# Small-Angle X-ray Scattering – a (mainly) practical introduction: "Where and how to measure SAXS?"

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# Outline

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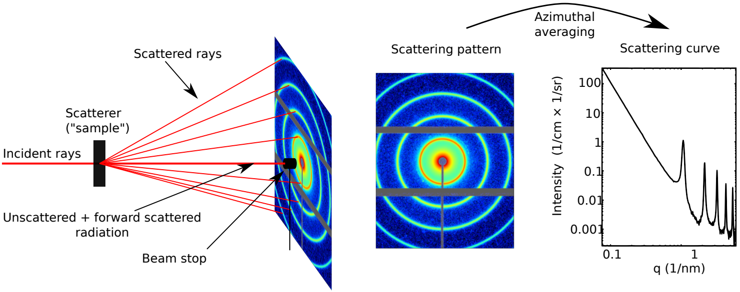
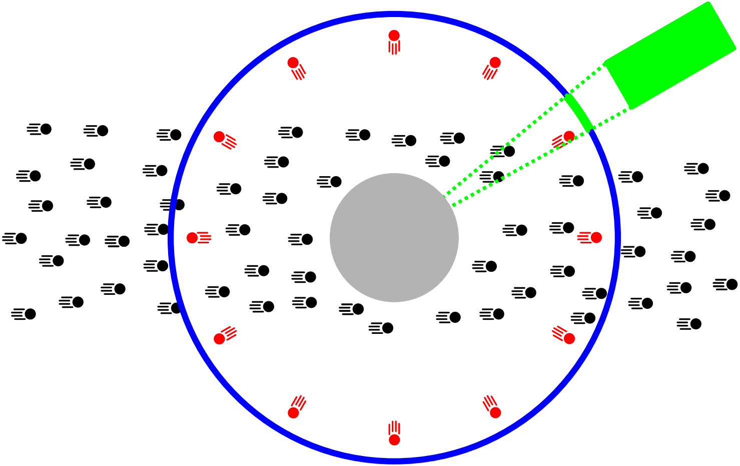
# Scattering experiments




- Scattering of X-rays on the matter
- Scattering pattern $\rightarrow$ scattering curve

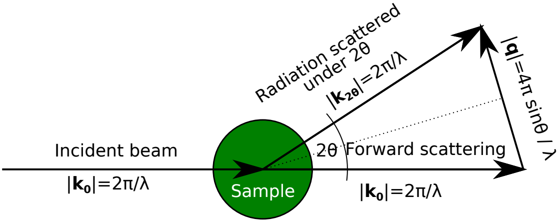
# Intensity



Relative intensity: the number of "event" detected during the measurement

Absolute intensity: differential scattering cross-section: $d\sigma/d\Omega = I_{\text{in}}/|\vec{j}_{\text{out}}|$

# Angle-dependence

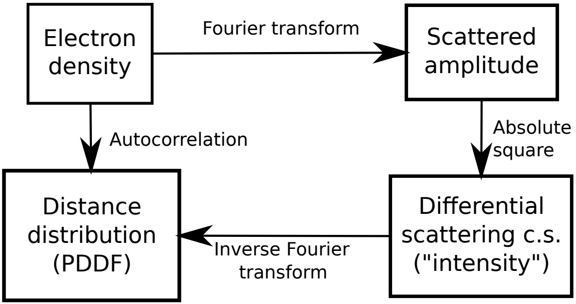



Scattering vector: $\vec{q} = \vec{k}_{2\theta} - \vec{k}_0$, $q = 4\pi \sin\theta/\lambda$

Physical meaning: The momentum acquired by the photon in the process ($h\lambda = p$)

Real-space equivalent: Periodic repeat distance via Bragg's equation: $q = 2\pi n/d$, $n \in \mathbb{N}$

# Connection between structure and scattering

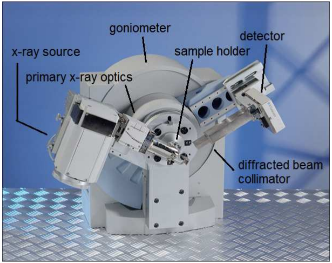



- Electron density function ($\rho(\vec{r})$)
- Fourier transform
- Absolute square $\rightarrow$ phase problem
- Back to the real space: PDDF

# Outline

# Small-angle scattering camera types

- Diffractometer
  - Point-by-point measurement in $q \rightarrow$ long measurement time
  - Well-defined angular resolution
- Line-focus (Kratky) camera
  - high intensity
  - instrumental smearing of the scattering curve
- Pinhole camera
  - low distortion
  - easily tunable
  - simple principle
- ...



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# Characteristic parameters

Final goal: measure weakly scattering samples in short times

## X-ray beam

- High flux
- Highly parallel (divergence $\ll 1$ mrad)
- Monochromatic ($\Delta\lambda/\lambda$)

## Angular resolution

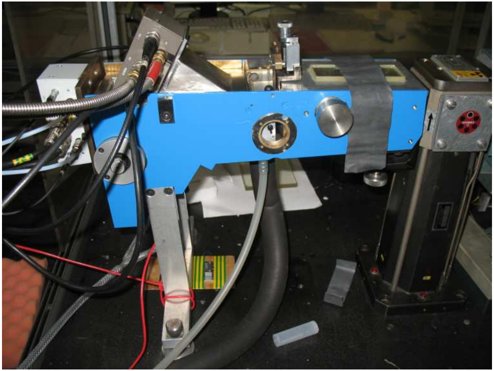
- Sample-to-detector distance
- Shape and size of the detector area
- Pixel size

## Noise

- Electronic noise from the detector
- Parasitic scattering (comes from the X-ray source, scattered, but not by the sample)
- External radiation

# Line-focus Kratky camera

- Compact, small footprint
- 1D position sensitive detector
- Fixed sample-to-detector distance
- No moving parts



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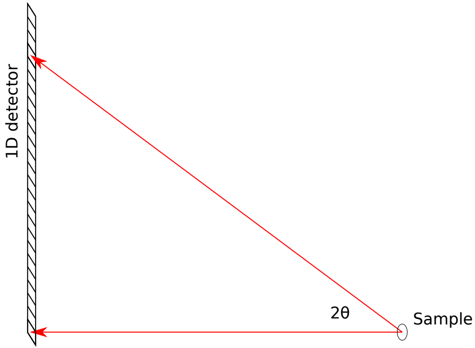

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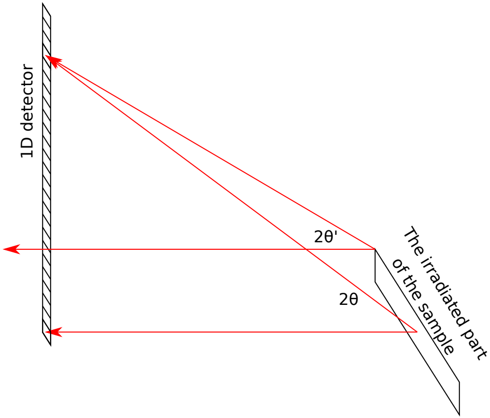

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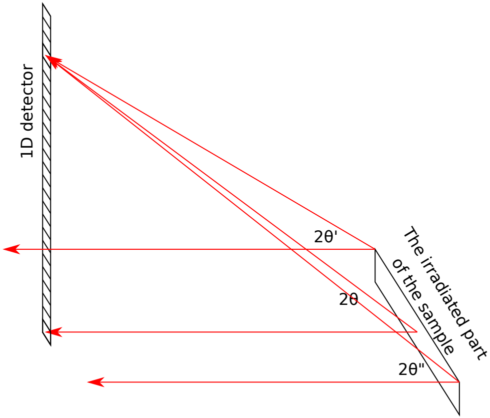

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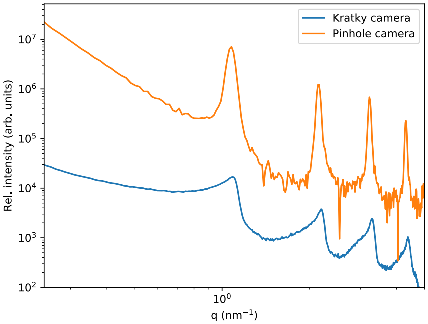
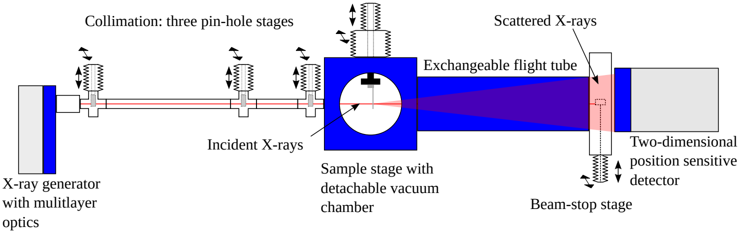


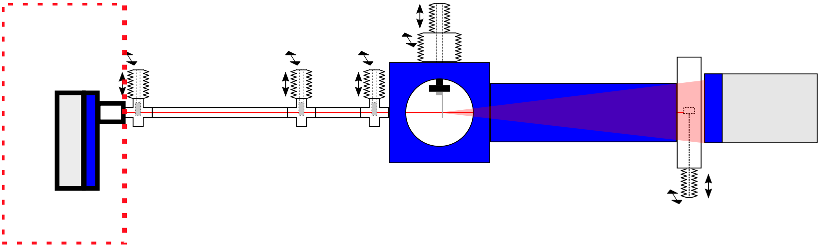
# Point focus (pinhole) camera



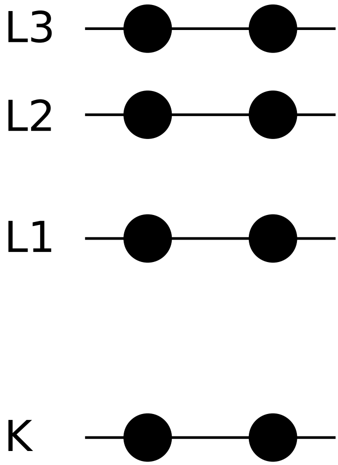

- Small beam size (<1 mm), low divergence
- Exchangeable sample-to-detector distance: tuning of the angular range
- Negligible smearing, typically no correction needed
- Easy to set up and align (compared to the Kratky block)
- Larger footprint but more possibilities
- Not (just) a “routine” instrument

# Outline

# Radiation source



# Characteristic X-radiation



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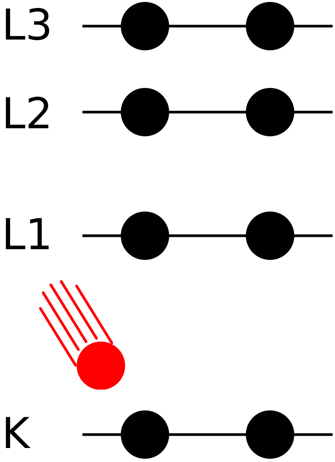

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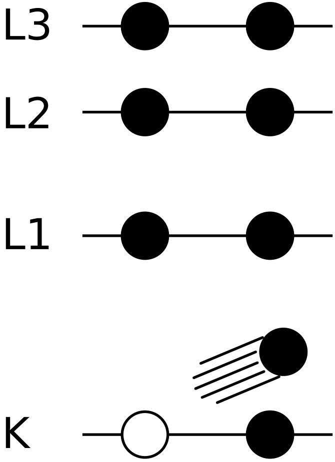

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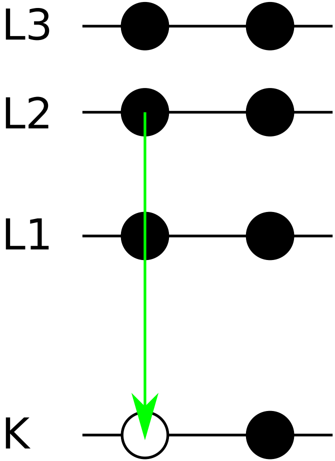

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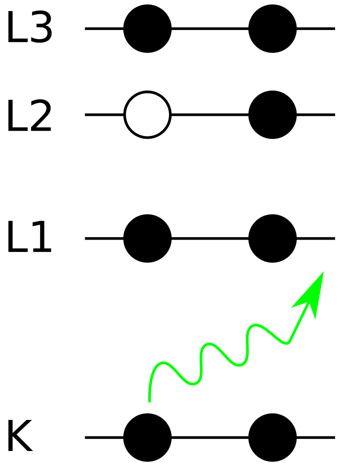

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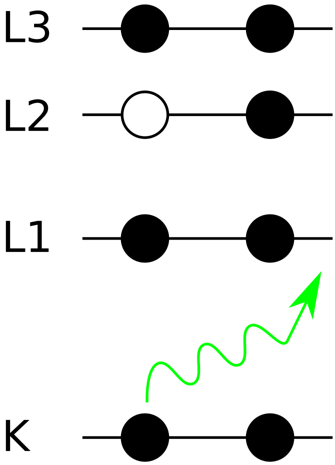

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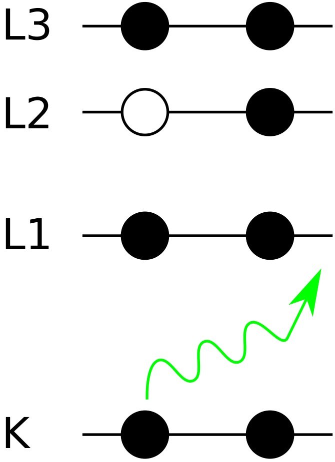

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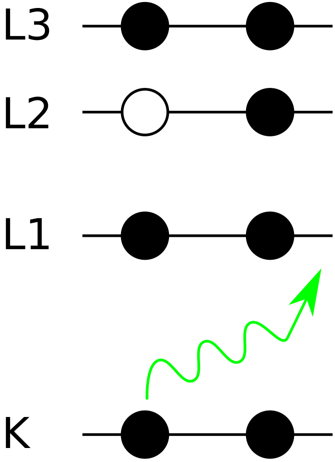
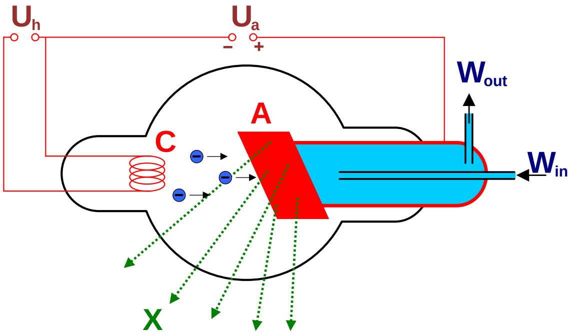
- $\Rightarrow h\nu < E_{\text{in}}$

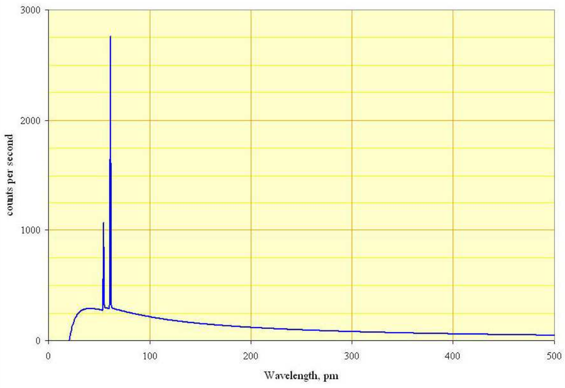


# How an X-ray tube works?




- Cathode (C): heated filament, electrons exit due to the thermionic effect
- $U_a$: accelerating voltage (40-100 kV): electrons accelerate towards the anode
- Anode (A): the incident electrons produce characteristic radiation (X)
- High heat load on the anode: cooling is needed! ($\mathrm{W_{in}}$, $\mathrm{W_{out}}$)

# Wavelength spectrum of the X-ray tube




- Peaks: characteristic radiation
- Continuous baseline: "Bremsstrahlung"

# Bremsstrahlung

- An accelerating charged particle produces electromagnetic radiation
- The total emitted power if the acceleration is parallel to the velocity:

$$P_{a\|v} = \frac{q^2 a^2 \gamma^6}{6\pi\varepsilon_0 c^3}; \qquad \vec{\beta} = \vec{v}/c; \qquad \gamma = \frac{1}{\sqrt{1-\beta^2}}$$

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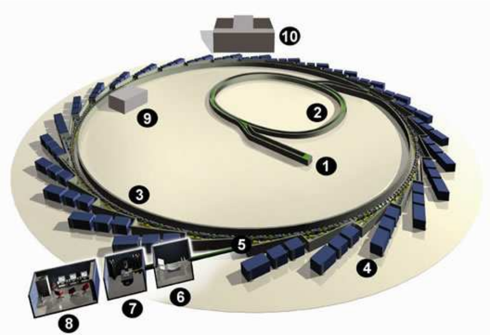
- $\Rightarrow$ orbiting charged particle emits electromagnetic radiation
- Where do we encounter orbiting charged particles?

# Synchrotron radiation

Electromagnetic radiation detected in the tangential direction of charged particles orbiting on a circular path

1. Electron gun and linear accelerator (linac)
2. Pre-accelerator ring (booster)
3. Storage ring
4. Experiment hall
5. Beamline
6. Optics hutch: mirrors, monochromators etc.
7. Experiment hutch
8. Experiment control room
9. Machine control room
10. Main building



# Storage ring

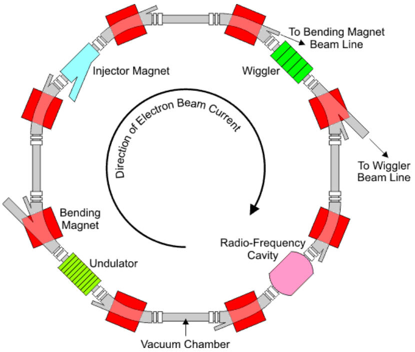
Vacuum chamber: $< 10^{-8}$ mbar

RF cavity: feeding back the emitted energy

Injector magnet: replacement of the absorbed electrons

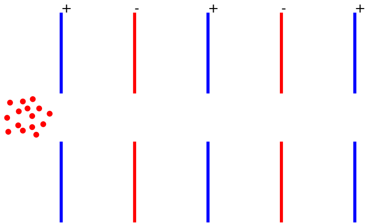
Bending magnets: Circular orbit, producing radiation

Wigglers and undulators: periodic magnets, producing high intensity radiation



# Radiation production in a synchrotron storage ring

- Charged praticles (typically electrons) orbit in bunches: *synchrotrons are pulsed sources*
- Feeding back lost energy and compacting bunches: RF cavities/resonators



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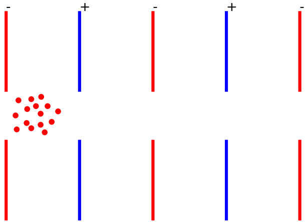

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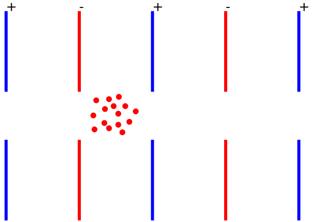

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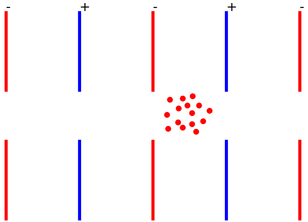

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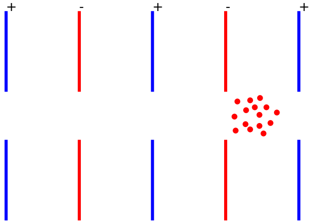

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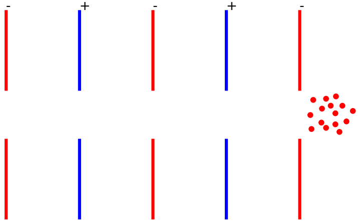

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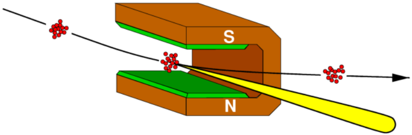

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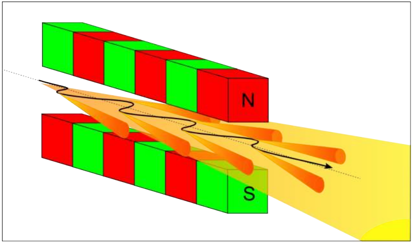

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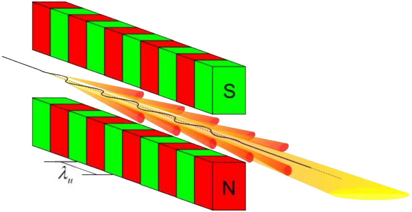

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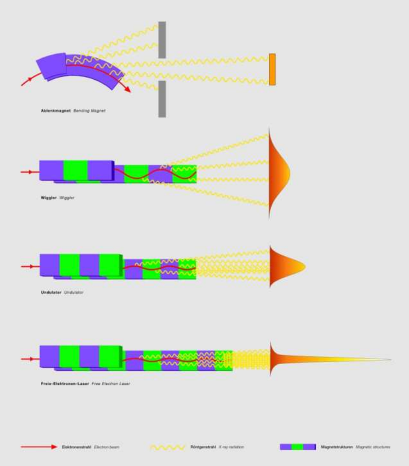

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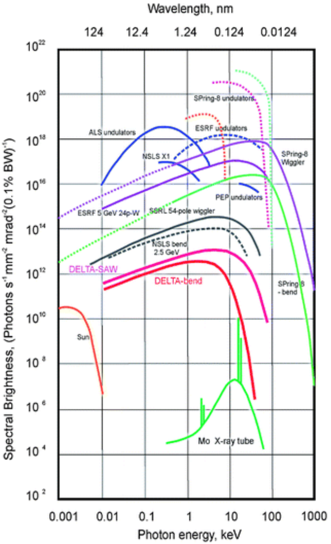
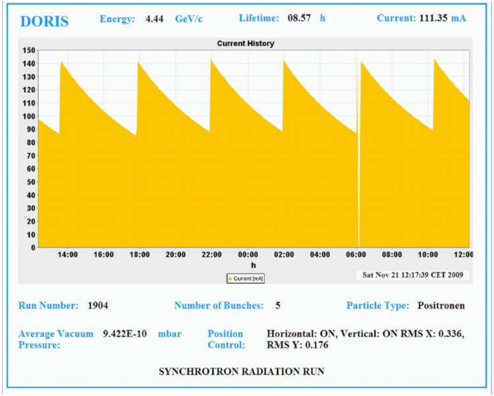


# Time structure of synchrotron radiation

- Intensity is not constant in time
- Long time scale: replacing the absorbed particles
  - Occasionally (4-8 hours): “injection”



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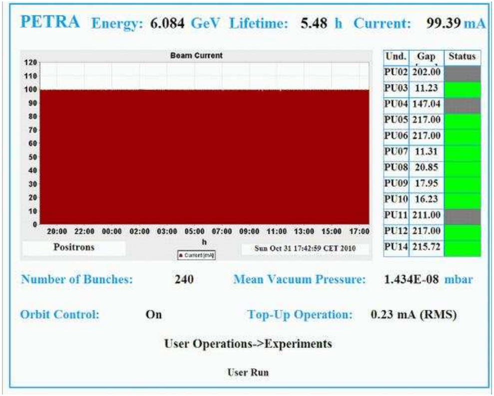

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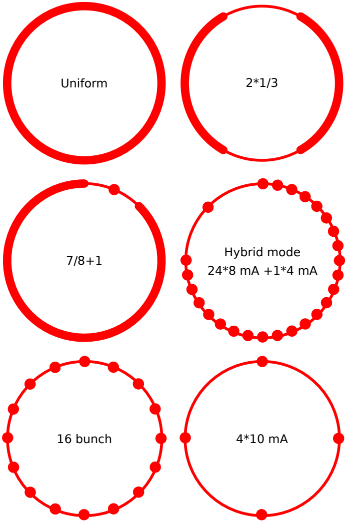
- Short time scale: dispersion of the bunches around the orbit
  - Time-resolved experiments
  - Pump-probe techniques



## Advantages of synchrotron radiation

High intensity: Short measurement times, SAXS imaging

Pulsed radiation: time-resolved experiments

Tunable wavelength: ASAXS

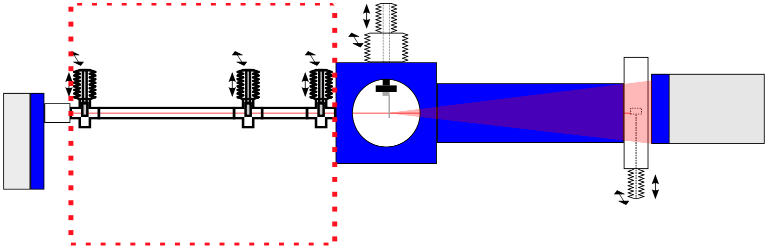
## Beamtime proposal system

1. Submit a beamtime proposal: scientific topic, relevance, why do you need a synchrotron. . .
2. Proposal gets refereed
3. Successful proposal: beamtime is scheduled
4. The experiments (1-5 days on-site)
5. Back home, evaluate the data (several GBytes)
6. "da capo al fine"

## Advantages of a laboratory SAXS instrument

- High availability (it is always there)
- Can be tuned or event rebuilt
- The same camera, just the source is different
- Chemistry lab, sample preparation is near
- Slow measurements, but "infinite" beamtime
- Preparation for synchrotron measurements
  - Better beamtime proposal
  - Preliminary characterization/screening

# Collimation

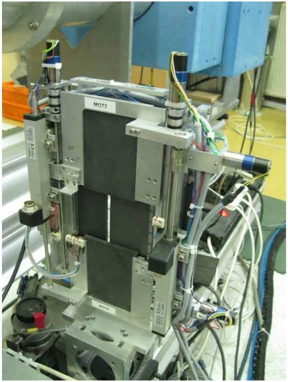


# Collimation: why do we need that?

- The scattering cross-section of X-rays is really small
  - the un-scattered intensity is more than $\times 1000$ as strong!
- The direct (not scatterd) radiation:
  - may damage the detector
  - a global read-out detector cannot detect the weaker scattered radiation
- Differentiation between scattered and non-scattered radiation:
  - Beam stop before the detector
  - Parallel beam with a small cross-section
- Beam shaping:
  - Optical elements: mirrors, capillaries, X-ray lenses
  - "Cutting" with slits

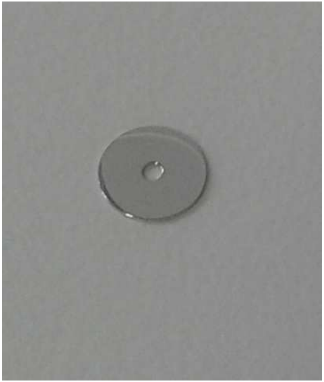
# Slits / pinholes

## Adjustable slit system



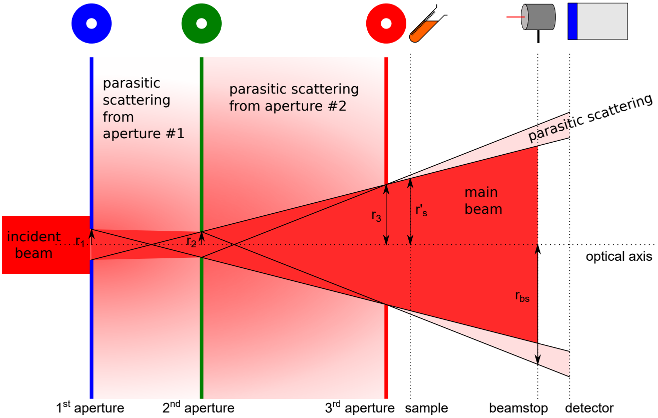
- Aperture changeable in two directions
- Rectangular beam shape

## Pinholes



- Simple / cheap
- Round beam shape

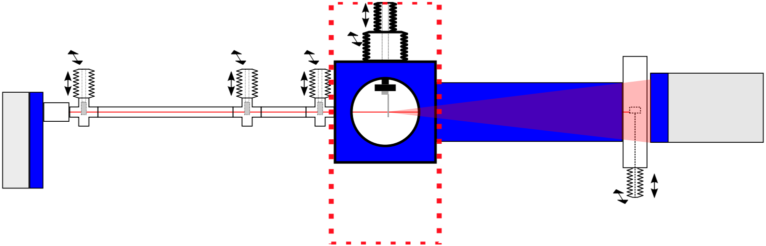
# Three aperture collimation scheme




Roles of the apertures

1. Limiting the size of the incoming beam
2. Limiting the divergence
3. Covering the parasitic scattering

# Sample environment



## Sample environment

- Air has small-angle scattering
  - In vacuum
  - Helium, hydrogen ($\Delta\rho$ small)
  - Minimizing the in-air beam path
- *In situ* measurements
  - Temperature
  - Shear
  - Magnetic field
  - Mixing

## Typical sample requirements

Self-carrying solids: homogeneous platelets, cross-section larger than the beam

Liquids: vacuum-safe sample holders (glass/quartz capillaries) $>20\ \mu$l

Powders: to be avoided (strong power-law scattering from the surfaces of powder particles)

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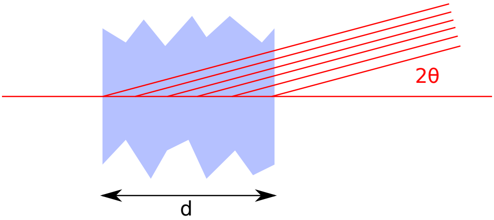
# Ideal sample thickness

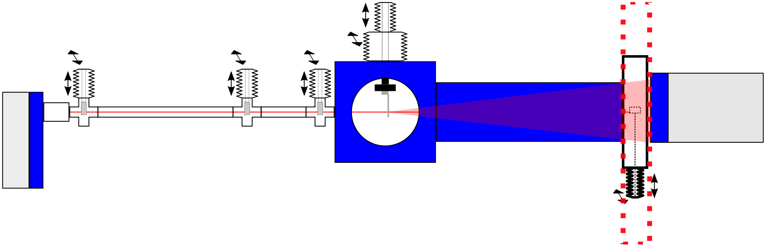



- Scattered and non-scattered rays are partially absorbed by matter
- Lambert-Beer law: $I(d) = I_0 e^{-\mu d} = I_0 T$
- Scattered intensity: $I(q,d) \propto I_0 e^{-\mu d} d$
- Maximizing the scattered intensity: $\frac{\partial I(q,d)}{\partial d} = 0$

$$
\begin{aligned}
\frac{\partial e^{-\mu d} d}{\partial d} = -e^{-\mu d}\mu d + e^{-\mu d} &= 0 \\
1 &= \mu d \\
d &= \boxed{1/\mu}
\end{aligned}
$$

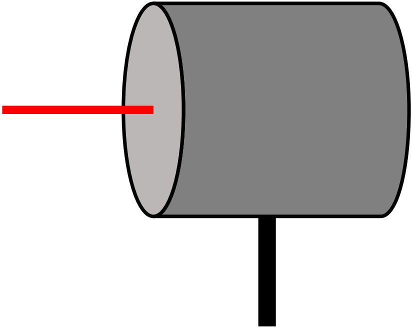
- Water: $1/\mu \approx 1$ mm with Cu K$\alpha$ radiation (8048 keV, 0.15418 nm)

# Beam stop

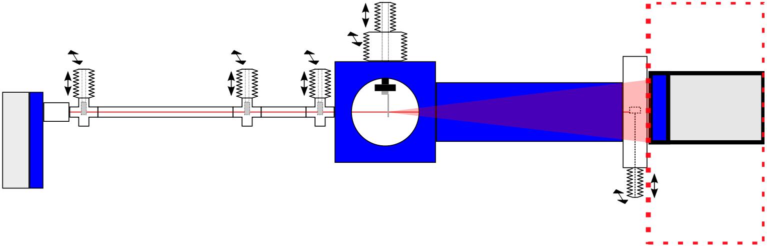


# Beam stop

- Absorbing X-rays not scattered by the sample
- Intensity: non-scattered $\gg$ scattered
- Reasons
  1. Spare the detector from high intensities
  2. Avoid the scattering of matter in the beam after the sample
- Opaque: absorbs all photons
- Semitransparent: beam spot on the detector: easy determination of the center



# Detector



# Detector

- Measuring the angle-dependence of the intensity
- Energy resolution ↔ **position resolution**
- Requirements:
  - Good position resolution (small pixel size, minimal pixel cross-talk)
  - Linearity (the detected signal is proportional to the incoming intensity)
  - High counting speed (frequency)
  - Low noise
  - No distortions
  - Large sensor shape
- Typical types

Gas-filled counters: linearity, low noise, energy selectivity, slow, global readout, large pixels, aging

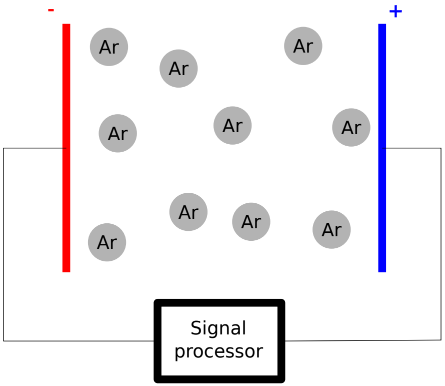
CCD detectors: fast readout, small pixel size, large electronic noise

CMOS detectors: fast readout, pratically no electronic noise, expensive

Image plate: linearity, slow readout, cumbersome handling, aging

# Operation principle of the proportional counter

1. Gas-filled counting chamber



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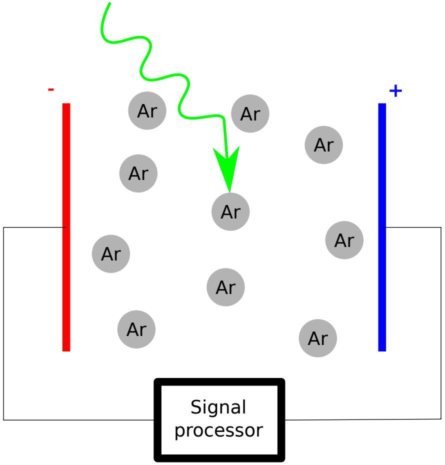
1. Gas-filled counting chamber
2. Primary ionization by the incoming X-ray photon



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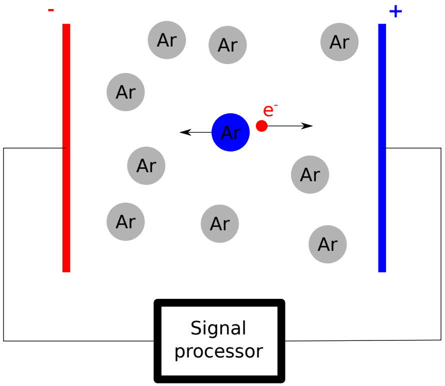

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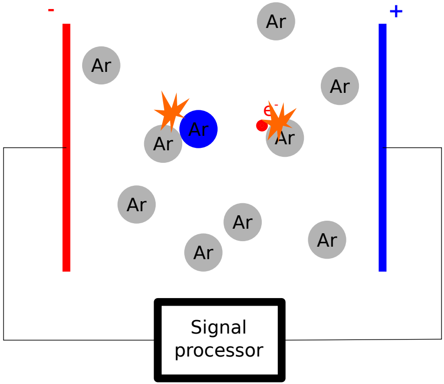
4. Secondary ionizations



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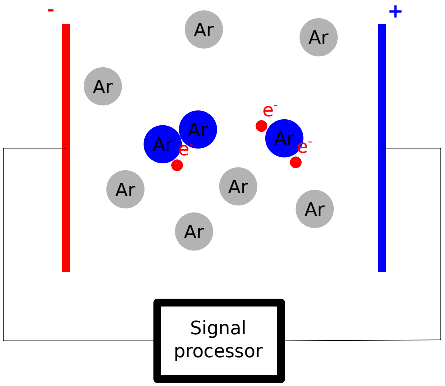

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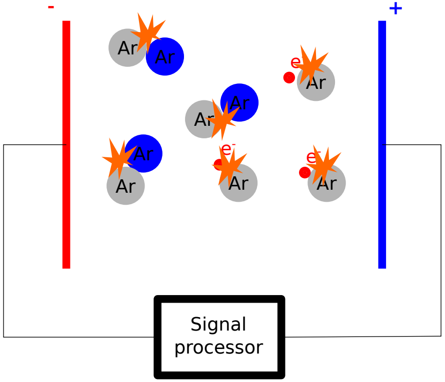

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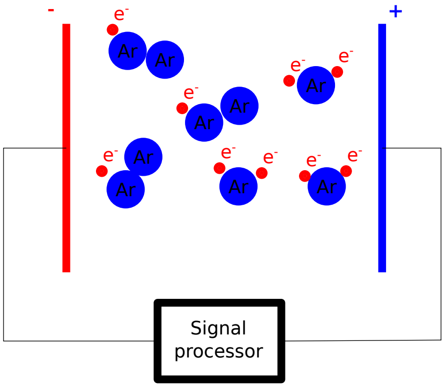

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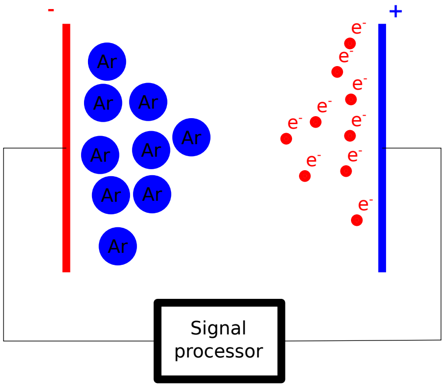
6. Ion- and electron avalanches hit the electrodes



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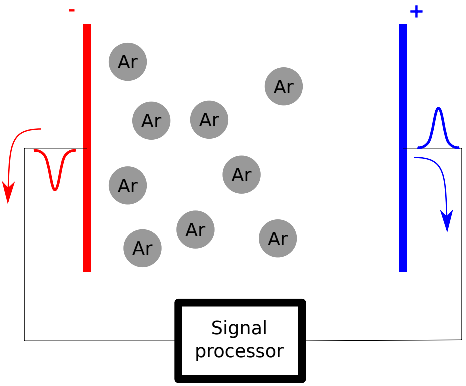
7. Recombination, pulse signals



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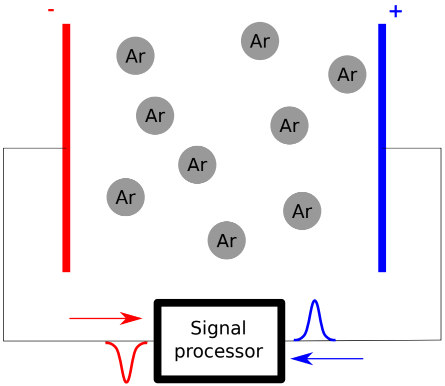

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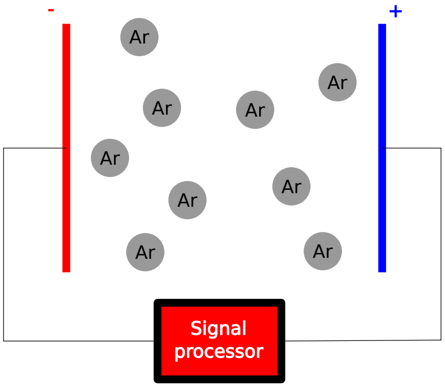
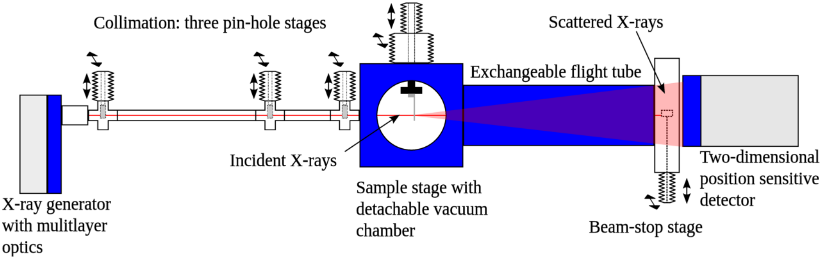
7. Recombination, pulse signals
8. Integral pulse height $\propto h\nu$
9. Detection of the pulses by the electronics

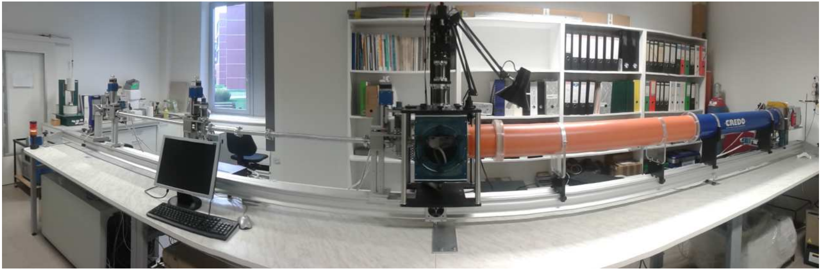


# Outline

# CREDO



Collimation: three pin-hole stages
Scattered X-rays
Exchangeable flight tube
Incident X-rays
X-ray generator with mulitlayer optics
Sample stage with detachable vacuum chamber
Two-dimensional position sensitive detector
Beam-stop stage

CREDO

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- GeniX$^{3D}$ Cu ULD X-ray generator (30 W, $\lambda = 0.154$ nm, divergence $<0.4$ mrad HW20%M)

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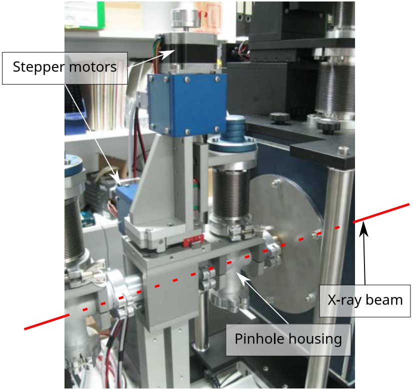

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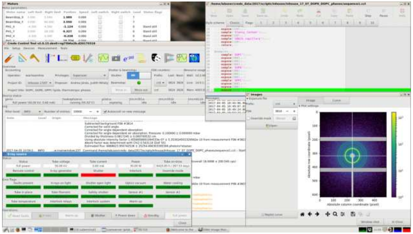
- Motorized sample stage, pinholes, beam stop
- Self-developed, automated data acquisition software
  - Instrument control
  - Carrying out the needed corrections and calibrations automatically

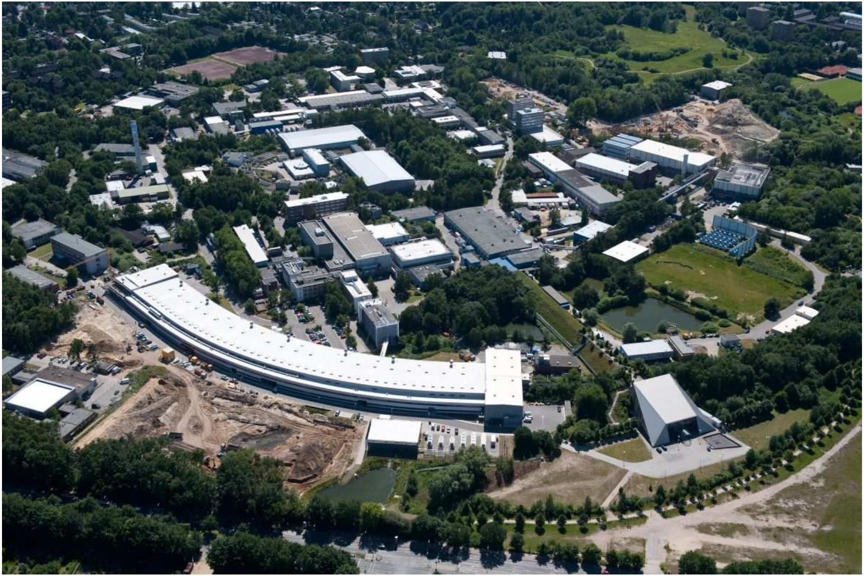


https://credo.ttk.mta.hu

# B1 („JUSIFA")

- **J**ülich's **U**ser-dedicated **S**cattering **F**acility
- Deutsches Elektronensynchrotron (DESY), Hamburg
- DORIS III storage ring
- dedicated instrument for ASAXS (anomalous SAXS)
- Gabriel MWPC, Pilatus-300k, Pilatus-1M detectors
- *1989 - †2012

# B1 („JUSIFA")



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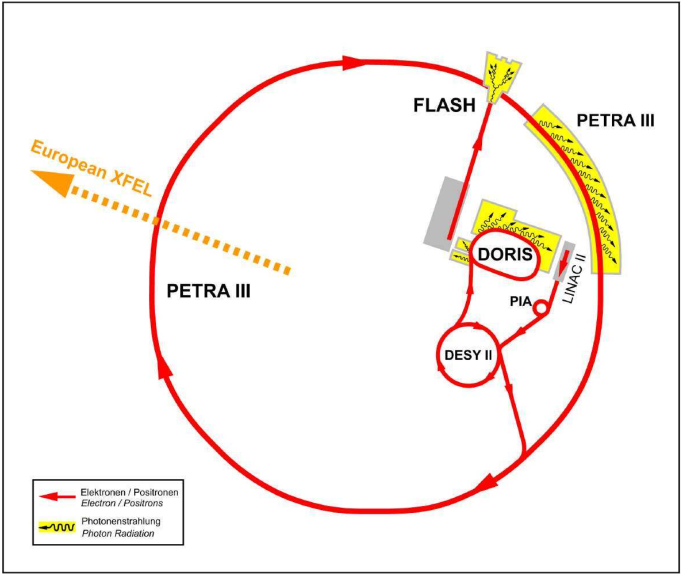
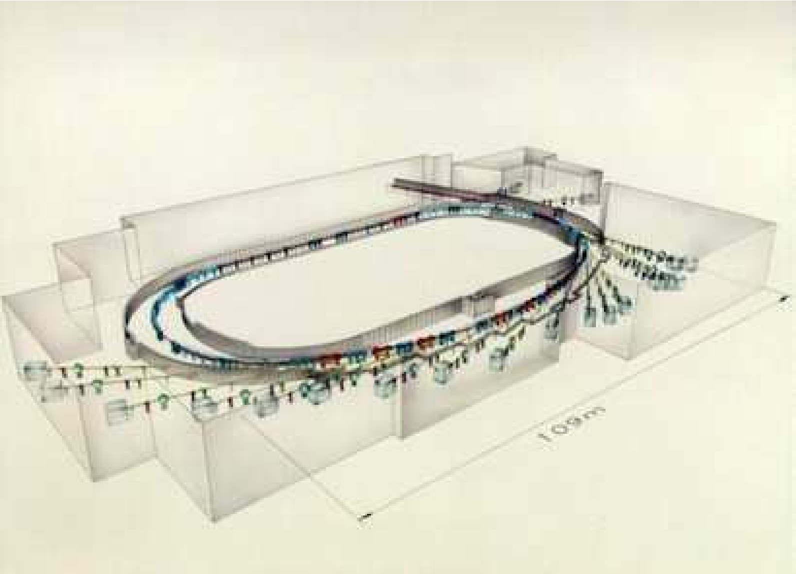


FLASH
PETRA III
European XFEL
DORIS
LINAC II
PIA
PETRA III
DESY II
Elektronen / Positronen
Electron / Positrons
Photonenstrahlung
Photon Radiation

# B1 („JUSIFA")



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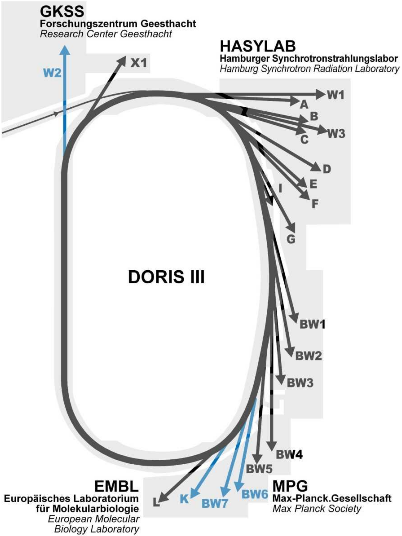

GKSS
Forschungszentrum Geesthacht
Research Center Geesthacht
HASYLAB
Hamburger Synchrotronstrahlungslabor
Hamburg Synchrotron Radiation Laboratory
W2
X1
W1
A
B
W3
C
D
E
I
F
G
DORIS III
BW1
BW2
BW3
BW4
BW5
EMBL
Europäisches Laboratorium
für Molekularbiologie
European Molecular
Biology Laboratory
L
K
BW7
BW6
MPG
Max-Planck.Gesellschaft
Max Planck Society

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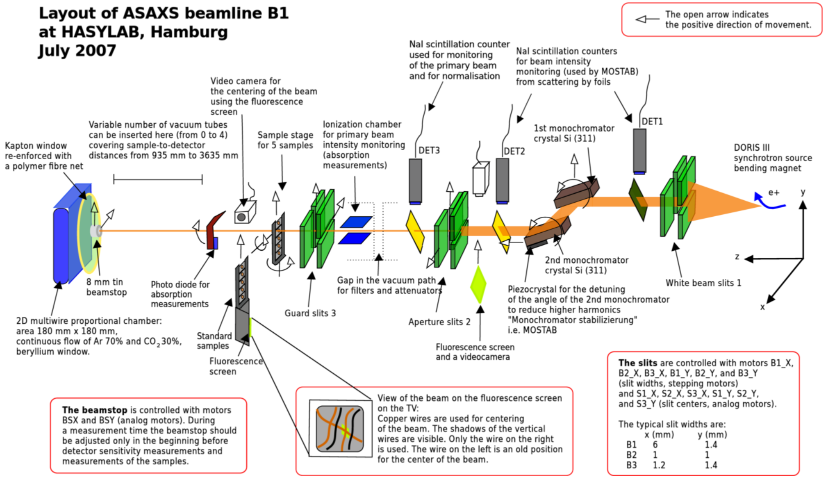
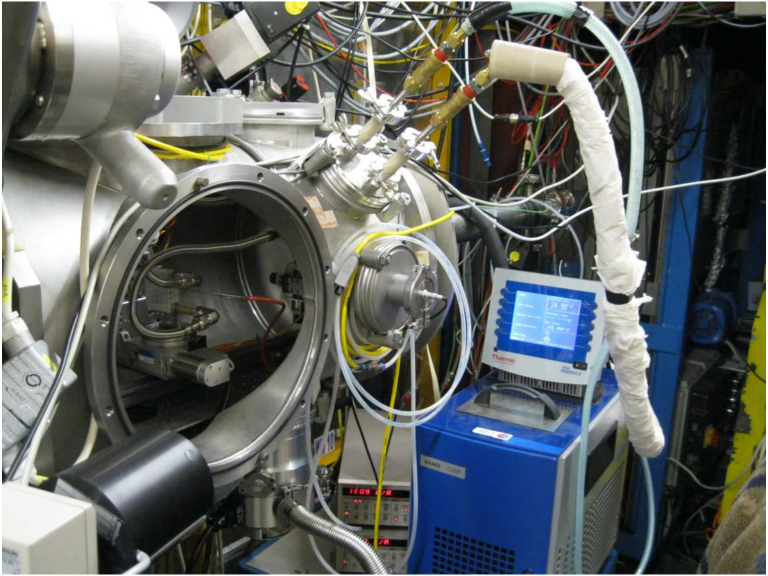


Layout of ASAXS beamline B1
at HASYLAB, Hamburg
July 2007
The open arrow indicates
the positive direction of movement.
NaI scintillation counter
used for monitoring
of the primary beam
and for normalisation
NaI scintillation counters
for beam intensity
monitoring (used by MOSTAB)
from scattering by foils
Video camera for
the centering of the beam
using the fluorescence
screen
Variable number of vacuum tubes
can be inserted here (from 0 to 4)
covering sample-to-detector
distances from 935 mm to 3635 mm
Sample stage
for 5 samples
Ionization chamber
for primary beam
intensity monitoring
(absorption
measurements)
DET3
DET2
DET1
1st monochromator
crystal Si (311)
DORIS III
synchrotron source
bending magnet
Kapton window
re-enforced with
a polymer fibre net
e+
y
z
x
8 mm tin
beamstop
Photo diode for
absorption
measurements
Gap in the vacuum path
for filters and attenuators
2nd monochromator
crystal Si (311)
White beam slits 1
Guard slits 3
Aperture slits 2
Piezocrystal for the detuning
of the angle of the 2nd monochromator
to reduce higher harmonics
"Monochromator stabilizierung"
i.e. MOSTAB
2D multiwire proportional chamber:
area 180 mm x 180 mm,
continuous flow of Ar 70% and CO2 30%,
beryllium window.
Standard
samples
Fluorescence
screen
Fluorescence screen
and a videocamera
The beamstop is controlled with motors
BSX and BSY (analog motors). During
a measurement time the beamstop should
be adjusted only in the beginning before
detector sensitivity measurements and
measurements of the samples.
View of the beam on the fluorescence screen
on the TV:
Copper wires are used for centering
of the beam. The shadows of the vertical
wires are visible. Only the wire on the right
is used. The wire on the left is an old position
for the center of the beam.
The slits are controlled with motors B1_X,
B2_X, B3_X, B1_Y, B2_Y, and B3_Y
(slit widths, stepping motors)
and S1_X, S2_X, S3_X, S1_Y, S2_Y,
and S3_Y (slit centers, analog motors).
The typical slit widths are:
x (mm) y (mm)
B1 6 1.4
B2 1 1
B3 1.2 1.4

# B1 („JUSIFA")



# B1 („JUSIFA")

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# B1 („JUSIFA")

# Outline

# Procedure of a “typical” SAXS experiment

1. Turn on the instrument, warm up the X-ray tube (45 minutes)
2. Optimize the geometry (30-45 mins)
   - Sample-to-detector size, beam stop size $\Rightarrow$ smallest attainable $q$
   - Select pinhole sizes and spacings: no parasitic scattering, highest intensity at the sample
3. Sample preparation, capillary filling (1/2-2 hours)
   - $\approx$ 1 mm borosilicate glass capillaries
   - Sealing: 2-component epoxy resin / melting
4. Preparation measurements: search for sample motor positions, measure transmissions (30-45 mins)
5. Automatic measurement sequence (several hours / overnight)
   - 5.1 Blank measurements (dark current, empty beam)
   - 5.2 Reference samples ($q$, $d\sigma/d\Omega$)
   - 5.3 Samples
   - 5.4 Repeat...

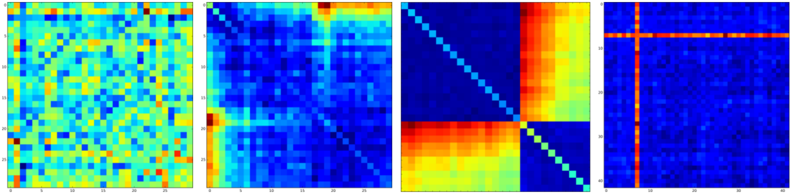
# Distribution of the measurement time

It is useful to measure many short exposures instead of one long

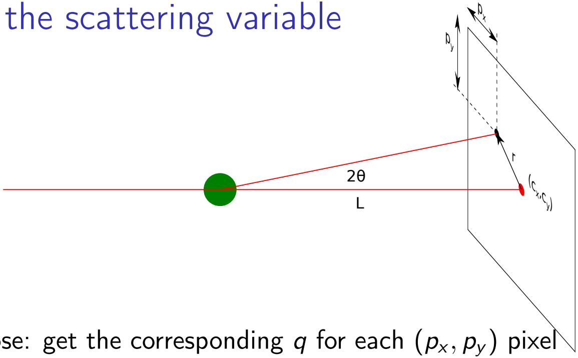
- Time resolution (TRSAXS)
- Frequent re-measuring of references
- Assessment of the stability of the samples and the instrument
- Excluding affected exposures with statistical tests:
  - Difference of the $j$-th and $j'$-th scattering curve:

$$\Delta_{jj'} = \begin{cases} \sum_k \left(I_j(q_k) - I_{j'}(q_k)\right)^2 & \text{if} \quad j \neq j' \\ \langle \Delta_{jl} \rangle_{l \neq j} & \text{if} \quad j = j' \end{cases}$$

  - Replacing the diagonal items with row averages: $\Delta_{jj} \to$ how much does the $j$-th curve differ from all the others



# Calibrating the scattering variable




- Purpose: get the corresponding $q$ for each $(p_x, p_y)$ pixel
- If the direct beam would hit the detector at $(c_x, c_y)$, the pixel size is $h$ and $L$ is the sample-to-detector distance:

$$2\theta = \tan^{-1}\frac{r}{L}; \quad r = h\sqrt{(p_x - c_x)^2 + (p_y - c_y)^2}$$

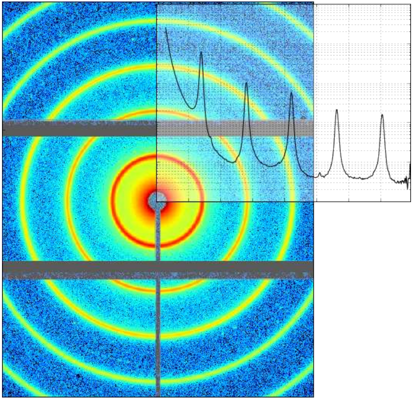
$$\boxed{q = \frac{4\pi}{\lambda}\sin\left(\frac{1}{2}\tan^{-1}\left(\frac{h\sqrt{(p_x - c_x)^2 + (p_y - c_y)^2}}{L}\right)\right)}$$

# Calibrating of the scattering variable

- The sample-to-detector distance is tricky to measure directly
- Calibration samples: silver-stearate, silver-behenate, SBA15, $LaB_6$, tripalmitine...
  - high intensity, sharp peaks in the scattering
  - stable: vs. time and vs. temperature
  - *peak positions are known*
- Principle of finding the sample-to-detector distance:
  - Peak positions are known ($q_i$)
  - Measured peak positions in *pixel* units ($p_i$)
  - Fitting of the function

$$q = \frac{4\pi}{\lambda}\sin\left(\frac{1}{2}\underbrace{\tan^{-1}(ph/L)}_{2\theta}\right) \rightarrow$$

determine $L$

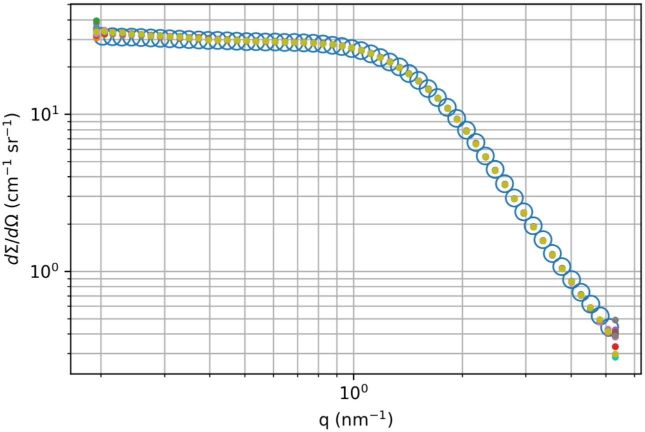


# Calibration of the scattering intensity

- Purpose: scale the count rates to differential scattering cross-section ($\mathrm{cm}^{-1}\ \mathrm{sr}^{-1}$)
- Independent from instrumental parameters:
  - Sample thickness
  - Beam shape and intensity
  - Quantum efficiency[1] of the detector
  - Measurement geometry
- Many parameters cannot be measured
- Reference sample:
  - Strong scattering
  - "Flat" scattering curve (not sensitive to miscalibrations in $q$)
  - Intensity is known in absolute units
    - Measured with other methods (e.g. glassy carbon, lupolen)
    - Known from theory (e.g. water, nanoparticle suspension)
- Measure the reference sample and your samples under the same conditions $\rightarrow$ the same intensity scaling factor applies

[1]The probability of the detection of an incoming photon

# Intensity calibration with glassy carbon




If the curves are appropriately corrected, the scaling factor is the inverse of the beam flux!

# Outline

# Summary

- Types of SAXS instruments
  - Line focus compact Kratky camera
  - Pinhole camera
- Operation principle of the X-ray generator
  - Characteristic radiation and bremsstrahlung
- Synchrotrons
  - Accelerators, storage ring
  - Insertion devices for beam production
  - Time structure
- Main parts of SAXS instruments
  - Source, detector, sample stage, collimation . . .
- Data collection
  - corrections, calibrations
  - data collection strategies (many a little makes a mickle. . . )

Next lecture: Interpretation of SAXS results with actual examples

# Thank you for your attention!