Australian Government

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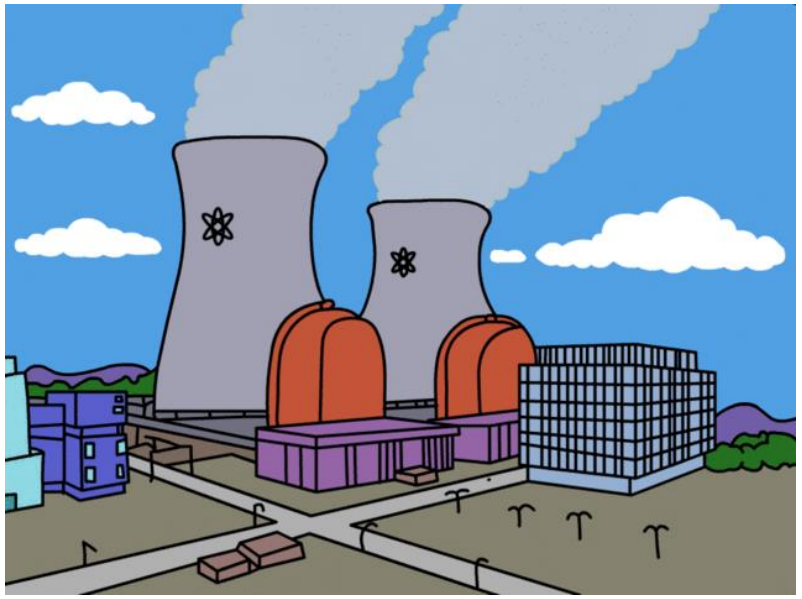
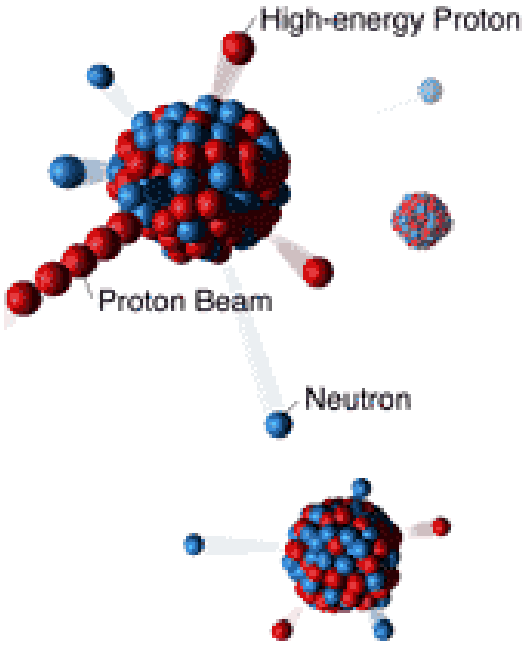
Neutron (X-ray) Reflectometry Round Robin

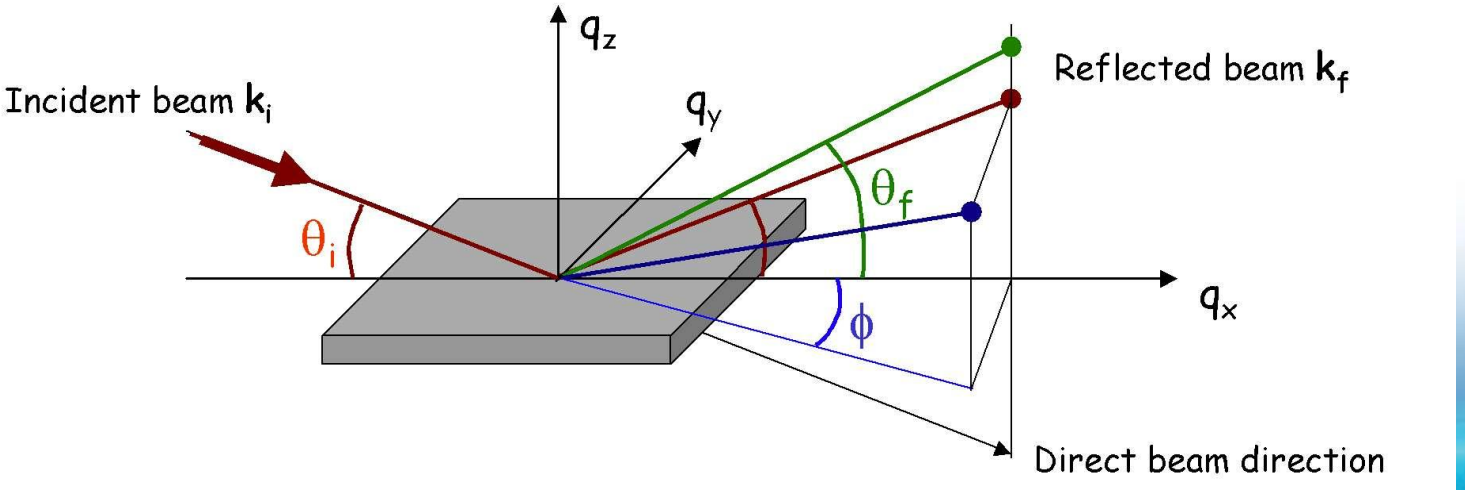
Andrew Nelson

Joseph Dura, Robert Newby, Jessie Zhange, Charles Majkrzak (NIST – NCNR and CNST)

Aim – Reproducible science







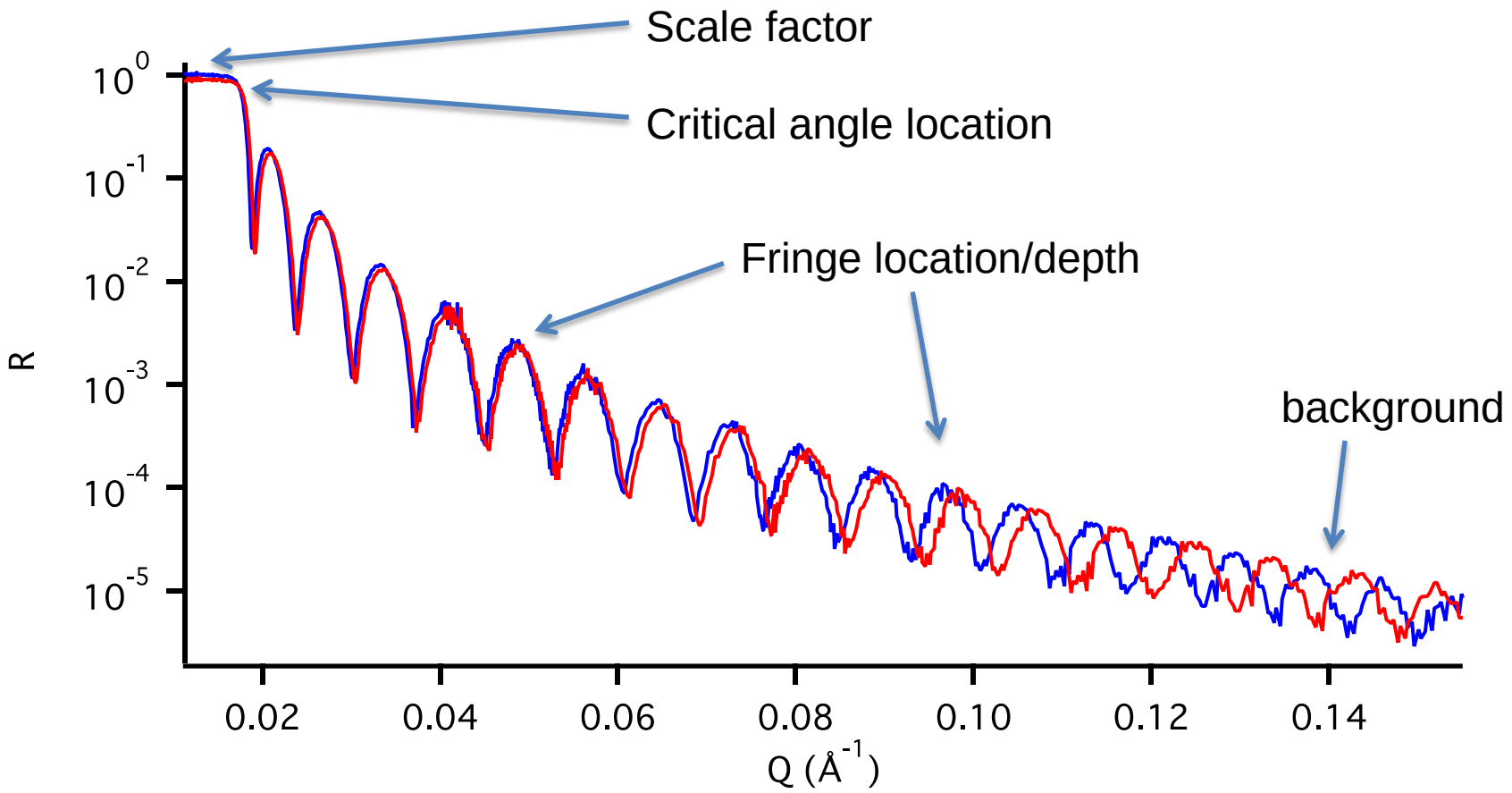
Different type of reflectometers

Platypus - ANSTO

MAGIK - NIST

SNS liquids reflectometer

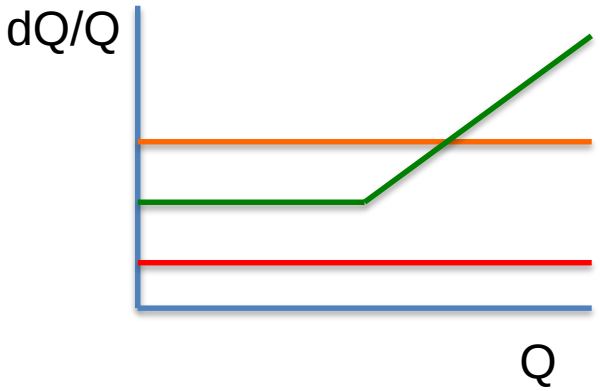
Aim – Reproducible Science



- Reproducible data
- Reproducible analysis

Reproducibility - we're all different

- Energy / Angle dispersive
- Different resolution functions
- Vertical / horizontal geometry
- Illuminated area
- Reduction algorithms
  - Background subtraction
  - Footprint correction
  - Rebinning
  - Scaling
  - Different configurations
  - TOF corrections
  - Resolution function calculation
  - Constant Q integration



Error propagation

SANS round robin

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Learning about SANS instruments and data reduction from round robin measurements on samples of polystyrene latex[1]

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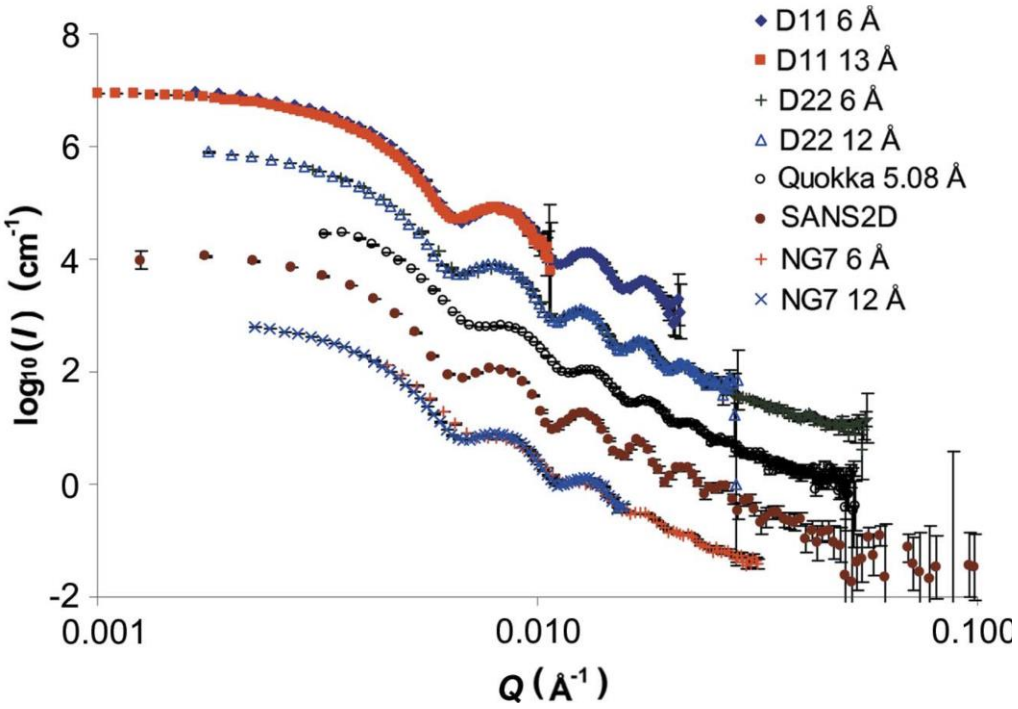
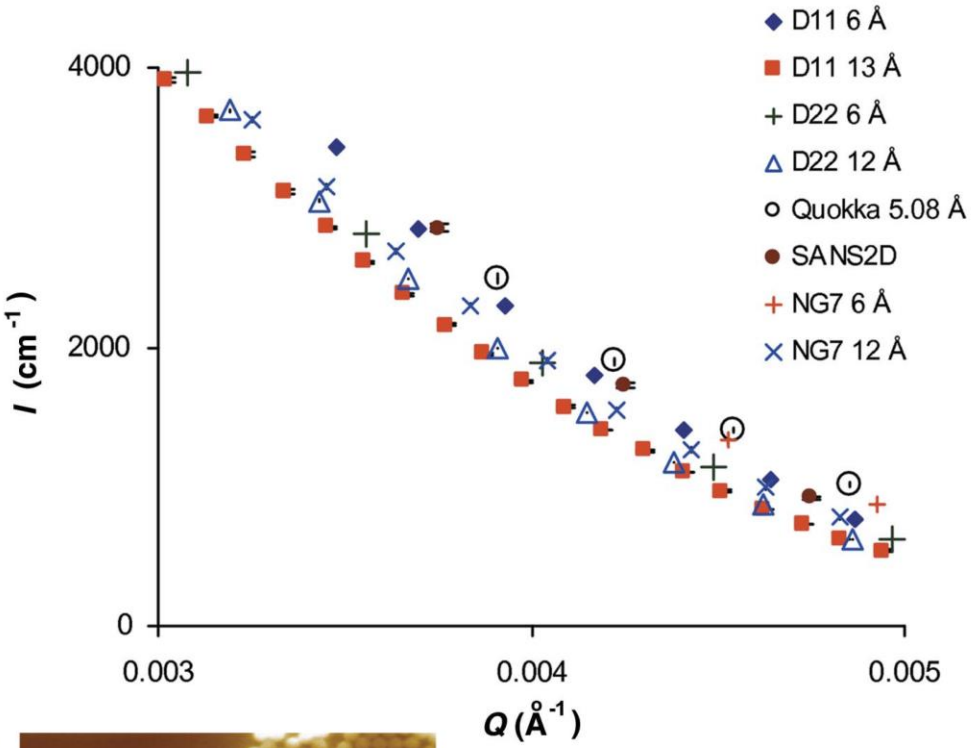
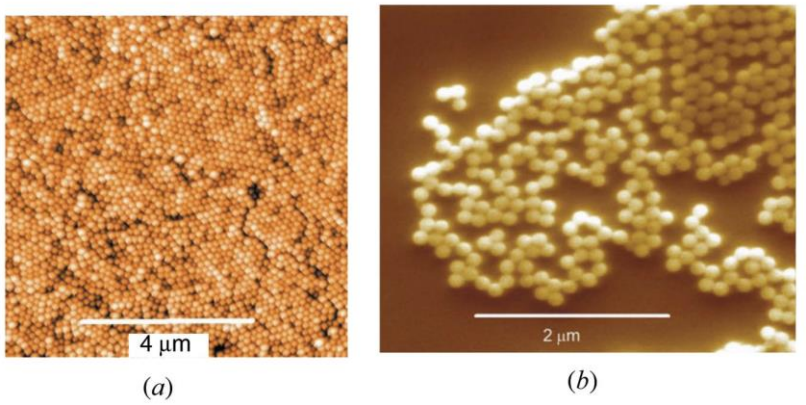
What was measured?







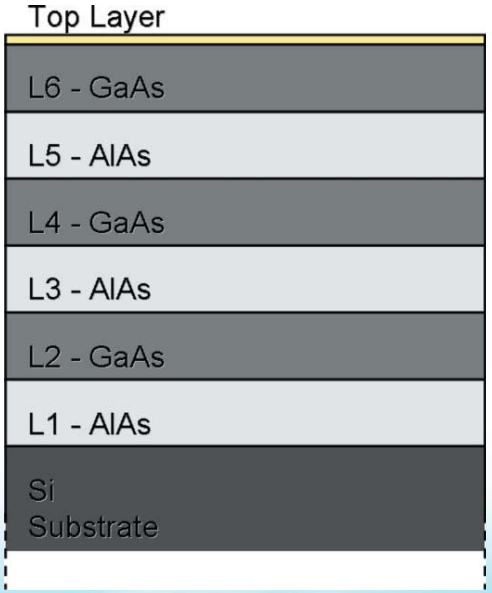
Rennie et al,. Learning about SANS instruments and data reduction, J. Appl. Cryst., **46** (2013)

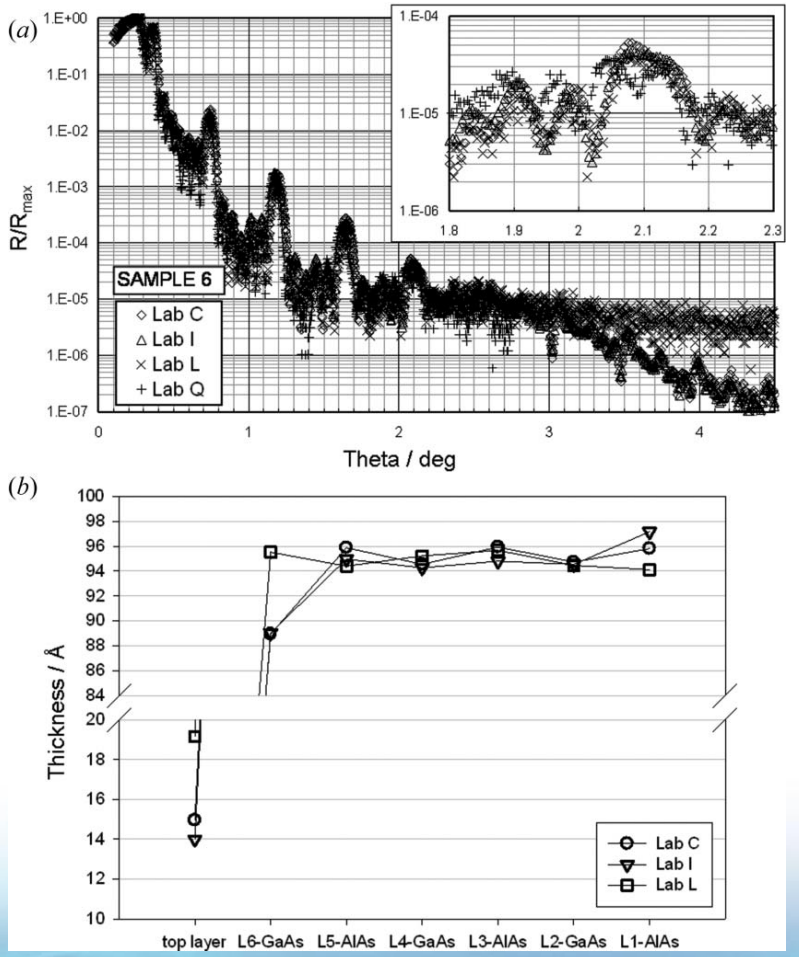
Lessons learnt

- Majority of I(Q) difference is due to resolution effects
  - Resist temptation to arbitrarily scale data at different instrument configurations.
- Deficiencies in metadata
  - Sometimes resolution smearing was underestimated
  - Sometimes a lack of resolution function information
  - Identified areas were improvements could be made (e.g. collimation and wavelength resolution are linked)
- Highlighted need for multiple scattering corrections
- Uncertainty in fitted parameters limited by systematic errors in calibration and modelling, not counting statistics.
- Standardised datasets can be used to document different sources of error.

Background - reflectometry

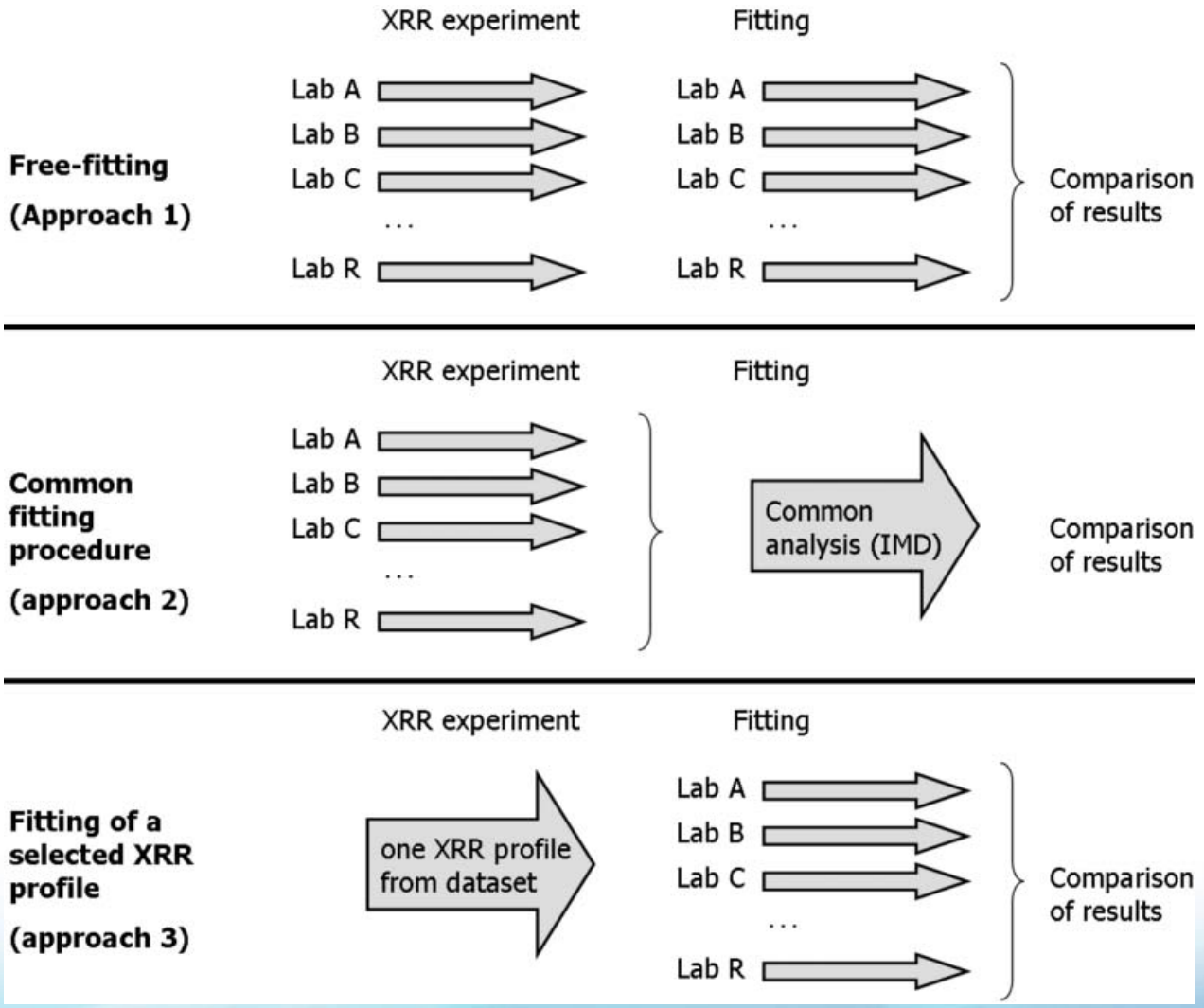
- X-ray reflectometry round robin, Colombi, 2007
  - GaAs / AlAs multilayer
  - 20 laboratories
  - Differences in analysis approach are non-negligible
  - Dynamic range important





Frederic Ott, 2005, http://neutronreflectivity.neutron-eu.net/main/RoundRobin

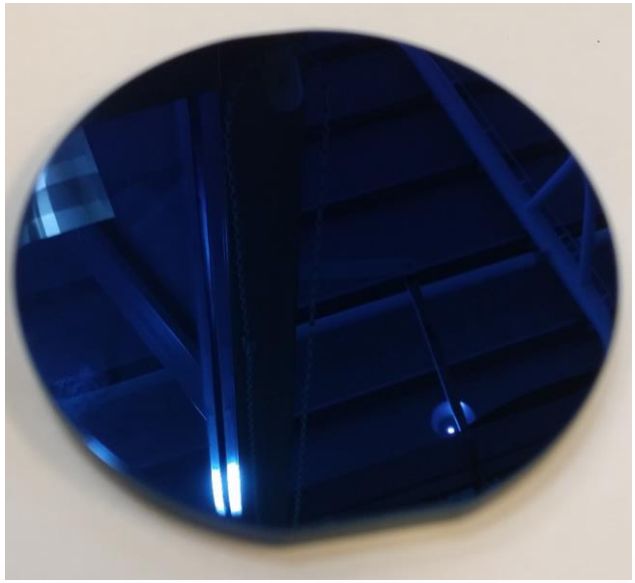
Comparative assessment



Colombi et al, *J. Appl.Cryst.* (2008), **41**, 143

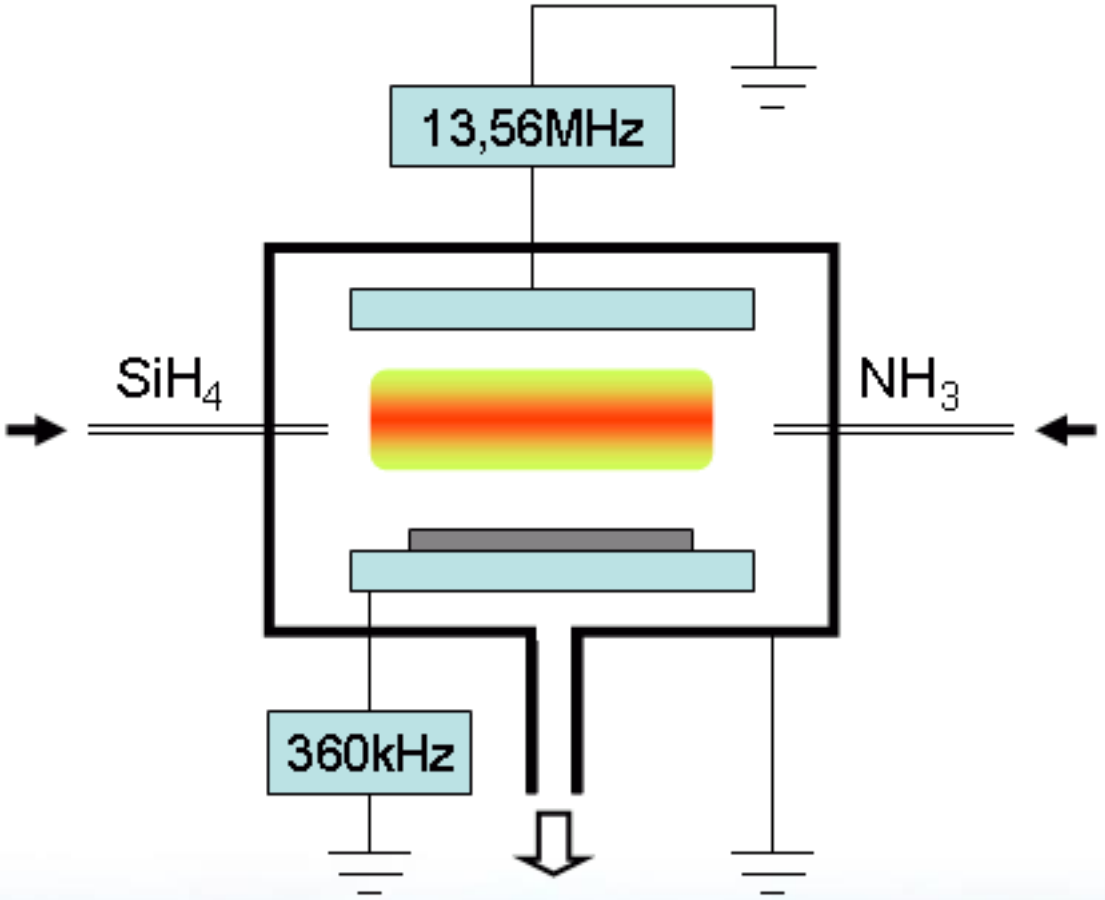
Sample choice

- Non magnetic
- Reflectivity insensitive to contaminants
- Robust
- Uniform film
- Low sample warp
- Test instrumental resolution
- Test scaling



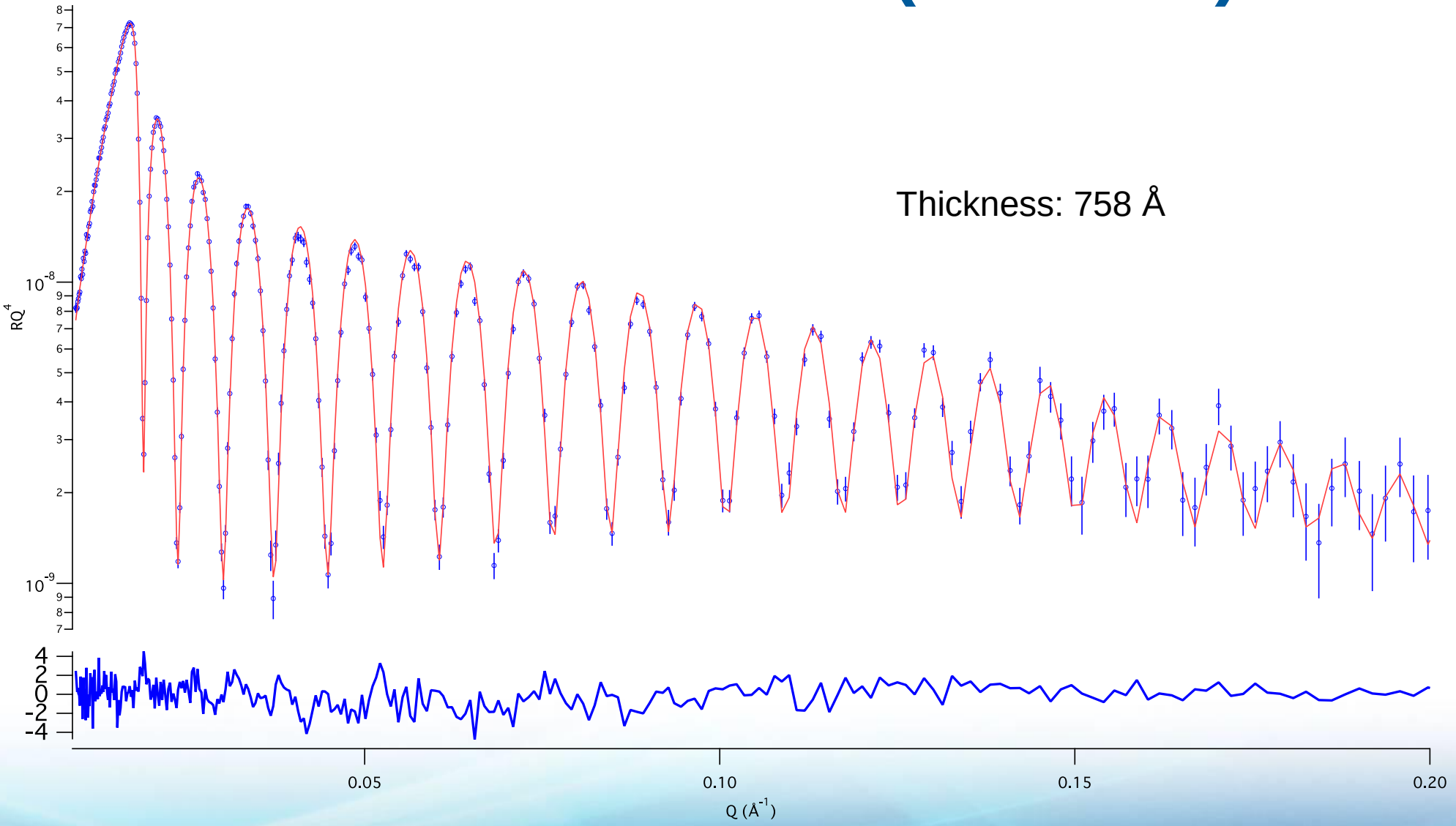
- **Si_3N_4**
- **600-700 Å**
- **SLD ~ 5.9×10^{-6} $Å^{-2}$**

Si3N4 deposition



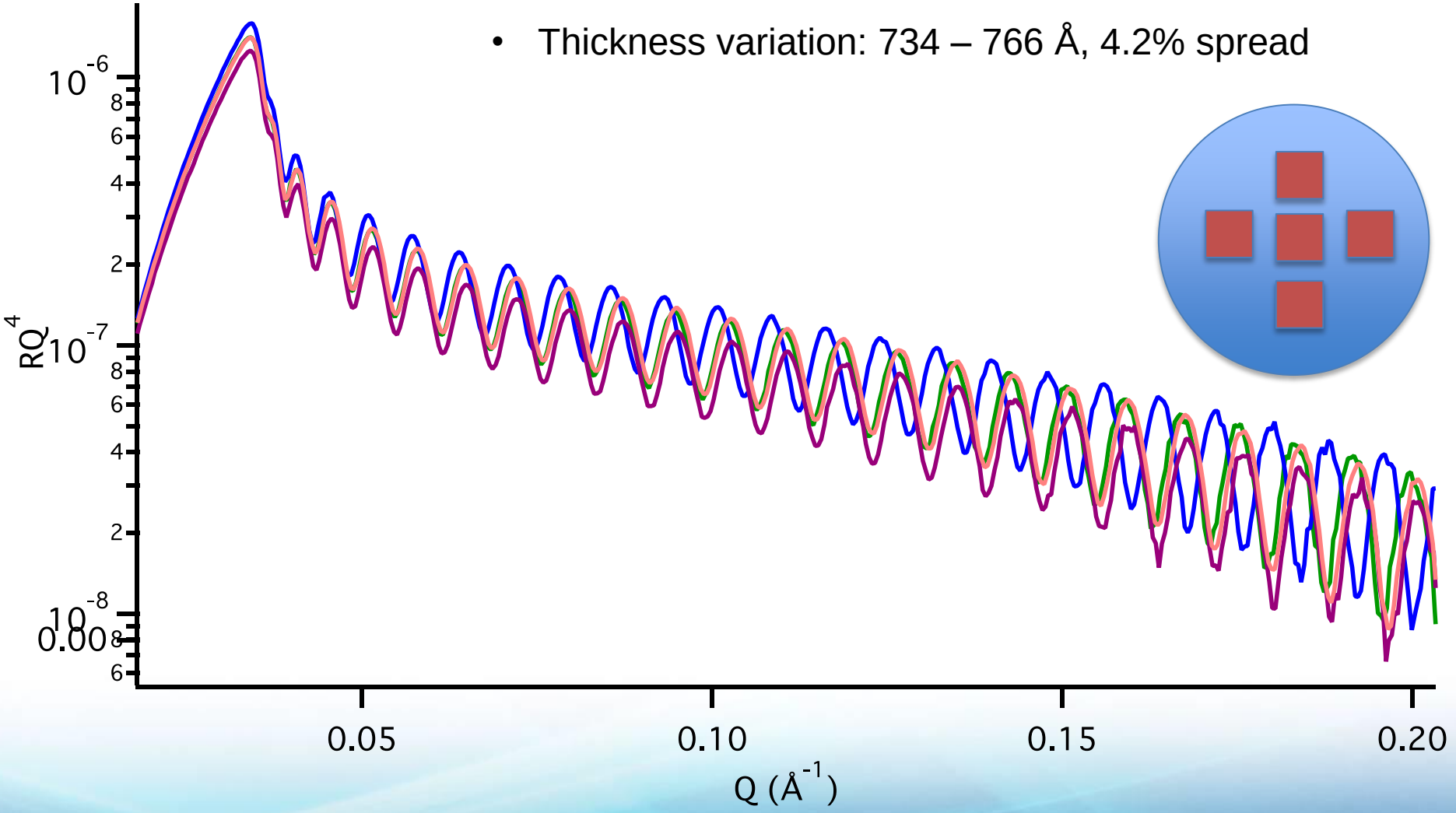
Schematic from www.crystec.com

Initial measurements (Neutrons)

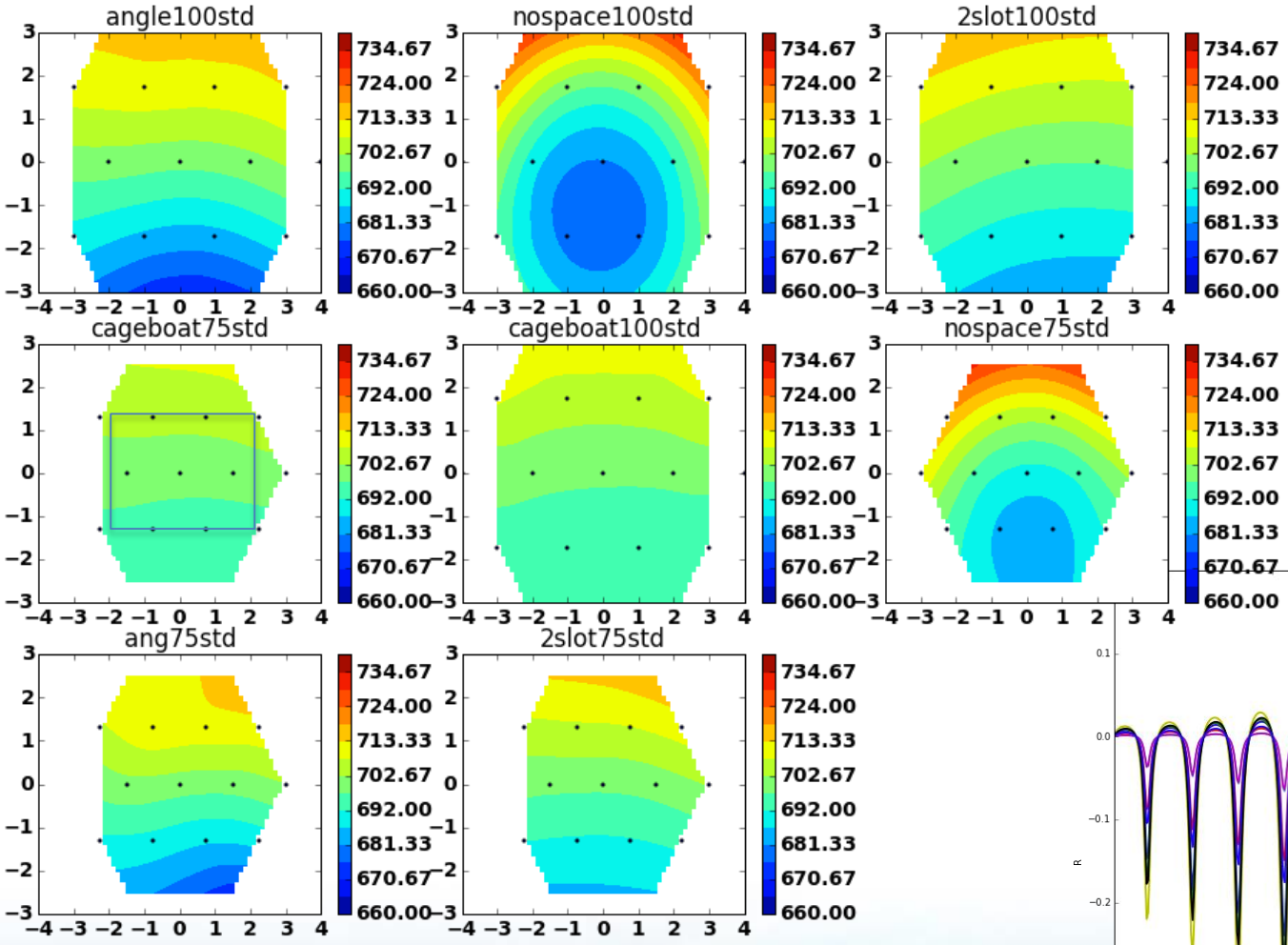


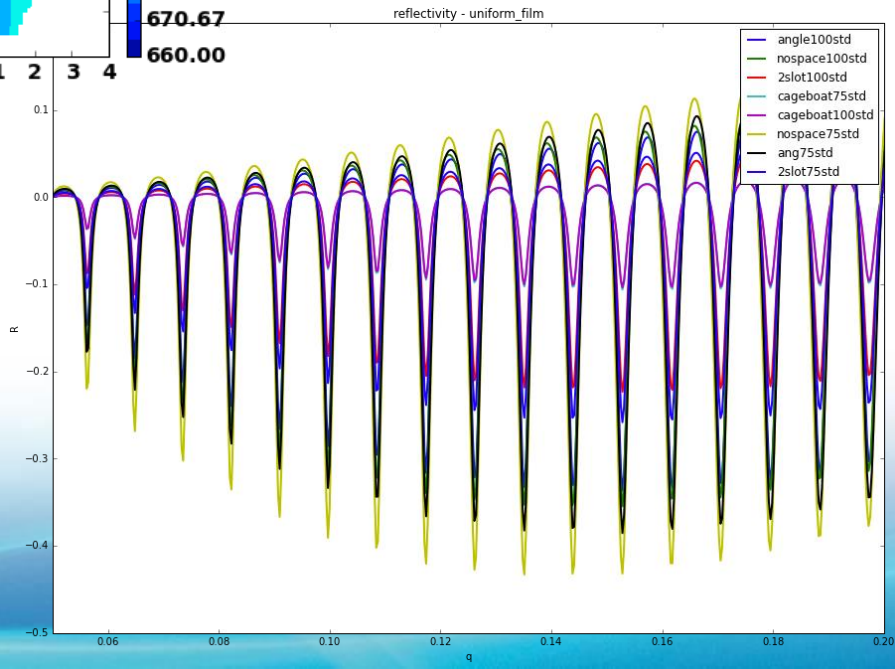
Initial measurements (X-rays)

- Thickness variation: 734 – 766 Å, 4.2% spread

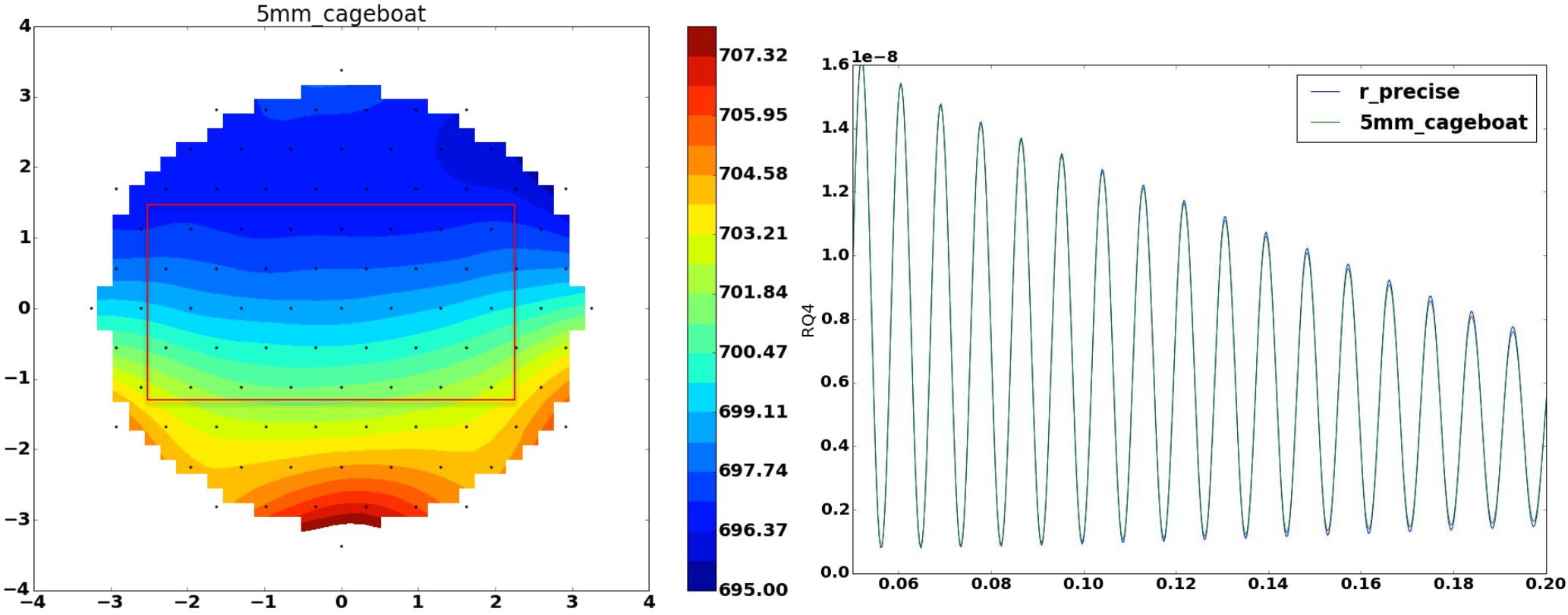


Deposition Characteristics



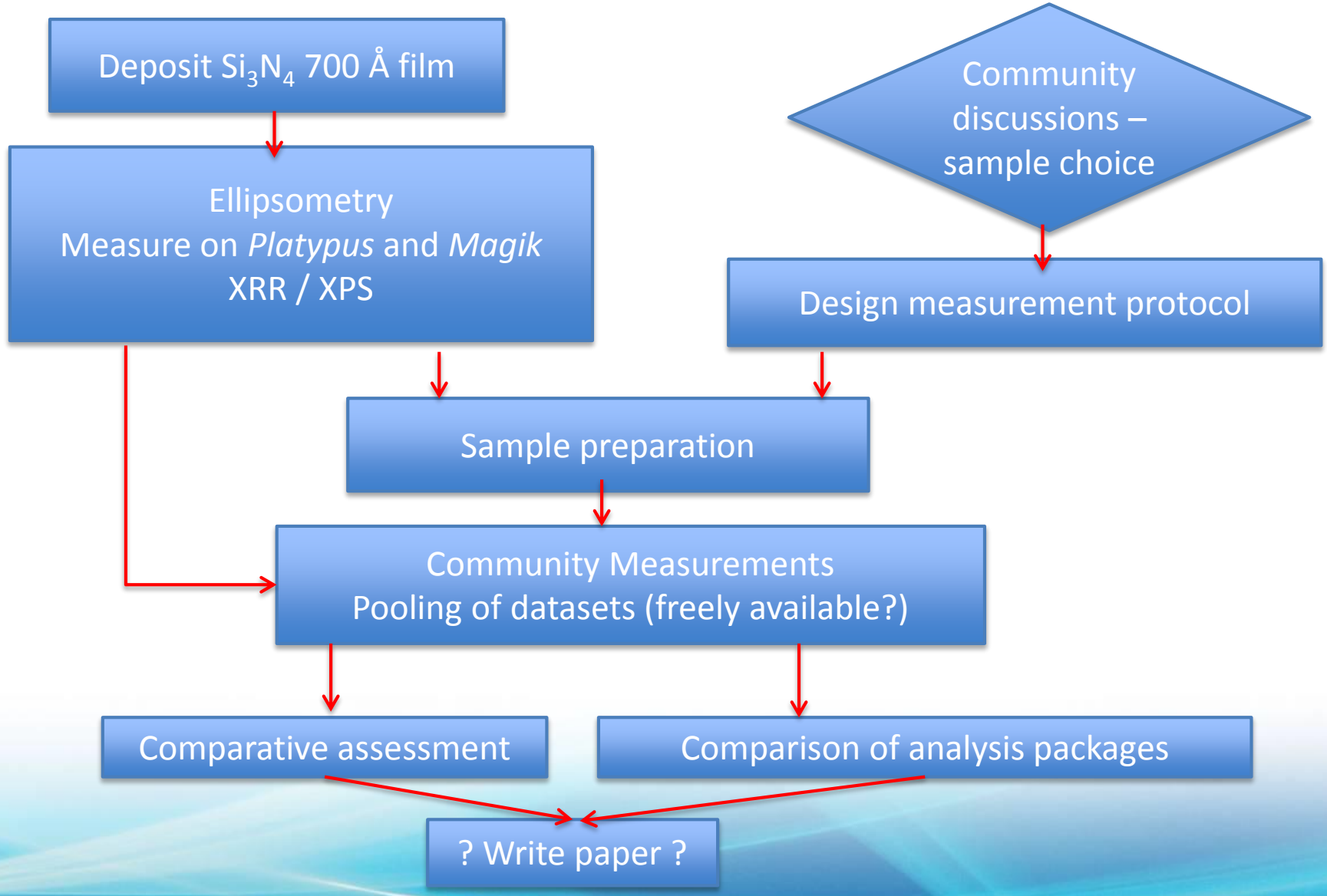


Best of the crop

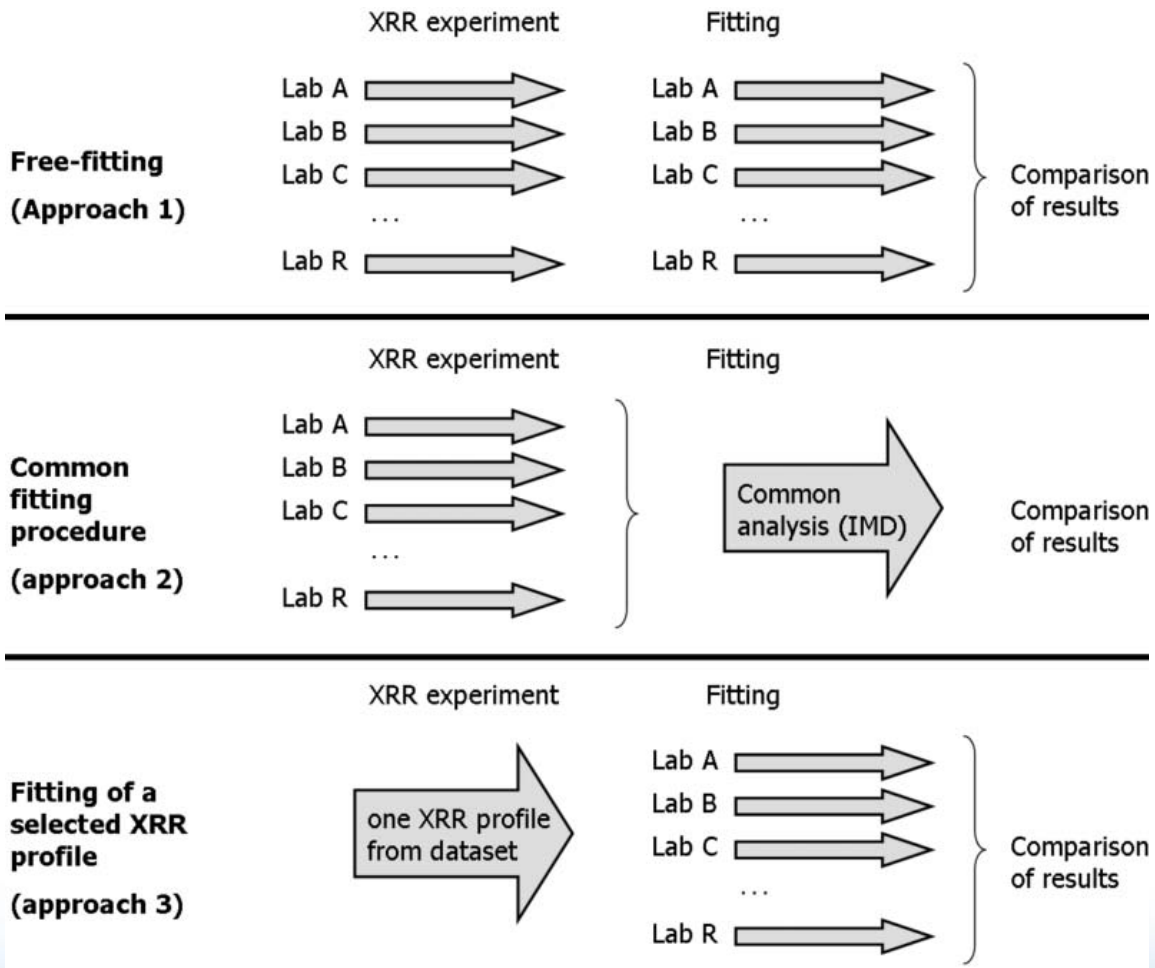


- 0.85% thickness variation over ‘sweet spot’

Roadmap


Deposit Si_3N_4 700 Å film
Ellipsometry
Measure on *Platypus* and *Magik*
XRR / XPS
Community discussions – sample choice
Design measurement protocol
Sample preparation
Community Measurements
Pooling of datasets (freely available?)
Comparative assessment
Comparison of analysis packages
? Write paper ?

Comparative assessment



Motofit

Program Y

Refl1D

Colombi et al, *J. Appl.Cryst.* (2008), **41**, 143

Advice please

- Community interactions
  - How does one maximise participation?
- What are the pitfalls (handling ‘skeletons in cupboard’)?
- At what stage should data be freely available?

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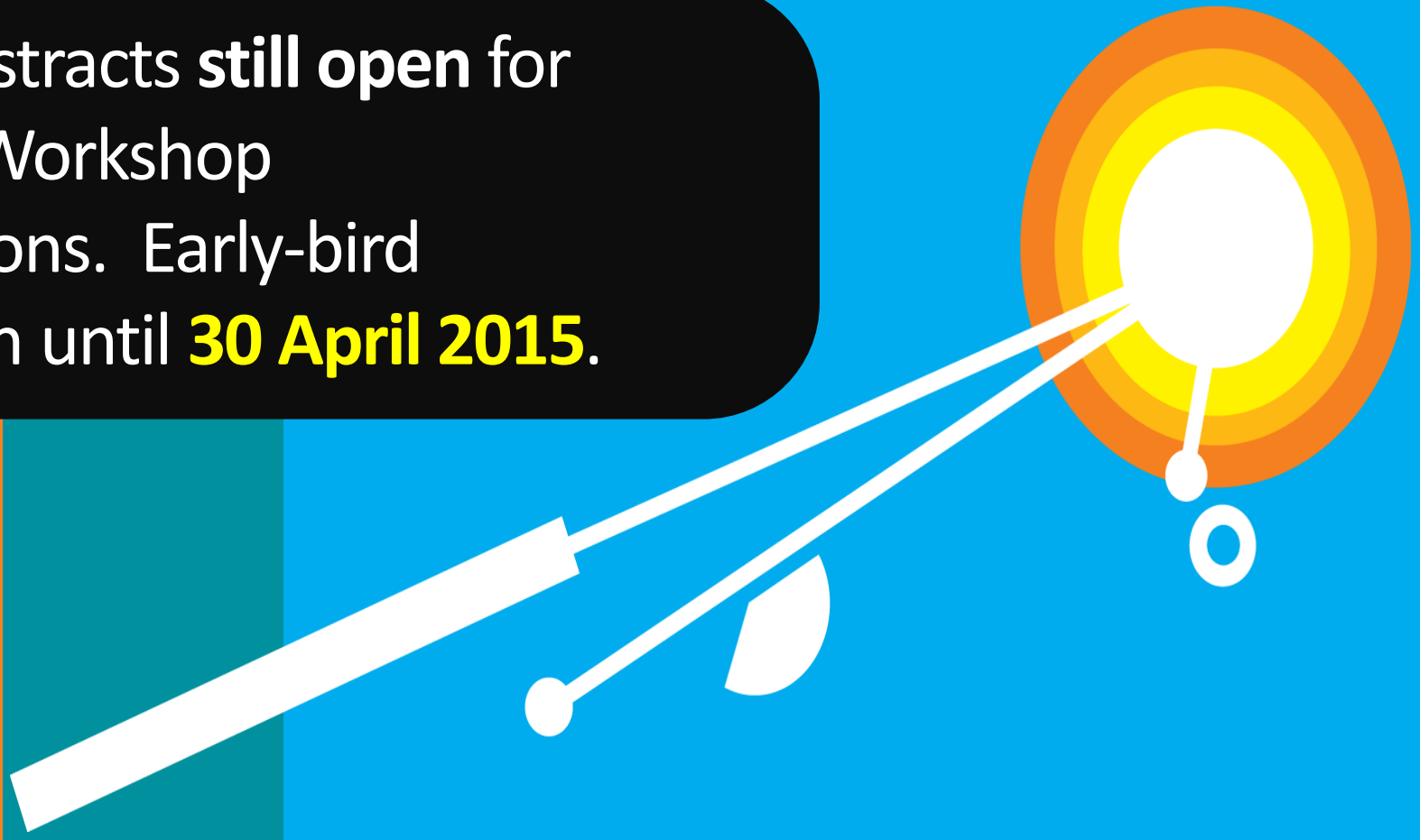

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