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What is learnt about SANS Instruments and Data Reduction from Round Robin Measurements? A Polymer Latex 'Standard'

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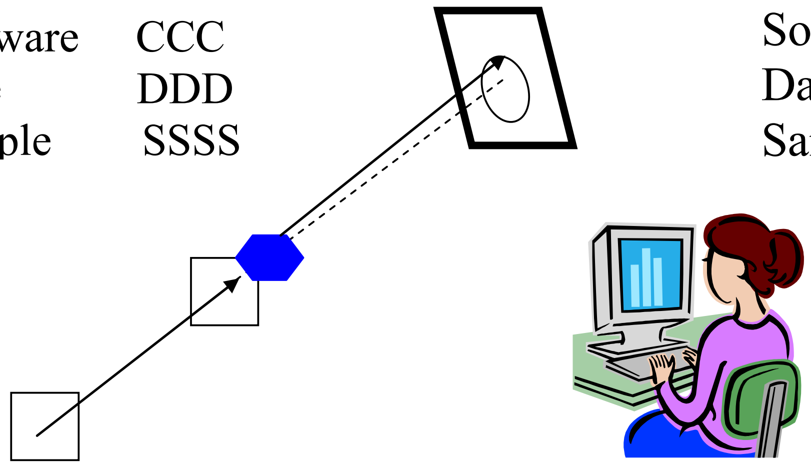
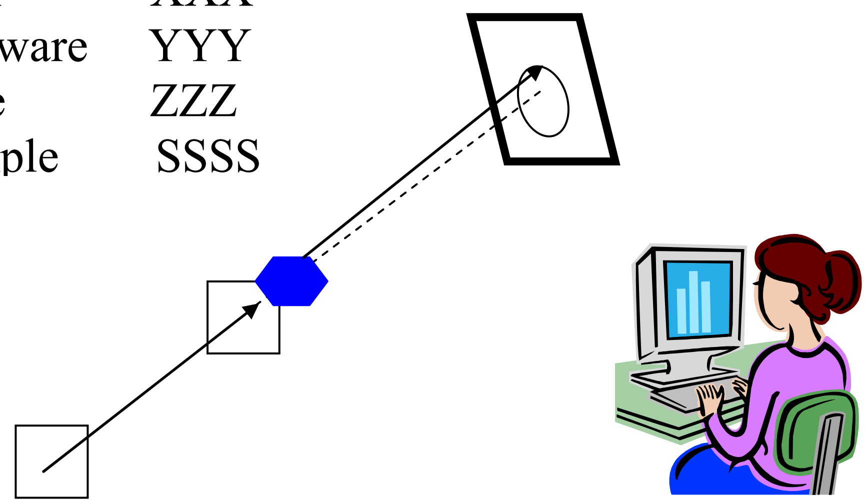
What is standardisation?

Instrument	AAA
User	BBB
Software	CCC
Date	DDD
Sample	SSSS



Instrument	WWW
User	XXX
Software	YYY
Date	ZZZ
Sample	SSSS



Do I get the same result? Has the sample changed?
How sure am I?

How do we obtain similar results?

Are results consistent?

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- Is the size (distribution) the same as that from electron microscopy, light scattering, GPC ?
- Does SAXS and SANS give the same result?
- Do I have the same conclusion from model fitting and inversion procedures?

Do we understand the differences?

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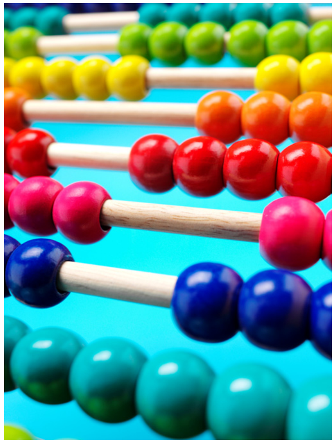
More than Calibration

- Wavelength
- Distance
- Angle
- Intensity
- Resolution
- Uniformity of detector
- etc.



How do I check these quickly?

Different Questions?

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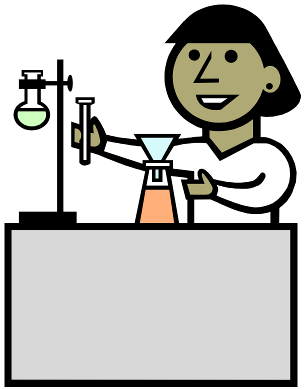

User: Do I understand the data? Are my results publishable?

Instrument scientist: Why are results different? Can the user publish the data?

Facility Manager:

My instruments are the best?

Everyone needs to understand better!

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Why Standardisation?

Comparisons:

- Samples
- Instruments
- Procedures
- Techniques
- Software

Provide understanding of small-angle scattering!

Co-operation and comparison helps this understanding

canSAS

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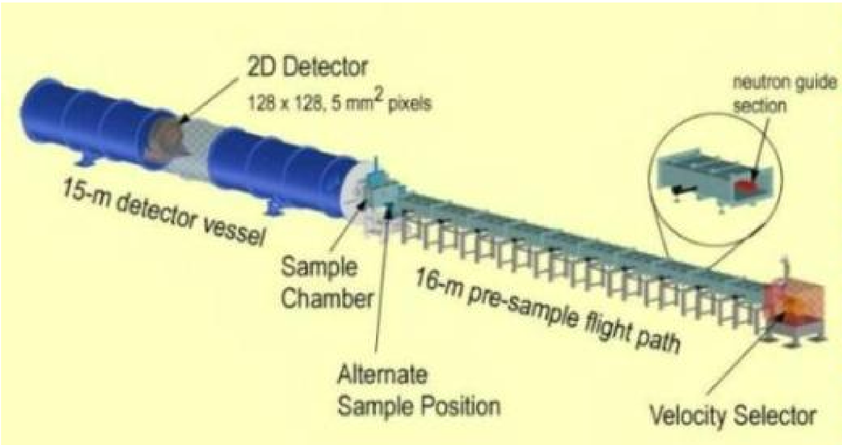
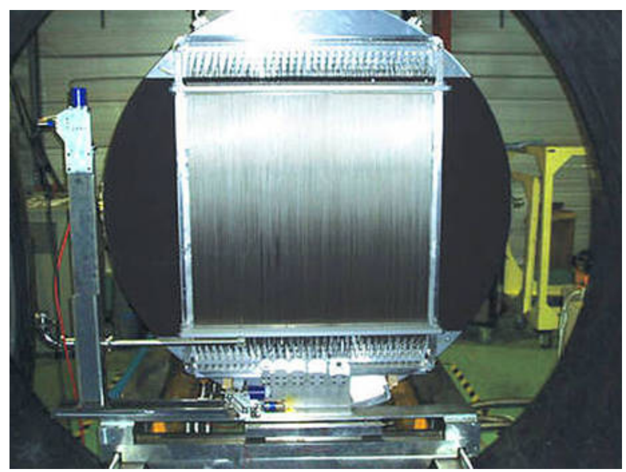
Instruments



NG7, NCNR, USA

Bragg Institute, Australia


SANS2D –ISIS, UK



D22 and D11, ILL, Grenoble, France

Round Robin Sample

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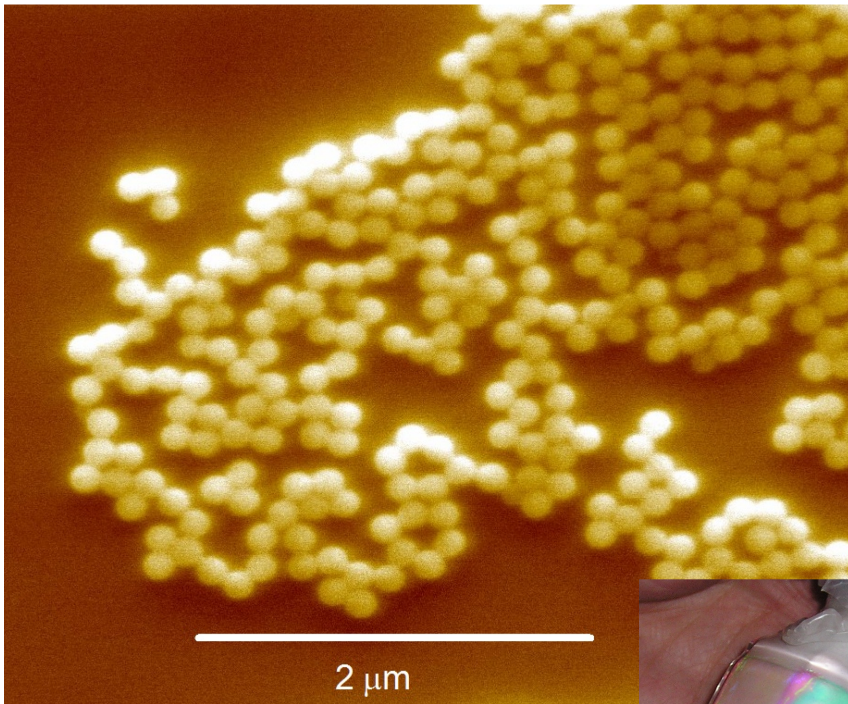
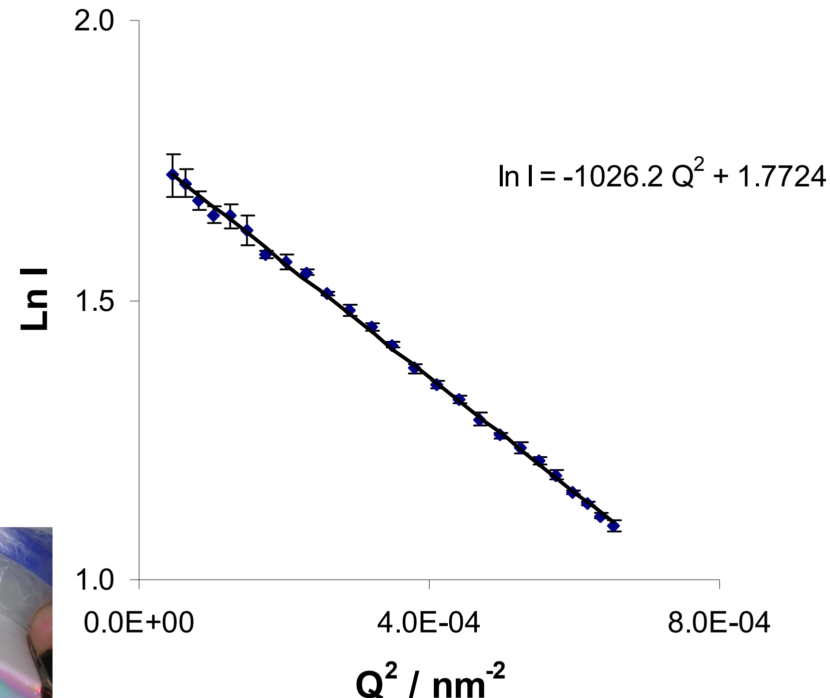


SEM 5 keV uncoated latex on Si wafer

8% - Diffracts light

PS3 Polystyrene latex in D_2O



Static light scattering – ALV HeNe laser R_g = 56 nm

R = 716 Å +/- 2 Å

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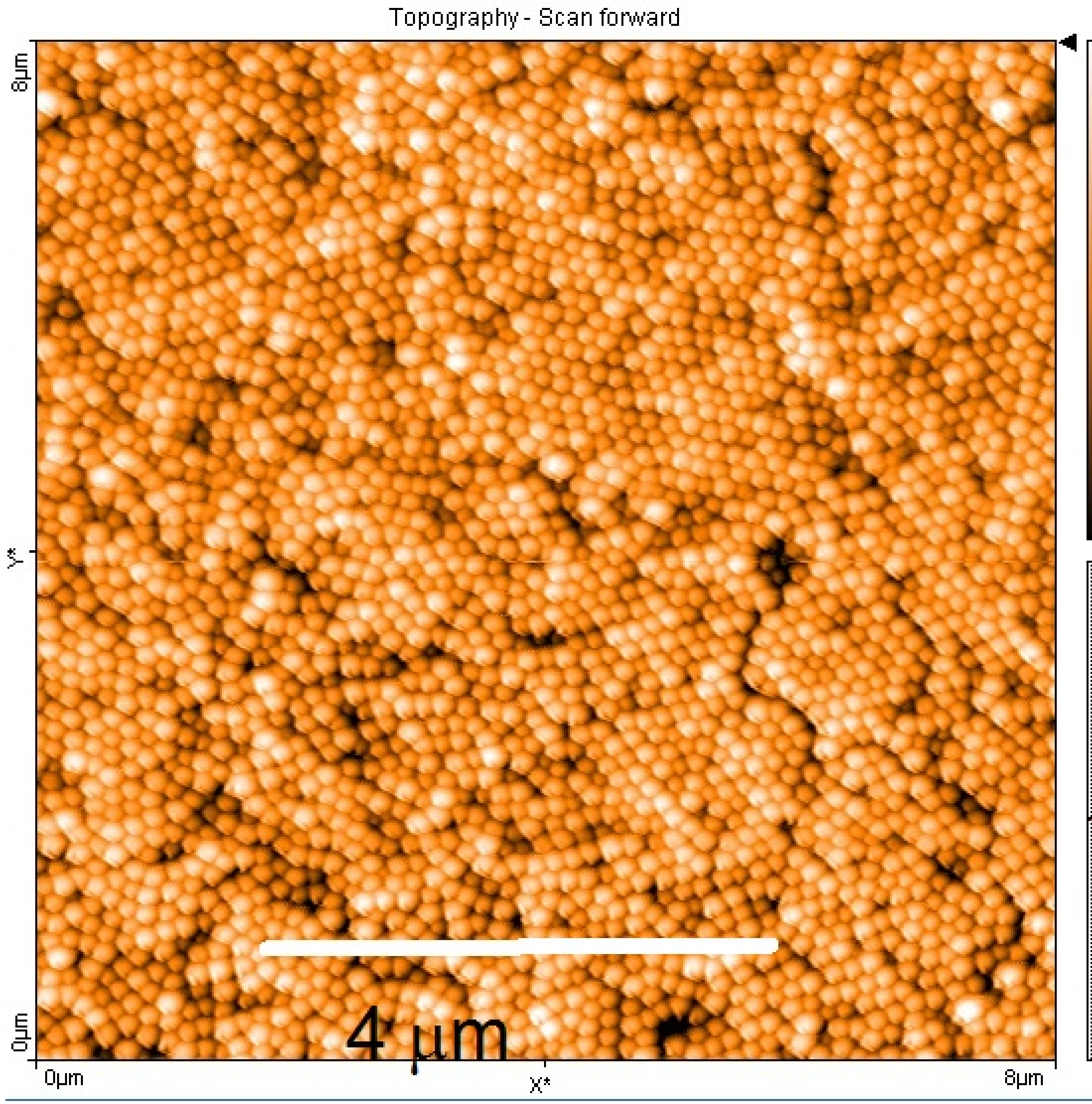
AFM

Orders on surfaces and in the bulk

Topographic mode
Nanoscope SM



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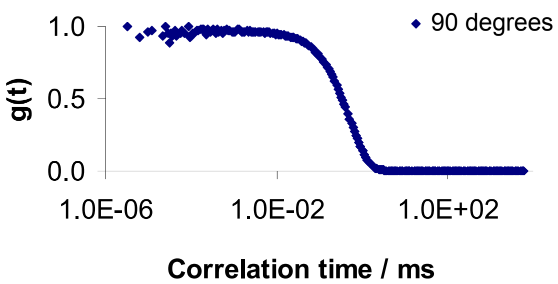
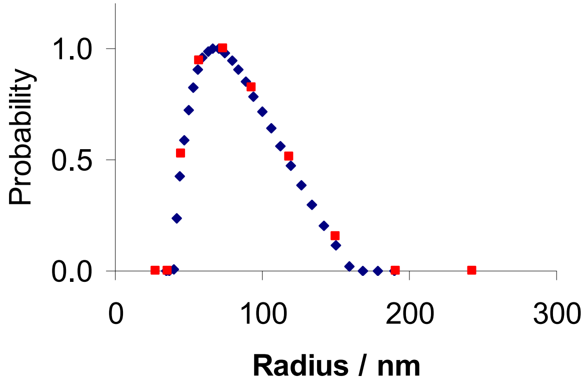
Dynamic Light Scattering





R_H = 71 to 73 nm

Polydispersity ~ 5%

Good agreement with SLS and SEM

λ = 633 nm, θ = 90° : data measured also at other angles

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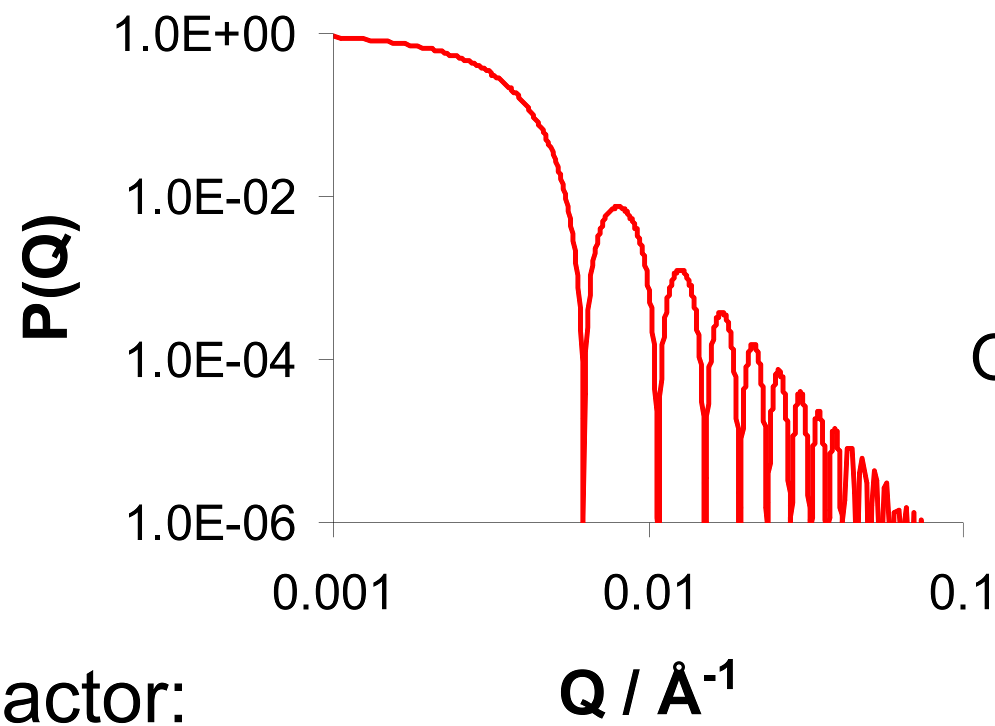
Scattering from Spheres



$Q = (4\pi/\lambda) \sin \theta/2$

Form Factor:

$$P(QR) = \{3(\sin(QR) - QR \cos(QR))/(QR)^3\}^2$$

Intensity:

$$I(Q=0) = (\Delta\rho)^2 \, n \, V^2$$

0.43% Latex in D_2O

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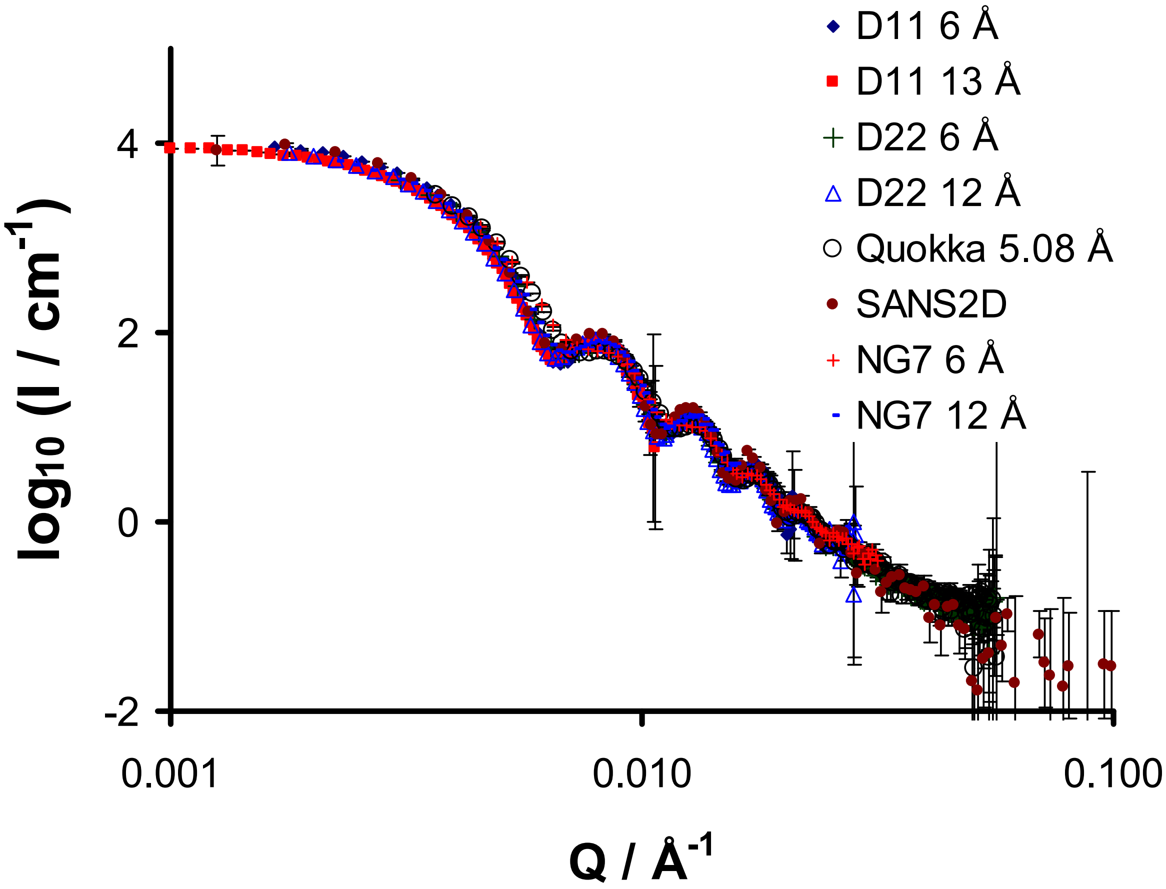
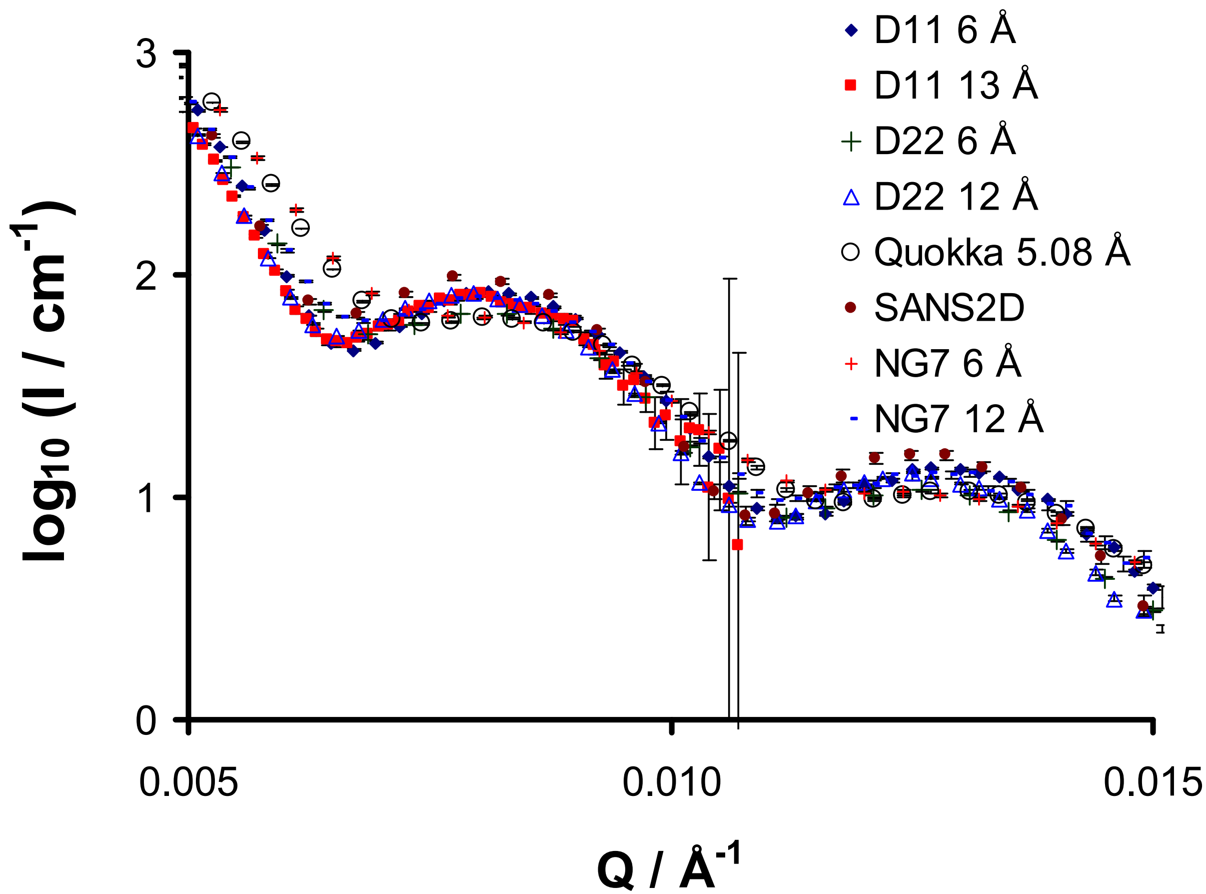
1 mM NaCl



Differences – Measured data

0.43% Latex in D_2O 1mM NaCl



Are some data wrong?

Differences – Measured data

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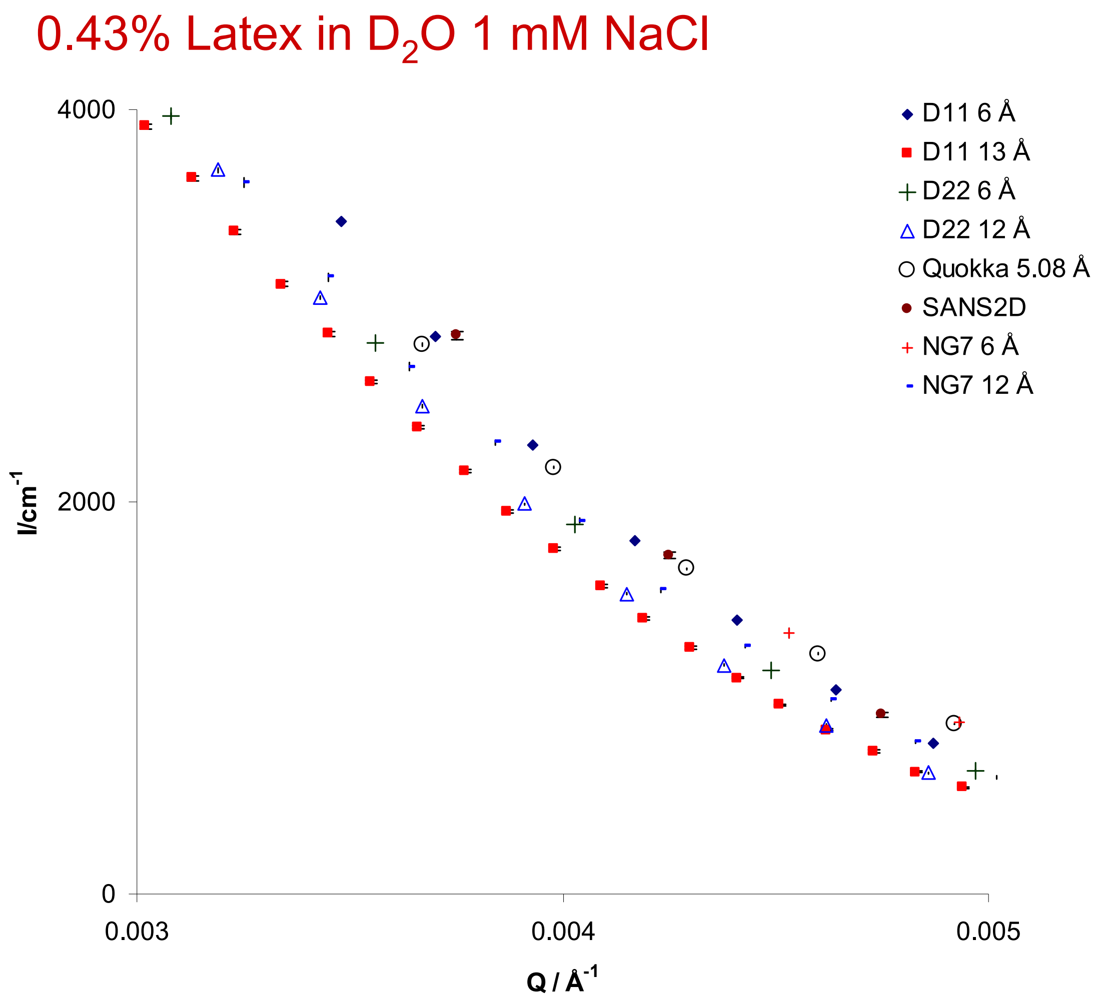

Are some data wrong?

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Presenting Data

Conclusions

- Logarithmic scales do not show everything well!
- Data are not necessarily wrong but perhaps misinterpreted
- Need more information – better description of metadata and uncertainties

Simple Fits – SasView Spheres

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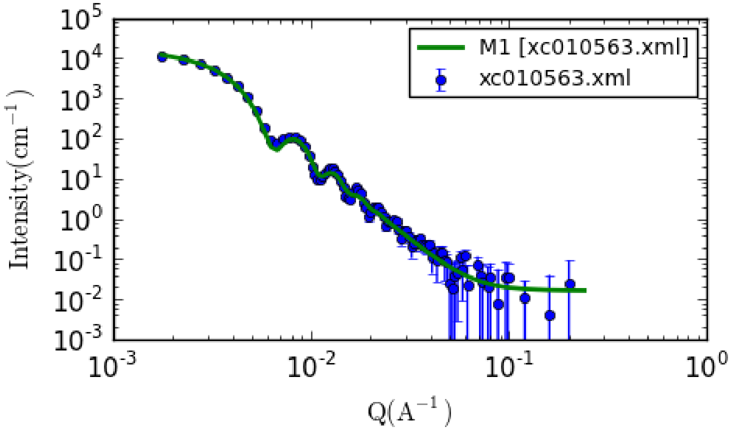
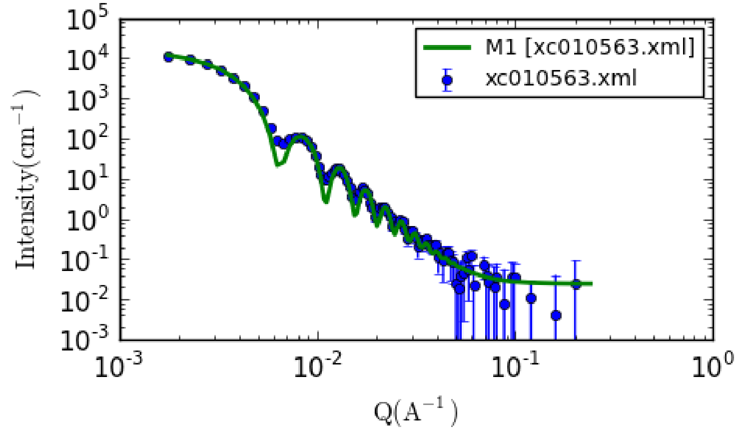
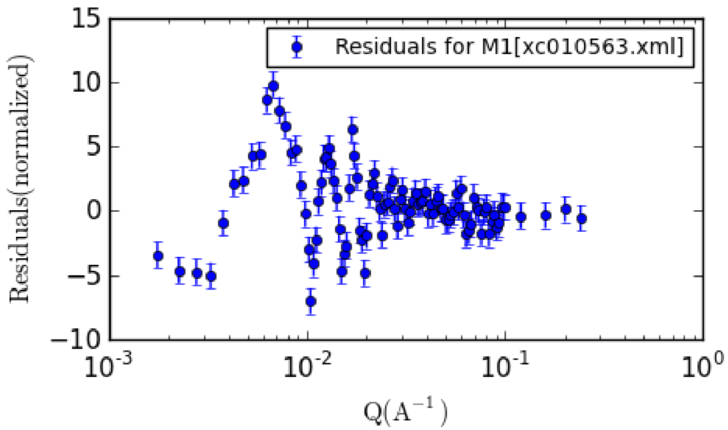
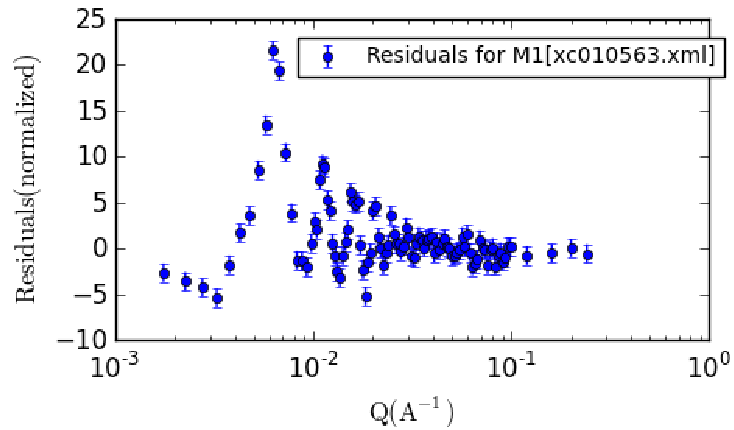




Polydispersity: 8%

3%





SANS2D data: Which fit is better? Both show systematic deviations!

Which fit is best?

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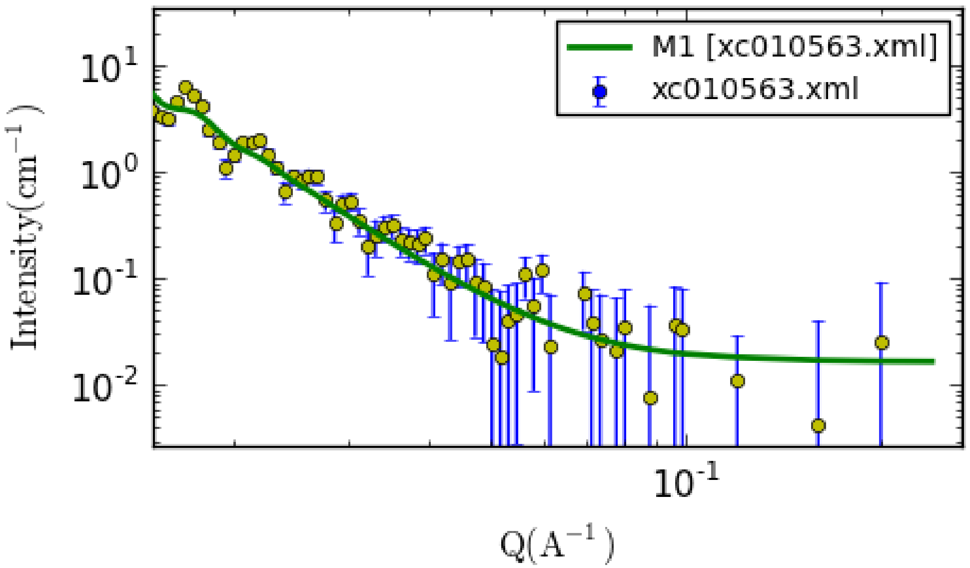
- Better (when choosing from 2) but neither is best!
- 8% polydispersity has smaller χ^2 but misses all large Q features
- Need more information



Fit with 8% polydispersity

R either 687 Å or 703 Å

(polydispersity 8% or 3%)

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Model Fitting

Need to include:

- Resolution
- Polydispersity
- Multiple scattering
- Interactions ?

Effects are similar but not identical

Variation with Q and concentration is different

Different Concentrations

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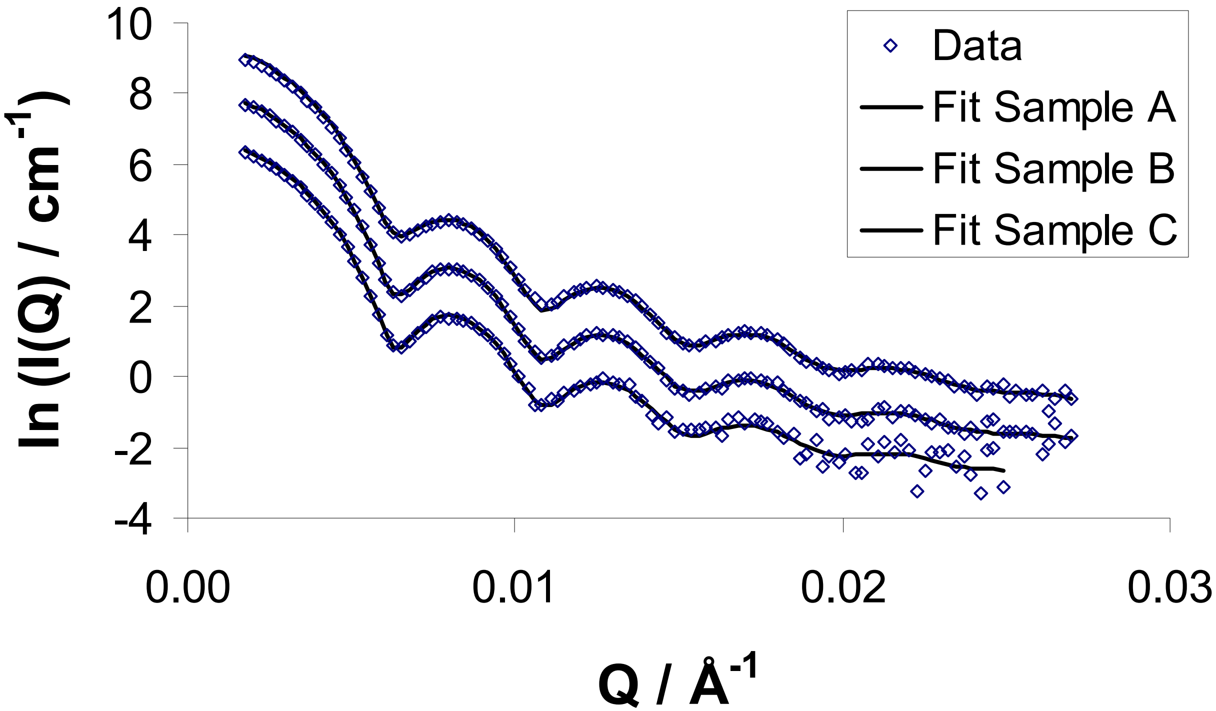
R = 721 Å

σ = 21 Å (~3%)

Sample	*Concentration / wt %*
A	0.43
B	0.11
C	0.03



D22 data: simultaneous fit hs2m

includes resolution and double scattering

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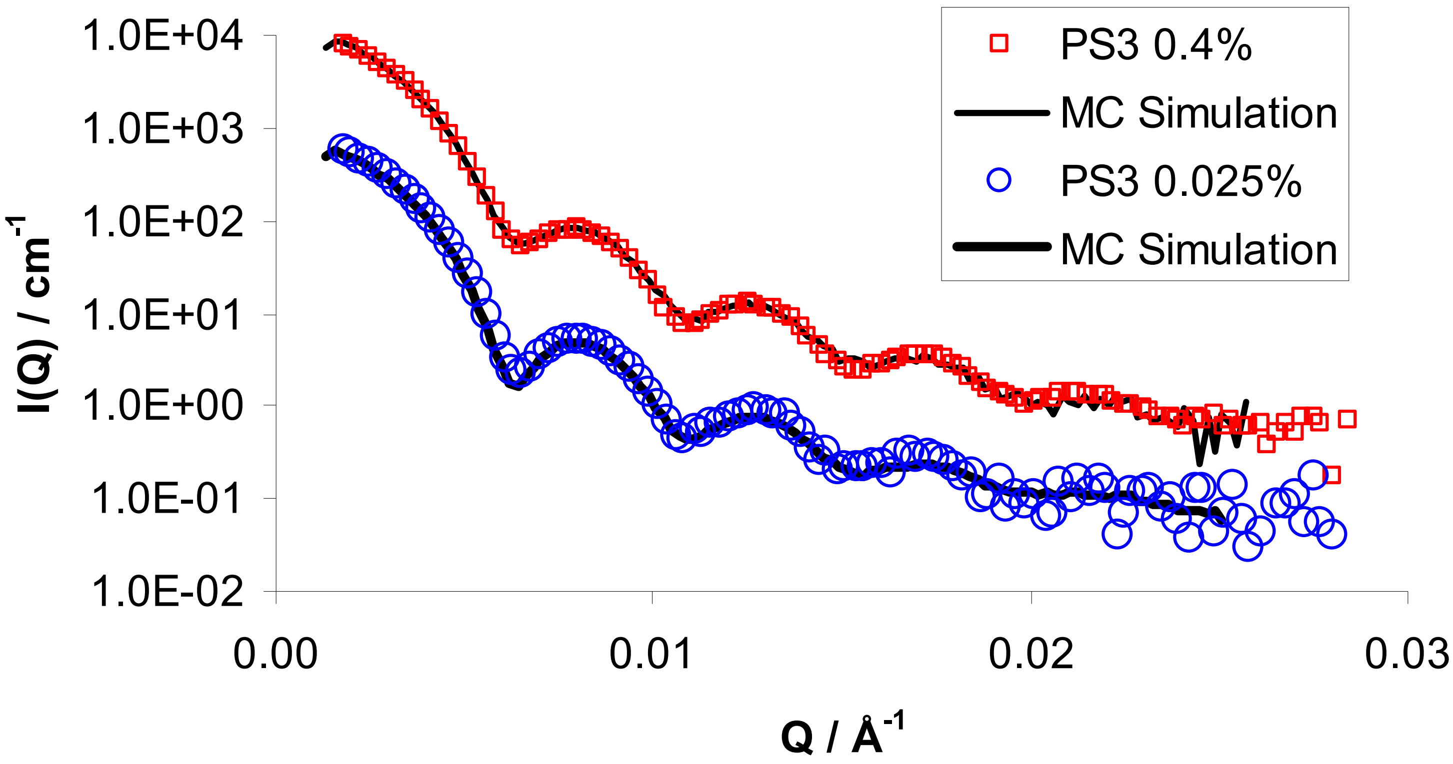
Monte Carlo Simulation



D22 data MC simulated with NCNR IGOR programs (J. G. Barker, S. G. Kline et al)

Multiple Scattering

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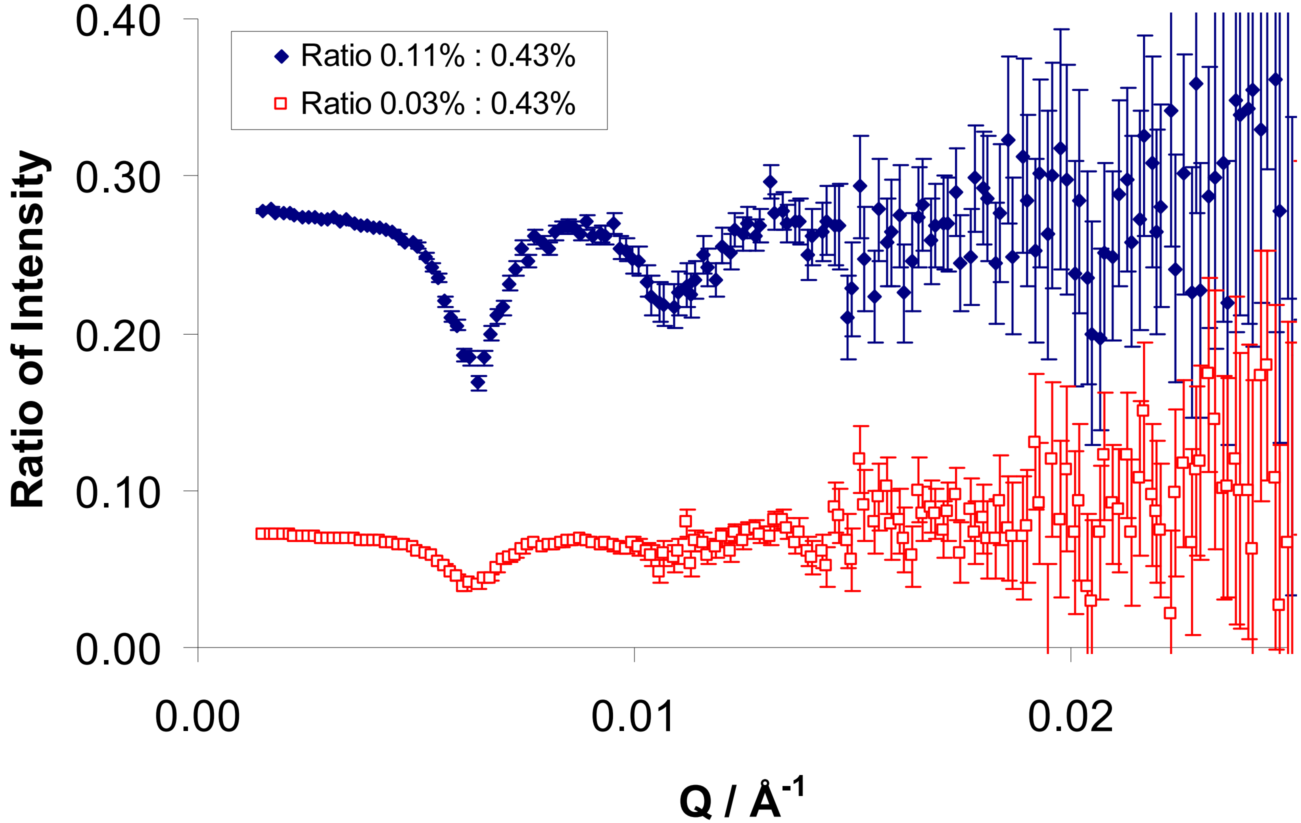
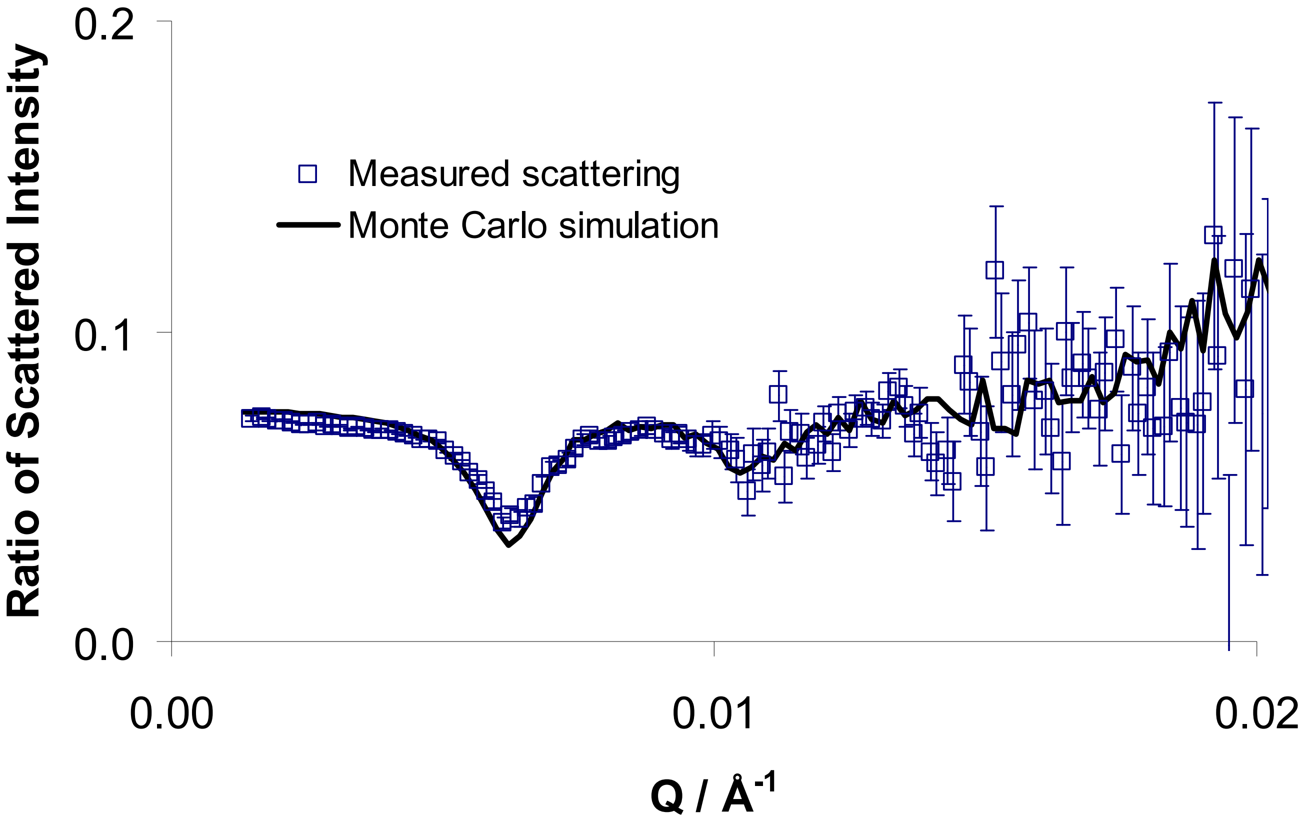


Data: D22, ILL 12 Å

Compare Ratio - Data & MC



Monte Carlo modelling can account for smearing by multiple scattering

Calculations for R = 705 A 4% polydispersity

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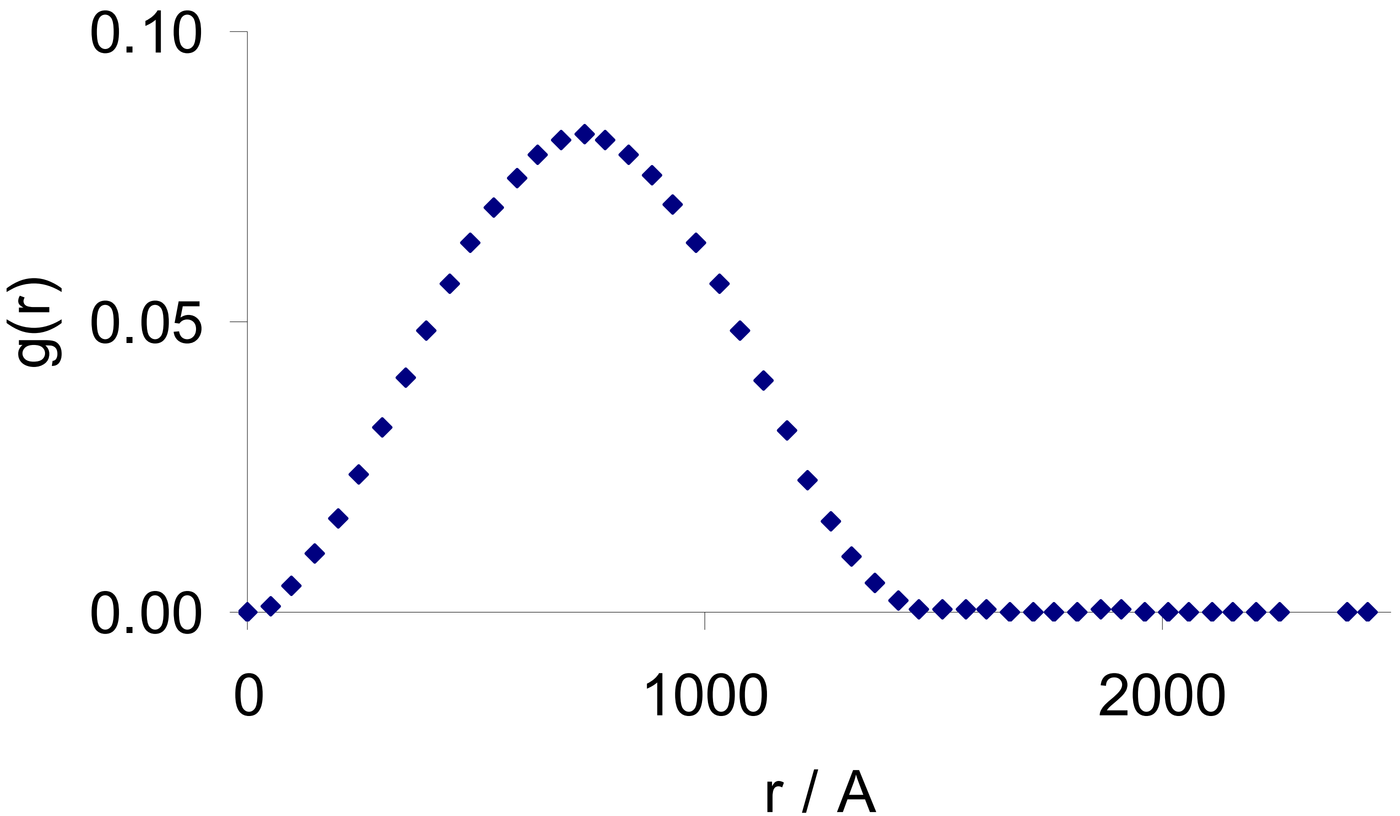
Analysis Methods

- Guinier analysis – limited fit information and needs low Q – no resolution
- Modelling scattering – multiple data sets and detailed knowledge of instrument/resolution needed. Only limited multiple scattering.
- Monte Carlo – needs precise instrument geometry. Background is difficult but MC can include coherent multiple scattering

Inversion to g(r)



Inversion works when data extends over adequate Q range but (a) resolution is rarely considered and (b) g(r) needs to be interpreted.

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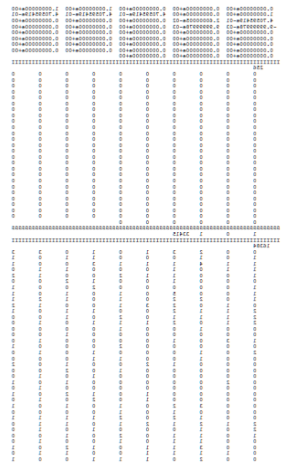
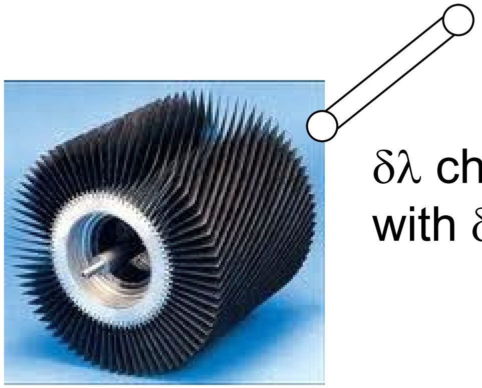
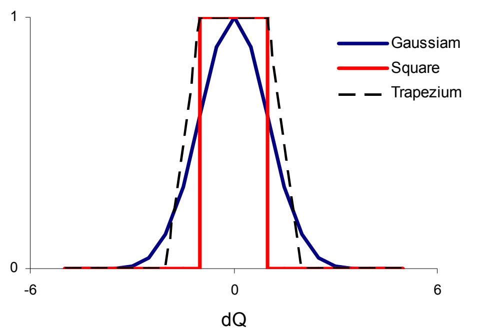
Challenges

Missing/incorrect metadata

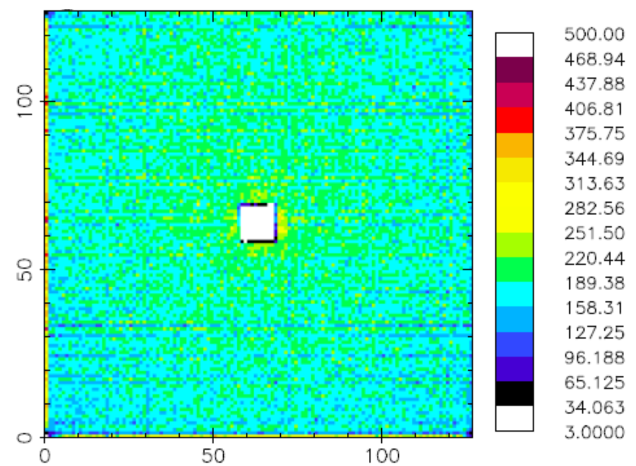




δλ changes with δθ



Resolution not Gaussian



Detector normalization changes with configuration

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Challenges

- Incorrect or missing metadata (e.g. to calculate resolution or multiple scattering)
- Wavelength resolution with velocity selector can depend on collimation
- Resolution function may not be a Gaussian – particularly on ToF instruments
- Detector normalization: inappropriate ‘flat field’ can distort data

Conclusions – What have we learnt?

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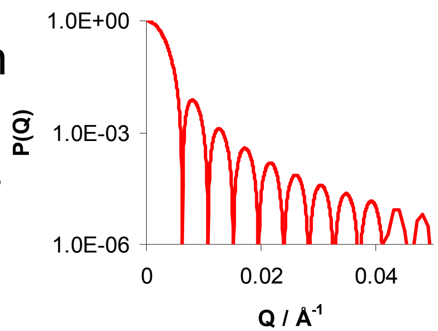
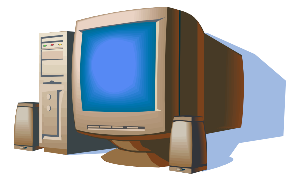
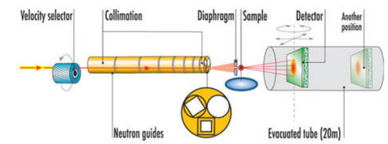
Data that can be modelled reliably helps comparisons

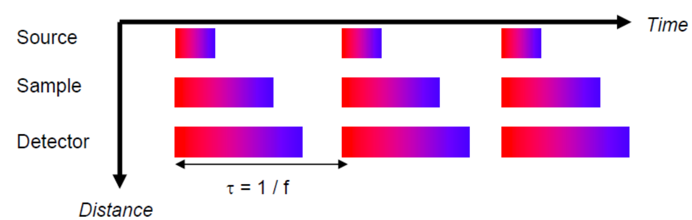






Compare instruments and software

Systematic deviations are often the largest source of uncertainty in interpretation



ToF and const λ measurements provide beneficial comparisons

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Conclusions

- Well-known form factor has identified problems with resolution, detector normalization and software
- Single wavelength data were easier to model in detail
- Time-of-flight SANS data with a wide Q-range and with good resolution highlighted multiple scattering
- Systematic deviations are often the largest source of uncertainty in interpretation
- Many other ‘unknown’ samples can show similar effects

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Recommendations

- Regular comparisons of instruments and procedures as well as software are helpful
- Data formats and publishing standards need to include uncertainty from systematic effects as well as counting statistics
- Do not be tempted to scale data to ‘match’ without allowing for resolution!
- Descriptions of data are essential - e.g. how is resolution described, σ, FWHM etc.?
- Density matched ‘sealed’ sample for long term reproducibility would be helpful

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Thanks

- Facilities and the Funding Agencies for the facilities
- Co-operation between many instrument scientists
- www.cansas.org

Thank you for listening

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Contact

Join in these activities?

Adrian.Rennie@physics.uu.se

Lunch time canSAS session at 12.40 today