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# Learning about Reproducibility, Reliability and Limits of Data Interpretation from uStudies

Adrian R. Rennie,

Materials Physics, Uppsala

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# Themes

- Introduction
- Standardisation
- Comparative Measurements
- Understanding SAS

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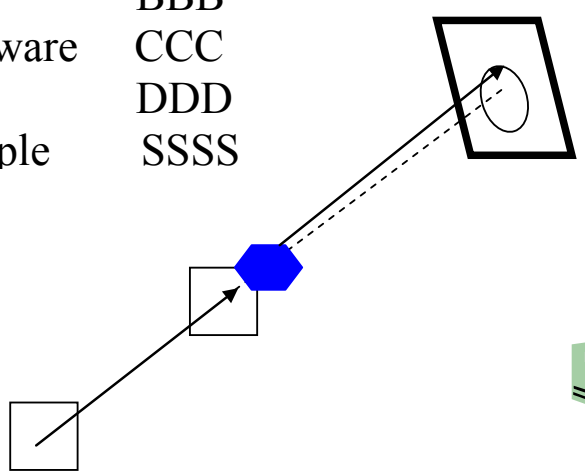
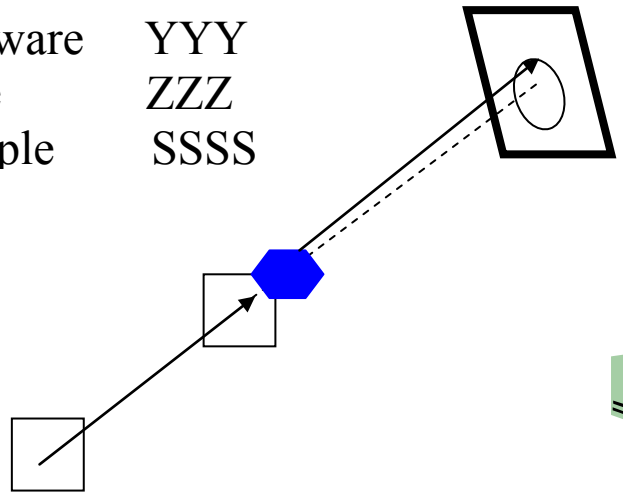
# What is reproducibililty?

| | |
|---|---|
| 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?

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# Reliability?

- Are we confident about our deductions from data?

Normally we need to communicate in terms of biology, chemistry, physics or materials science

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# Are results consistent?

- 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?

More than calibration!

Do we understand the differences?

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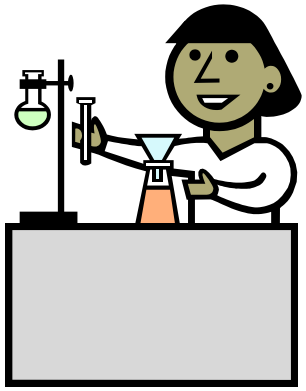
# Different Questions?



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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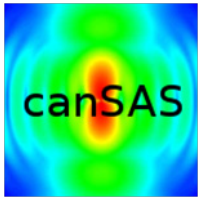
# Why Standardisation?

## Comparisons:

- Samples
- Instruments
- Procedures
- Techniques
- Software

**Provide understanding of small-angle scattering!**

**Co-operation and comparison helps this understanding**



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# canSAS – up to now

Round Robin present samples

Glassy carbon

Polystyrene latex

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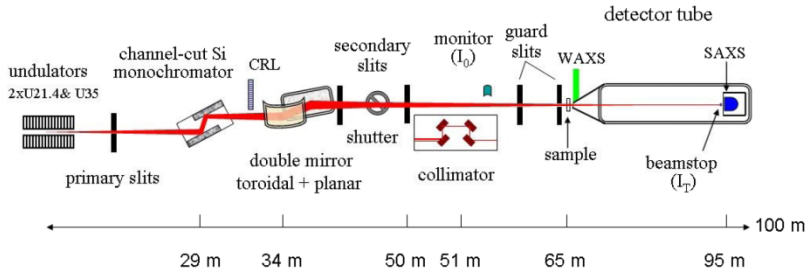
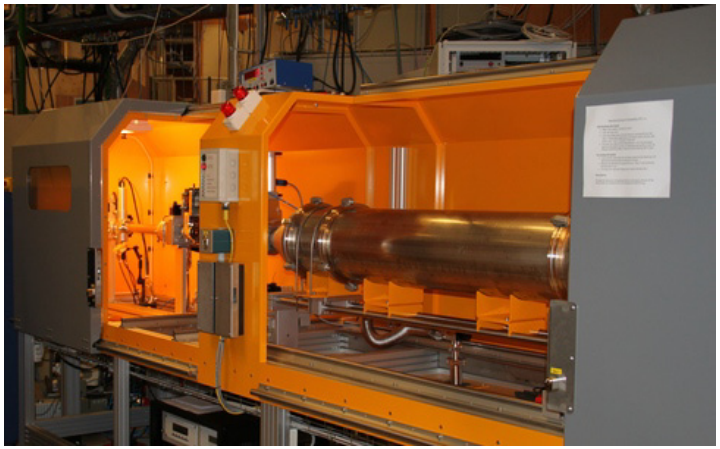
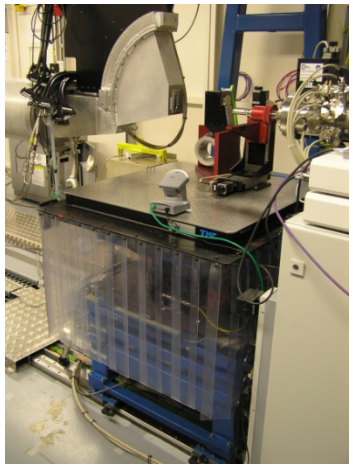
# SAXS Instruments




ID02, ESRF, France



I9-11 Max Lab, Sweden



I22, Diamond,UK

Australian Synchrotron

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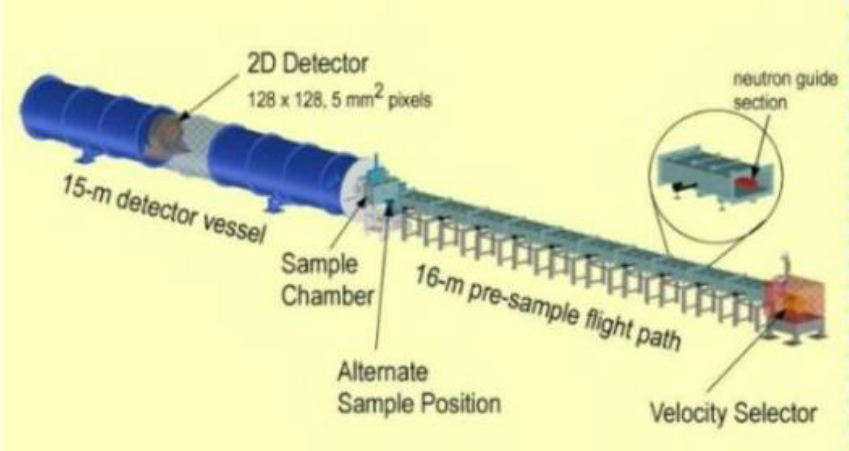
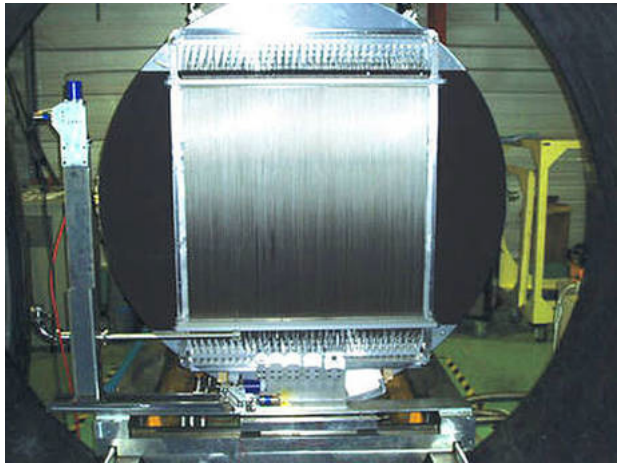
# Neutron Instruments

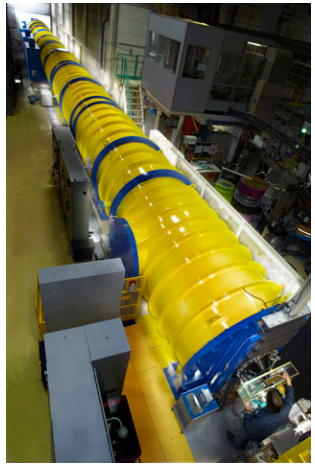



NG7, NCNR, USA


Bragg Institute, Australia

SANS2D –ISIS, UK





D22 and D11, ILL, Grenoble, France

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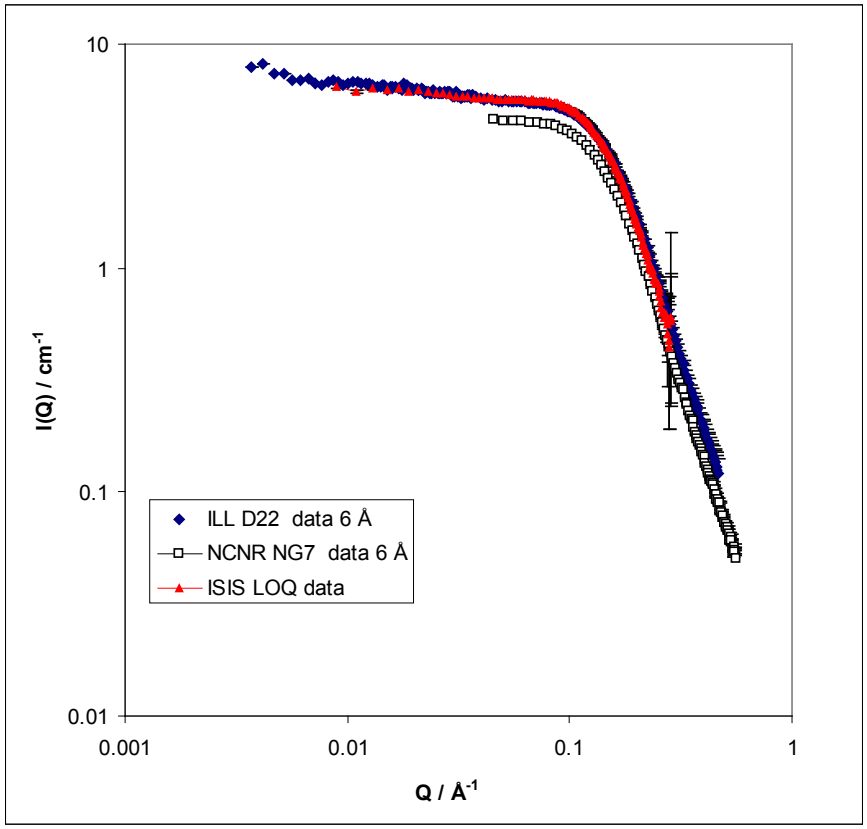
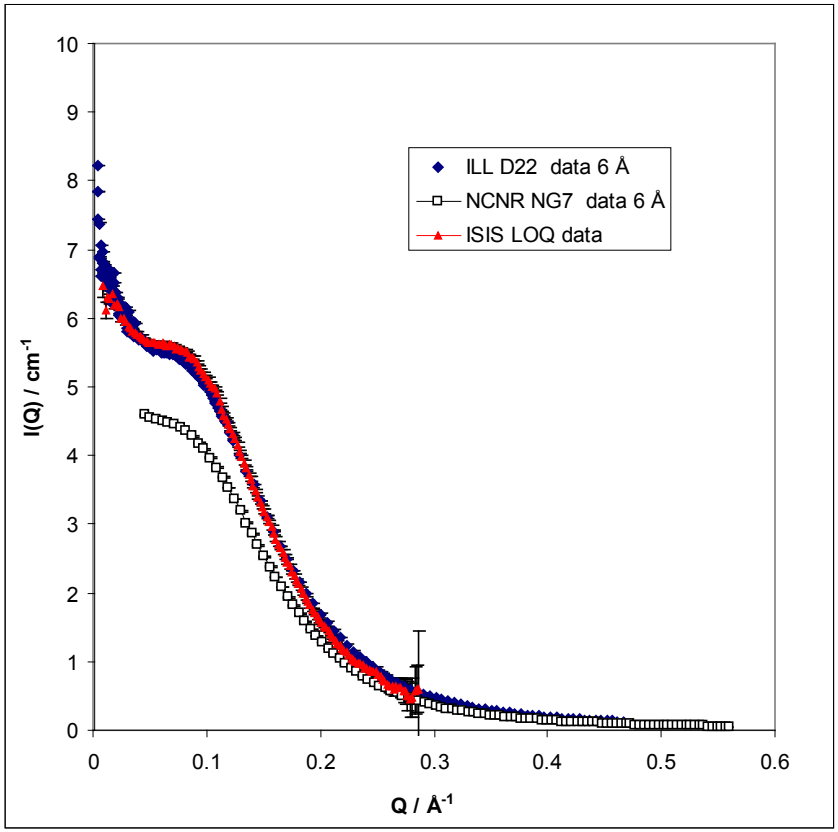
# Different Samples – Different Comparisons




C4 – Glassy Carbon

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# Contribution from Scott Barton

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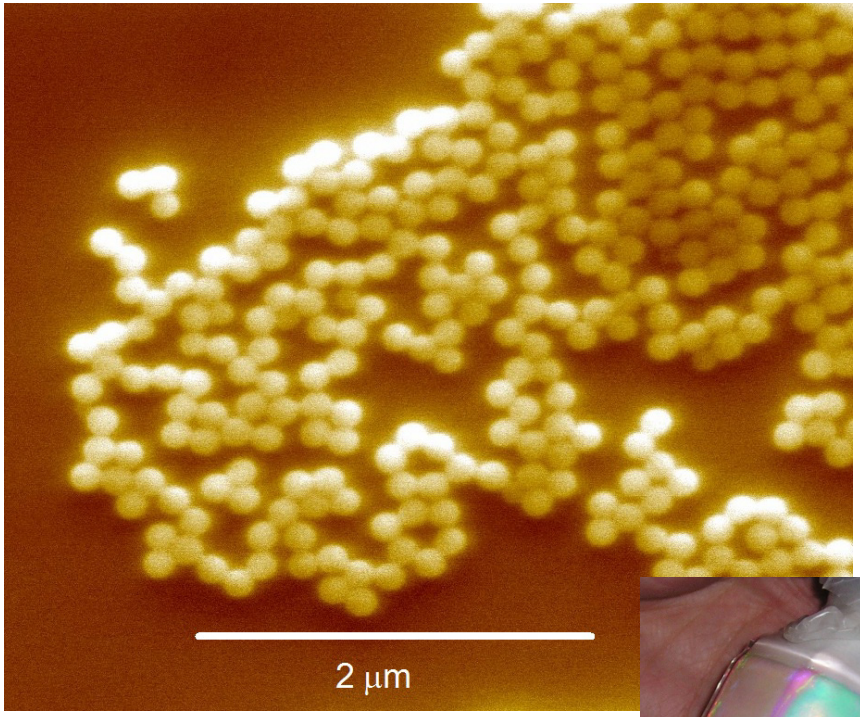
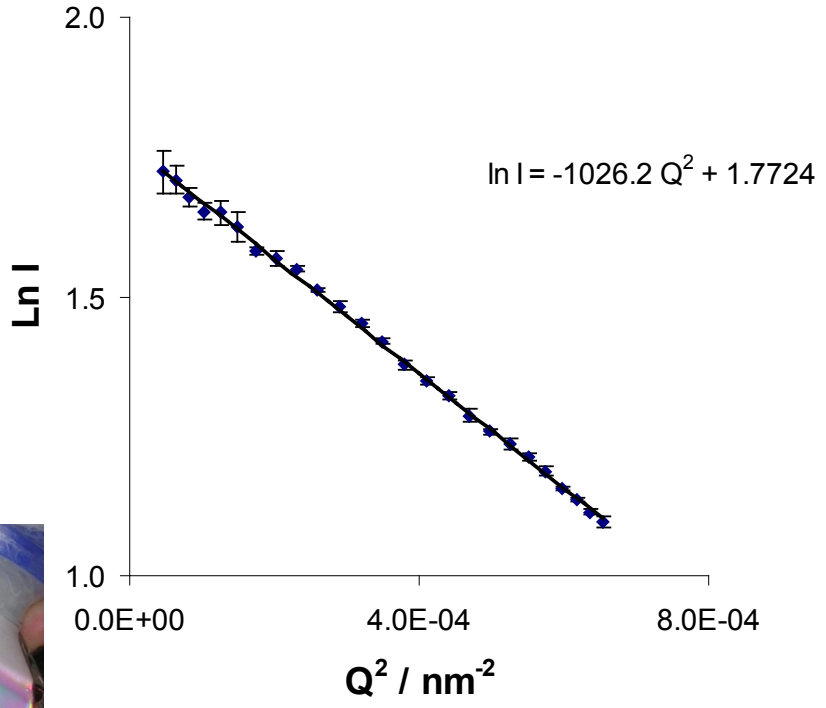
# Round Robin Sample




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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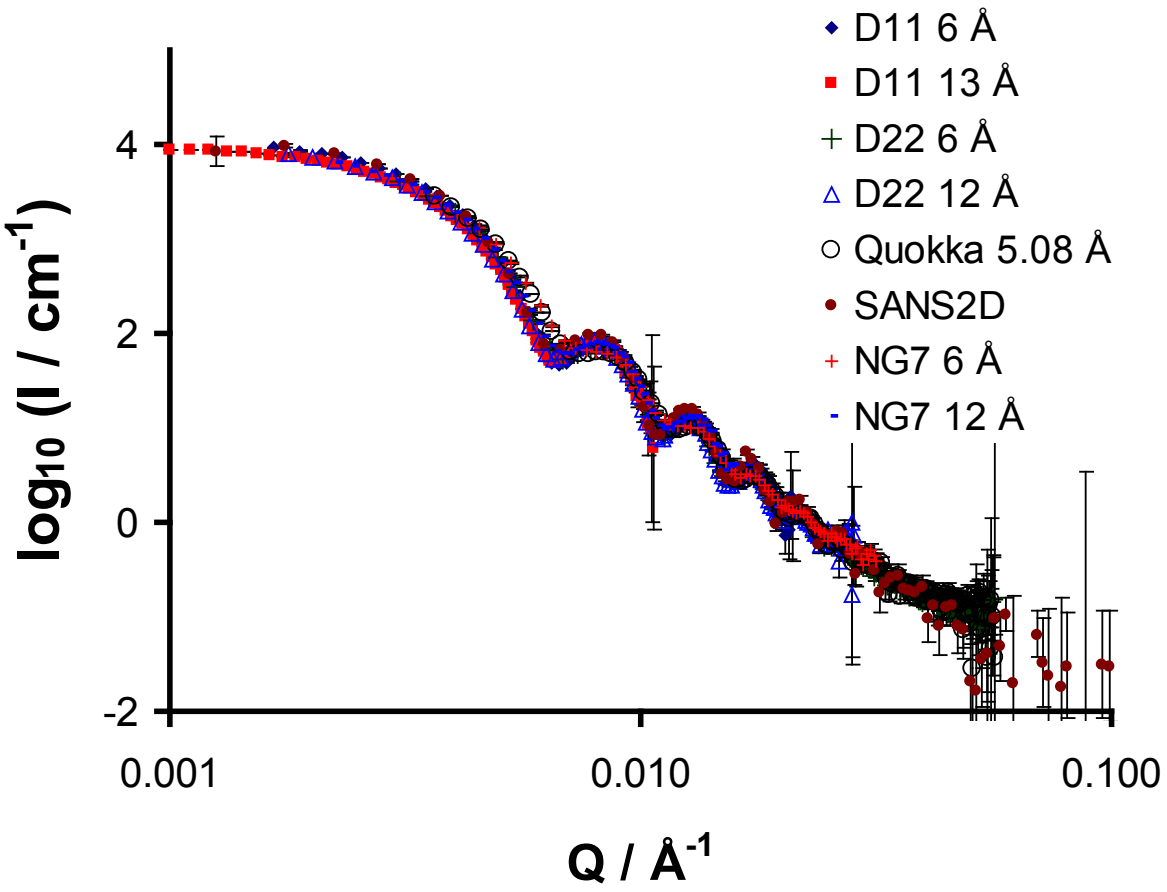
# 0.43% Latex in $D_2O$

1 mM NaCl



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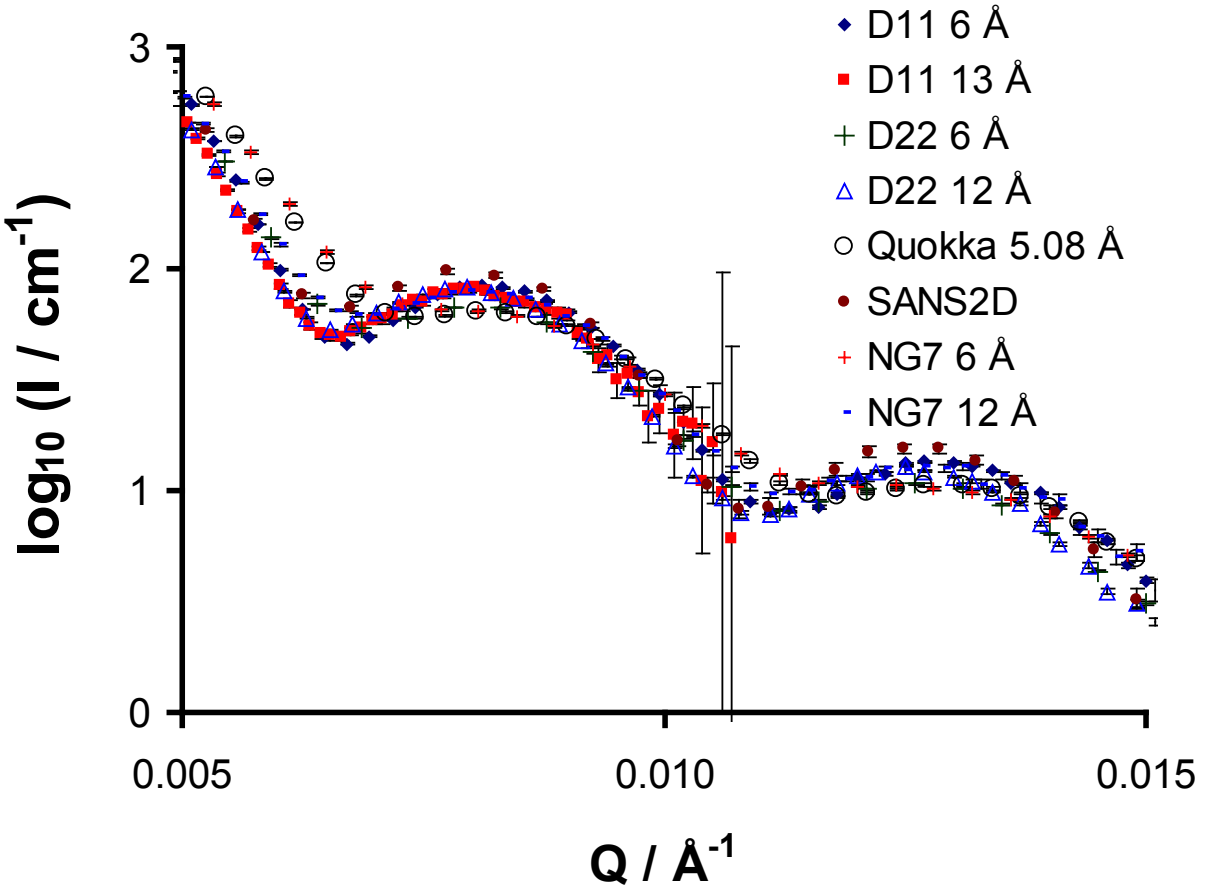
# Differences – Measured data

0.43% Latex in $D_2O$ 1mM NaCl




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

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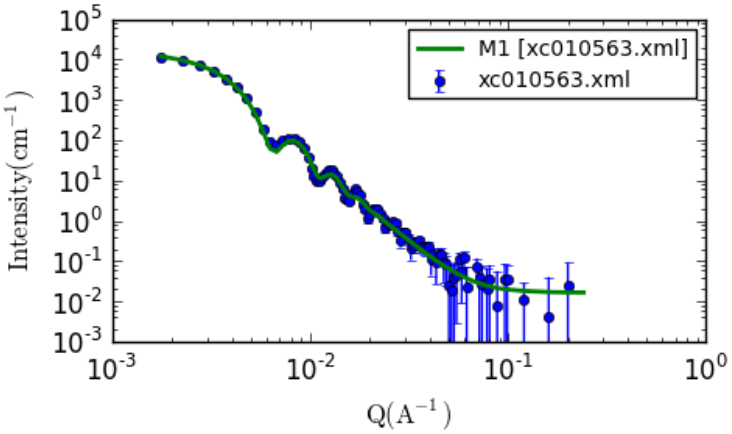
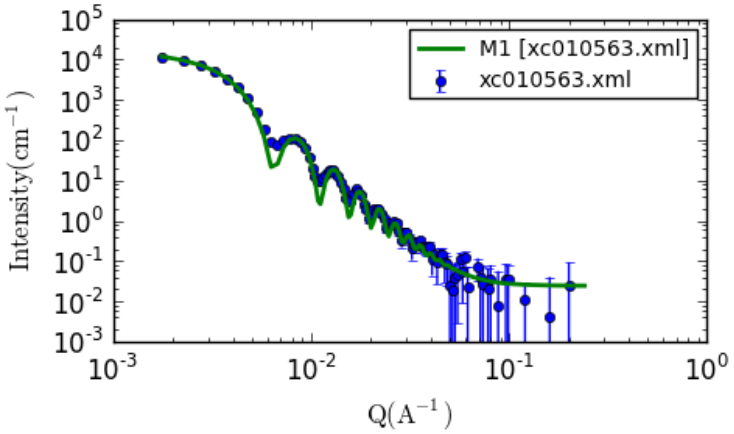
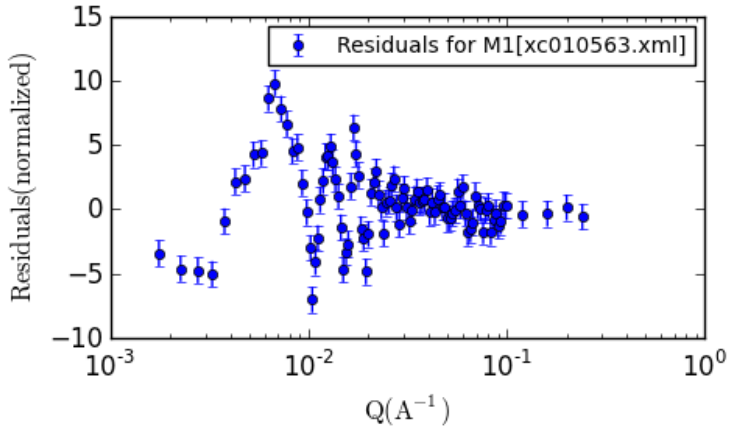
# Simple Fits – SasView Spheres

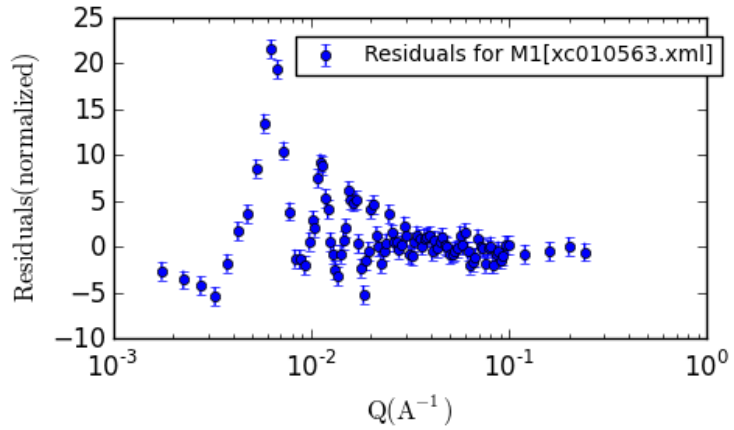



Polydispersity: 8%

3%




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

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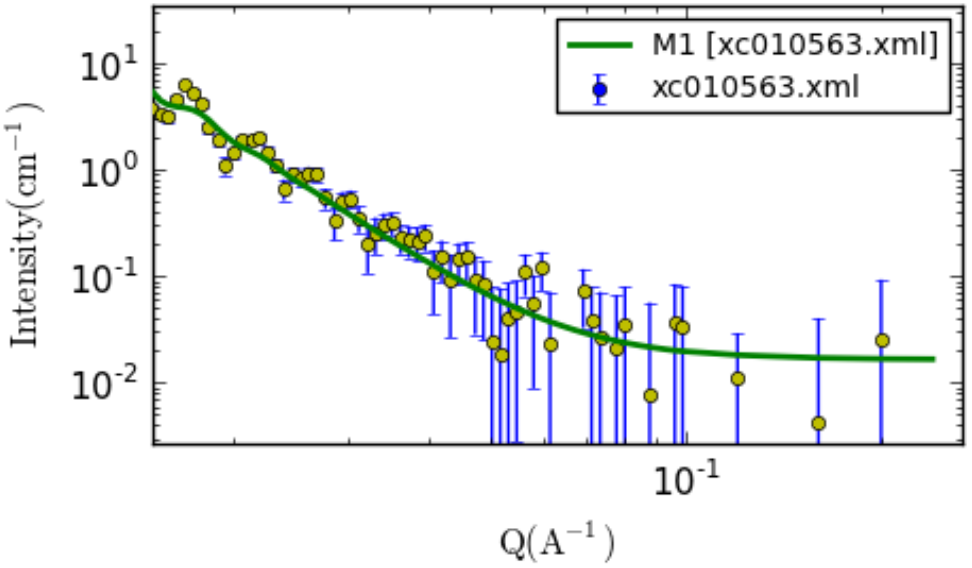
# Which fit is better?

- Better (when choosing from 2) but neither is best!
- 8% polydispersity has smaller $\chi^2$ 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

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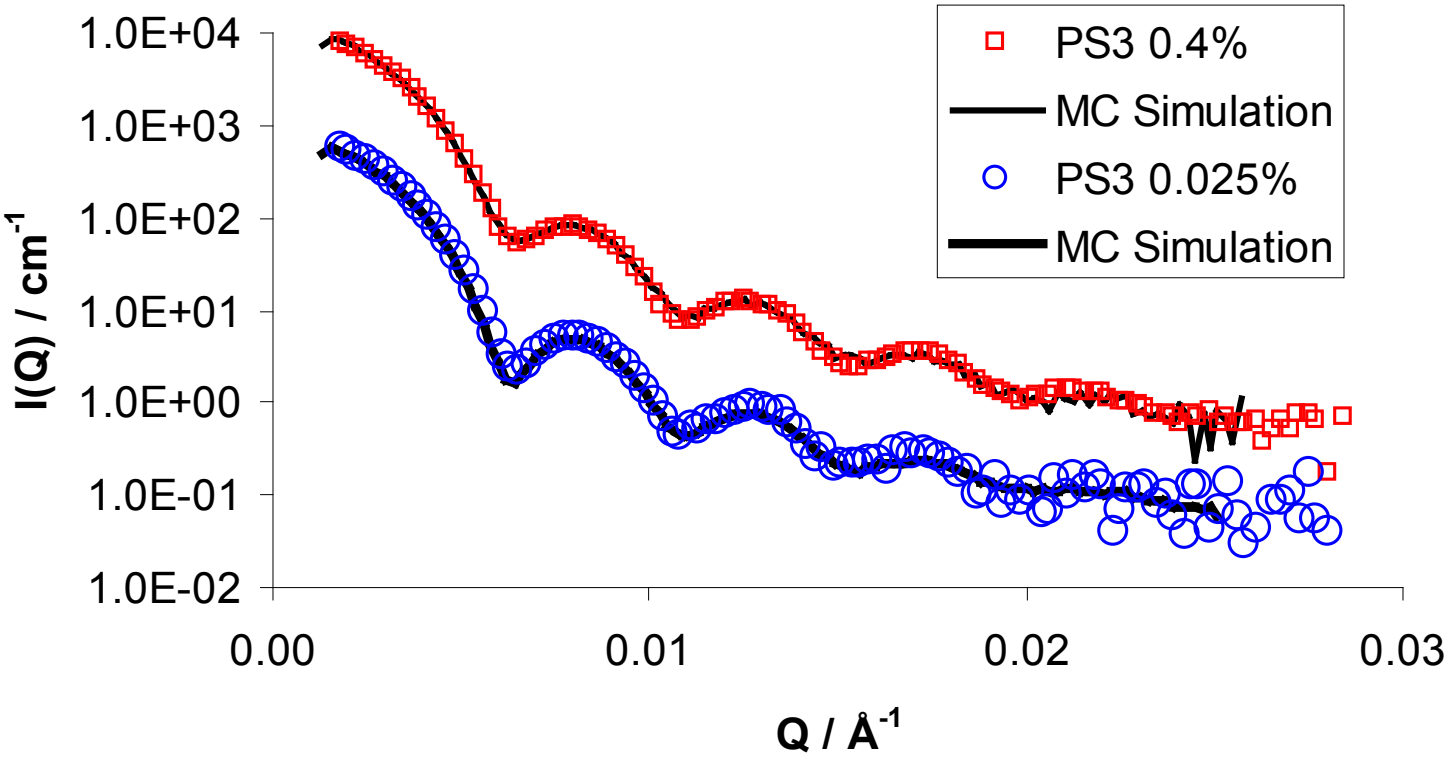
# Monte Carlo Simulation




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

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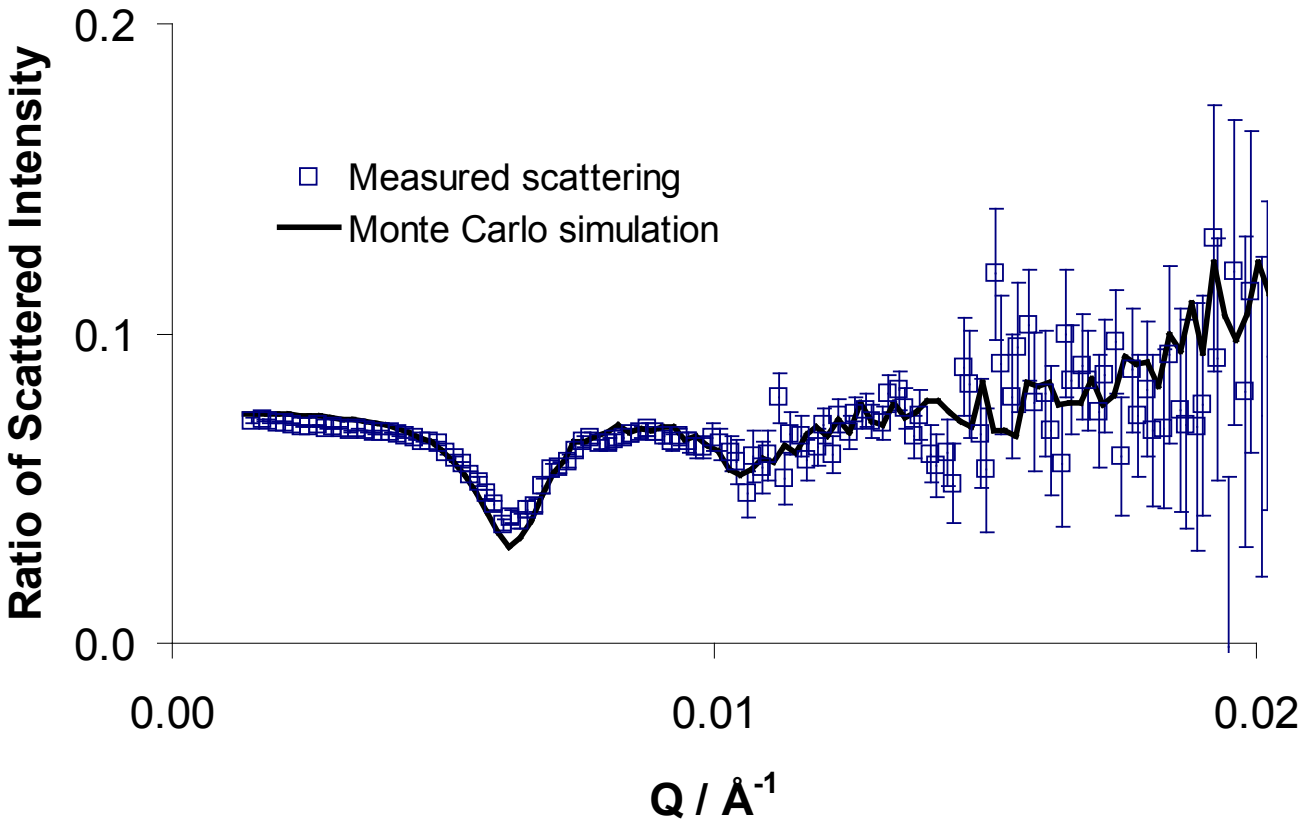
# Compare Ratio - Data & MC




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.
- Modeling 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.

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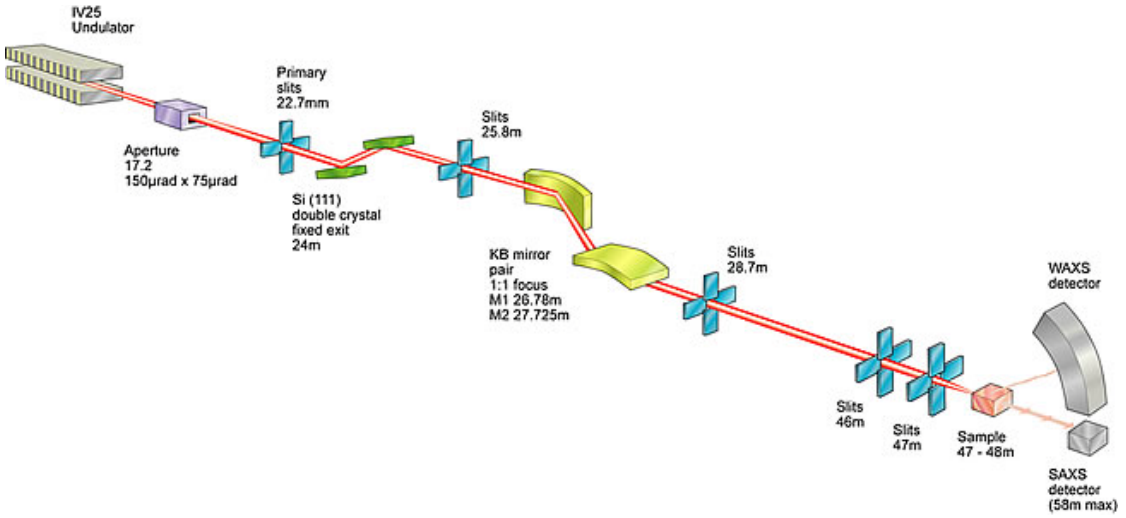
# SAXS – Is it different?




## Diamond Light Source

- I22 12.4 keV Pilatus 2M detector
- Sample detector distance 9.2 m

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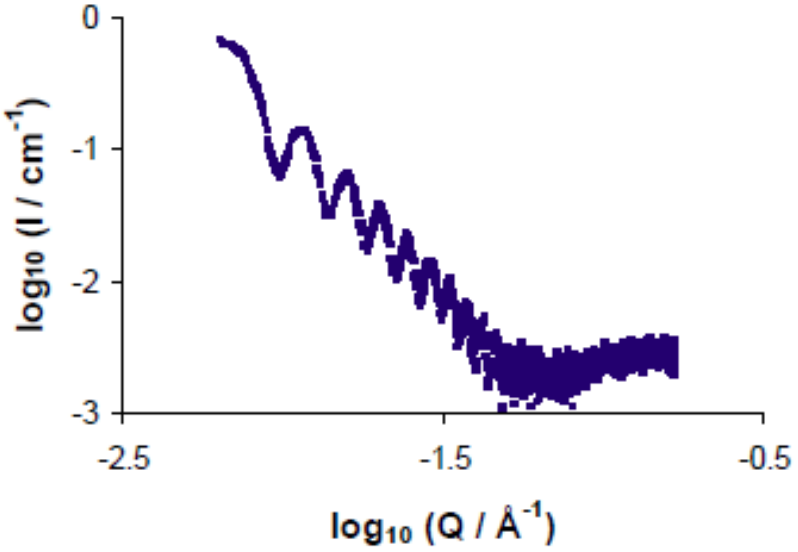
# SAXS – I22 Diamond

- PS3 Latex in $D_2O$
- 0.5 wt%

- Contrast between polystyrene and water is small ~ 0.2 x $10^{-6}$ $Å^{-2}$



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# SAXS

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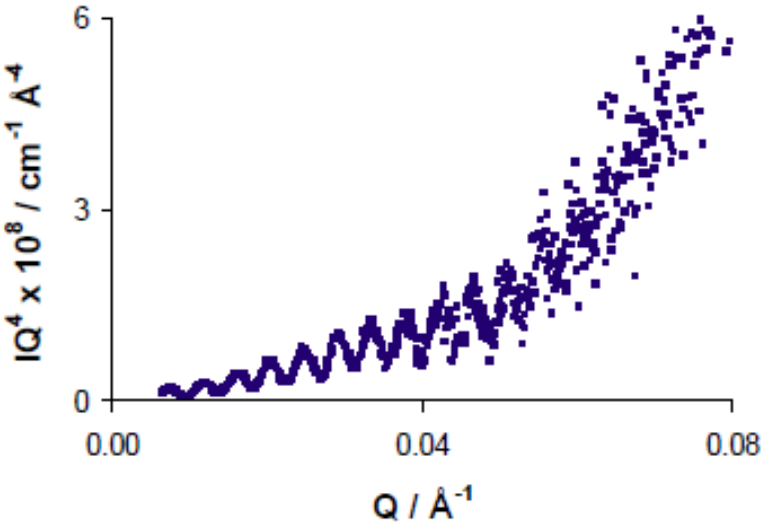


$$I \sim Q^{-3}$$

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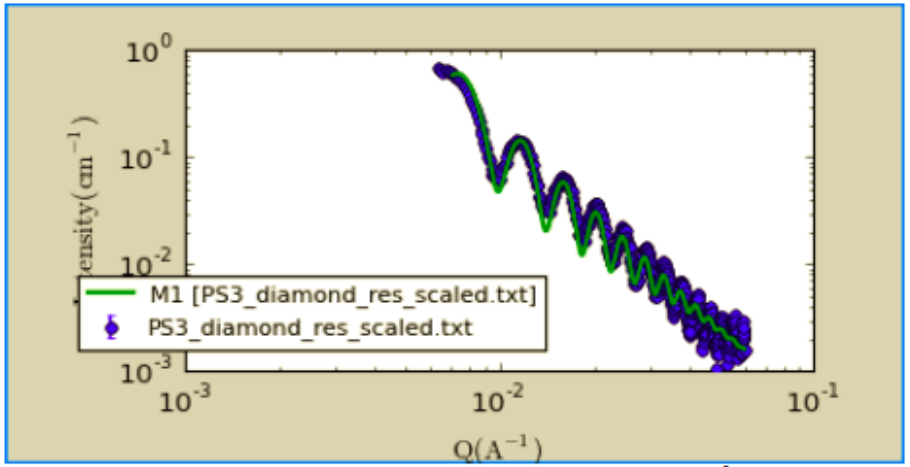
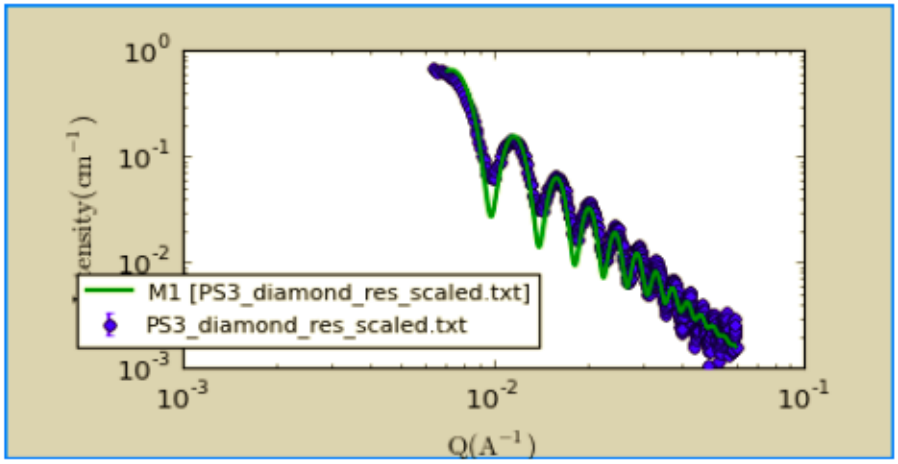
# Fitting SAXS Data

- 20 Å shell with 13% higher electron density
- Ionisable groups from synthesis



Fit with constant dQ resolution of 0.00042 Å-1



Calculated model with same parameters and no resolution smearing

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# SAS Lessons & Conclusions

- Resolution
  - Often poorly measured & documented
  - Become important as people use monodisperse samples
- Multiple Scattering
  - Can be confused with resolution & polydispersity
  - Simple Double Scattering calculations useful

# Conclusions – What have we learnt?

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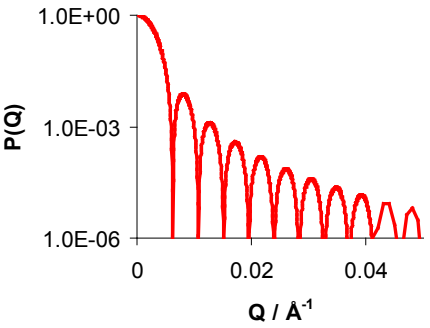
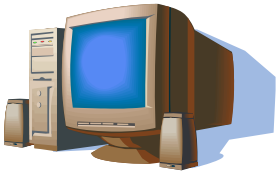
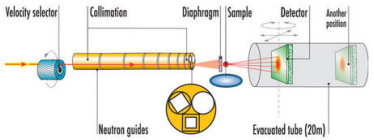
Data that can be modelled reliably helps comparisons

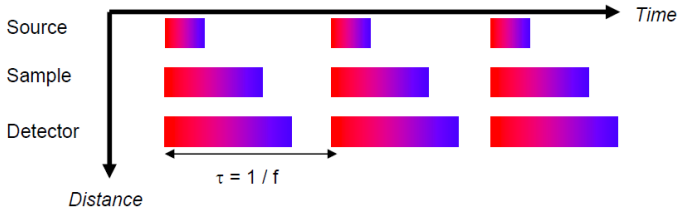





Compare instruments and software

Systematic deviations are often the largest source of uncertainty in interpretation




- ToF and const λ measurements provide beneficial comparisons
- SAXS & SANS comparison desirable

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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, $\sigma$, FWHM etc.?

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# People who do the work:

Maja S. Hellsing, Adrian Hawley, Peter Boesecke, Tomas Plivelic, Anna Labrador, Katy Wood,

Elliot P. Gilbert , Lionel Porcar,

Ralf Schweins, Charles D. Dewhurst,

Peter Lindner, Richard K. Heenan,

Sarah Rogers, Paul D. Butler, Jeff Krzywon, Ron E. Ghosh, Andrew J. Jackson,

Marc Malfois

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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