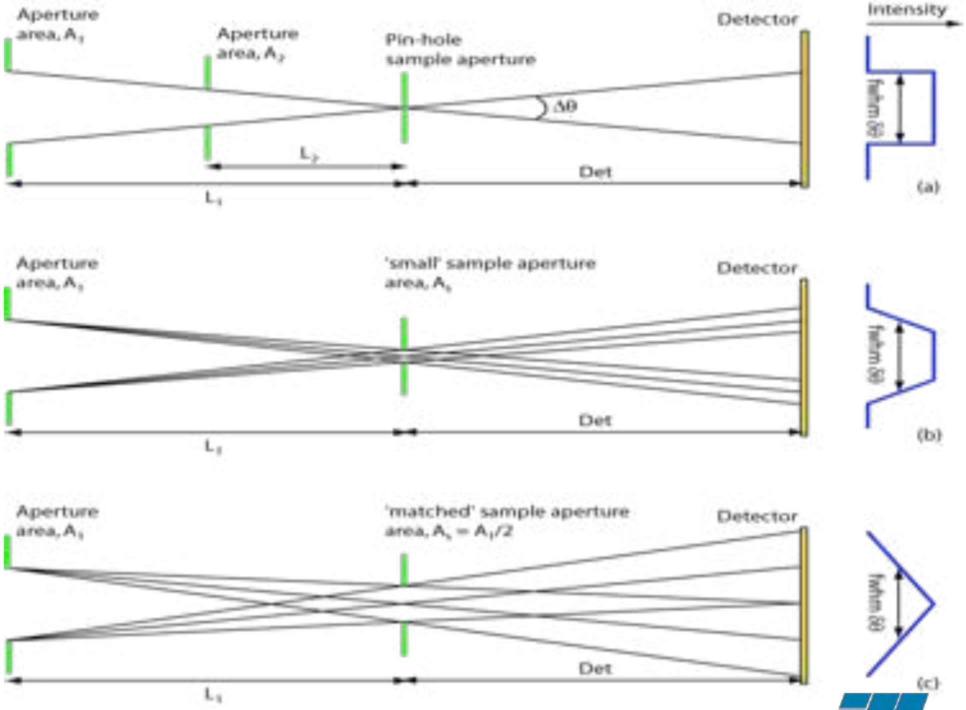
- **Optimised SANS flux & resolution conditions:**

  - Pin-hole optimisation allows sample sizes up to ½ the source size before dominating the angular resolution of the instrument

  - Source aperture controls beam divergence
    - Remains valid as long as $A_s < A_1/2$

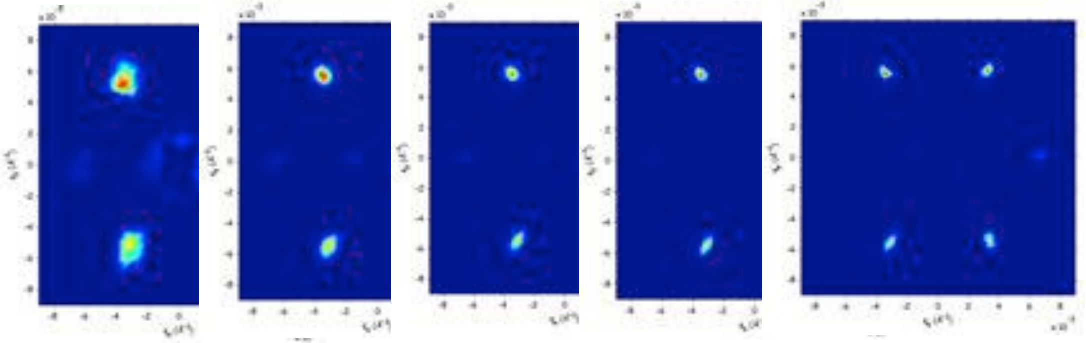
  - What is the largest realistic sample size?

  - Implications for the size of the instrument



WHAT ARE WE TRYING TO MEASURE AND WITH WHAT ACCURACY?

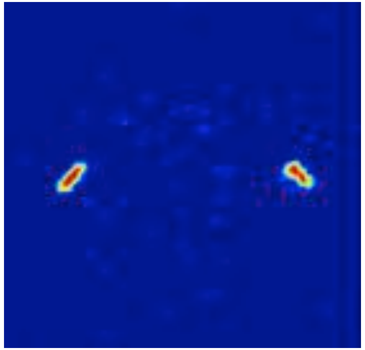
- Effects of beam divergence



40x55 mm 30 mmØ 20 mmØ 10 mmØ 5 mmØ

e.g. Bragg peaks from the vortex lattice in superconductors can be resolution limited

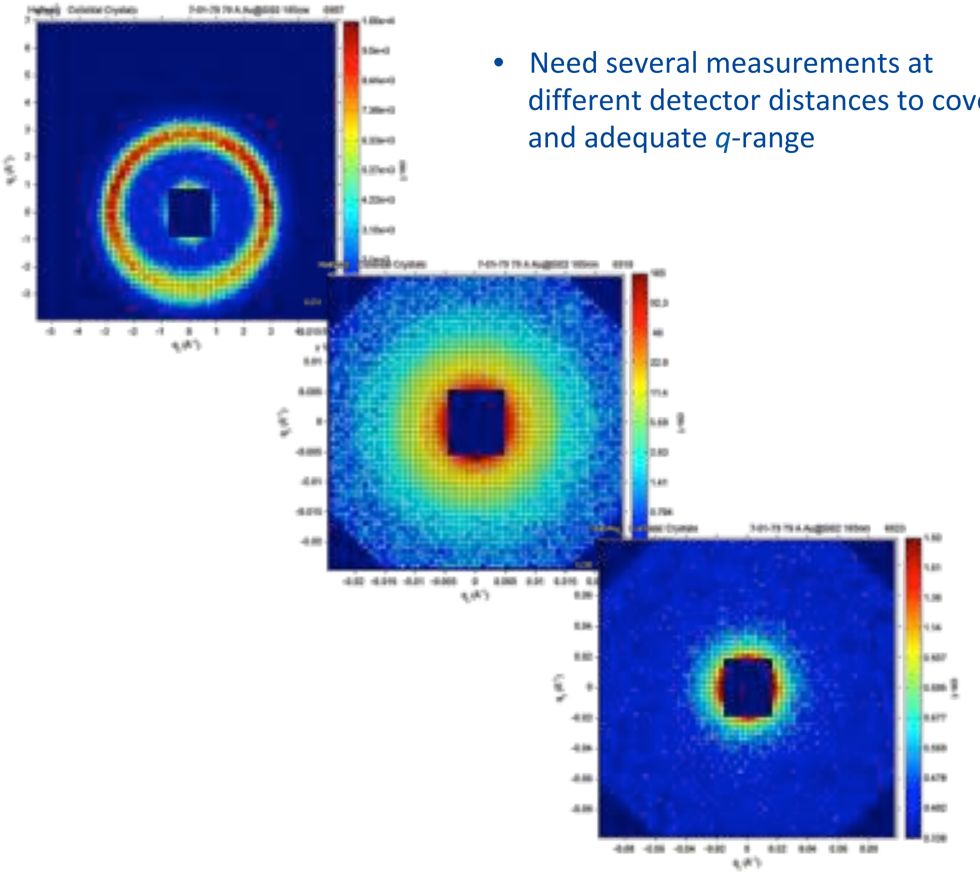
- Effects of wavelength spread (and gravitational dispersion)



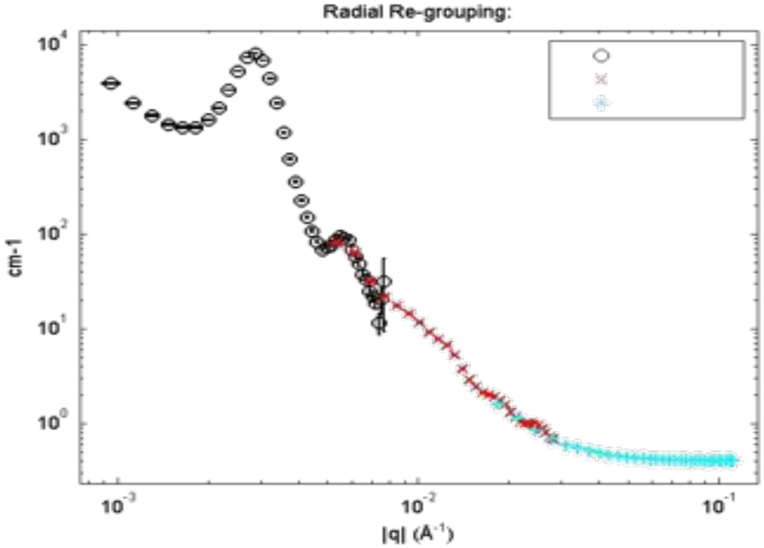
e.g. Bragg peaks from a diffraction grating, d = 2500 Å, formed by laser holography in an optically sensitive polymer. λ = 18 Å

- Radial broadening of the Bragg peaks is due to 10% wavelength spread
- Further distortion due to gravitational dispersion

What are we trying to measure and with what accuracy?



- Need several measurements at different detector distances to cover and adequate *q*-range



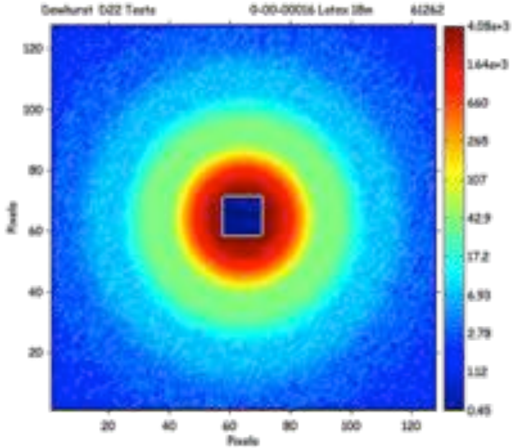
- Data treatment procedures:

  - Transmission corrections
  - Background subtractions
  - Detector efficiency correction
  - Parallax corrections
  - Absolute scaling
  - Re-grouping procedures
  - Fitting & modeling

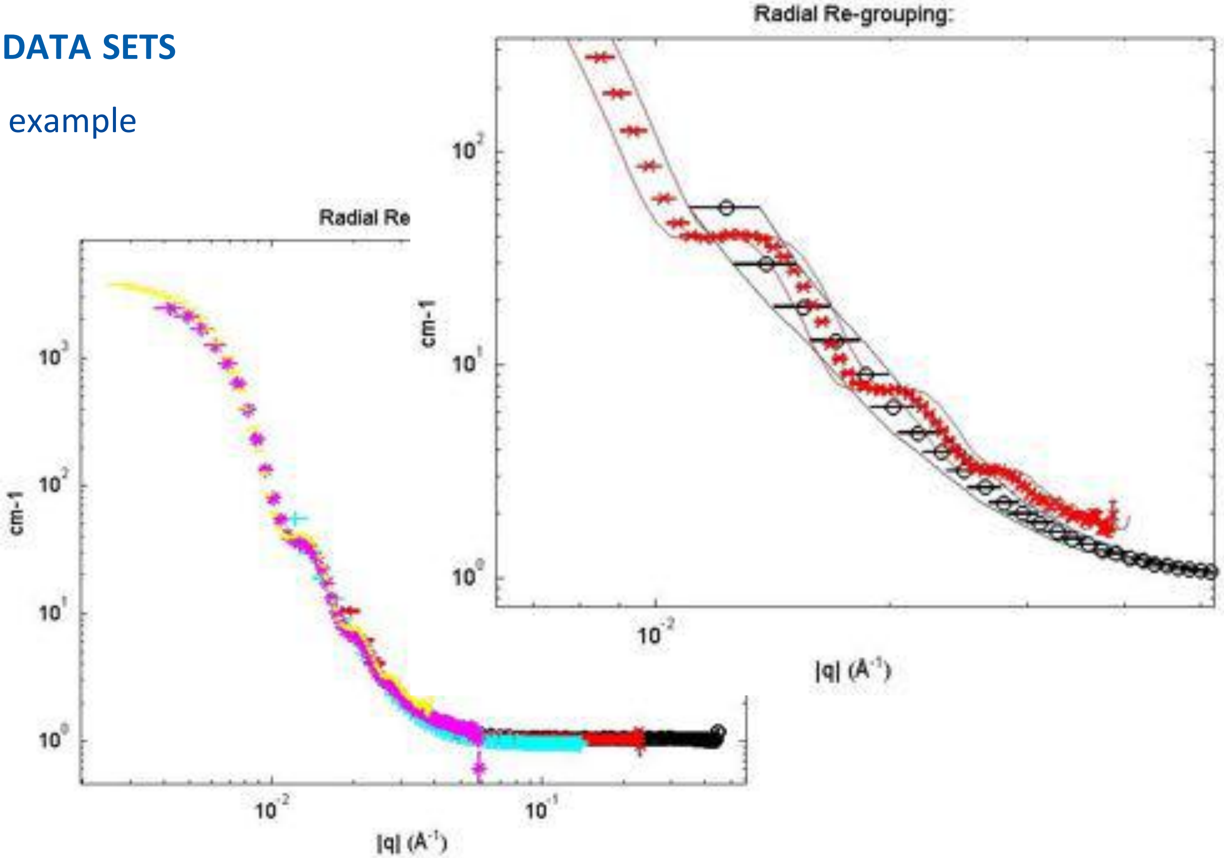
……..Etc.

RESOLUTION EFFECTS: OVERLAPPING DATA SETS

- Resolution overlap: Latex spheres example



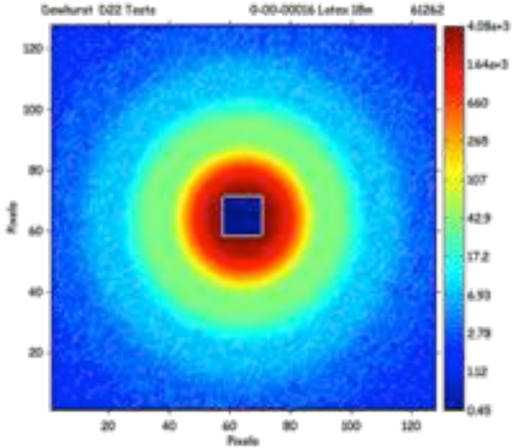
2D Scattering from latex spheres in solution



- The full scattering pattern measured in ‘decent’ resolution takes between 4-6 instrument configurations and measurements

RESOLUTION EFFECTS: BEAM DIVERGENCE AND WAVELENGTH SPREAD

- Data fitting including instrument resolution



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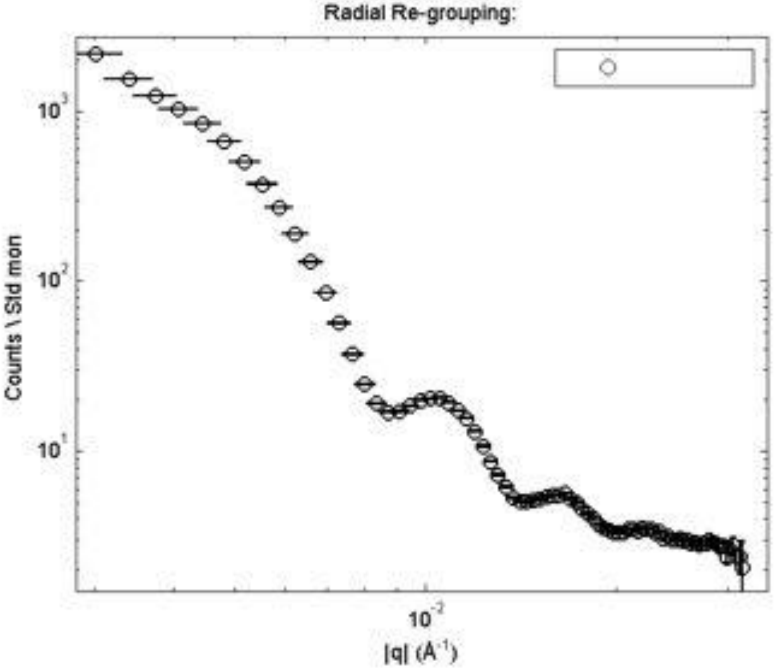
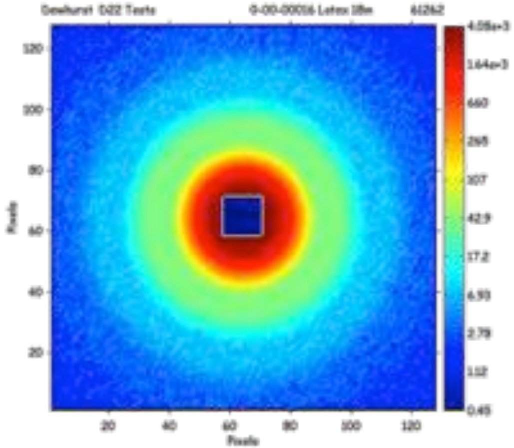


Reduced I vs. Q data: Sphere Form-Factor convoluted by instrument resolution

RESOLUTION EFFECTS: BEAM DIVERGENCE AND WAVELENGTH SPREAD

- Data fitting including instrument resolution



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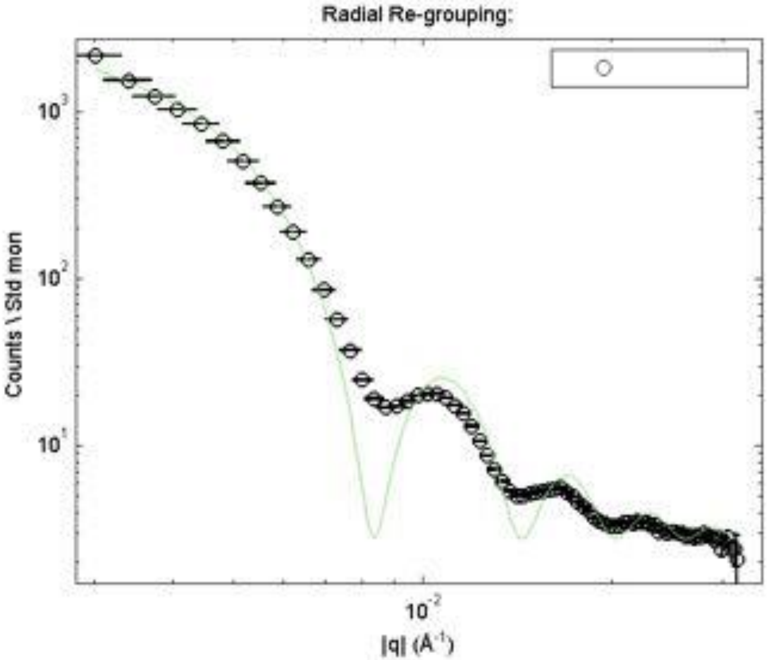
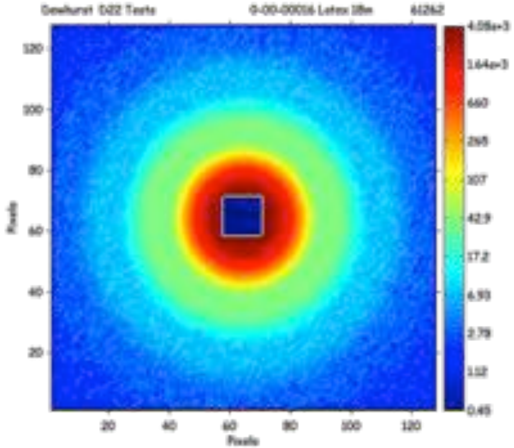


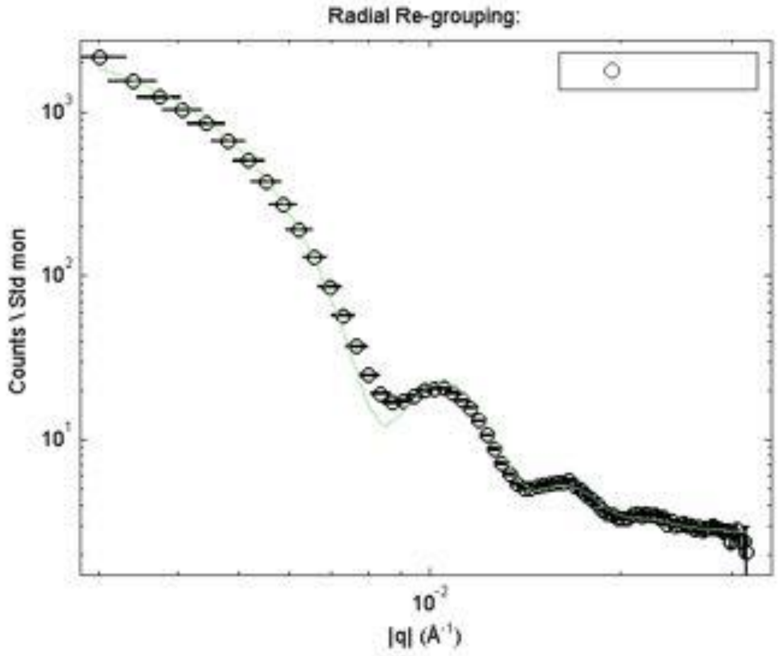
Sphere Form-Factor fit

RESOLUTION EFFECTS: BEAM DIVERGENCE AND WAVELENGTH SPREAD

- Data fitting including instrument resolution



2D Scattering from latex spheres in solution



Sphere Form-Factor fit convoluted by instrument resolution

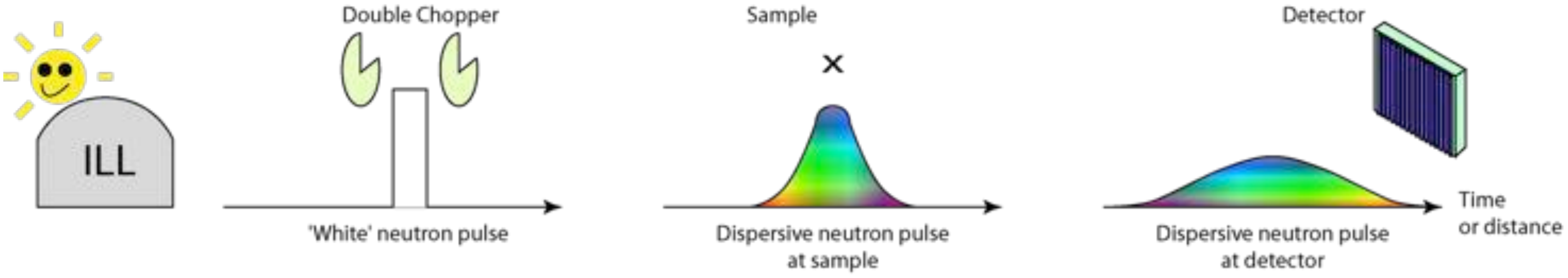
CONCLUSION: Pay attention to the instrument resolution

SANS: Traditionally Monochromatic
What about Time-Of-Flight instruments?

- Why TOF SANS?
  - Large dynamic *q*-range, q_{max} / q_{min}
  - Sometimes the only choice!

- Spallation sources: ISIS, IPNS, JPARC, SNS, other

- Continuous sources: ILL (D33), ANSTO (Bilby)
  - Inspired by white-chopper reflectometers (D17, Figaro)



D33:

Project Overview & Goals:

To build a new user instrument, D33, for Small-Angle Neutron Scattering

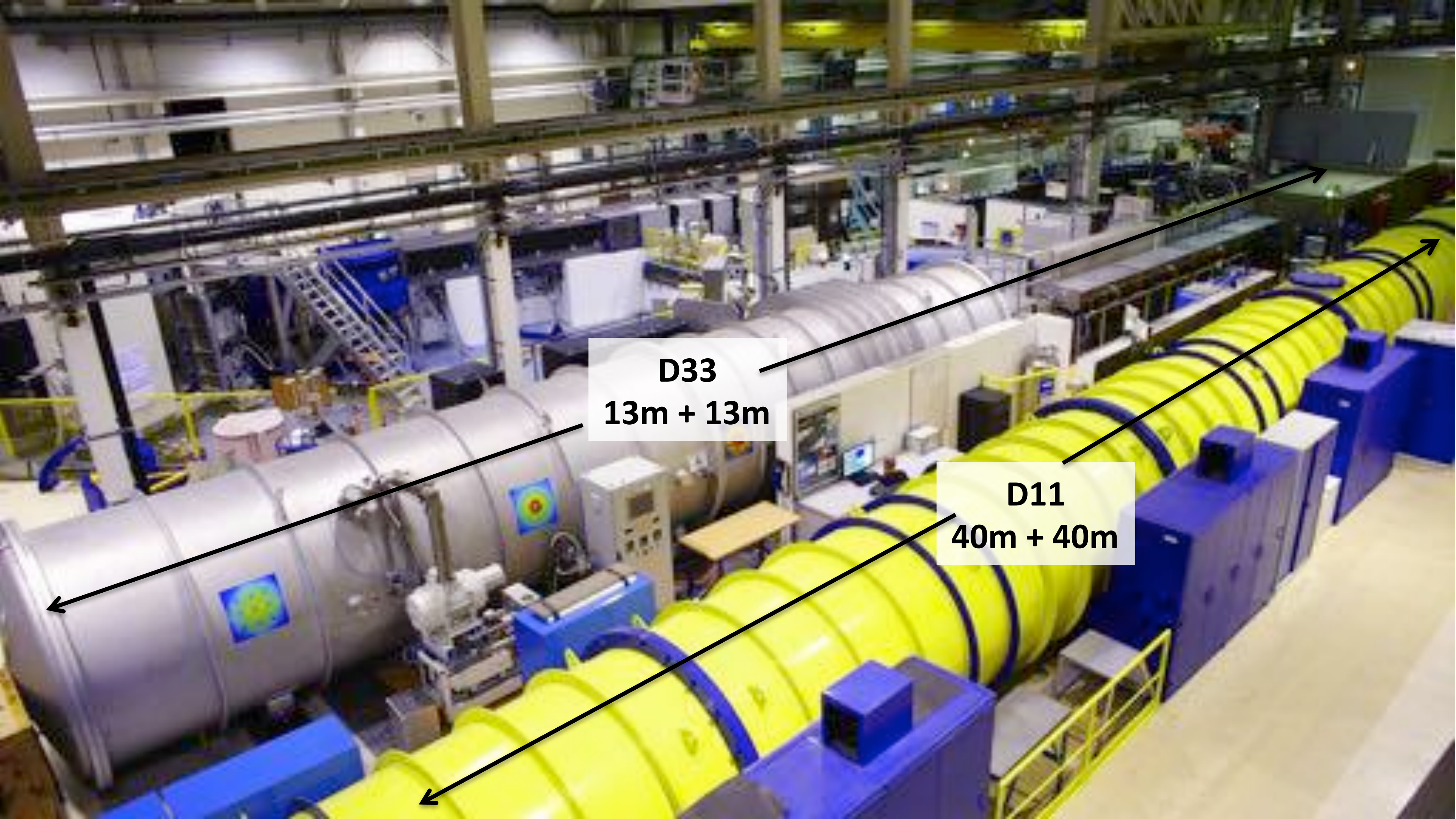
D33 has the following characteristics:

- **Large dynamic *q*-range: compete with spallation sources**
- **Monochromatic & Time-Of-Flight modes of operation**
- **Extremely flexible and optimised instrument resolution**
- **Polarised neutron beam and spin analysis**
- **High magnetic field capability**
- **High neutron flux – given by the ILL's HFR**

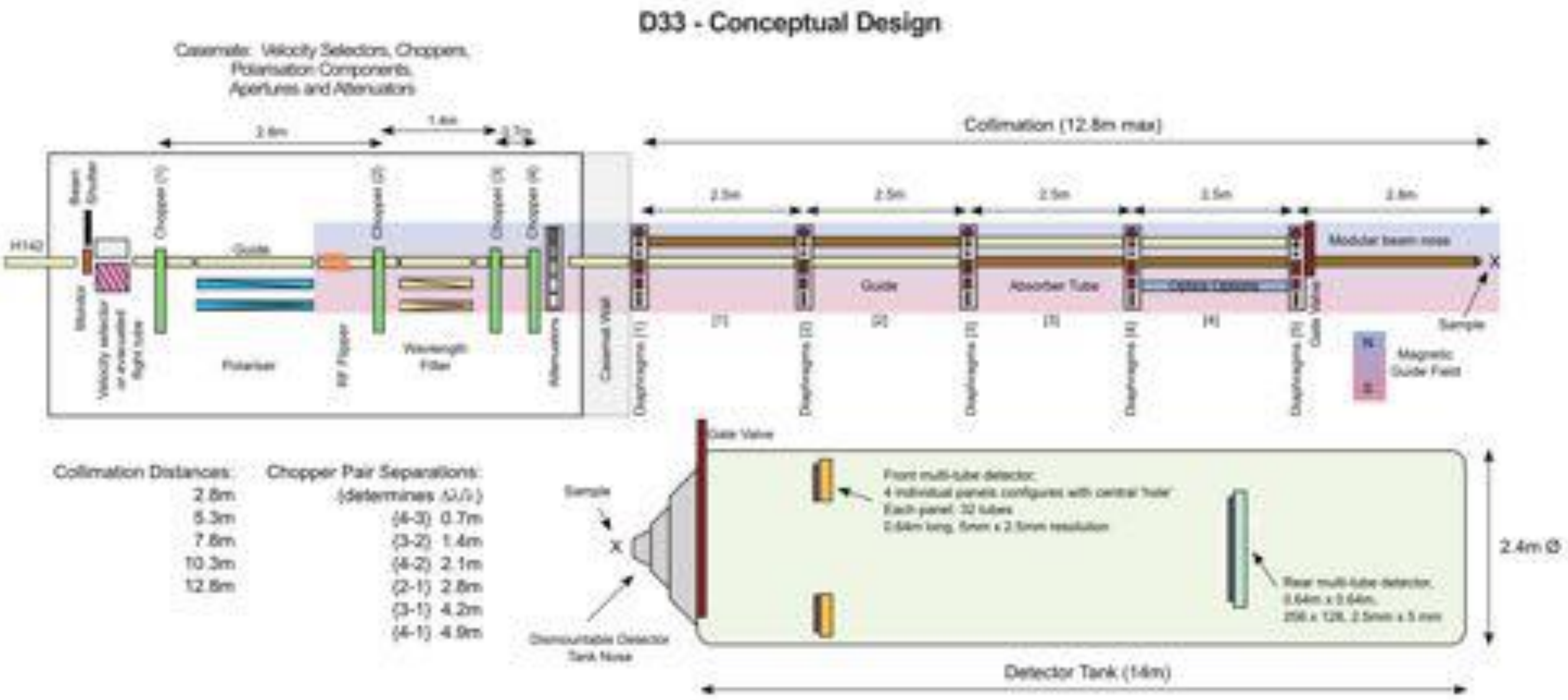
Construction costs : ~ 3.3 M€
+ H14 guide rebuild and ILL7 guide hall

Commissioned 2012

Concepts developed for D33 are inspiring new instrumentation approaches elsewhere: e.g. VSANS (NIST), Bilby (ANSTO)

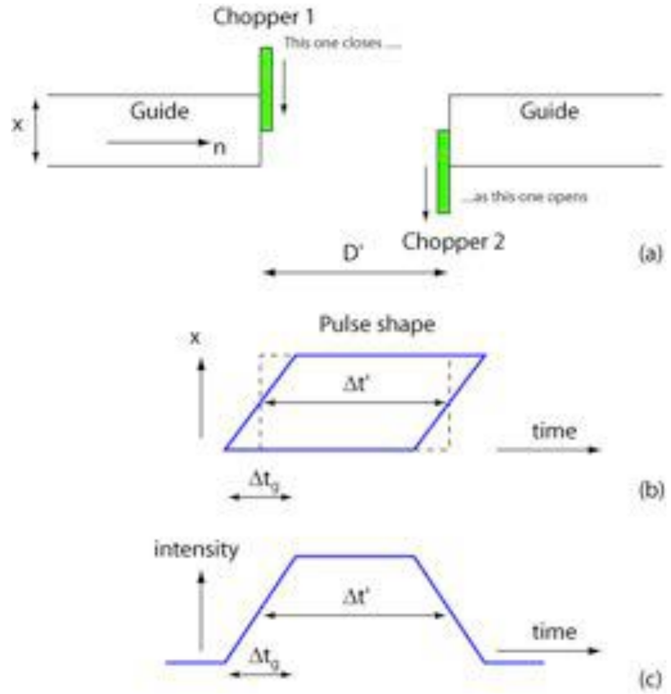

D33
13m + 13m
D11
40m + 40m

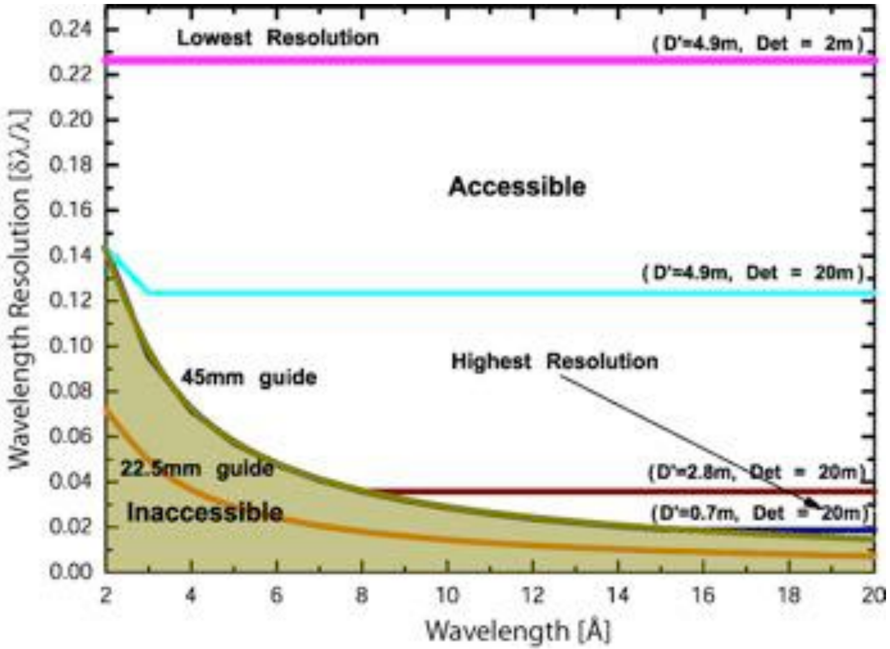
D33:


D33 - Conceptual Design
Casemate: Velocity Selectors, Choppers, Polarisation Components, Apertures and Attenuators
Collimation (12.8m max)
H142
Guide
Polariser
Wavelength Filter
Absorber Tube
Modular beam nose
Sample
Magnetic Guide Field
Gate Valve
Collimation Distances:
2.8m
5.3m
7.8m
10.3m
12.8m
Chopper Pair Separations:
(determines Δλ/λ)
(4-3) 0.7m
(3-2) 1.4m
(4-2) 2.1m
(2-1) 2.8m
(3-1) 4.2m
(4-1) 4.9m
Front multi-tube detector
Rear multi-tube detector
Dismountable Detector Tank Nose
2.4m Ø
Detector Tank (14m)

D33: How to make TOF-SANS work on a continuous source

- Problem with D33 V1.0 - TOF mode unable to achieve highest wavelength resolutions
- Nominal chopper speeds are very slow ~ few 100's RPM
- Cannot achieve $\delta\lambda/\lambda$ ~ 2% with slow choppers and large guide cross-section

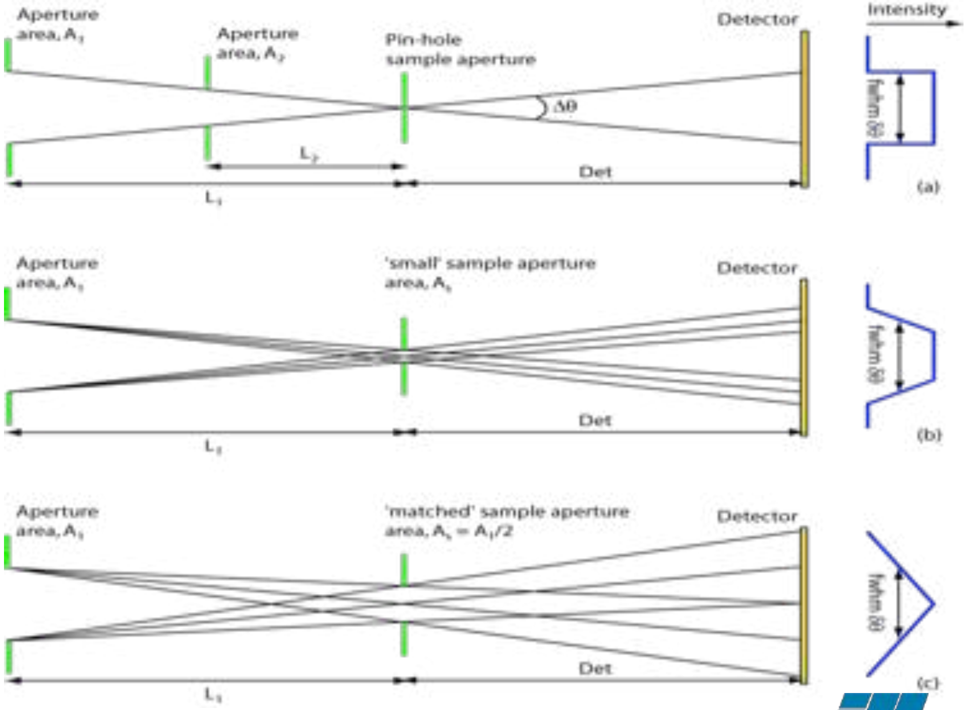




D33: HOW TO MAKE TOF-SANS WORK ON A CONTINUOUS SOURCE

TOF timing – re-evaluation of the SANS pin-hole geometry

- Why are SANS instruments so large?
- Neutron flux ? ~ 10^8 n/cm^2/s
- Guide sizes ? ~ 50 x 50 mm
- Sample Sizes ? ~ 1cm^2
- ....SANS instruments still too big!



Be brave ....make D33 <u>smaller</u>!

- Smaller guide
- Shorter instrument
- Faster choppers
- Smaller samples
- Less flux

....but same brightness!

D33: How to make TOF-SANS work on a continuous source

TOF timing − Spin the choppers faster

- Need more choppers to make frame selection?
- Smart solution: Configuration of the existing choppers

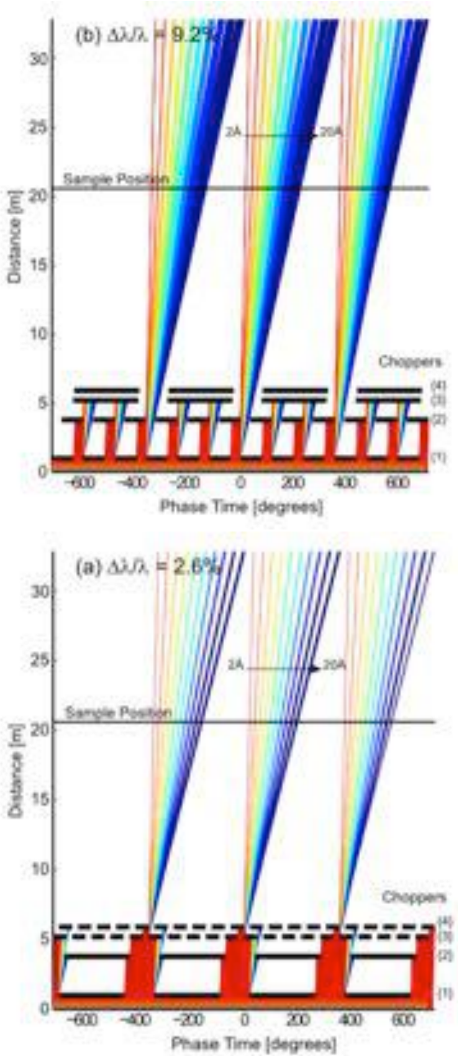
Be brave ....make D33 <u>smaller</u>!

- Speed multiplication up to x6 f_0
- Smaller guide and shorter instrument

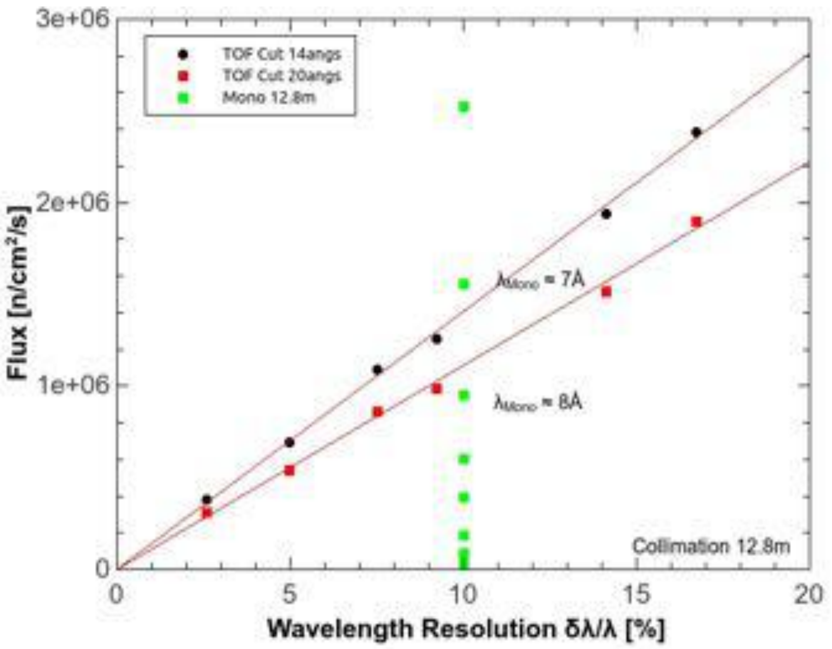
  → Higher f_0

- Reduce guide cutting time by factor > 10

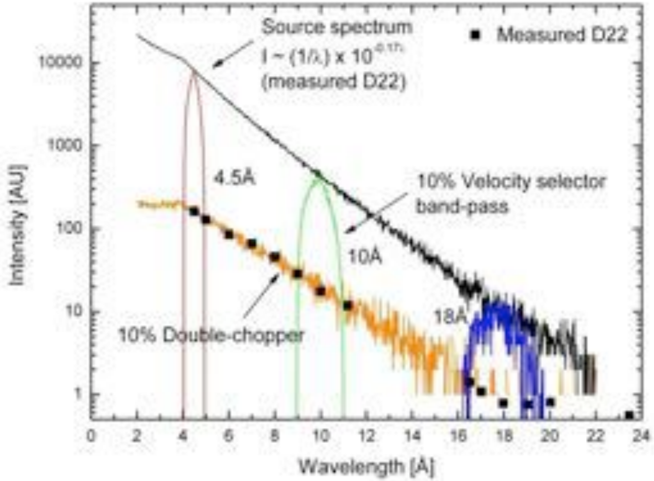
  → High resolution mode possible



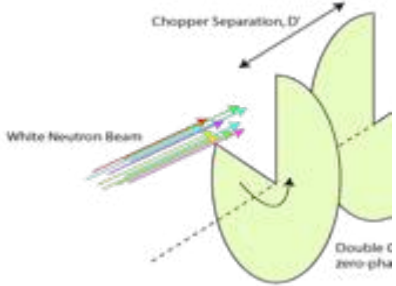
D33: TOF FLUX & RESOLUTION



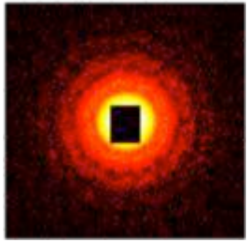
- 6 $\delta\lambda$ resolutions between 2% → 25%
- Flux is as expected
- TOF 10% ~ Mono 8Å 10%
- Remember: Same flux but spreading same number of neutrons more 'thinly' over *q*-space



vs.

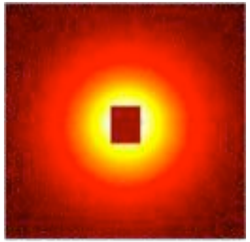


D33: MONOCHROMATIC SANS VS. TOF SANS



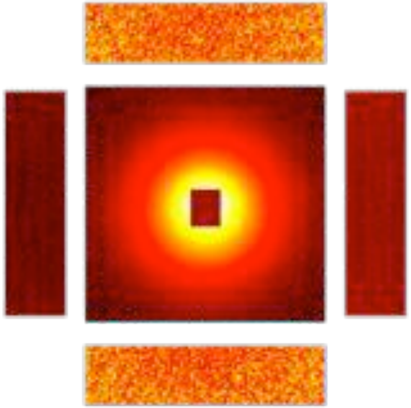
- **Monochromatic: 1 Detector**
  $q_{max} / q_{min} \sim 10$

x10



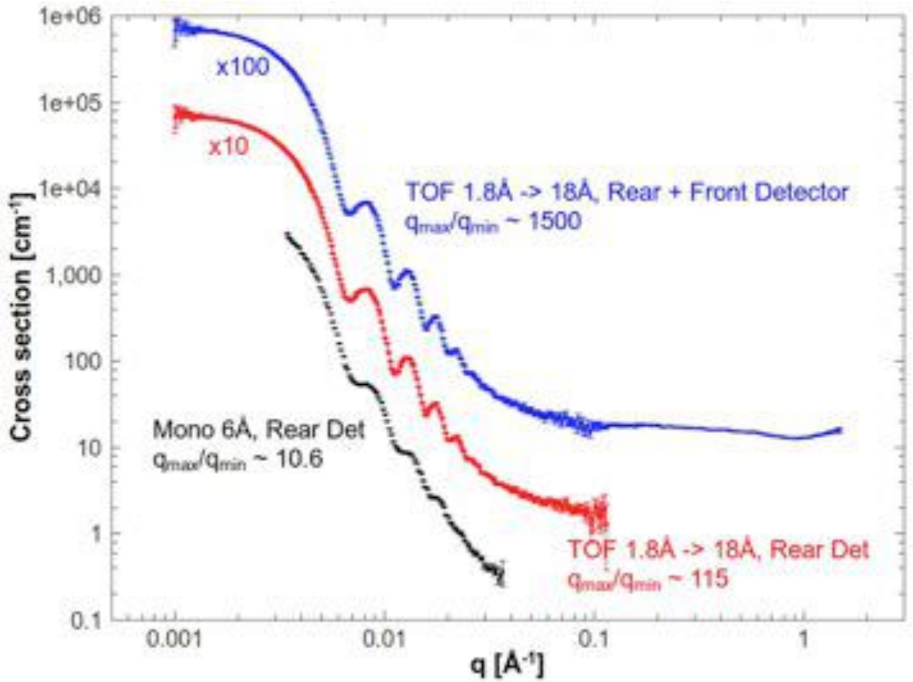
- **TOF: 1 Detector**
  $q_{max} / q_{min} > 100$

x10



- **TOF + Front & Rear Detectors**
  $q_{max} / q_{min} > 1000$

Massive dynamic q-range, q_{max} / q_{min}



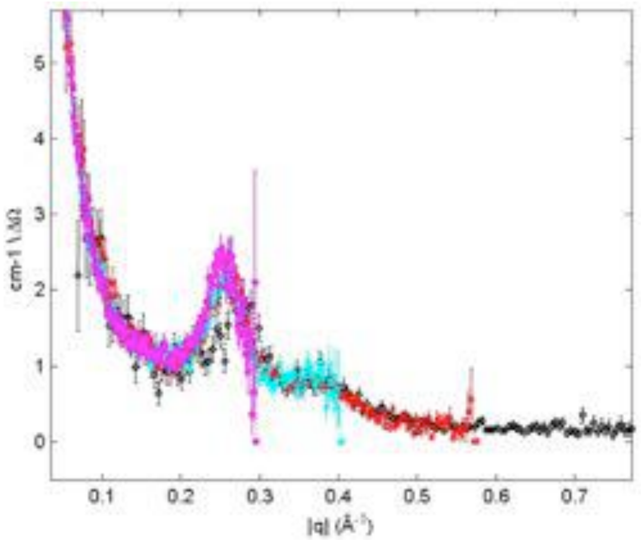
Latex Spheres in D_2O, r ~ 700Å

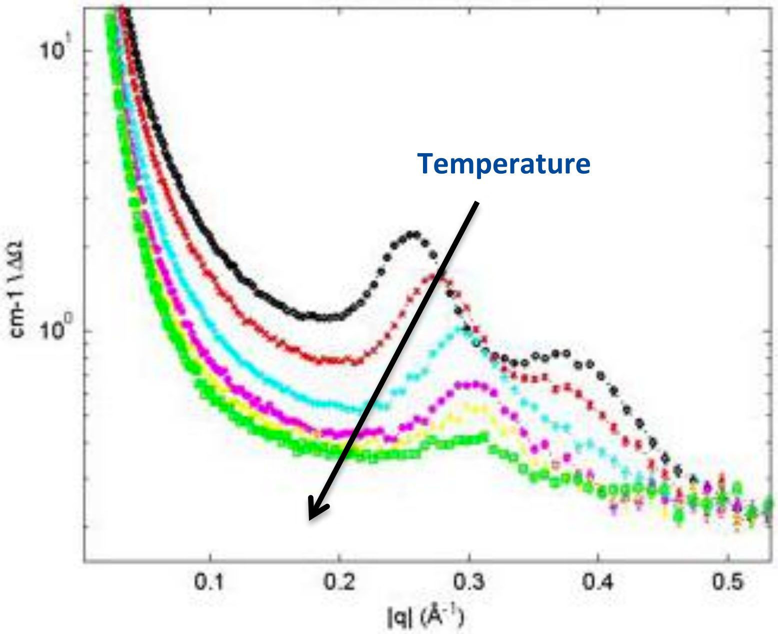
Det1 = 1.2 m, Det2 = 12.8 m, 9.2% $\delta\lambda / \lambda$
20 minute count time

D33: MONOCHROMATIC SANS VS. TOF SANS

Hard matter & magnetism

- Wide dynamic q-range
- Access to higher q's using small λ
  (useful for restricted sample environments)
- No problems of inelasticity



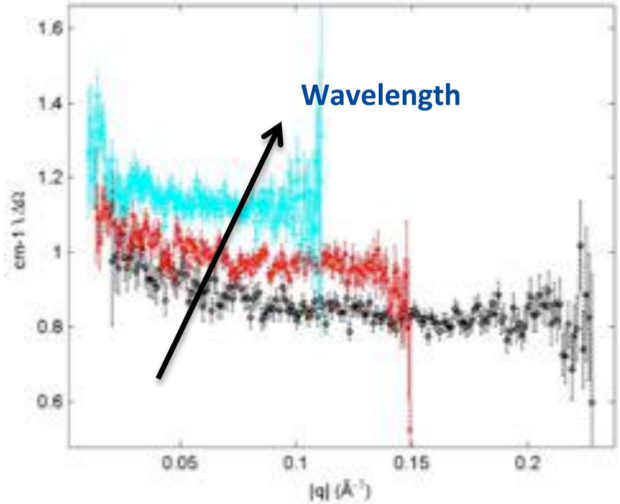


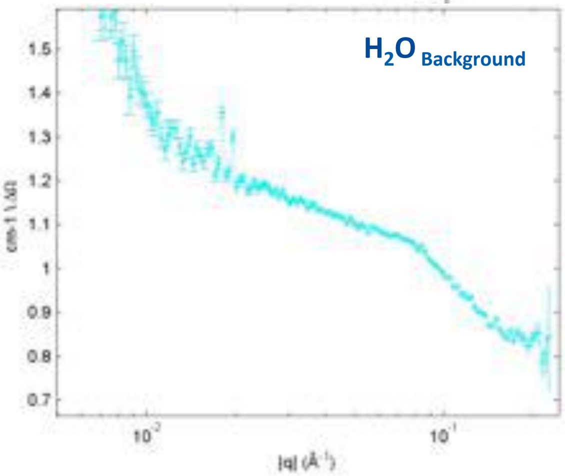
Nano-crystalline Holmium

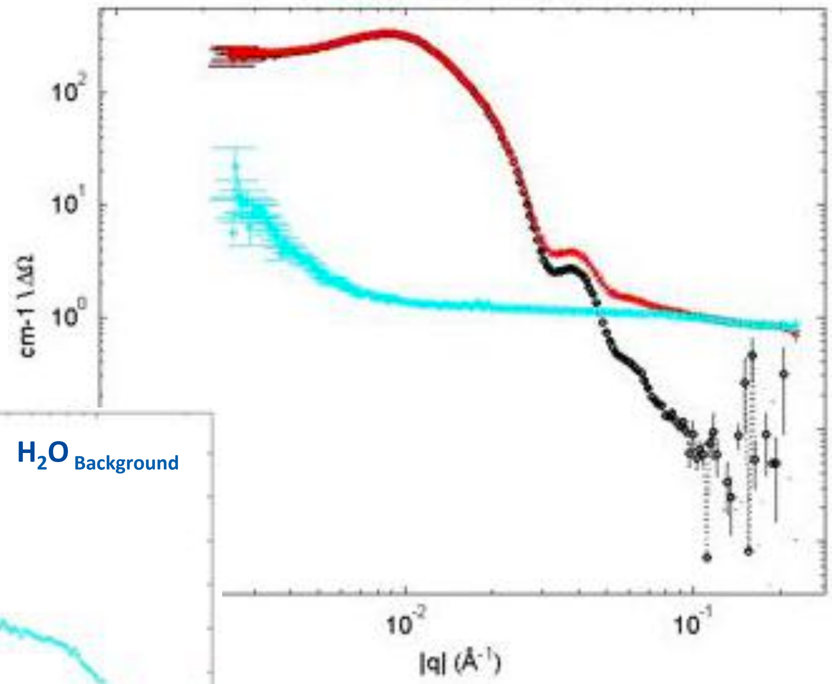
D33: Monochromatic SANS vs. TOF SANS

Hydrogenated materials & inelastic scattering

- Inelastic scattering from H − shows up in ‘wrong’ time channels
- Cross-section of H_2O not constant with λ
- OK for dilute samples − background







Silica Spheres in H_2O

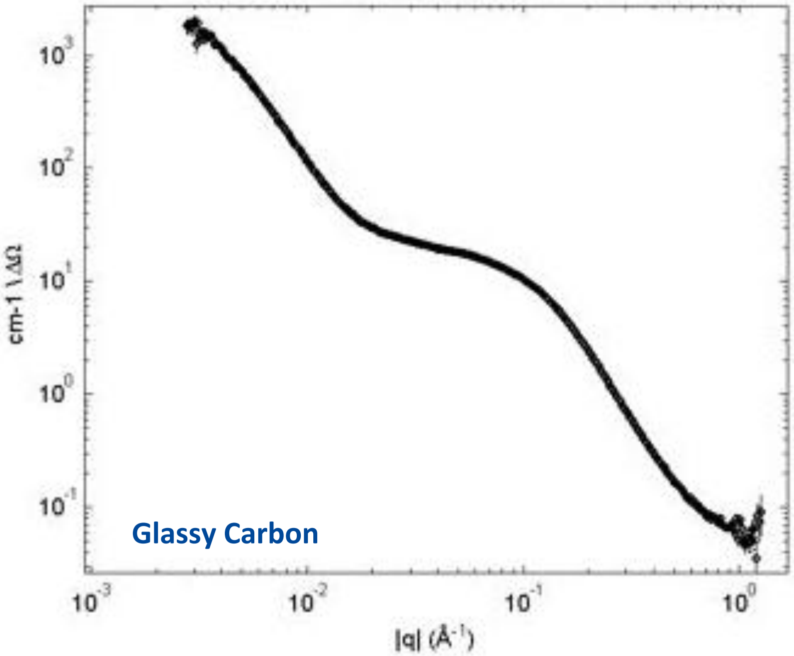
D33: MONOCHROMATIC SANS VS. TOF SANS

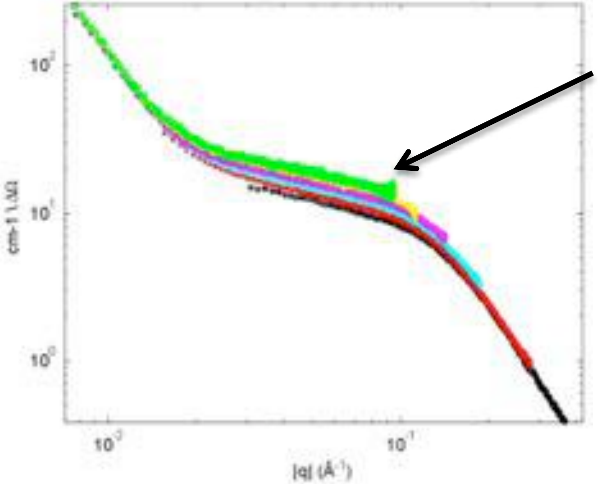
Glassy-carbon 'round-robin' standard sample

- Really great data in TOF
- Massive dynamic *q*-range

But beware:

- Samples need to be well adapted to measurement in TOF (or Mono)
- More 'aware' of inelastic & multiple scattering

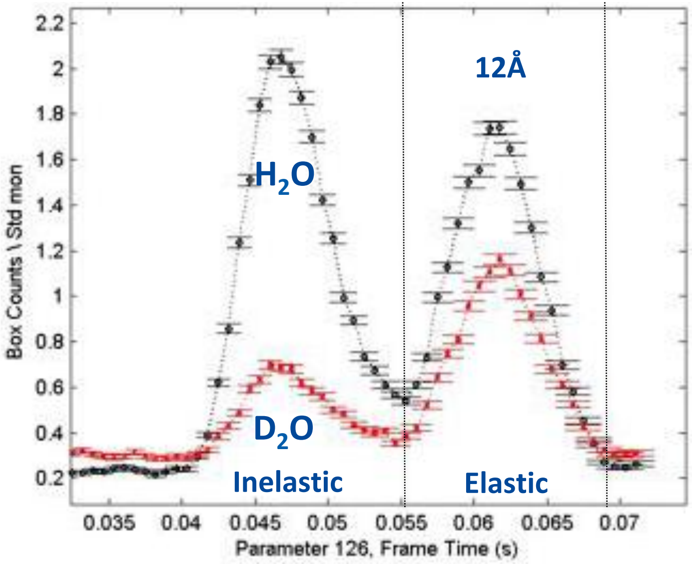




TOF time frames: 2Å, 4Å, 6Å, 8Å, 10Å, 12Å

Note sample transmission:
0.93 (0.9Å) → 0.51 (17.8Å)

D33: Enormous flexibility = New possibilities

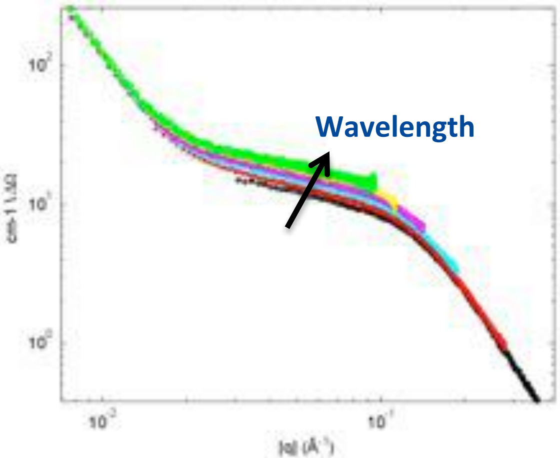


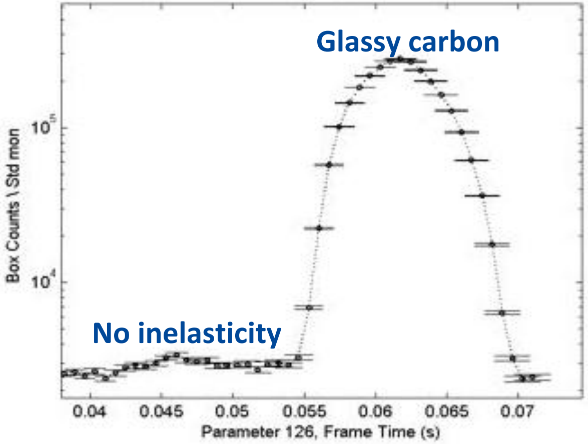
- With a monochromator (selector) AND chopper system can easily look at inelastic effects, i.e. thermalisation

- Can use TOF + Mono to remove much of the inelastic (incoherent) background from light elements, specifically in solution

...or at least ....

- Diagnose issues with data, samples, measurements, might come from (...and de-bug spallation sources!)

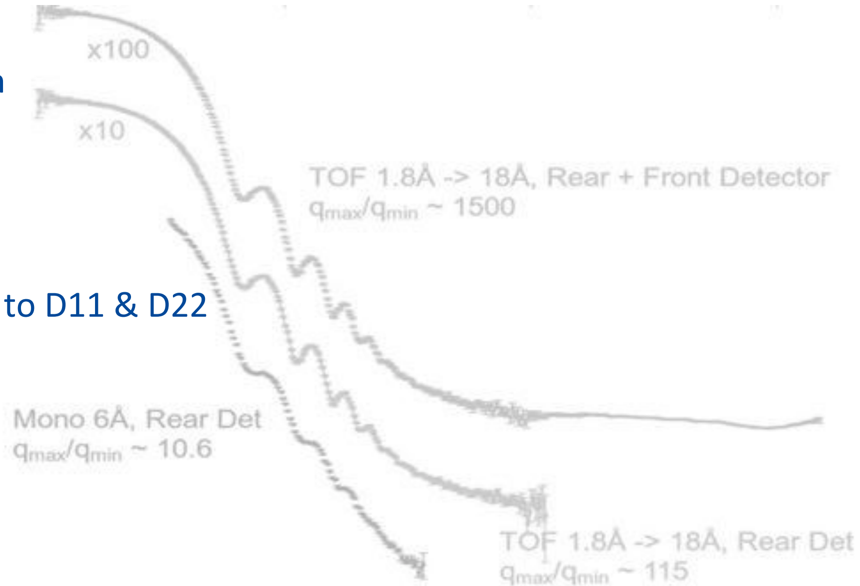




CONCLUSIONS:

SANS:

- What's important & why: Understanding resolution



D33:

- Neutron Flux: Brightness ~ same as D22
- Monochromatic mode optimised further compared to D11 & D22
  - Detectors (2x larger solid angle coverage)
  - Fast configuration changes
- Polarisation & Analysis
- Large & flexible sample zone – Really useful
- No magnetic field interference with neighbours

Time-of-Flight SANS:

- Massive dynamic *q*-range (TOF + 2 Detectors): Up to q_{max} / q_{min} ~ 1000
- Useful for kinetics
- Access to the highest *q*'s & short wavelengths or restricted sample environments
- Learning a lot about scattering by comparison between mono & TOF SANS data on same samples
- Still lots of challenges in understanding TOF / Mono data & data treatment