

PAUL SCHERRER INSTITUT



EPFL

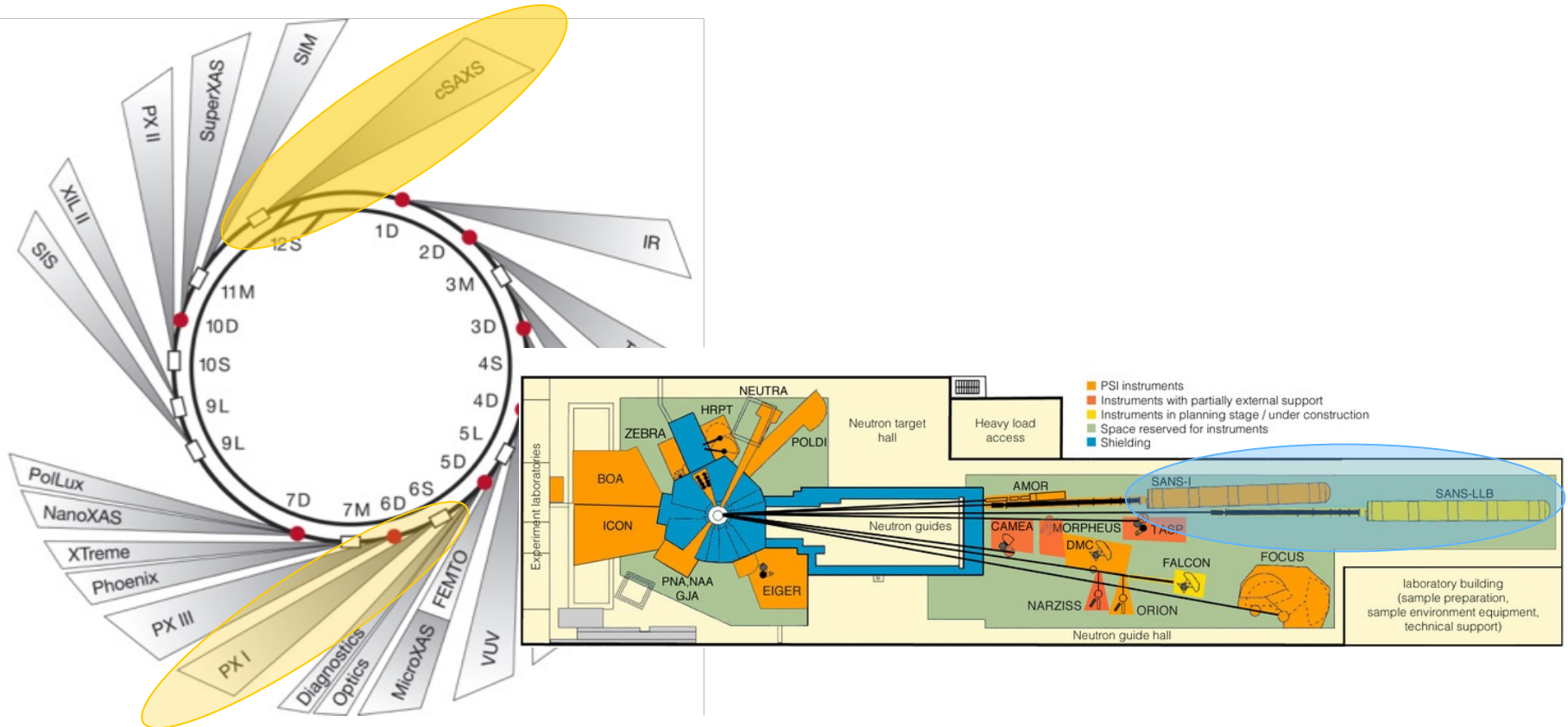


Marianne Liebi– Material Science at Large Scale Facilities

Small-angle scattering (SAXS/SANS)

EPFL Master Course 2024 MSE435

Small angle scattering at PSI: SAXS and SANS



X-ray vs. neutrons (vs. light)

what size range is of interest → q-range: detector distance and energy

beamsize: resolution vs. flux-density and beam damage

exposure time: signal to noise, detector speed

detector saturation

thickness of sample, for X-rays energy: ideally transmission = $1/e$

Why is the ideal transmission in SAXS assumed to be equal to $1/e$?

this value arises because of the exponential decay of X-ray intensity as it passes through matter. The intensity of X-rays passing through a sample decreases exponentially with sample thickness, and the attenuation coefficient, often denoted as " μ ", is a measure of this attenuation.

In SAXS, the ideal transmission corresponds to the limit of zero sample thickness, where the transmitted intensity is equal to the incident intensity. At finite sample thickness, the transmitted intensity is given by $I/I_0 = \exp(-\mu t)$, where t is the sample thickness.

For a sample of thickness t , the ideal transmission is obtained by taking the limit as t approaches zero, which gives:

$$\lim_{t \rightarrow 0} \exp(-\mu t) = 1$$

However, this ideal transmission is not achievable in practice, as all samples have a finite thickness, and some degree of scattering is always present. In experimental practice, the transmission is typically measured as the fraction of X-rays that pass through the sample without being scattered by more than a certain angle.

if you ask ChatGPT what the ideal transmission is for SAXS...and if you insists why 30%

- In practice, it is difficult to achieve an ideal transmission of 1 or 100%, and a transmission of 30% is often used as a practical limit in SAXS experiments. This value is chosen because it represents a compromise between the desire for high transmission and the need to avoid saturation of the detector due to high incident intensities.

X-ray vs. neutrons (vs. light)

what size range is of interest → q-range: detector distance and energy

beamsize: resolution vs. flux-density and beam damage

exposure time: signal to noise, detector speed

detector saturation

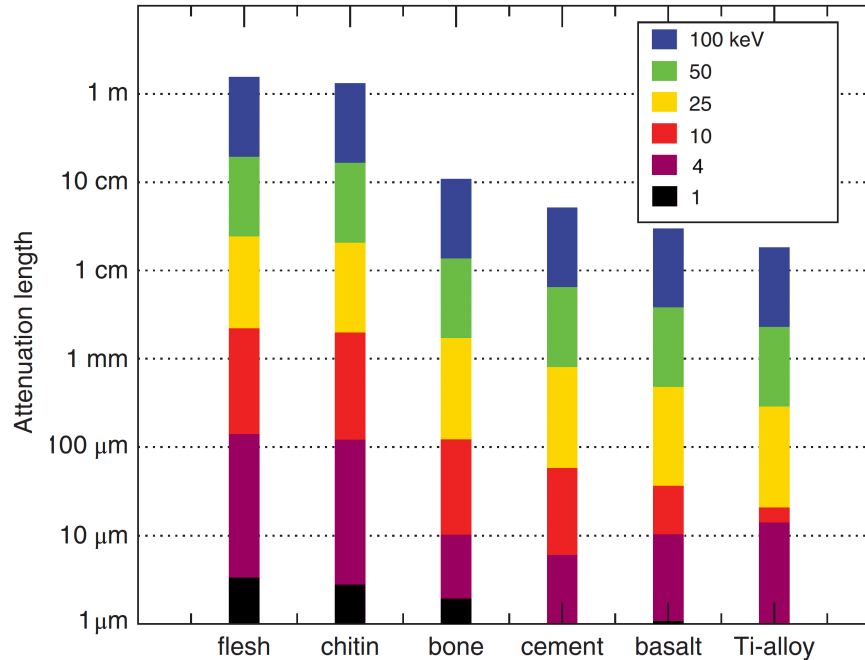
thickness of sample, for X-rays energy:

number of scatterer proportional to intensity $I(q) = (\rho_P - \rho_M)^2 N_P V_P^2 P(q) S(q)$

but absorption (Lambert-Beer law): intensity decays exponentially with thickness $I = I_0 e^{-N_i \sigma z}$

maximum at the absorption length i.e. where transmission is 1/e, ~ 30%

Sample thickness and X-ray energy



use online calculators for specific materials

for example: https://henke.lbl.gov/optical_constants/filter2.html

You want to measure your sample with SAXS for that you need to

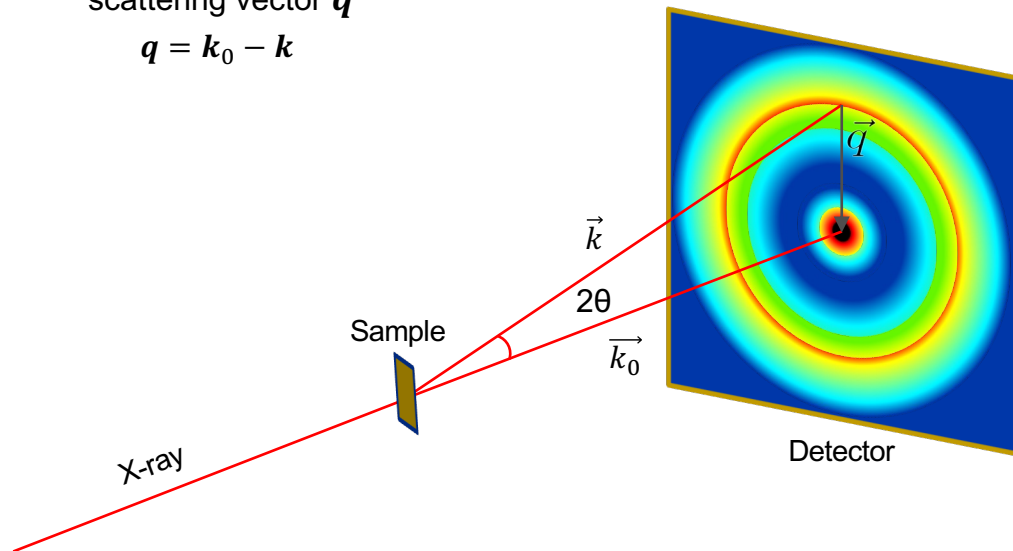
- choose the beamline
 - define X-ray energy and sample-to-detector distance
-
- what size (range) do you need to probe? → define q-range
 - energy depends on sample transmission and thickness → use online calculator
 - check with 1mm and 10 μm sample thickness
 - chose a beamline which covers the energy range you need
 - what is the minimum distance you need to place your detector for measuring your relevant size, when you have a 1mm beamstop blocking the direct beam, mounted 1cm in front of the detector
 - what is the maximum q you will reach at this position considering the detector area available?

some SAXS beamline specifics (from beamline webpages)

- cSAXS@SLS
 - 4.4 – 17.9 keV
 - Pilatus 2M, pixel size 172 μm x 172 μm , active area: 254 x 289 mm²
 - 7200 m or 2100 mm
- PX-I@SLS
 - 4.4 – 17.9 keV
 - EIGER X 16M, pixel size: 75 μm x 75 μm , active area: 311.1 x 327.2
 - max 2000 mm
- P62@DESY
 - 3.5 keV - 35.0 keV
 - Eiger2 X 9M (in vacuum), pixel size: 75 μm x 75 μm , active area: 233.1 mm x 244.7 mm
 - 1.5m up to 12.0m
- ForMAX@MAXIV
 - 8-25 keV
 - EIGER2 X 4M, pixel size: 75 μm x 75 μm , active area: 155.1 mm x 162.2 mm
 - detector distance. 800 – 7600 mm

scattering vector $\mathbf{q}$

$$\mathbf{q} = \mathbf{k}_0 - \mathbf{k}$$



$$|\vec{q}| = q = \frac{4\pi\sin(\theta)}{\lambda}$$

light $\lambda = 400$ to 600 nm

X-ray tube $\lambda = 1$ to 2 Å

Cu $K\alpha = 1.5406$ Å

synchrotron $\lambda = 0.1$ to 5 Å

thermal neutrons $\lambda = 1$ to 10 Å

electrons $\lambda = 0.025$ Å

X-ray energy mostly given in keV

Electronvolt = eV

Energy of an electron after being accelerated from rest in a potential of 1 V

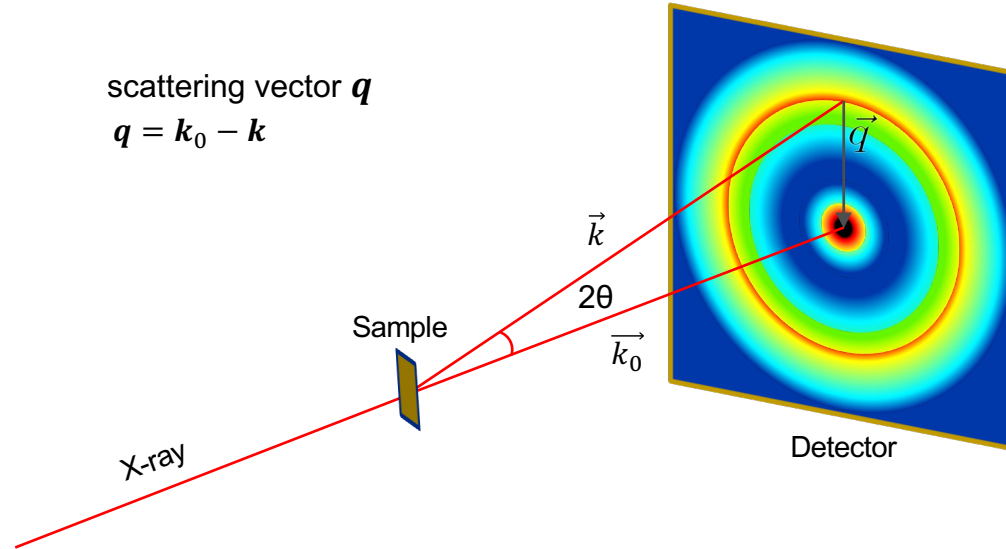
$$1 \text{ eV} = 1.6022 \times 10^{-19} \text{ J}$$

$$E = hc / \lambda$$

h is Planck's constant (6.6261×10^{-34} Js)

c is the speed of light (2.9979×10^8 m/s).

$$\lambda [\text{Å}] = 12.3984/E [\text{keV}]$$



scattering vector $\mathbf{q}$
 $\mathbf{q} = \mathbf{k}_0 - \mathbf{k}$

$$|\vec{q}| = q = \frac{4\pi\sin(\theta)}{\lambda}$$

Bragg's law

$$d = \frac{2\pi}{q}$$

SAXS: scattering from variation in electron density distribution, NOT from single atoms as in XRD

larger structures $\rightarrow$ smaller angles
 XRD/WAXS: 10 cm detector distance
 SAXS: several m detector distance

X-ray vs. neutrons (vs. light)

what size range is of interest → q-range: detector distance and energy

beamsize: resolution vs. flux-density and beam damage

exposure time: signal to noise, detector speed

detector saturation

thickness of sample, for X-rays energy:

number of scatterer proportional to intensity $I(q) = (\rho_P - \rho_M)^2 N_P V_P^2 P(q) S(q)$

but absorption (Lambert-Beer law): intensity decays exponentially with thickness $I = I_0 e^{-N_i \sigma z}$

maximum at the absorption length i.e. where transmission is 1/e, ~ 30%

C) Experimental method(s), specific requirements.

The proposed experiment plans to measure simultaneous scanning SAXS/WAXS with a beamsize of $25\mu\text{m}$ and a photon energy of 12.4 keV. We aim to measure samples extracted from the inferior and superior sides of each femoral neck, yielding a dataset of 180 samples in total. In order to obtain the full characterization of bone scattering parameters, we need a q -range of $0.02 - 2 \text{ nm}^{-1}$ in the SAXS regime in order to capture the collagen equatorial scattering and sufficient mineral scattering, as well as a WAXS q -range of $10\text{-}40 \text{ nm}^{-1}$ in the aim of obtaining the hydroxyapatite 002 peak ($\sim 18.2 \text{ nm}^{-1}$) in the full azimuthal range. Previous experiments at the beamline have proven the feasibility of the suggested setup in using sample-to-detector distances of 7m and 130mm for the SAXS and WAXS respectively.

