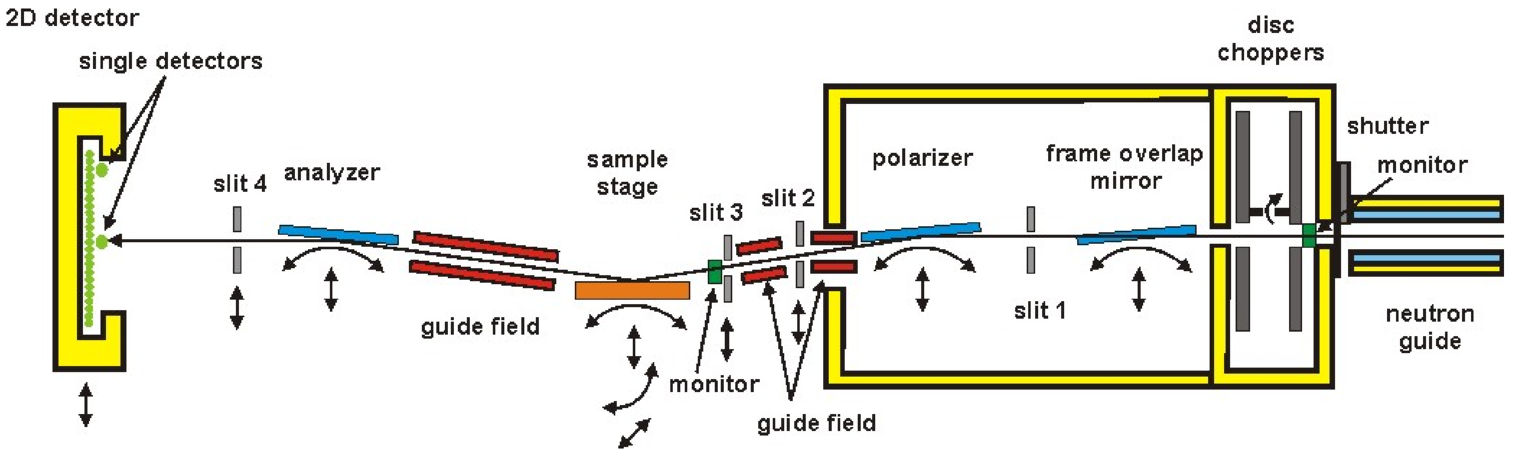
Reflectometry with X-rays and Neutrons

The Practical

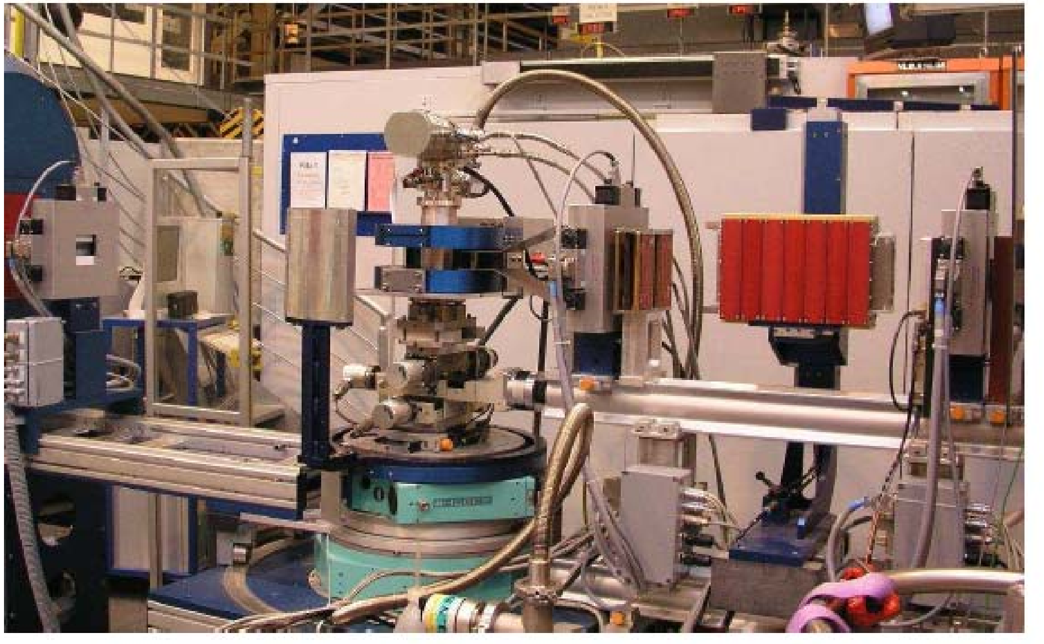


T. Gutberlet
LNS, PSI & ETHZ, Villigen

Outline

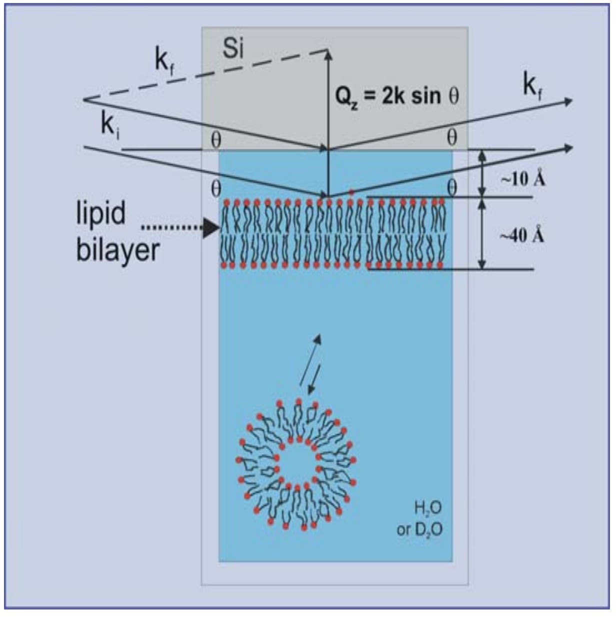
- Setting up a measurement
  - sample
  - alignment
  - measurement

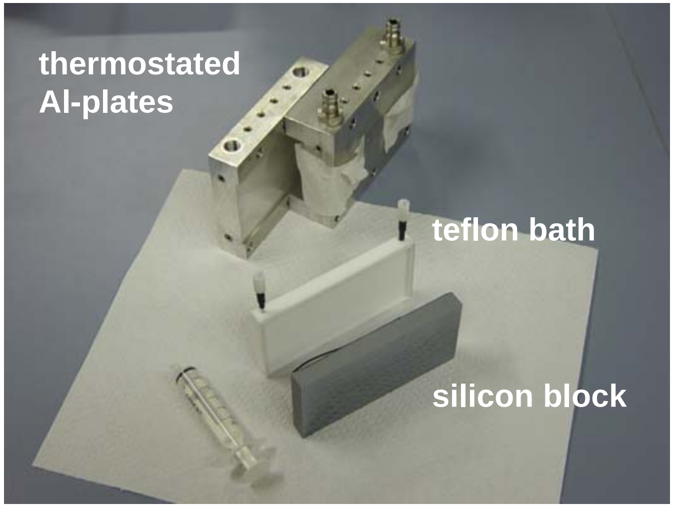
- Data refinement and analysis
  - data reduction
  - fitting
  - Parrat32

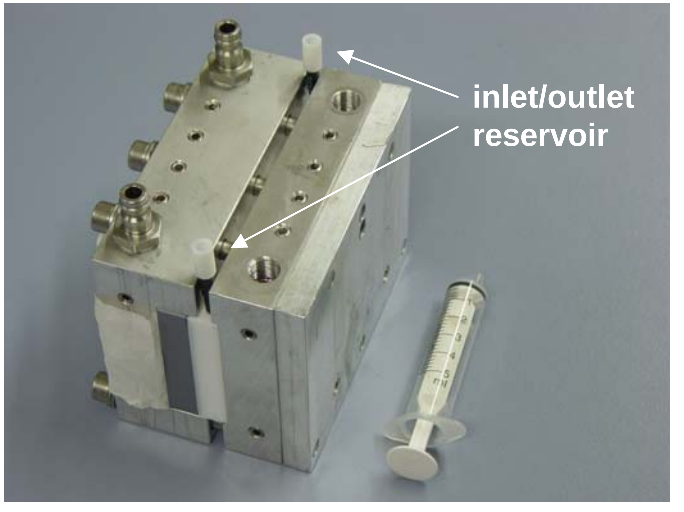


Sample

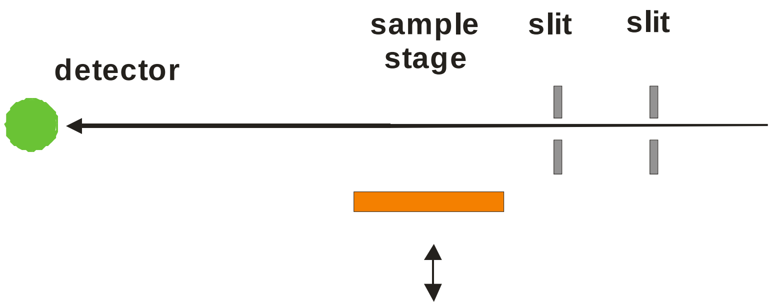
Example: preparation of solid/liquid sample cell

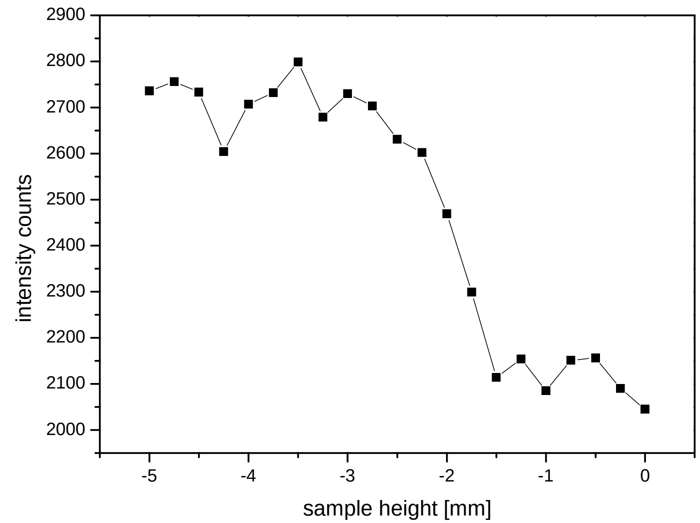


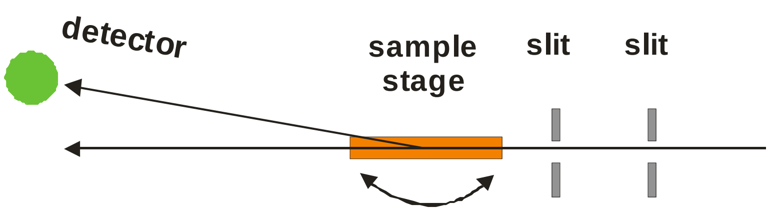


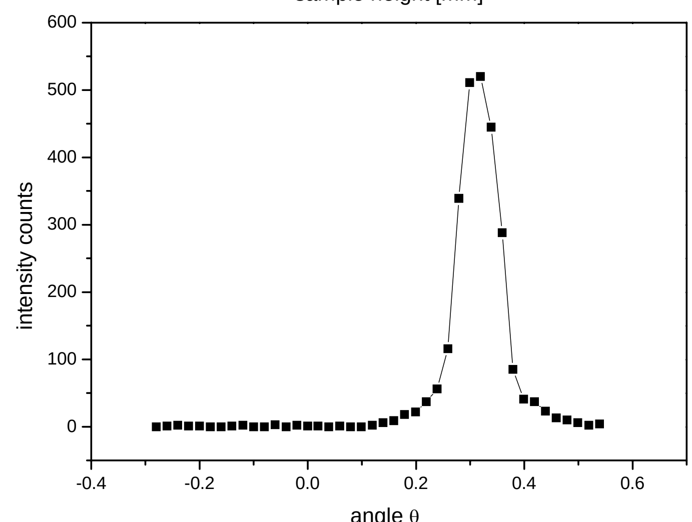


Alignment


detector
sample
stage
slit
slit

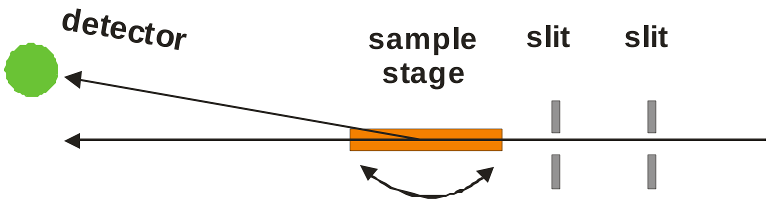

intensity counts
2900
2800
2700
2600
2500
2400
2300
2200
2100
2000
-5
-4
-3
-2
-1
0
sample height [mm]

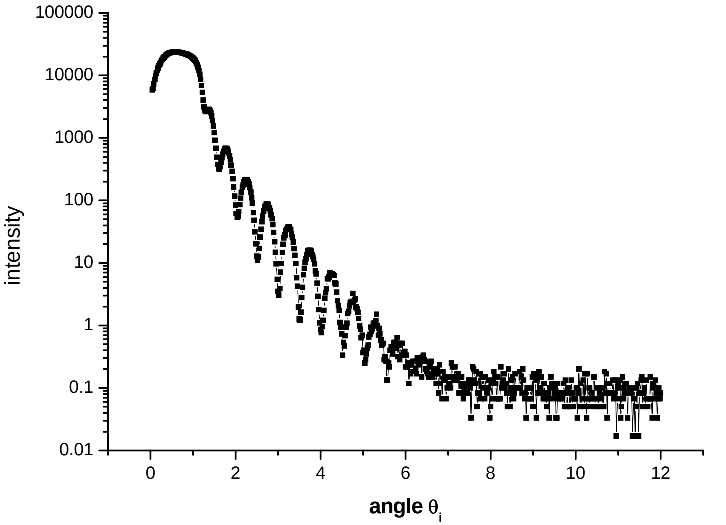

detector
sample
stage
slit
slit


intensity counts
600
500
400
300
200
100
0
-0.4
-0.2
0.0
0.2
0.4
0.6
angle θi

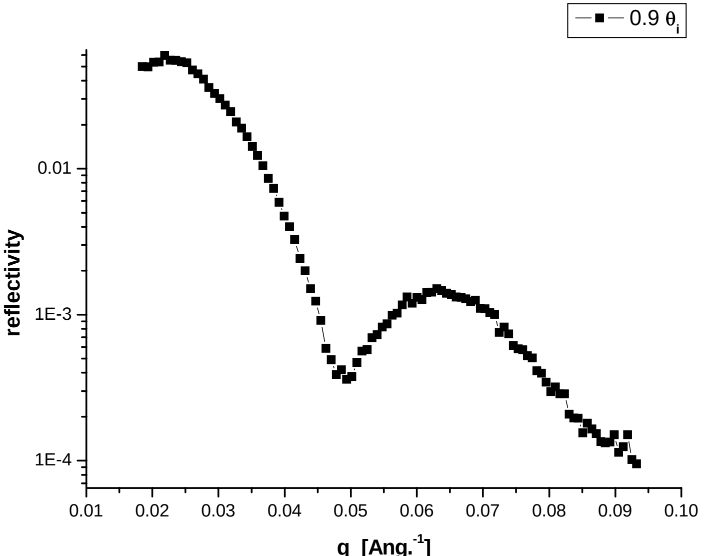
Measurement

$R(q_z),\ q_z = 4\pi \sin\theta_i/\lambda$





monochromatic mode: fixed λ, variable θ



time-of-flight mode: variable λ, fixed θ

$$\lambda = \frac{h}{m_N}\frac{t_{tof}}{d} = \frac{6.6252 \cdot 10^{-34}}{1.6747 \cdot 10^{-27}} 10^{3} \frac{t_{tof}}{d}$$

Data reduction

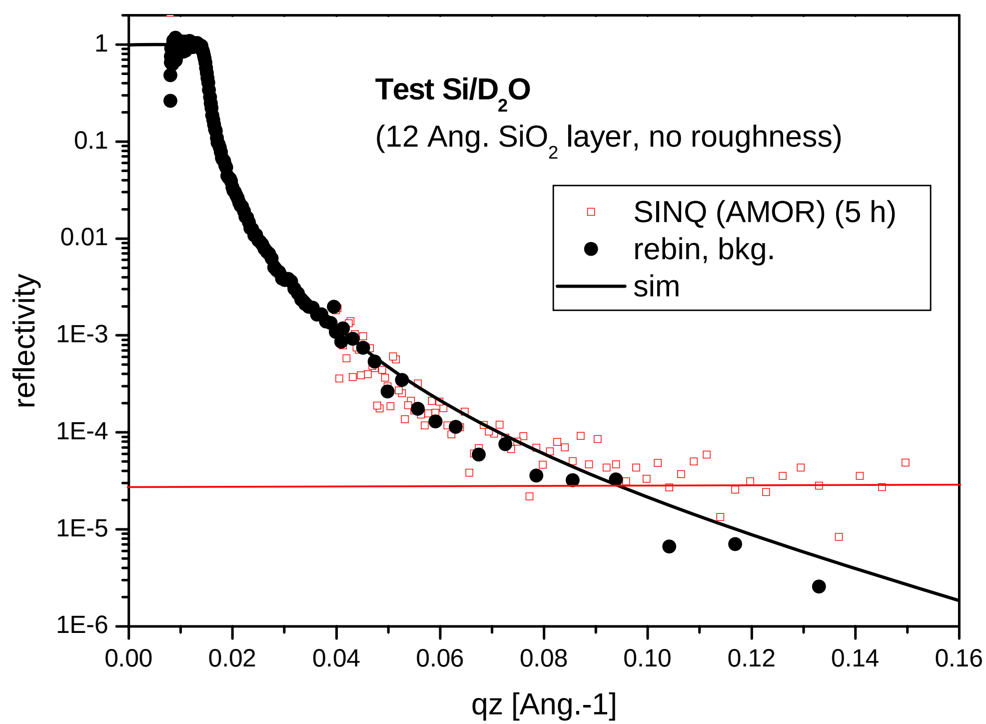
- Transformation of θ (and λ) into q_z

- Background subtraction

    - For time-of-flight:

$$R(q_z) = \frac{I_{ref} - b}{I_{dir} - b}$$

- Illumination



Data reduction

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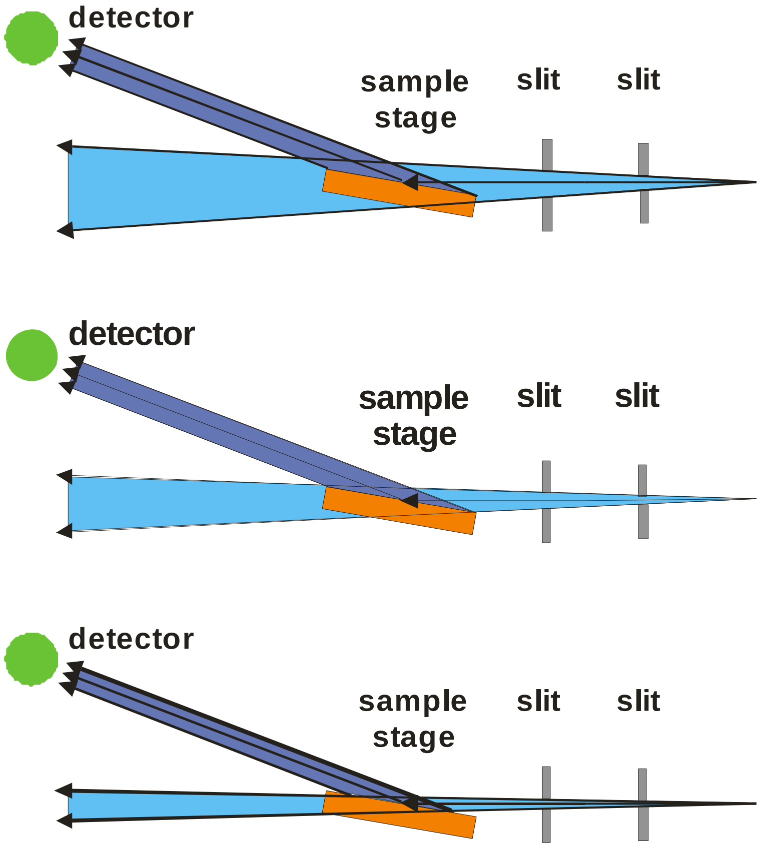
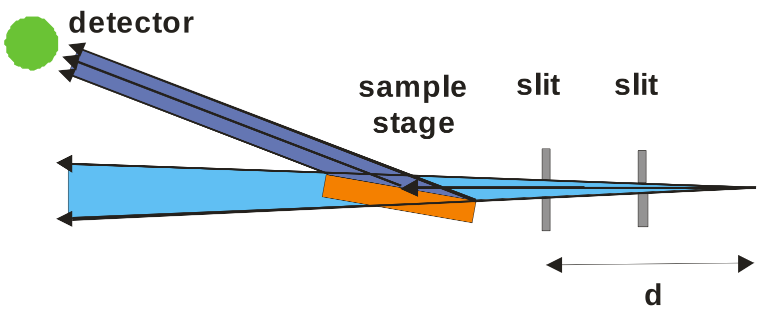


Resolution

- angular resolution δθ

$$\delta\theta = \frac{\Delta\theta}{\theta} \quad \text{with} \quad \Delta\theta = \arctan\frac{h}{d}$$

d, distance source to slit
h, half height of slit



- wavelength resolution δλ

$$\delta\lambda = \frac{\Delta\lambda}{\lambda}$$

Δλ, either wavelength spread by monochromator or,
for time-of-flight data by time spread

$$\delta\lambda \equiv \delta t = \frac{t_{pulse} + t_{bin}}{t_{tof}}$$

determines q_z resolution

Simulation of Data

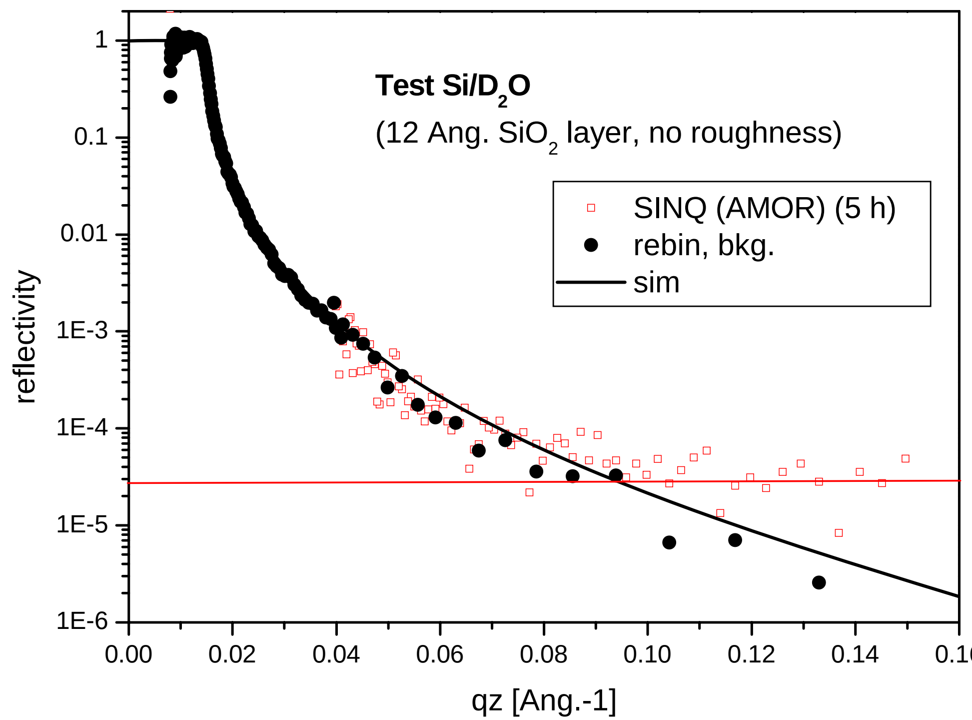
$$R(q) = \left(\frac{q_c}{q_1}\right)^4 \left|\int \rho(z) e^{2iq_1 z} dz\right|^2$$

$$= R_F(q) \left|\int \rho(z) e^{2iq_1 z} dz\right|^2$$

for each layer/interface

- scattering length density
- thickness
- roughness

are requested.



Simulation of Data

scattering length density

$$SLD \equiv \rho b = \frac{\sum nb_i}{v_M} = \sum nb_i \frac{N_L \rho}{\sum nM_i}$$

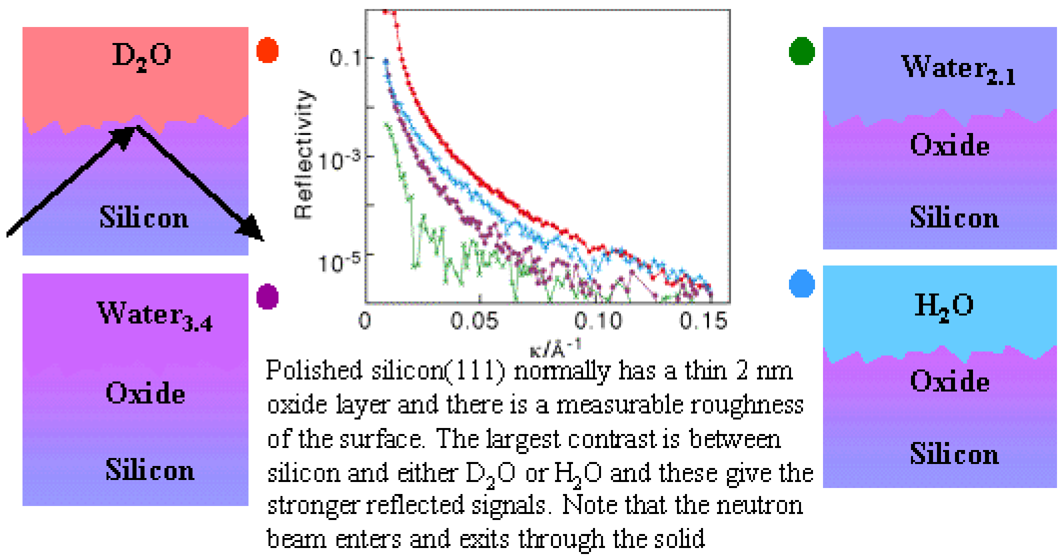
with b_i atomic incoherent scattering length,
M_i atomic mass,
ρ substance density,
N_L Avogadro's number

Neutron Scattering Lengths and Cross Sections
http://www.ncnr.nist.gov/resources/n-lengths/

Scattering Length Density Calculator
http://www.ncnr.nist.gov/resources/sldcalc.html

Simulation of Data

- simulations of the reflection of light
- simulation of reflectivity curve
- contrast variation in neutron reflectometry



http://ptcl.chem.ox.ac.uk/~rkt/techniques/nrmain.html

Fitting of Data

available programs:

Parratt32, HMI, Berlin
Reflfit and Reflpol, NIST, Gaithersburg
Motofit, A. Nelson, ANSTO, Sidney
SimulReflec, LLB, Saclay
SURFace, ISIS, Didcot
…

For each layer/interface

- scattering length density
- thickness
- roughness

will be obtained.

Fitting of Data

Example for Parratt32

2. Conducting Polymer

$C_{20}H_{13}N_3O_6S_2$

ρ: 1.3 g cm^{-3}

Z = (6x20)+(1x13)+(7x3)+(8x6)+(16x2) = 234

M = (12.011x20)+(1.008x13)+(14.0067x3)+(16*6)+(32.06*2)

= 455.464 g mol^{-1}

X-rays:

SLDx = 1.14x10^{-5} + 7.46x10^{-8}i Å^{-2} (from NIST SLD calculator)

λ = 1.54056 Å

δ = 4.306x10^{-6}; β = 2.818x10^{-8}

Neutrons:

SLDn = 2.63x10^{-6} Å^{-2} (from NIST SLD calculator)

b = (6.646x20) + (-3.739x13) + (9.36x3) + (5.803x6) + (2.847x2)

= 152.905 fm

Abs. X-sect.=(0.0035x20)+(0.3326x13)+(1.9x3)+(0.00019x6)+(0.53x2)

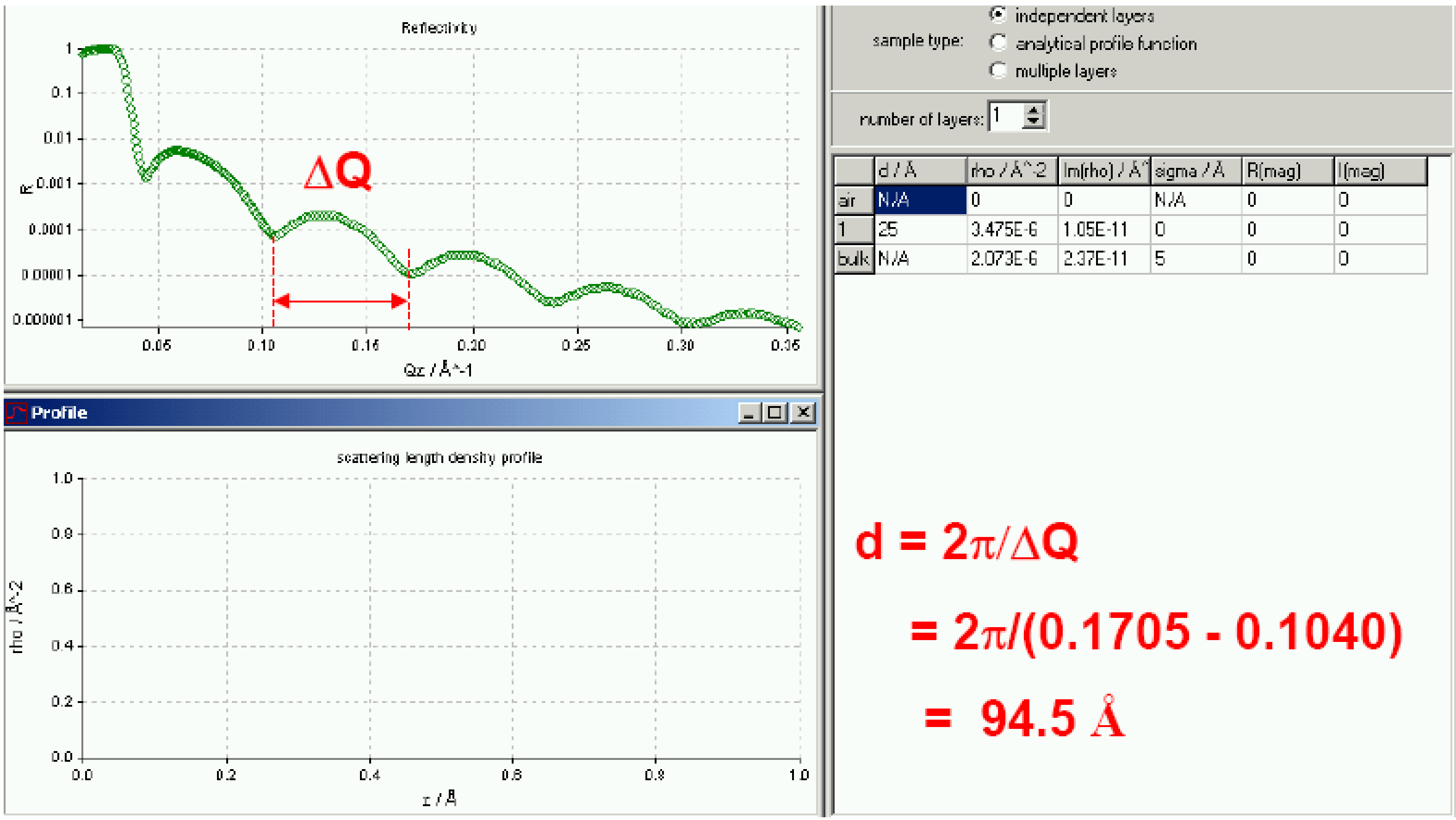
= 11.155 barn

Parratt SLD = 6.18x10^{-6} + 5.24x10^{-13}i Å^{-2}

by M. James, ANSTO
http://www.ansto.gov.au/ansto/bragg/symposium/talks/fri_james.pdf

Conducting Polymer

Estimating Film Thickness



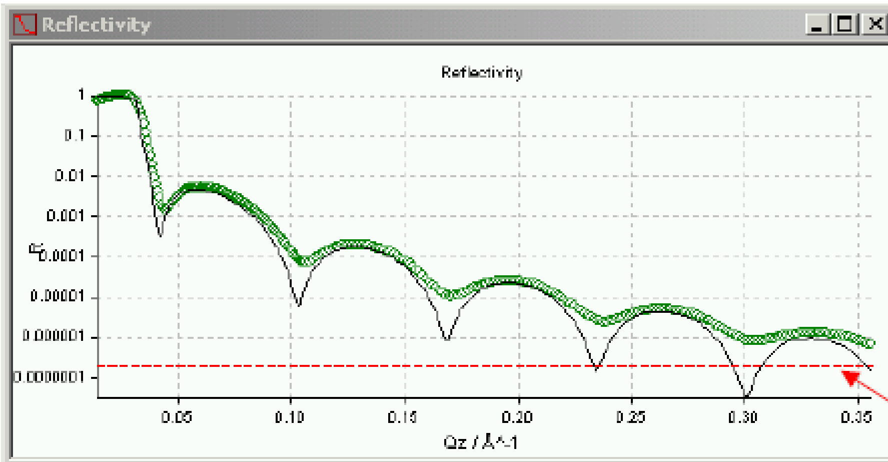
$$d = 2\pi/\Delta Q$$

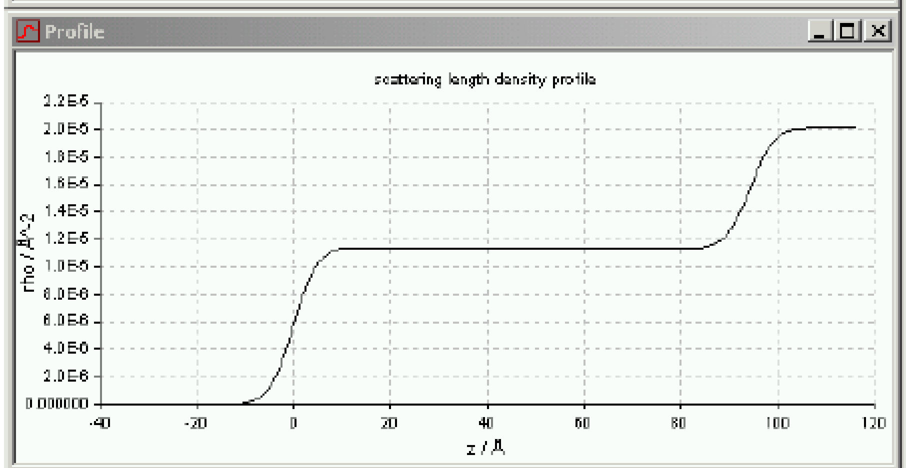
$$= 2\pi/(0.1705 - 0.1040)$$

$$= 94.5\ \text{Å}$$

Conducting Polymer

Estimating Background





Model

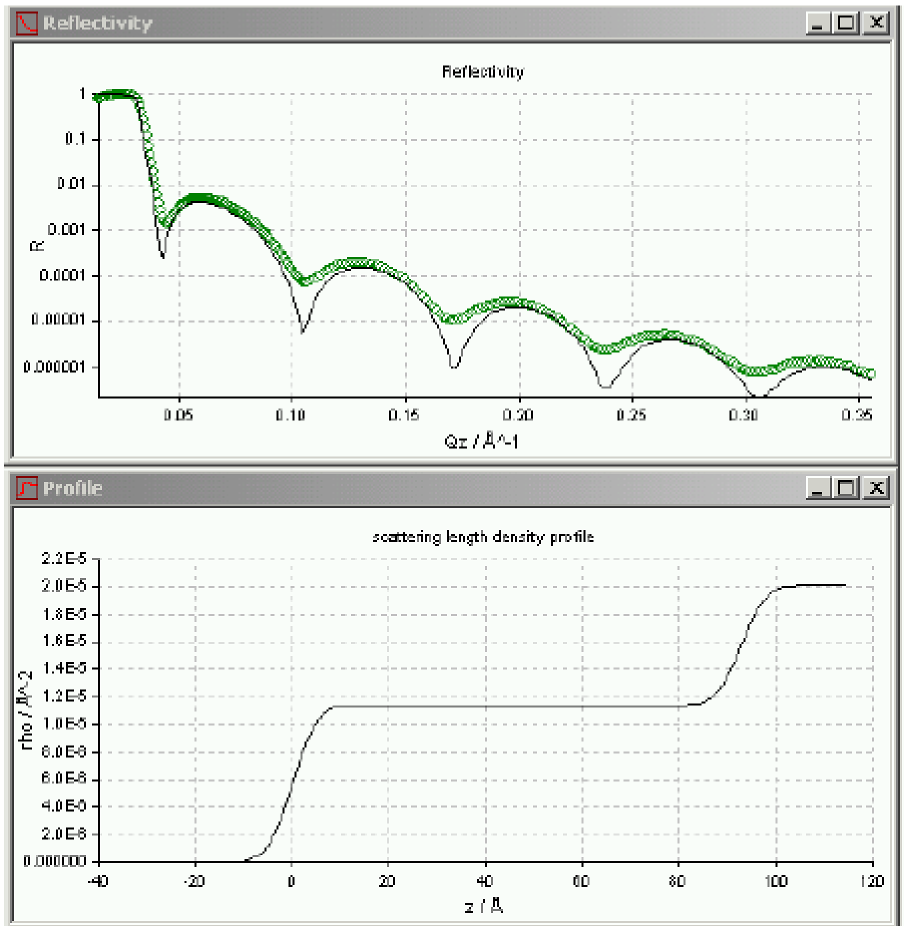
sample type: (•) independent layers ( ) analytical profile function ( ) multiple layers

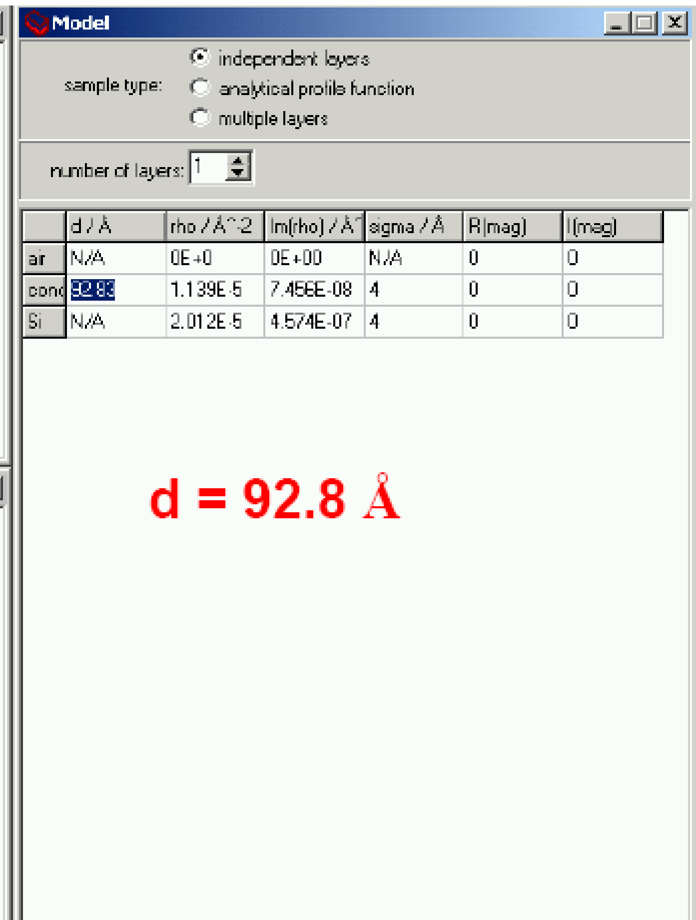
number of layers: 1

	d / Å	rho / Å^-2	Im(rho) / Å^	sigma / Å	R(mag)	I(mag)
air	N/A	0E+0	0E+00	N/A	0	0
cond	94.5	1.139E-05	7.456E-08	4	0	0
Si	N/A	2.012E-05	4.574E-07	4	0	0

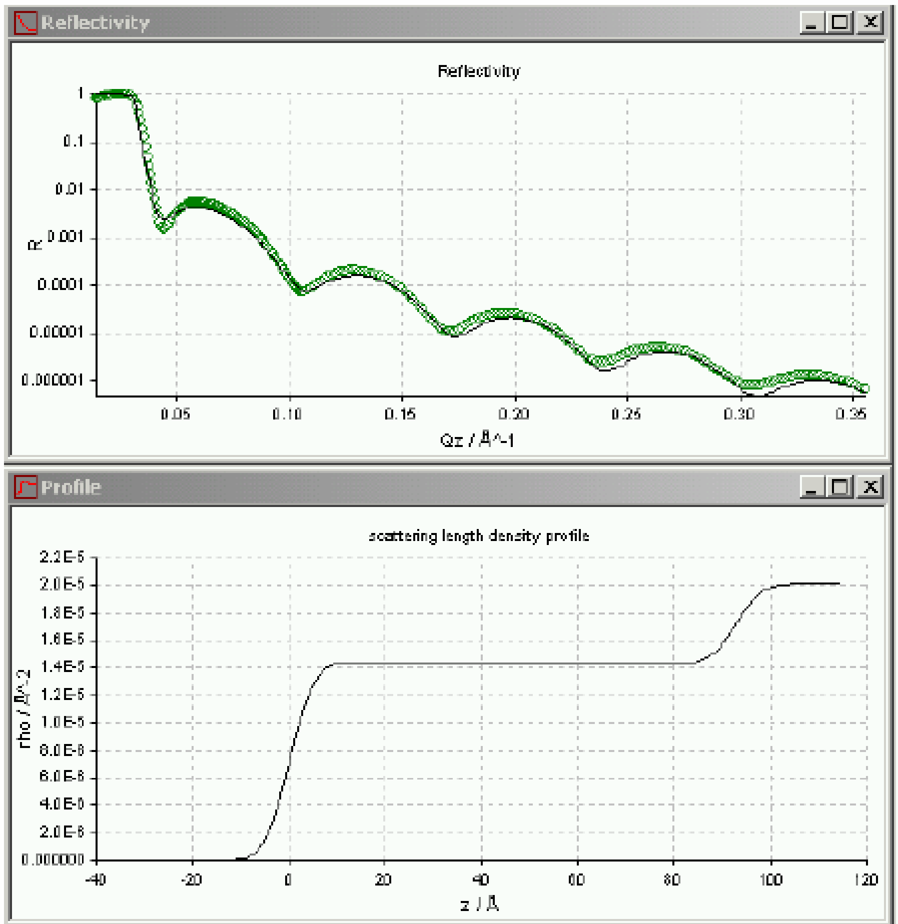
Background ~ 2x10^{-7}

Conducting Polymer
Refining Film Thickness





Conducting Polymer
Refining Film SLD



Model

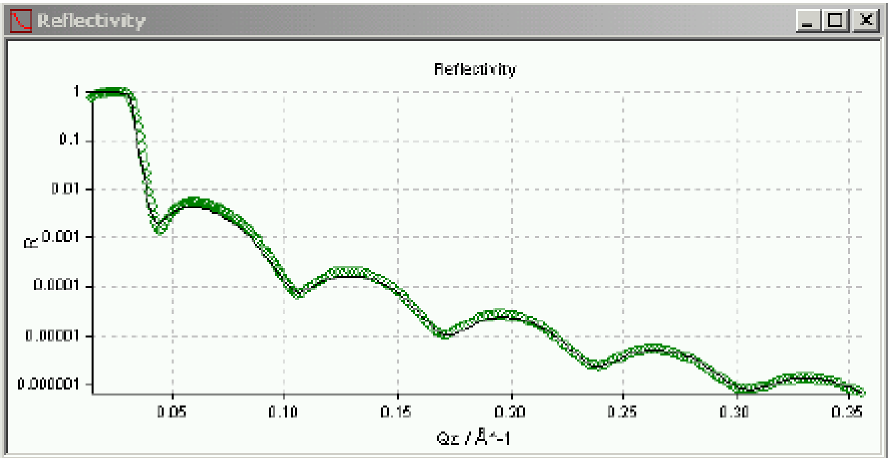
sample type: independent layers / analytical profile function / multiple layers

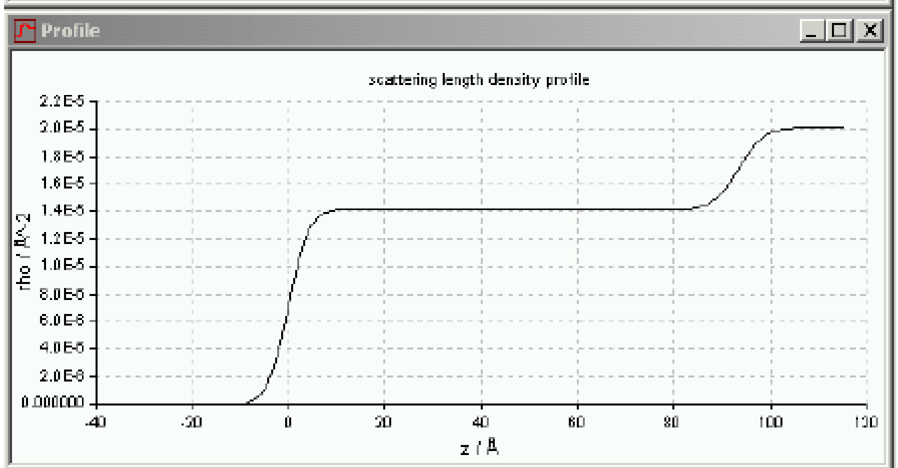
number of layers: 1

	d / Å	rho / Å^-2	Im(rho) / Å^	sigma / Å	R(mag)	I(mag)
air	N/A	0E+0	0E+00	N/A	0	0
cond	92.83	1.437E-5	7.456E-08	4	0	0
Si	N/A	2.012E-5	4.574E-07	4	0	0

SLD = 1.44x10^{-5} Å^{-2}

Conducting Polymer
Refining Film & Substrate Roughness





Model

sample type: independent layers / analytical profile function / multiple layers

number of layers: 1

	d / Å	rho / Å^-2	Im(rho) / Å^	sigma / Å	R(mag)	I(mag)
air	N/A	0E+0	0E+00	N/A	0	0
cond	93.55	1.415E-5	7.456E-08	3.422	0	0
Si	N/A	2.012E-5	4.574E-07	4.23	0	0

D = 93.6 Å

SLD = 1.42x10^{-5} Å^{-2}

σ_{film} = 3.4 Å

σ_{Si} = 4.2 Å