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Small-angle scattering (SAXS/SANS)

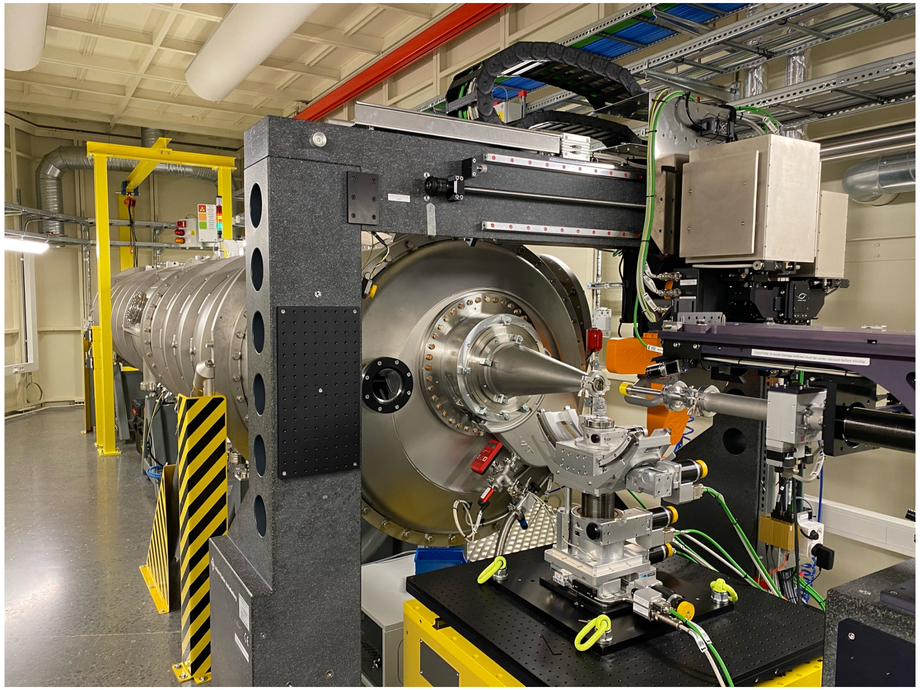
EPFL Master Course 2025 MSE435

X-ray scattering at synchrotrons

state-of the art detectors

shared sample environments (e.g. Rheo-SAXS)

Large flight tubes and low emittance: Ultra small angle X-ray scattering



X-ray scattering at synchrotrons

High Brilliance allows for

Low divergence ⇒ high angular resolution scattering/diffraction patterns

Tight focus ⇒ small sample sizes e.g., protein crystals ~ 1 mm^3

Low emittance ⇒ large working distance between focussing optics and sample ⇒ bulky sample environments

High flux ⇒ rapid data acquisition, time-resolved studies down to ms regime or shorter

X-ray scattering at synchrotrons

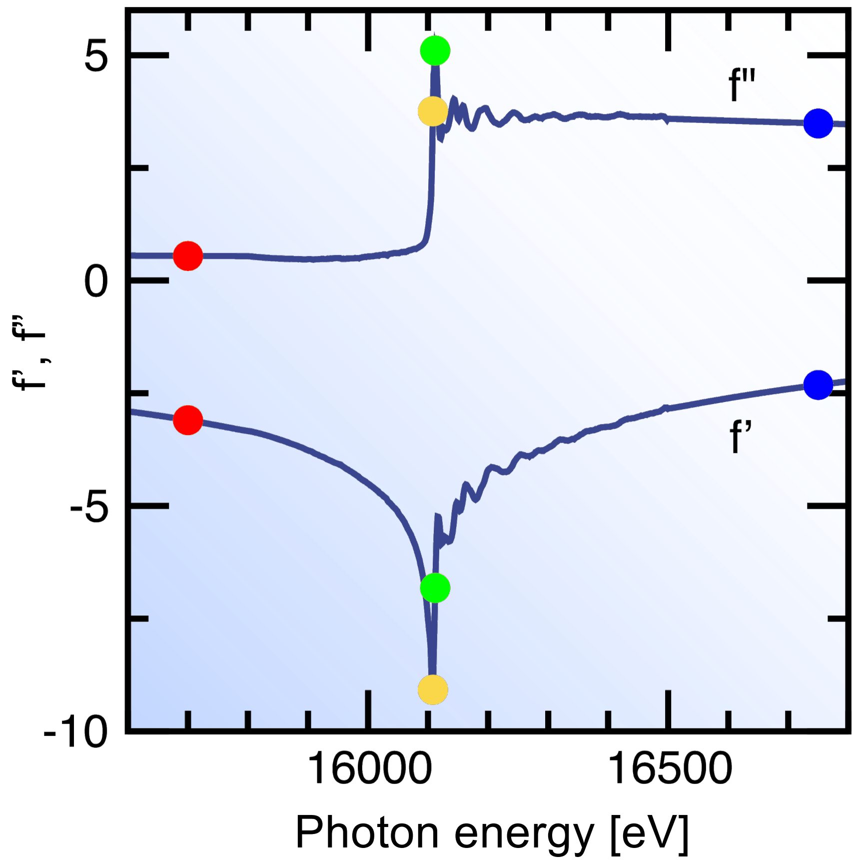
Acess to high photon energies:

Penetrate deep into samples, e.g., aeronautical components, large fossils, concrete, etc.

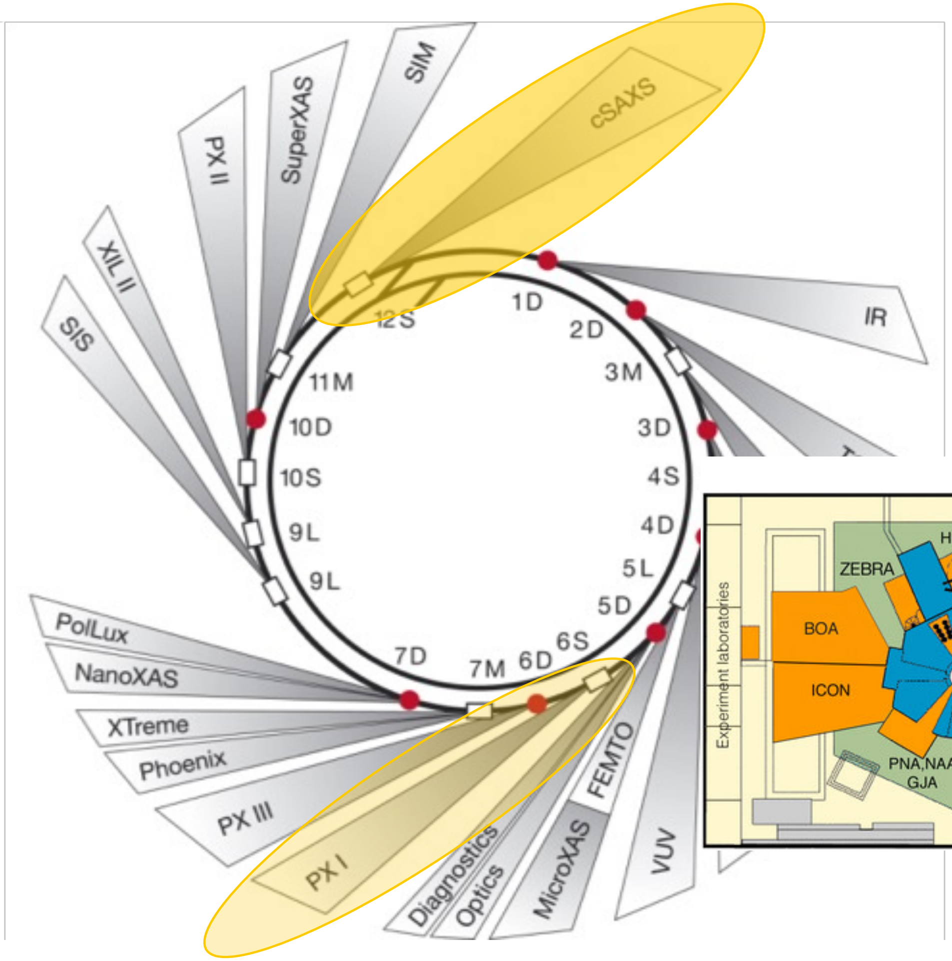
Tunability of energy:

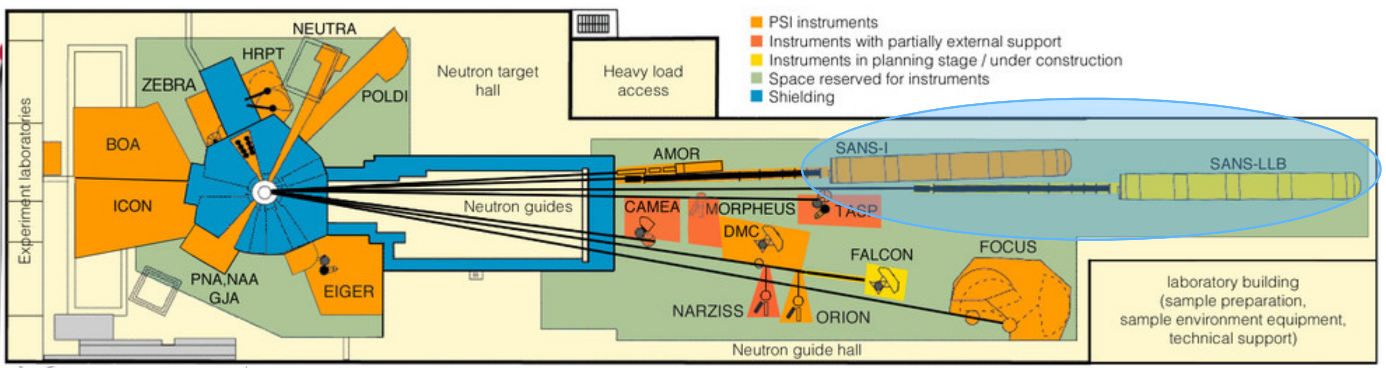
Abrupt changes to atomic scattering amplitudes as one crosses an absorption edge

"Anomalous" signal



Small angle scattering at PSI: SAXS and SANS





Practical consideration for an experiment

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

if you ask ChatGPT…

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 $I/I0 = \exp(-\mu t)$, 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 tranmission 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.

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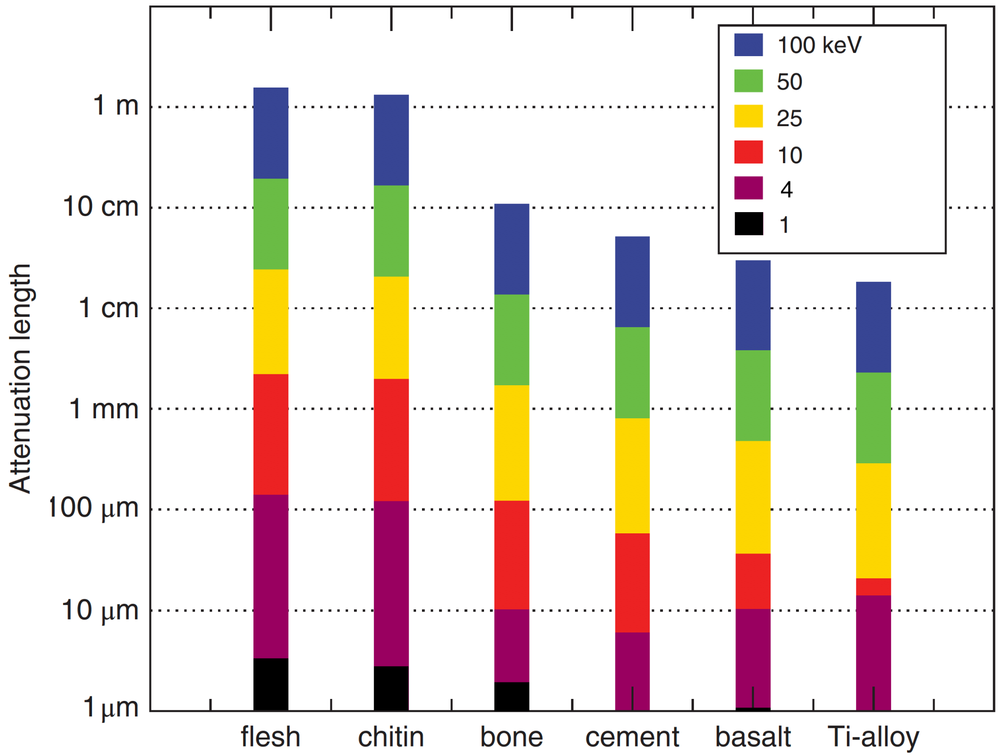
detector saturation

thickness of sample, for X-rays energy:

- number of scatterer proportional to intensity $I(q) = (\rho_\mathrm{P} - \rho_M)^2 \boldsymbol{N_P} V_P^2 P(q) S(q)$
- 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 calculatators for specific materials

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

Exercise case study

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 um 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 mm^2
  - 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

You want to measure bone using small-angle X-ray scattering with an energy of 12.4 keV, the interesting features you expect to be the spacing along the collagen fiber with a d-period of 67 nm, what is the minimum distance you need to place your detector at for measuring the collagen peak when you have a 1mm beamstop blocking the direct beam mounted 1cm in front of the detector?

Using $q = 4\pi \sin(\theta)/\lambda$ and $d = 2\pi/q$, calculate the scattering angle $2\theta \approx 0.0855°$

The horizontal distance needed between sample and detector can be calculated from $dist = r \tan(2\theta) + 10mm$, with $r = 0.5mm$ for the beamstop radius. This gives $dist \approx 345mm$.

1.If you want to also measure the diameter of the collagen fiber which you expect to be in the range of 150 – 200 nm, do you need to move the detector closer or further away?

Larger structures scatter at smaller angle, thus the detector needs to be moved further away

1.How large do you need the detector area to be, to also measure the (002) peak of the hydroxy-apatite crystal which has a lattice parameter c = 6.88 Å?

Using $q = 4\pi \sin(\theta)/\lambda$ and $d = 2\pi/q$, calculate the scattering angle $2\theta \approx 8.336°$

The detector size can be calculated from $l = \tan(2\theta) * dist$ using dist calculated in (1), we have $l = 50.55mm$. Then $area = (2l)2 \approx 102cm2$

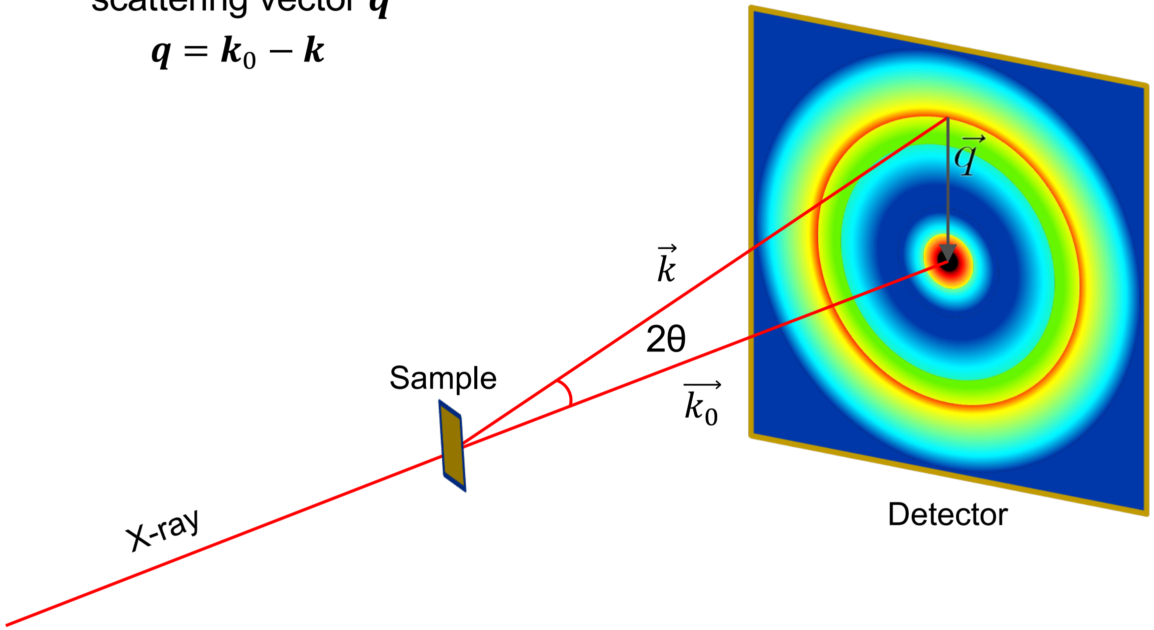
EPFL PSI

Scattering/Diffraction

scattering vector $\boldsymbol{q}$

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

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



light λ = 400 to 600 nm

X-ray tube λ = 1 to 2 Å

Cu Kα = 1.5406 Å

synchrotron λ = 0.1 to 5 Å

thermal neutrons λ = 1 to 10 Å

electrons λ = 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 eV = 1.6022×10^{-19} J

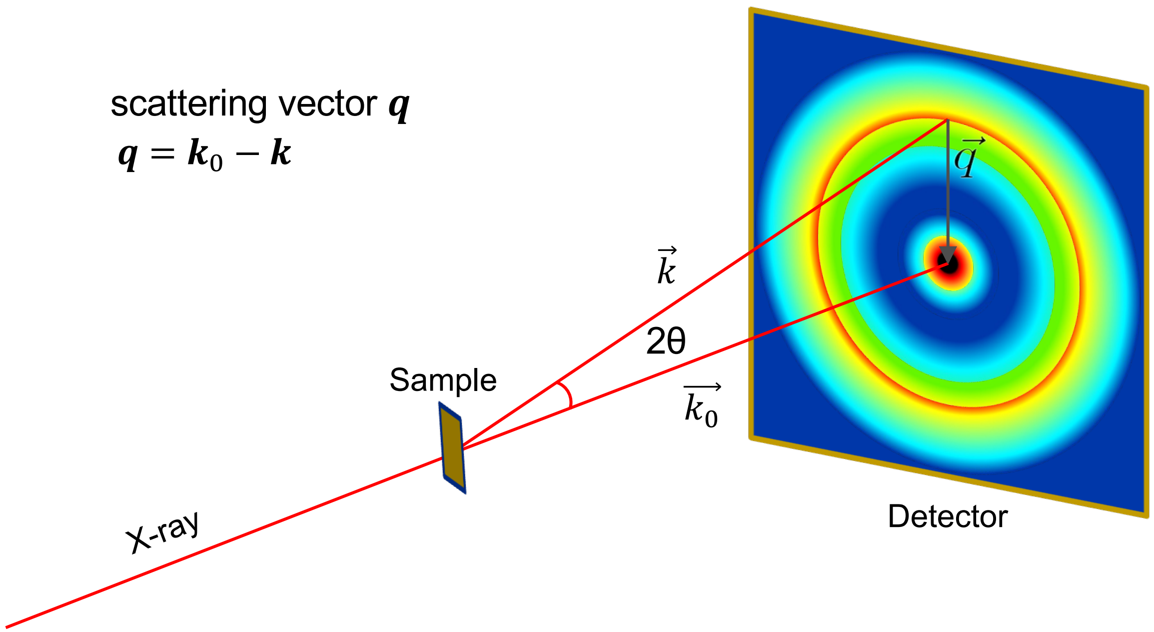
$E = hc / \lambda$

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

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

λ [Å] = 12.3984/E [keV]

Scattering/Diffraction



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

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 → smaller angles
XRD/WAXS: 10 cm detector distance

SAXS: several m detector distance

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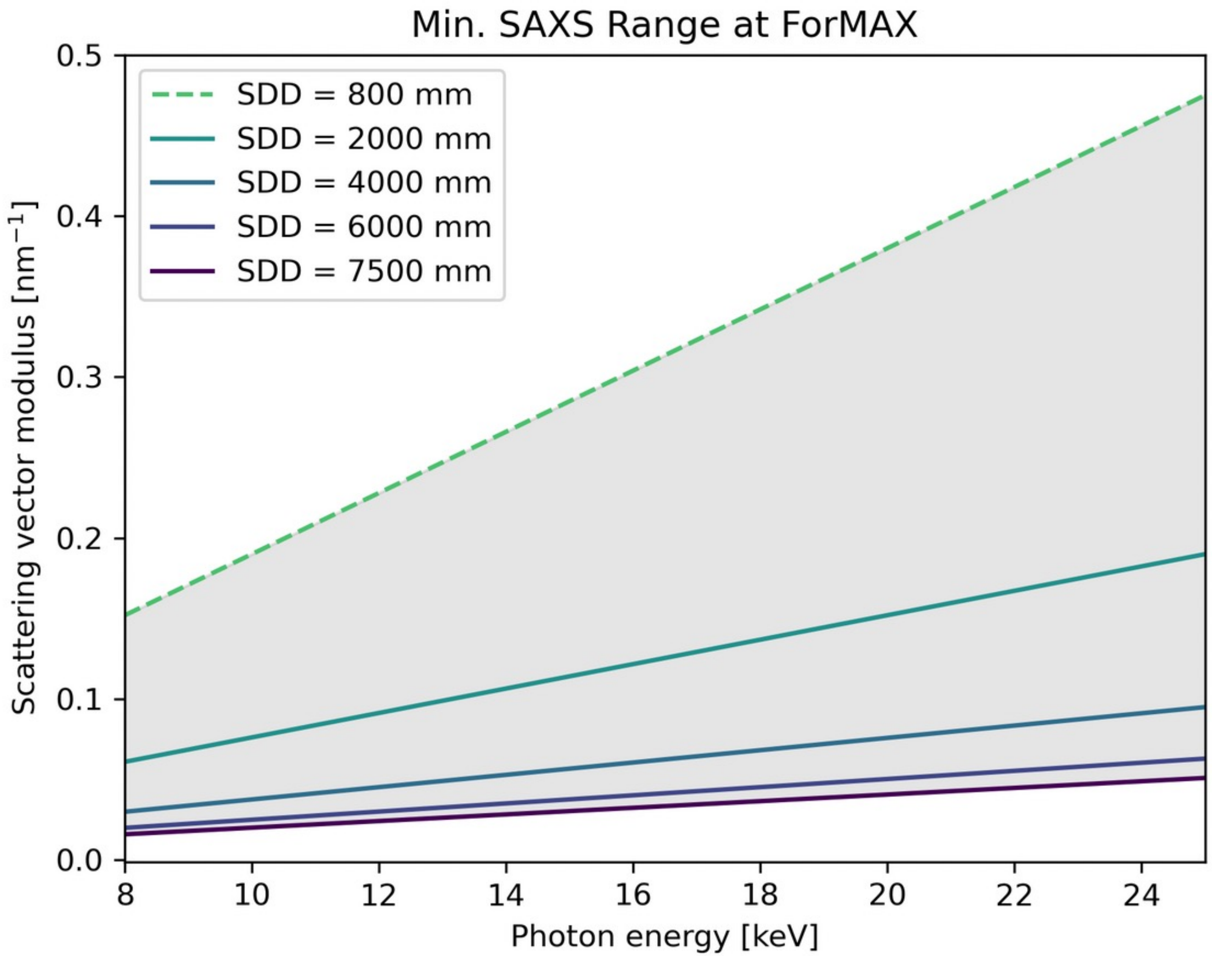
Example from proposal to ForMAX@MAXIV

C) Experimental method(s), specific requirements.

The proposed experiment plans to measure simultaneous scanning SAXS/WAXS with a beamsize of $25\mu m$ 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 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 10-40 nm^{-1} in the aim of obtaining the hydroxyapatite 002 peak (~18.2 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.



Min. SAXS Range at ForMAX
SDD = 800 mm
SDD = 2000 mm
SDD = 4000 mm
SDD = 6000 mm
SDD = 7500 mm
Scattering vector modulus [nm^{-1}]
0.0
0.1
0.2
0.3
0.4
0.5
8
10
12
14
16
18
20
22
24
Photon energy [keV]