Synchrotron SAXS Instrumentation

Ivan Rajkovic

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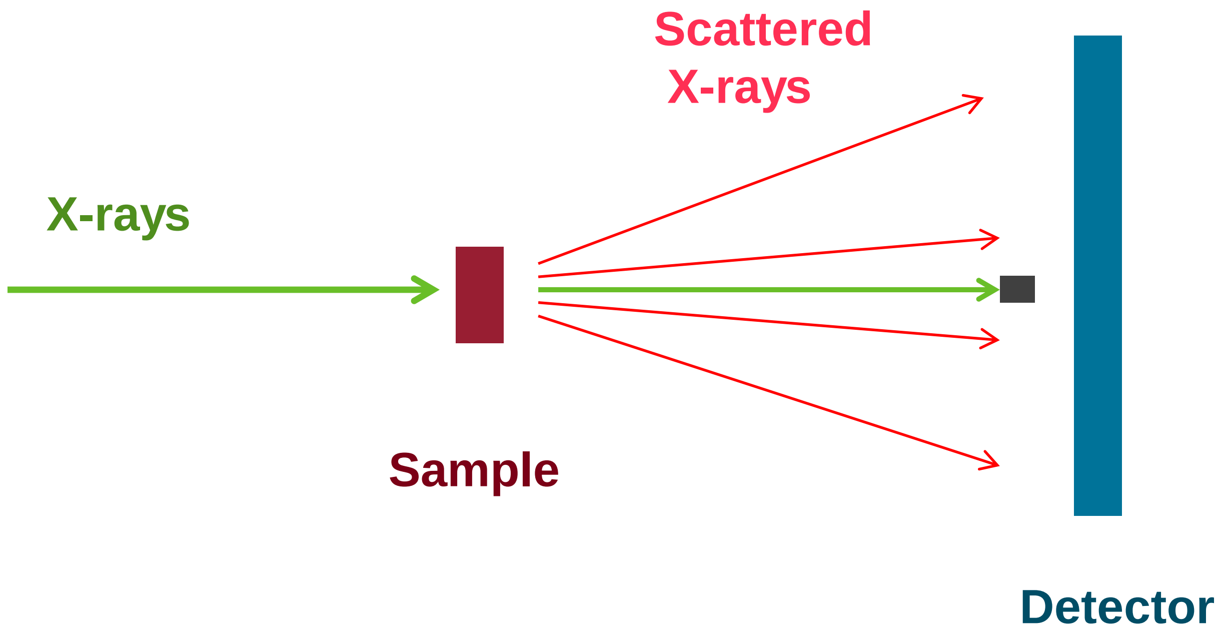
U.S. DEPARTMENT OF ENERGY
Office of Science

SLAC NATIONAL ACCELERATOR LABORATORY

What do we need?



X-ray Sources

SLAC

Requirements for SAXS:

- Small beam divergence/convergence
- Small beam (source) size
- Clean beam intensity profile
- monochromatic beam (wide bandpass okay)
- high beam intensity desirable
- ***high positional/angular stability***

- X-ray tube (focused)

- Rotating anode (focused)

- Synchrotron radiation (dipole/ID)
  - 1st-2nd generation SR dipole/wiggler
  - 3rd generation SR dipole/wiggler/undulator (small source size)

Synchrotron Radiation

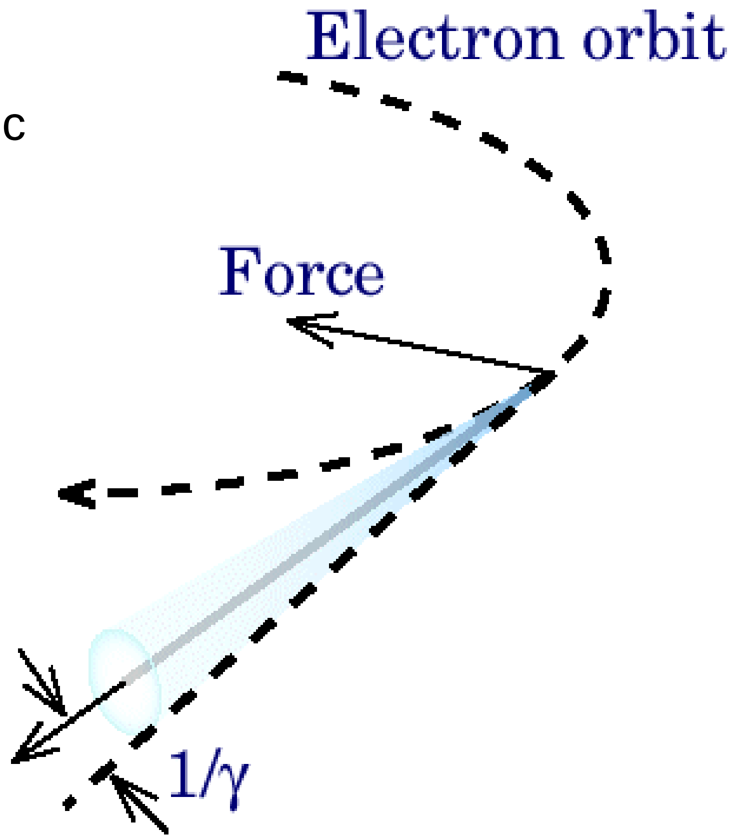
- an accelerated charge emits electromagnetic radiation

- the charge is kept on a circular orbit using magnetic dipole fields

- at relativistic speeds ($v/c \approx 1$) the radiation is collimated in a forward cone

- for SPEAR3:

  - e^--energy = 3GeV
  - v/c = 0.9999999
  - 270 bunches
  - revolution time 781ns
  - bunch length 20ps rms (6mm)



- additional magnetic fields involved (quadrupoles, sextupoles) for beam conditioning

Bending Magnet

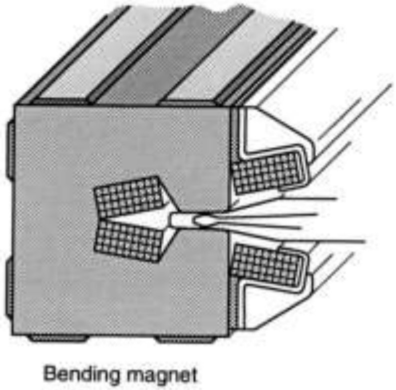
- synchrotron ring is strictly circular, but has straight sections and bening sections

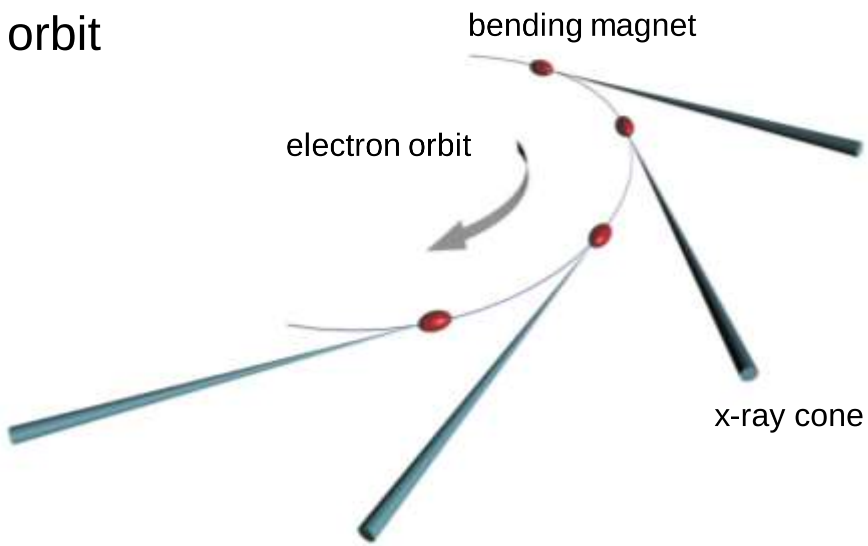
- at each bend x-rays will be emitted that can be used for a beamline

- continous spectrum of emitted x-rays is up to some critical energy (proportional to the electron energy and magnetic field)

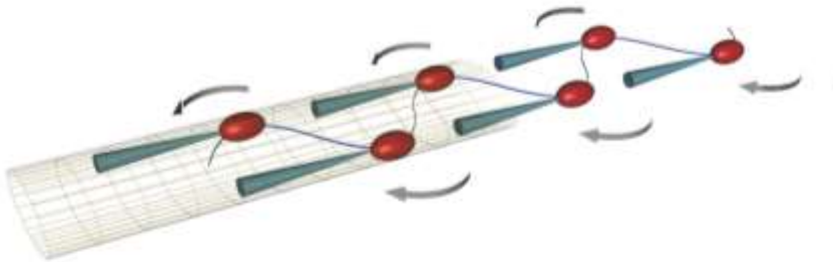
- radiation is polarized in the plane of the orbit

- at SSRL: beamlines 1, 2, 8 and 14





Insertion Devices: Wiggler & Undulator



$$K = \gamma\theta$$

$\gamma = E / m_0 c$

$\theta = angle\ in\ each\ pole$

Wiggler : K >> 1

- incoherent superposition
- intensity proportional to N (# poles)
- continous spectrum
- higher flux, small divergence

Undulator : K << 1

- coherent interference (high intensity, N^2)
- quasi monochromatic spectrum
- fundamental + higher harmonics
- small source size, low divergence

BL 4: 20 poles, 2.0T

SAXS instrumentation: wish list

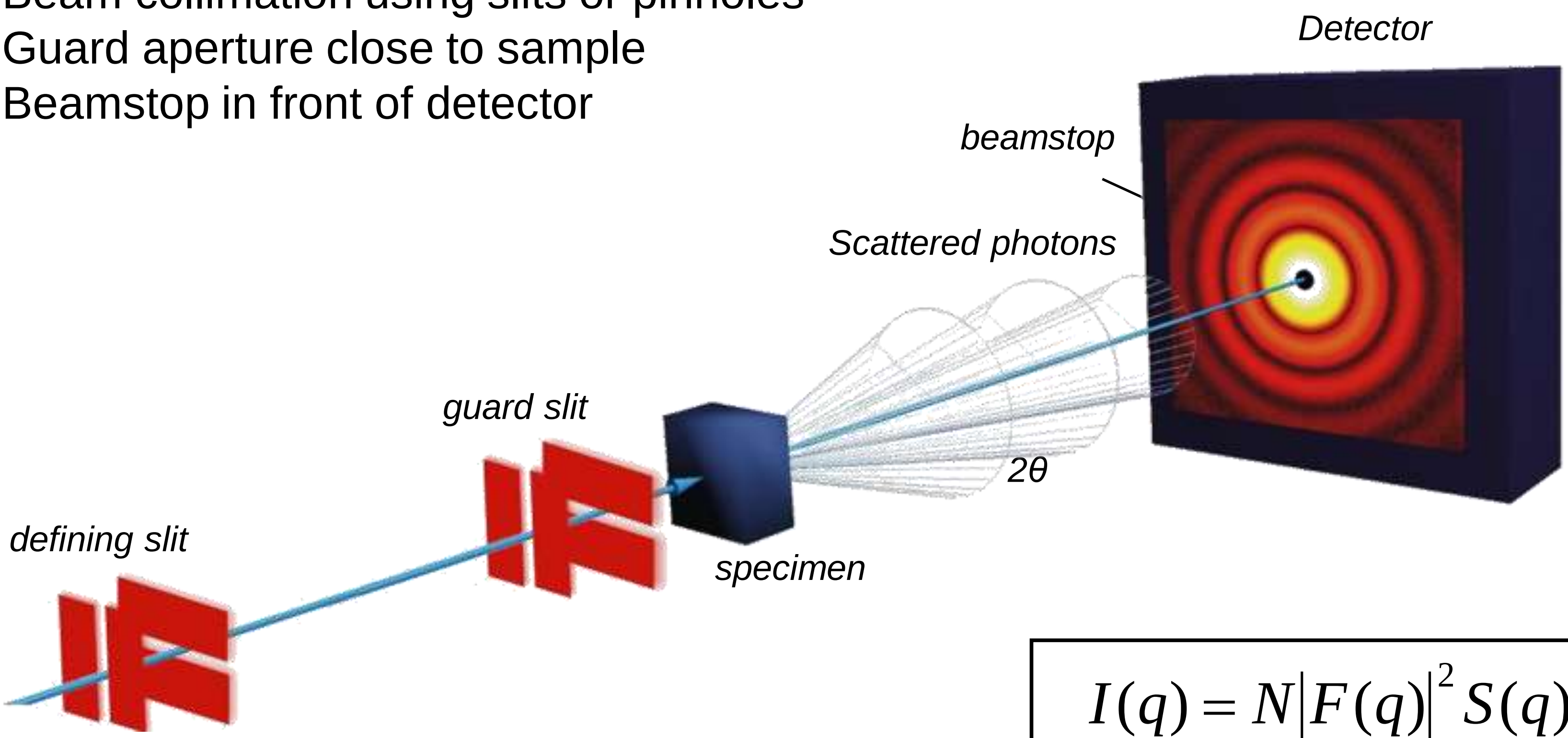
- High beam brightness (source/focus)
- Low background
- Sensitive detectors (photon counting)
- **Wide Q range coverage**

  Qmax~0.6Å^{-1}, occasionally ≥ 1.0 Å^{-1}

  Increasing demand for Qmin< 0.01 Å^{-1}
- **User friendly (hardware/software automation)**
- **Efficient data collection**
- **Reliable operation and good maintainability**
- *Good sample economy*
- *Versatility (time-resolved studies, size-exclusion etc.)*
- *Variety of sample holders*

Pinhole SAXS Setup

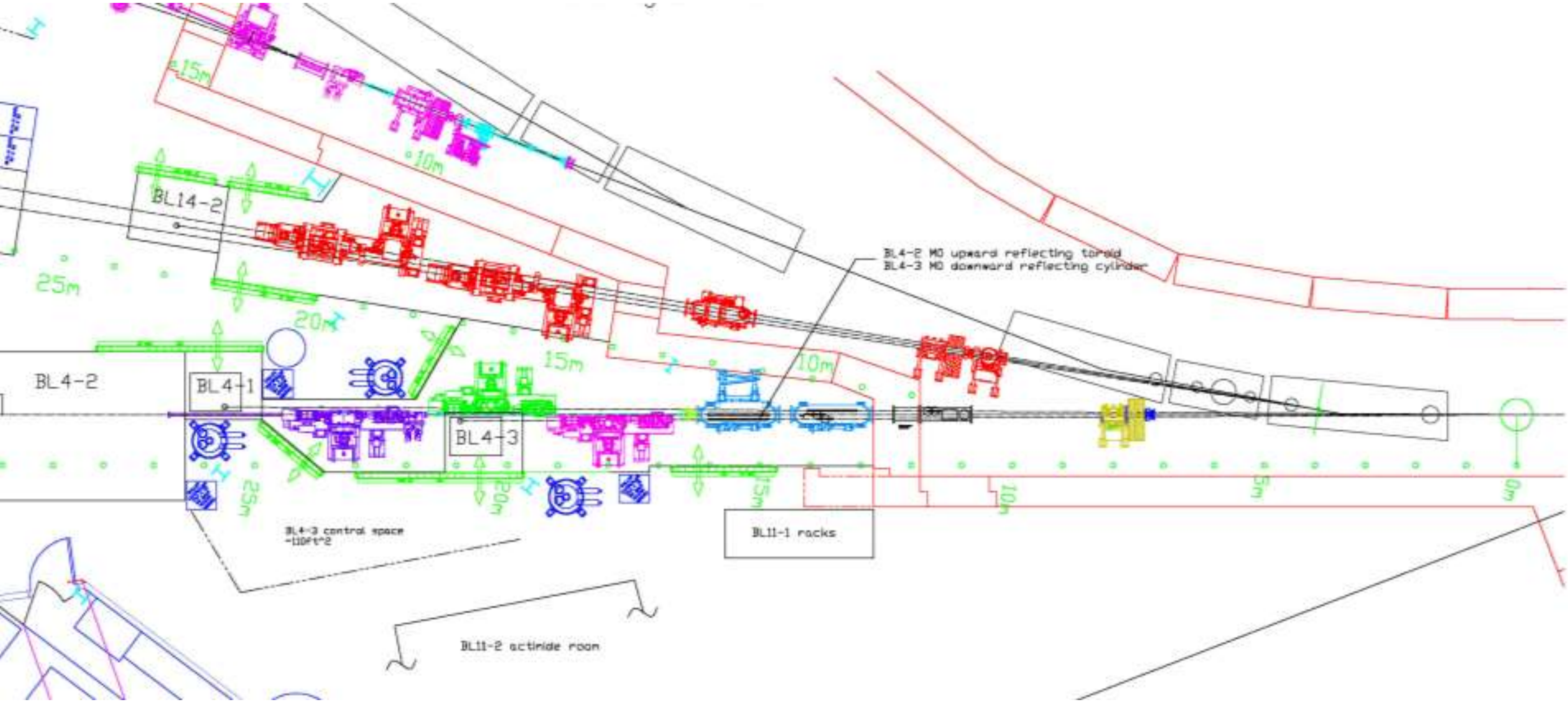
- Beam collimation using slits or pinholes
- Guard aperture close to sample
- Beamstop in front of detector



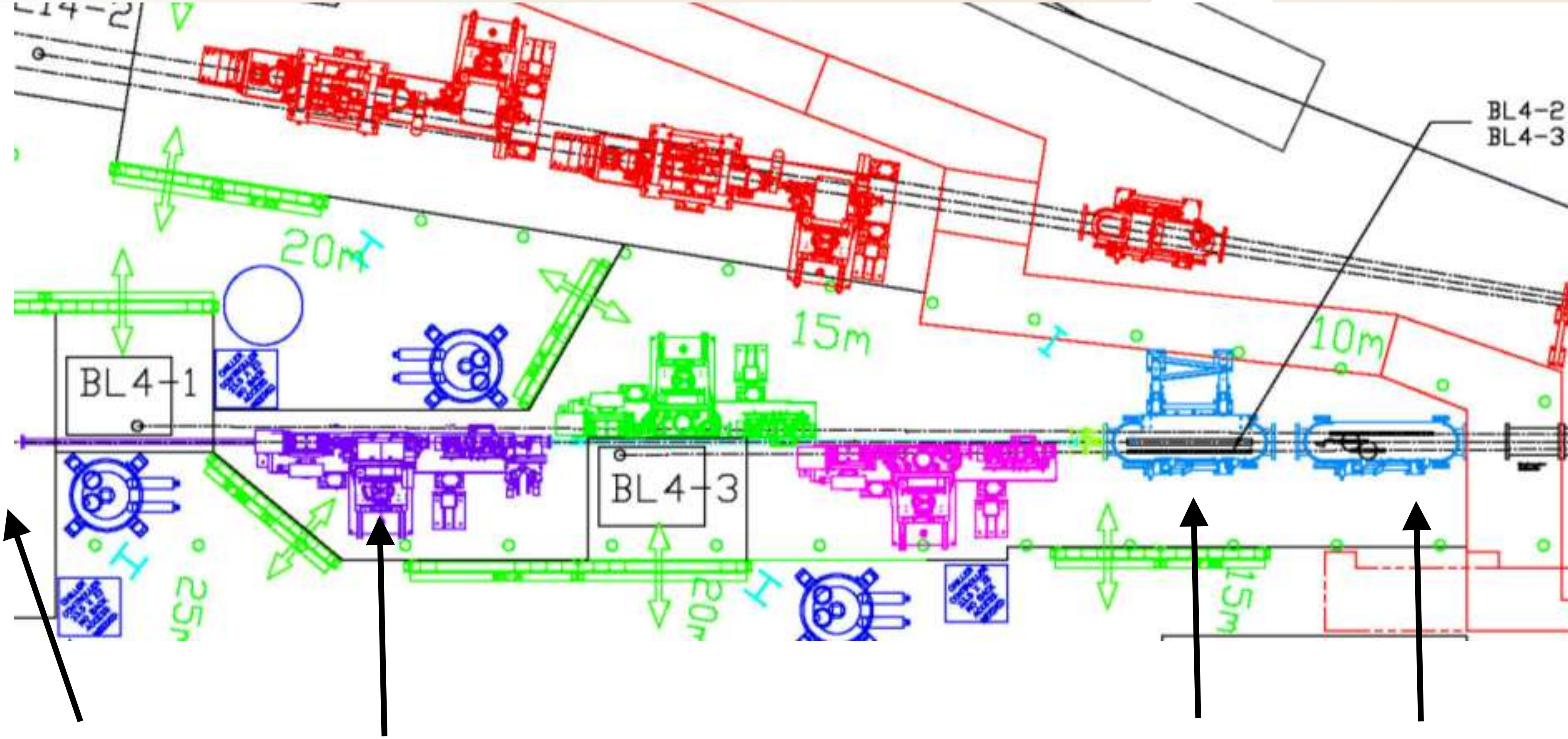
$$I(q) = N\left|F(q)\right|^2 S(q)$$

$$q = \frac{4\pi \sin\theta}{\lambda}$$

BL 4-2 Layout: Overview



BL 4-2 Layout: Overview



Exp. Hutch

Mono
23.2m

Mirror
15.4m

MPS
14m

X-ray Mirror

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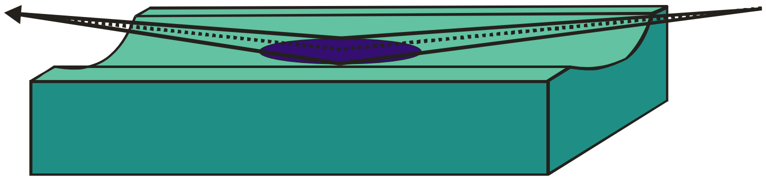


x-ray refractive index: $n = 1 - \delta$

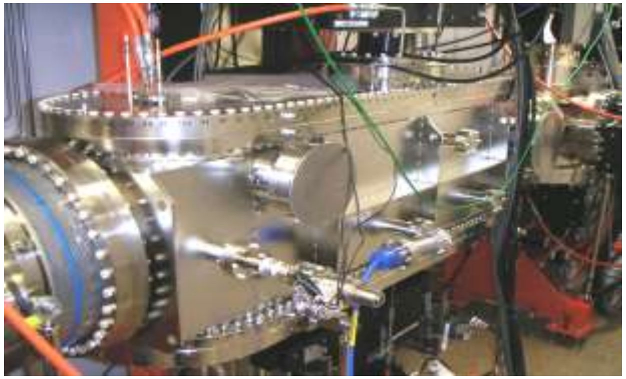
<u>Function:</u>

- focussing
- power filter
- harmonic filter

➡ *grazing angle, total reflection*

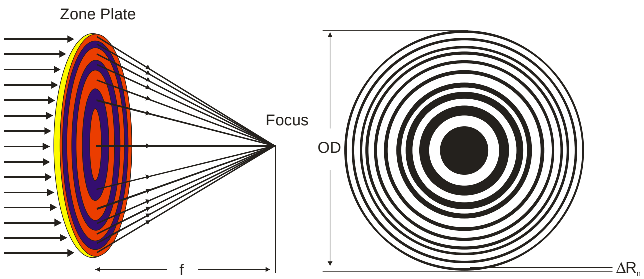
<u>Mirror at Bl4-2:</u>

- water cooled torroidal mirror (at z=15.4m)
- 1:1 focus the beam in horz. and vert.
- pitch 3.6 mrad
- typical 0.2 x 1.0 mm^2 (v x h) FWHM
- acceptance max: 1.25mrad (typ: 0.5mrad)
- cut-off energy 17keV (Rh on Si)

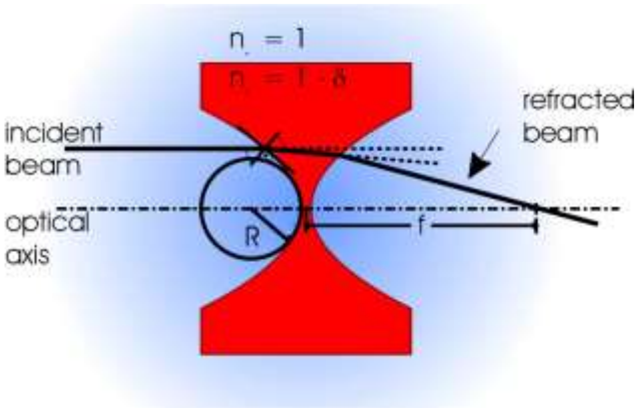


mirror tank in the optics hutch

Other focusing devices for x-ray

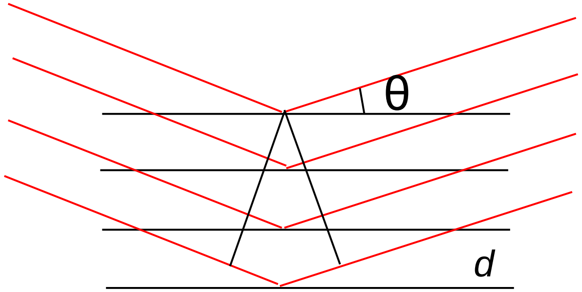


Frenel zone plates



refractive Be lenses

Monochromator



Bragg equation

$$2d \sin\theta = n\lambda$$

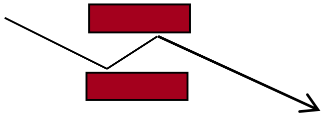
<u>Function:</u>

- wavelenth selection

- selection of specific wavelength depending on angle
- higher harmonics also pass through the monochromator

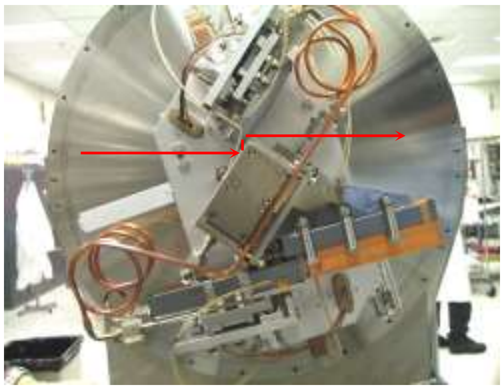
commonly used monochromators

- single reflection or multiple reflection (channel cuts)
- Si crystal for high energy resolution
- multilayer for high flux (broader bandwidth)



Monochromator @ BL4-2

Si(111) option:

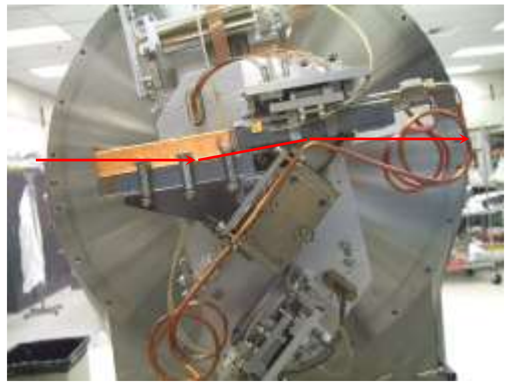


$$\frac{\Delta E}{E} \approx 10^{-4}$$

flux ≈ 2-3 x 10^{12} ph/s

@ 500mA, (0.2 x 1.0)mm^2

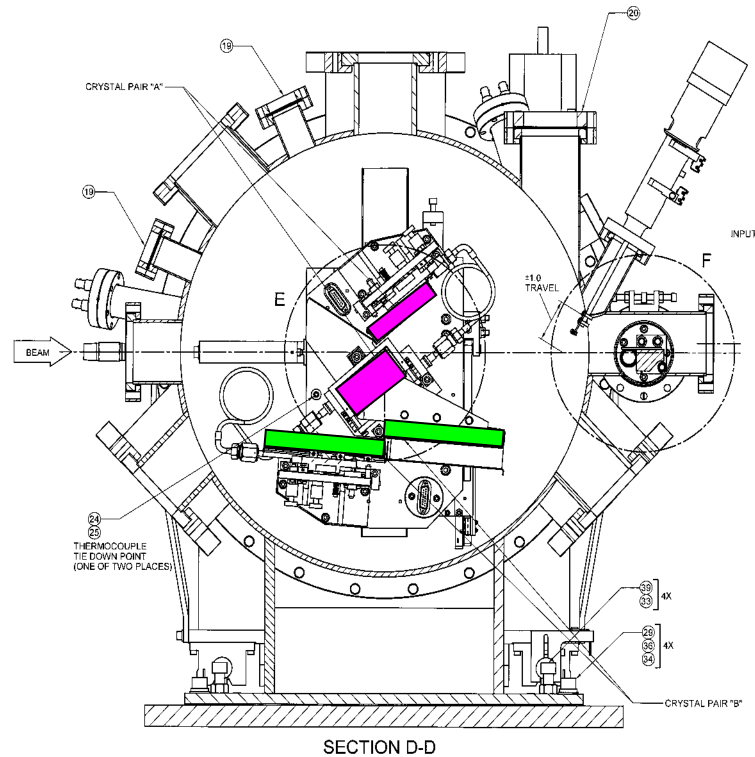
Mo/B_4C option:



$$\frac{\Delta E}{E} \approx 2\times 10^{-2}$$

flux ≈ 10^{14} ph/s

@ 500mA, (0.2 x 1.0)mm^2



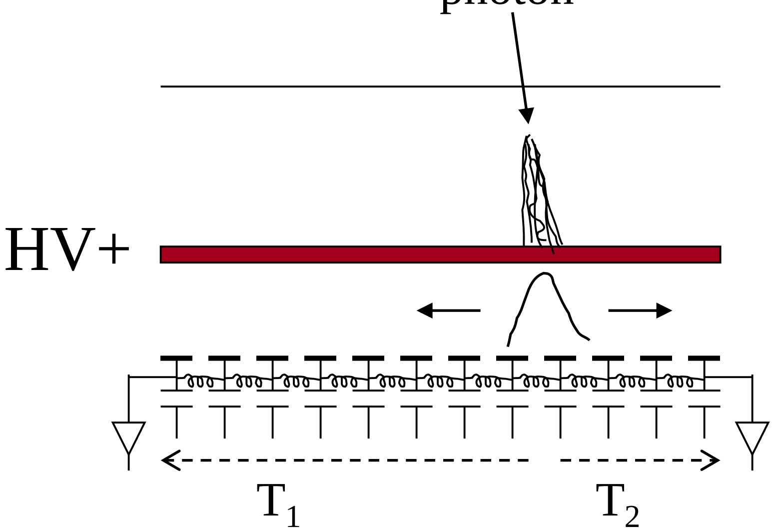
liquid N_2 cooling

user-selectable between:

- Si(111) for high energy resolution
- Mo/B4C multilayer for high flux

Commonly used detectors

Gas chamber detectors

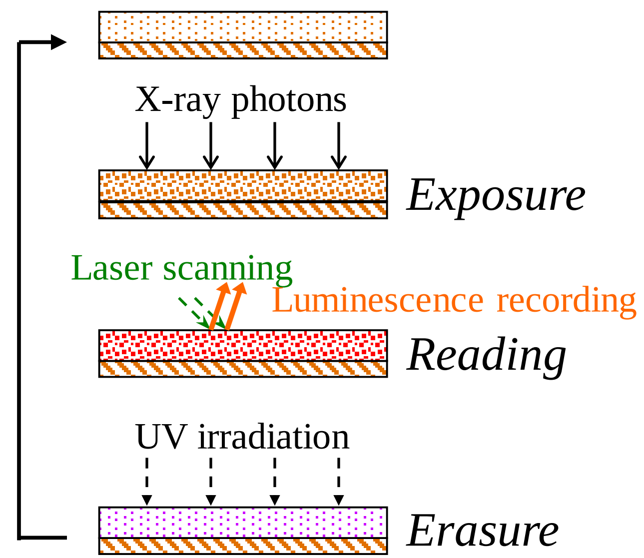


Photon counting $dT = T_1 - T_2$

Photon events encoded in time-domain

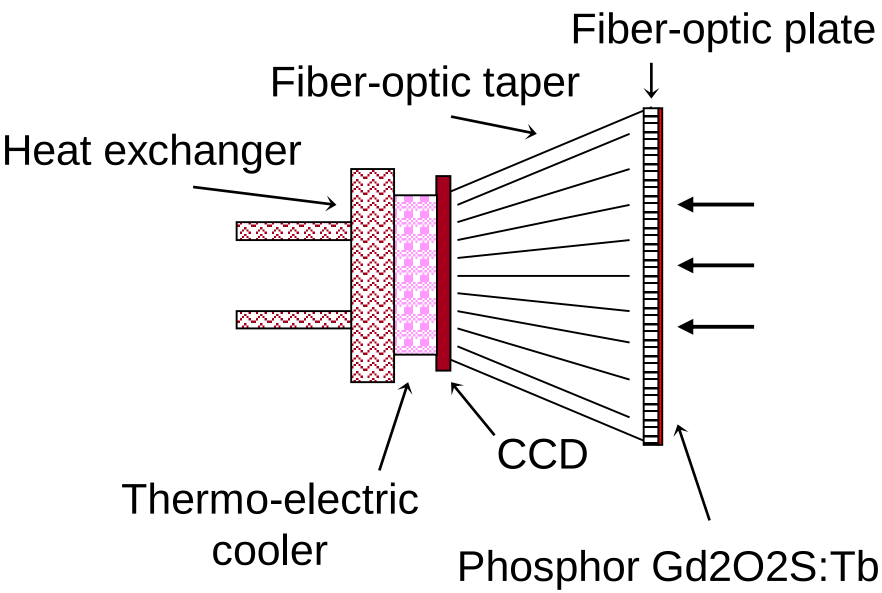
Space charge effects

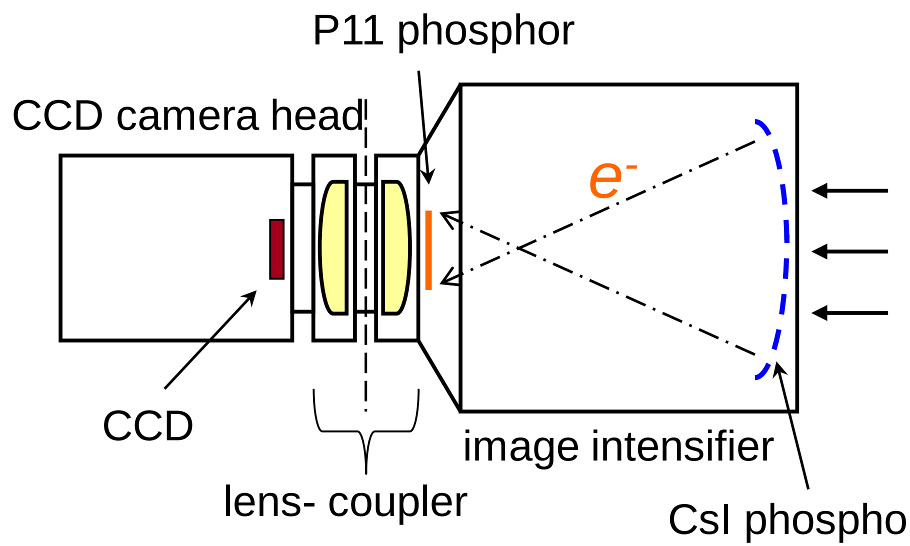
- Phosphor imager (Image Plate)



- *Large area*
- *Good sensitivity & linearity*
- *Slow readout (minutes)*
- *Intensity decay*

Opto-electronic detectors





Fiberoptic CCD

Moderately large area

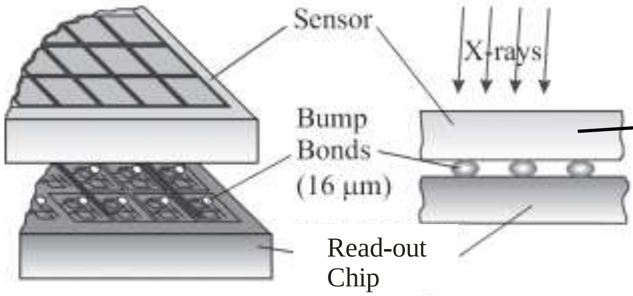
Small distortion & non-unformity

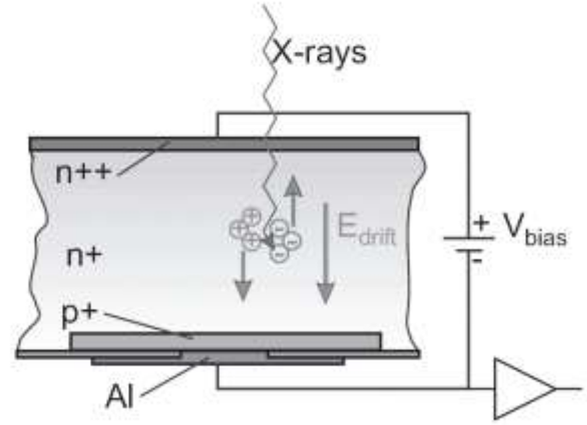
Wide dynamic range

- Image Intensified CCD
- *Moderately large area*
- *High sensitivity & stability*
- *Available fast readout CCD's*

Pixel Array Detectors

Pilatus = **Pi**xel **A**ppara**tu**s for the **S**LS





- photon counting = zero noise
- 20 bit dynamic range
- 200 Hz framing rate
- count rate per pixel > 2×10^6 ph/s

Detectors @ BL 4-2

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Rayonix MX 225-HE

- HE = **H**igh photon detection **E**fficiency
- 9 back-illuminated 1kx1k CCD chips
- fiber optic taper 2.85:1.0
- New low-noise readout electronics
- 16 bit
- active area 225x225 mm^2

Pilatus 300k

- photon counting = zero noise
- 20 bit dynamic range
- 200 Hz framing rate
- active area 84x106 mm^2
- count rate per pixel > 2x10^6 ph/s

SAXS Instrument

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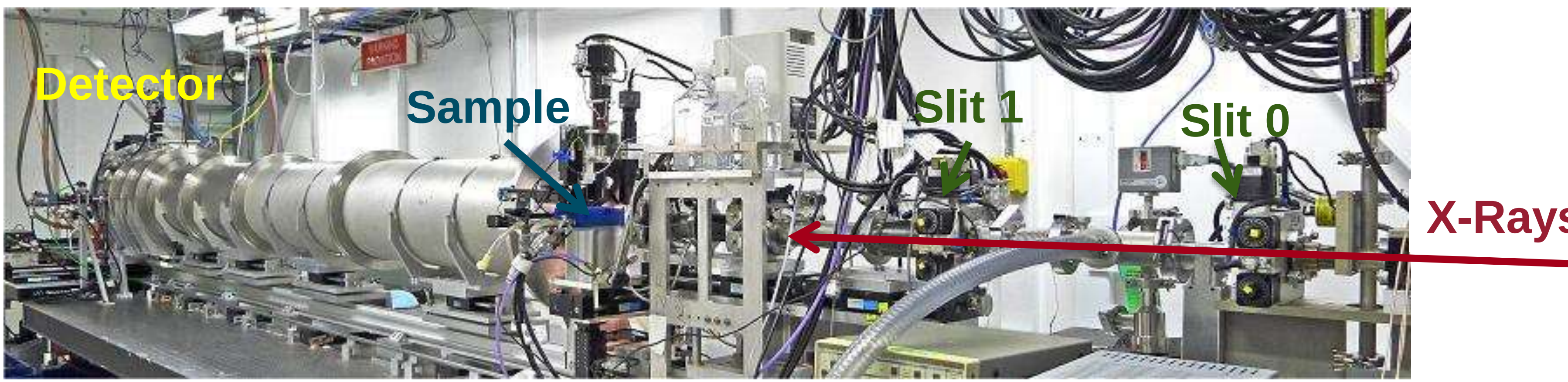
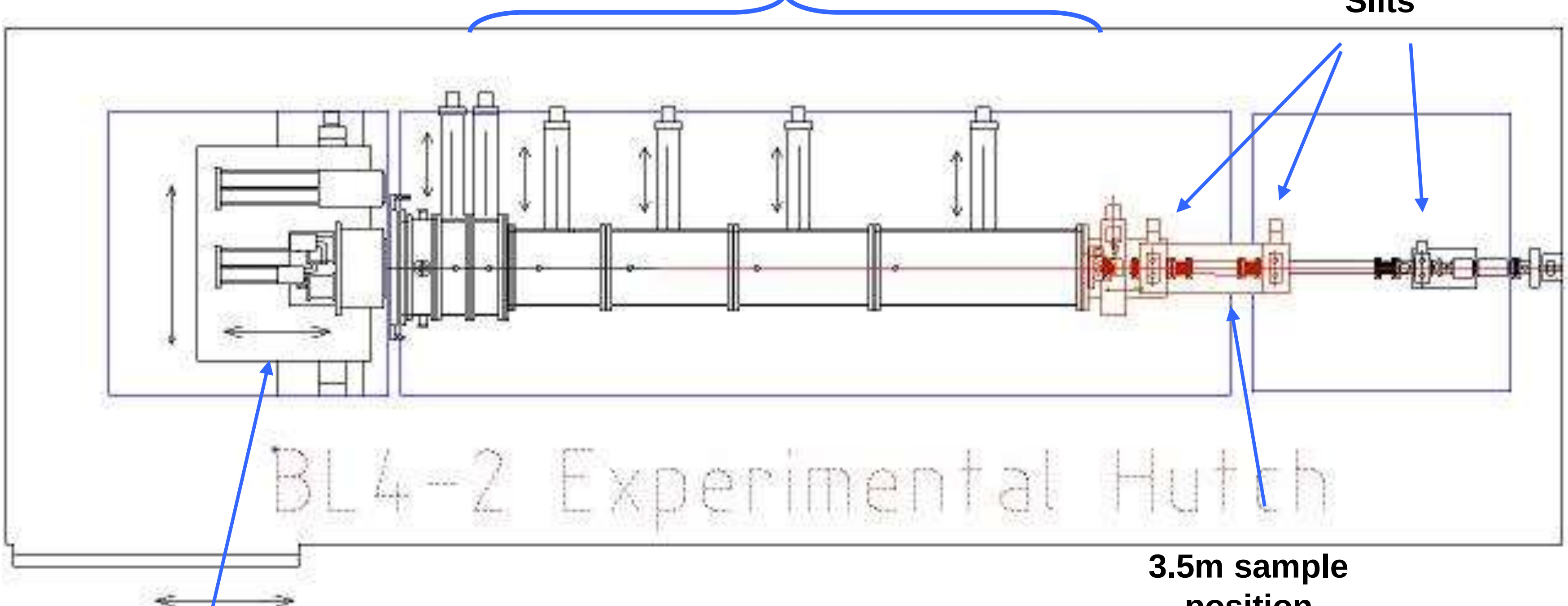
- pinhole camera with variable sample – detector distance
- evacuated flight path (0.25m – 3.5m)
- semi-automatic distance change
- beamstop with photodiode (measuring transmitted intensity)
- various sample cells/handling devices
- CCD detectors (optimized for static and time-resolved experiments)



SAXS Instrument

adjustable flight pass

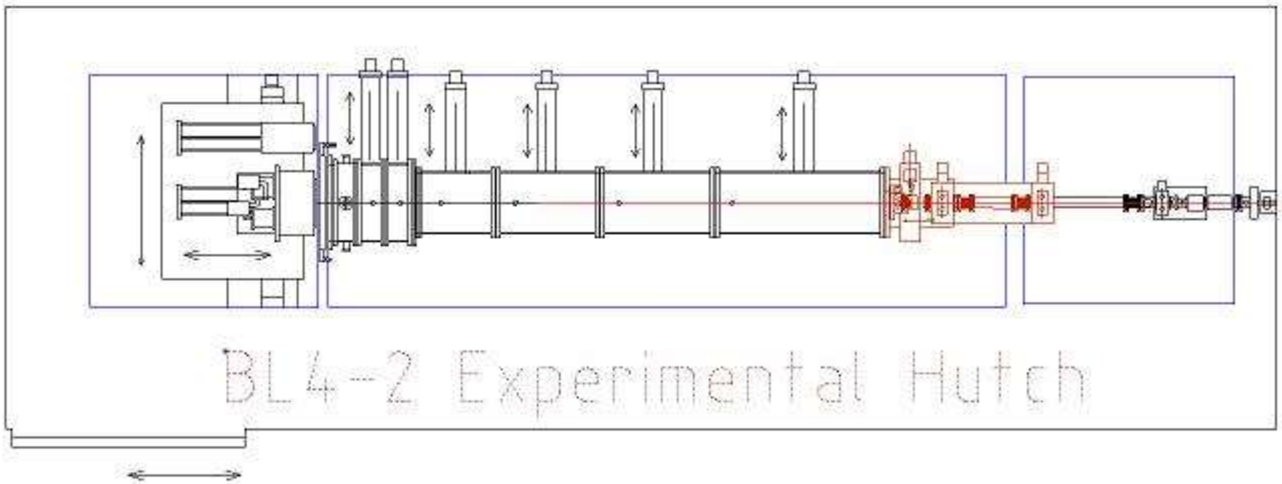
Slits



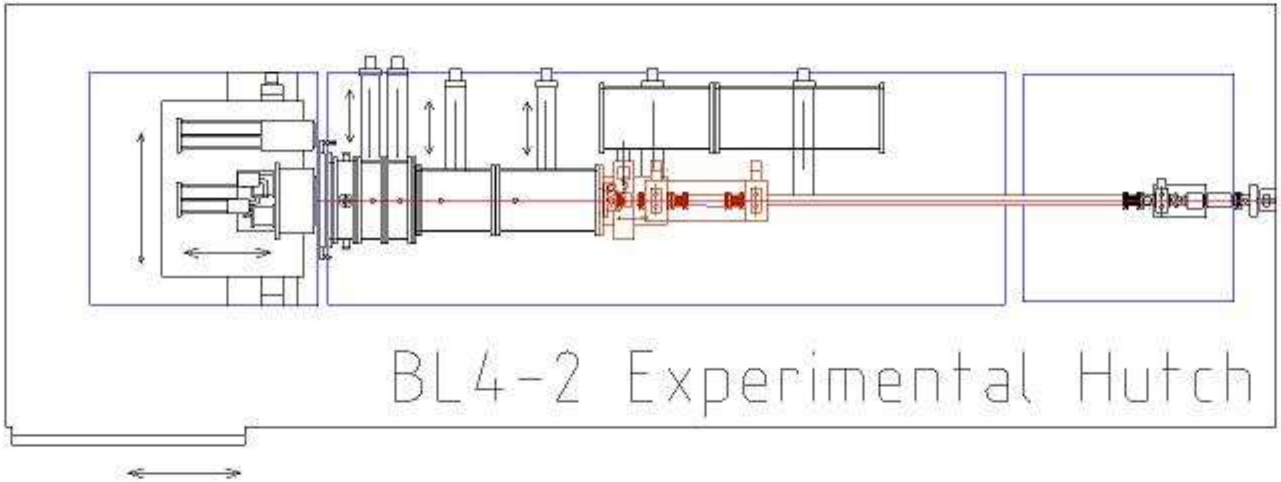
3.5m sample position

motorized Detector stage
easy exchange between detectors

Automated distance change



3.5 m



1.7 m

Software

Beamline Control Software

- entire beamline is controlled via a customized version of BluIce
- easy to use and intuitive graphical user interface (point & click)

Data reduction Software

- fast radial averaging and intensity scaling
- frame averaging and rad. damage detection
- batch mode or GUI
- automatic buffer subtraction
- can be synchronized to data collection for online processing and display of measured data

Sample environments

A large variety of sample cells are available for the experiments:

Autosampler

manual capillary flow cells

stopped-flow rapid mixer (5ms)

FPLC-SAXS (size-exclusion)

Autosampler

- 11 samples

- Each sample:
  Buffer + 7 concentrations

- 30 µl sample consumption

- Temperature controlled (5-75°C)

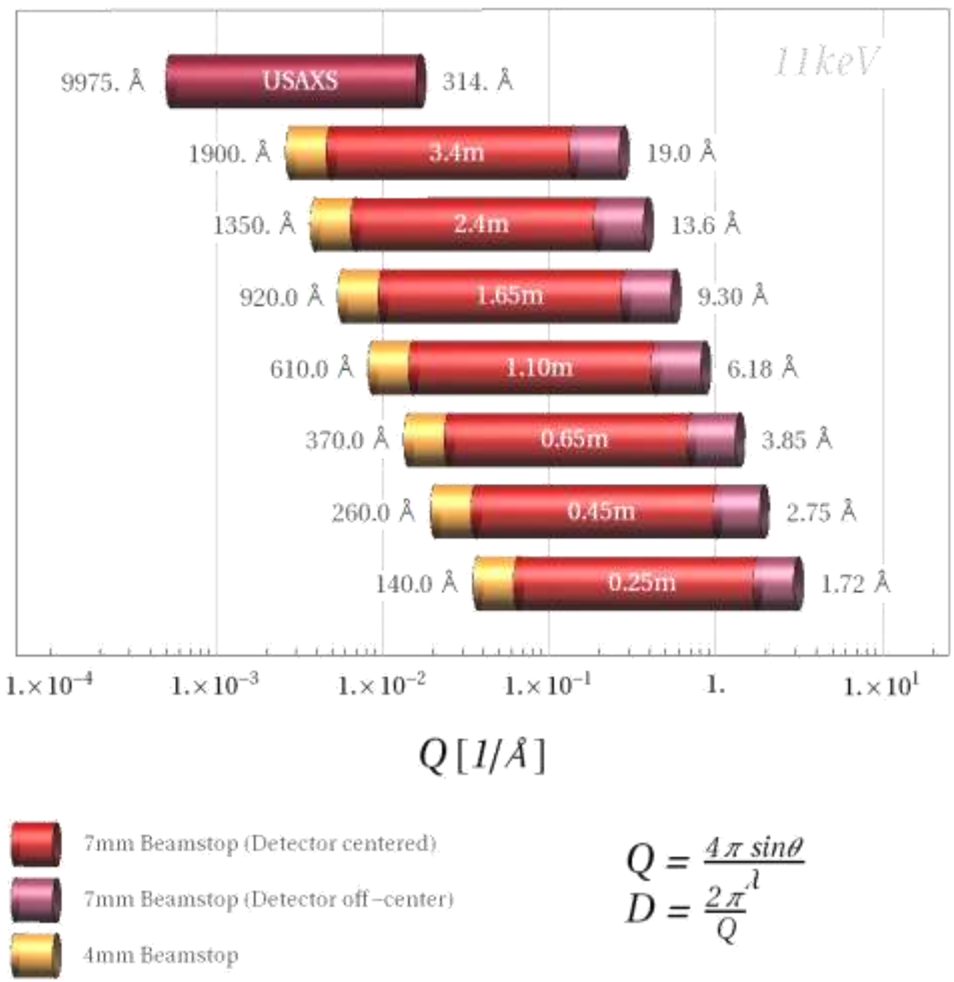
- Automatic capillary washing

- Total experiment time:
  11 * 9 * 5 min ~ 8 hours

BL 4-2 Basic Characteristics

- variable distances (0.25 m – 3.5 m)
- semi-automatic distance change
- small volume autosampler (30 µl)
- high sensitivity detector
- accessible energy range: 5keV - 17keV
- beamsize FWHM typical 0.2mm x 1.0mm
- quick monochromator change between Si and Multilayer
- photon flux: ~2 x 10^{12} (Si111), ~10^{14} (Multilayer)

Accessible q-range



$$Q = \frac{4\pi \sin\theta}{\lambda}$$
$$D = \frac{2\pi}{Q}$$

- large accessible q-range:

  $q = 0.0035$ Å^{-1} ... 3.65 Å^{-1}

- choice of seven different sample-detector distances

- overlapping q-range