Instrumentation for Small-Angle X-ray and Neutron Scattering
and
Instrumental Smearing Effects

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➤ Limited to most common instruments……
Outline

- Instrumental Smearing
- SAXS Instrumentation
- SANS Instrumentation

Keywords:
- Long-slit
- Pin-hole
- Focussing

Litterature


Schematic setup for small-angle scattering

\[ q \equiv \frac{4\pi \sin \theta}{\lambda} \]

C. Glinka
Flux and resolution

Well-defined wavelength and well-collimated beam

⇒ low flux

⇒ relax resolution,

usually wavelength or collimation
or one of the directions of collimation
(out-of-plane or focusing)

Often not an issue for synchrotron radiation due to extreme flux!
Increase flux:

- Relax instrumental resolution
  - Long-slit geometry (x-rays)
  - Increase wavelength spread
- Focus beam
  - Mainly used for x-rays

Focussing at detector

\[ q \equiv \frac{4\pi \sin \theta}{\lambda} \]

Fig. 3. Scattering angles for a setup with the beam focused at the detector.

Does not lead to smearing for SAXS from non-crystalline samples
Long-slit geometry

\[ q \equiv \frac{4\pi \sin \theta}{\lambda} \]
Long-slit geometry

\[ q \equiv \frac{4\pi \sin\theta}{\lambda} \]
Distributions of $q$

(a) long-slit geometry           (b) pinhole geometry.

Constant $q$ contours

Distributions of $q$: 
X-ray sources I

- Laboratory
  - Sealed tube
  - Rotating anode (distribute heat)
  - Micro source (reduce heat, increase peak intensity)

- Isotropic emission!
- Characteristic lines!
- Source size: 10µm to 1 mm
X-ray sources II

- **Synchrotron**
  - Bending magnet
  - Wiggler (series of magnet, incoherent addition of rays) $I \propto N$
  - Undulator (series of magnet, coherent addition of rays) $I \propto N^2$

- Collimated emission!
- Continuous spectrum (except Undulator)!
- beam size: 10µm to 1 mm

Als-Nielsen and MacMorrow:
**SAXS principle**

Guard pinhole/slit or anti-scatter pinhole/slit
Kratky block camera (Long slit)

Q range to 0.003 1/Å
Sealed tube $10^7$-$10^8$ ph/s
Non-oriented sample
- or perfectly oriented
Data from block copolymer micelles

1% (w/w) P-85 solution at 50 C:

Smeared data
Ideal data

Otto Glatter, Gunther Scherf, Karin Schillen and Wyn Brown,

Characterization of a Poly(ethylene oxide)-Poly(propylene oxide) Triblock Copolymer (E027-PO39-EO27) in Aqueous Solution
Long-slit geometry: Smearing

\[ q \equiv 4\pi \sin \theta / \lambda \]

Source \hspace{1cm} Sample \hspace{1cm} Detector

\[ w_{\parallel}(q_{\parallel}) \hspace{1cm} w_{\perp}(q_{\perp}) \hspace{1cm} w_{\lambda}(q_{\lambda}) \]
Long-slit smearing

\[ I(q) = \int \int \int \frac{d\sigma}{d\Omega} \left( \frac{\lambda_0 \sqrt{(q - q_\parallel)^2 + q_\perp^2}}{\lambda} \right) W_\parallel(q_\parallel) W_\perp(q_\perp) W_\lambda(\lambda) \, dq_\parallel \, dq_\perp \, d\lambda, \]

\[ \frac{d\sigma}{d\Omega}(q) \] is ideal cross section

Note: 3 numerical integrations !!!
**SAXSess camera**

**Improved Kratky camera:**

Rotating anode + Göbel mirrors: $10^{10}$ ph/s !

*(monochromatic beam)*

→ Short acquisition times/deadtime effects

→ Image plate detector

with less than 0.1 mm resolution

*('photographic’ linear detector)*

*Note: Also wide-angle option and pseudo-pinhole geometry*

A. Bergmann, D. Orthaber, G. Scherf and O. Glatter

*Appl. Cryst.* (2000). 33, 869-875  Improvement of SAXS measurements on Kratky slit systems by Göbel mirrors and imaging-plate detectors
Göbel mirrors

Si-W multi-layers on a parabolic substrate

Lattice spacing of multilayer varies with position, so that divergent beam comes out parallel!

Length about 5 cm, Bragg angle a few degrees
Bonse-Hart camera = Ultra SAXS

Two multiple bounce perfect crystals
⇒ Good collimation in scattering plane of crystal
  'long-slit' smearing in perpendicular direction
  • Large beam size (3 x 3 mm²)
  • q down to 10⁻⁴ Å⁻¹
  • Rotating anode: 10⁶ ph/s
  • Sequential data collection
  • Always subtract two very large signals to obtain scattering from sample

• Zemb et al.: Göbel mirrors gives factor of 10 increase in flux
• Synchrotrons allows double Bonse-Hart geometry with pinhole smearing
Perfect crystal

Extinction gives effectively a finite number of layers contributing to the diffraction

(a) symmetric Bragg

\[ Q = mG(1 + \zeta) \]

\[ \Delta k \]

\[ k \]

\[ k' \]

\[ d \]
Perfect crystal: Darwin curve

Width at 1 Å is about 0.1 mrad

Figure 5.5: The Darwin reflectivity curve calculated from Eq. (5.21). For values of $x$ between $-1$ and $1$ the reflectivity is 100%. This is known as the region of total reflection. For large values of $|x|$ the intensity decays as $1/(2x)^2$. At $x = 1$ the X-ray wavefield has its maxima on the atomic planes, whereas for $x = -1$ the nodes of the wavefield coincide with the atomic planes.
Bonse and Hart, 1965

Single bounce $R(\theta) = (\theta - \theta_o)^2$

m-bounce $R(\theta) = (\theta - \theta_o)^{2m}$

-but limitations due to thermal diffuse scattering and imperfections

- note also influence of absorption
Simple pin-hole camera

• Lower flux, but use 2D detector
• In general: $q_{\text{min}} = 0.01 - 0.005$ 1/Å at lab sources
  $q_{\text{min}} = 0.001$ 1/Å at synchrotron sources
• Can be used for any sample!

Oak Ridge 10 m camera:

Graphite monochromator

6kW rot. anode

sample

beamstop

2D detector

sample 1x1 mm²; $10^6$ ph/s

Graphite: Mosaic crystal: Broad rocking curve
Huxley-Holmes SAXS

EMBL Hamburg, bending magnet $10^{12}$ photons/s/100mA
Rotating anode: $10^7$ ph/s

Detector

- Mirror: 1m!
- Source-monochr.: 20 m
- Monochr.-detector: 10 m
- Sample size 5 x 2 mm$^2$

Franks-type set-up: Two mirrors + filter at conv. source
Developed by P. Fraztl, Bruker AXS and Anton Paar
NanoSTAR for solution scattering

The Small Angle X-ray Scattering (SAXS) setup consists of:

- A rotating anode
- Göbel mirrors
- A three pin-hole collimation
- An integrated vacuum
- A 2D-gas detector

- Standard: 0.01 to 0.35 1/Å with 2 x 10⁷ ph/s
- Beamsize 1 mm diameter

High flux
Low background

Aarhus SAXS
New NanoSTAR
SAXS data from Aarhus NanoSTAR

- Sodium dodecyl sulphate (SDS) micelles
- 1 wt% in water.
- 2 hrs acquisition time

\[ q \text{ [Å}^{-1}\text{]} \]
\[ I(q) \text{ [cm}^{-1}\text{]} \]

\[ r \text{ [Å]} \]
\[ \Delta \rho (r) \text{ [a.u.]} \]

\[ \Delta \rho (r) \text{ for SDS micelle model} \]

Data Fit

\[ 0 \quad 0.1 \quad 0.2 \quad 0.3 \]
\[ 0.001 \quad 0.01 \quad 0.1 \]
\[ 0 \quad 0.2 \quad 0.4 \]

\[ 0 \quad 0.1 \quad 0.2 \quad 0.3 \quad 0.4 \]
\[ -1.2 \quad -1.0 \quad -0.8 \quad -0.6 \quad -0.4 \quad -0.2 \quad 0.0 \quad 0.2 \quad 0.4 \]

\[ 0 \quad 10 \quad 20 \quad 30 \quad 40 \quad 50 \]
Neutron sources

• Steady-state fission reactors
  - cold sources for long wavelength neutrons

Institute Laue-langevin, Grenoble

• Spallation sources (accelerators based)
  - Moderators
  (time of flight technique!!)

ISIS, UK:
Steady-state SANS

Broad band monochromator: mechanical velocity selector

Typically 10 – 20 % (FWHM) wavelength spread
Low q – high q

0.001 – 0.5 1/Å

$\lambda = 3 – 30 \text{ Å}$

Long wavelength $\Rightarrow$ low q

Short wavelength $\Rightarrow$ high q
Risø SANS: now at the PSI
Smearing contributions

Nominal $q$: $<q> = 4\pi \sin <\theta>/<\lambda>$
Smearing contributions

Nominal q: \( <q> = 4\pi \sin \frac{\theta}{<\lambda>} \)
Actual q: \( q = 4\pi \sin \frac{\theta}{\lambda} \)
SANS resolution function I

\[ q = 4\pi \sin \theta / \lambda \]

Derivative with respect to \( \lambda \):

\[ \Delta q = -q \Delta \lambda / \lambda \]

Derivative with respect to \( \theta \):

\[ \Delta q = \frac{4\pi}{\lambda} \cos \theta \Delta \theta \approx \frac{4\pi}{\lambda} \Delta \theta \]

Assume independent distributions described by Gaussians:

\[ \sigma (\langle q \rangle)^2 = \langle q \rangle^2 \sigma (\Delta \lambda / \lambda)^2 + (4\pi / \lambda)^2 \sigma (\Delta \theta)^2. \]
The various contributions can be measured, simulated or calculated

\[ \sigma (\langle q \rangle)^2 = \langle q \rangle^2 \sigma (\Delta \lambda / \lambda)^2 + (4\pi / \lambda)^2 \sigma (\Delta \theta)^2. \]

\[ I(\langle q \rangle) = \int R(\langle q \rangle, q) \frac{d\sigma(q)}{d\Omega} \, dq \]

Collimation and detector contributions

ILL D11: (Timmins, Svergun)

Direct beam (attenuated): Azimuthally averaged
Source: 5.0 x 3.0 cm²
Sample: 1.0 x 0.7 cm²
Source-sample: 5 m
Sample-detector: 3 m

- Includes also detector smearing
Analysis with smearing

\[ I(\langle q \rangle) = \int R(\langle q \rangle, q) \frac{d\sigma(q)}{d\Omega} \, dq \]

\[ \frac{d\sigma}{d\Omega}(q) = \Delta \rho^2 V^2 \left[ \frac{3[\sin(qR) - qR \cos(qR)]}{(qR)^3} \right]^2 \]

Data from Wiggnal et al.
TOF SANS

优势:
一切都在一个设置下被记录。

主要问题:
低通量在低q处。

图4.3  LOQ装置的示意图，ISIS裂变源，Didcot, UK [2]。经过样品与样品（典型的中子通量为样本 = 2 × 10^5 cm^-2 s^-1），束流通过一个真空管，管中包含一个^3H气体填充探测器（有效面积为64 × 64 cm^2，像素尺寸为6 × 6 mm^2）放置在4.5 m处的样品。入射波长范围为~2.2 - 10 Å，且散射角度< 7°，提供有用的Q范围为0.009 - 0.249 Å^-1。
Neutron Bonse-Hart set-up

Low flux
Large sample (cm²)
q = 2x10⁻⁵-3x10⁻³ 1/Å

Summary

**SAXS:** Kratky camera
Bonse-hart setup
Simple pinhole camera
Huxley Holmes camera
Franks setup
NanoSTAR with Göbel mirrors
+ many others

**SANS:** Steady state pinhole camera
Time-of Flight pinhole camera
Bonse-Hart setup
+ more

**Instrumental smearing:** - is routinely included in state-of-the-art data analysis