



# Instrumentation for SAXS and WAXS

- the Xeuss

FERNÁNDEZ Manuel, PhD Physics  
Application Scientist at Xenocs

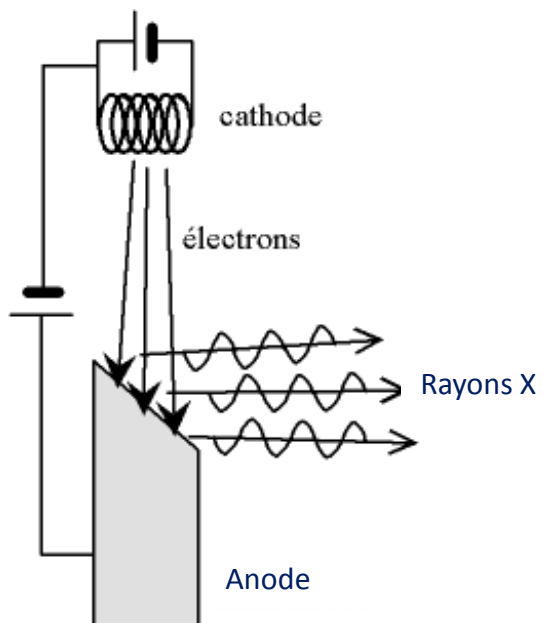
# X-ray Instrumentation

- X-ray generation
  - Sealed Tubes, rotating anodes ...
  - Synchrotron
- SAXS geometries
  - Pinhole camera
- X-ray Optics and Collimation
  - Crystal monochromators
  - Mirrors
  - Multilayers
- Detectors
  - PIN-diode
  - Image Plate
  - Pixel Detectors
- Xeuss

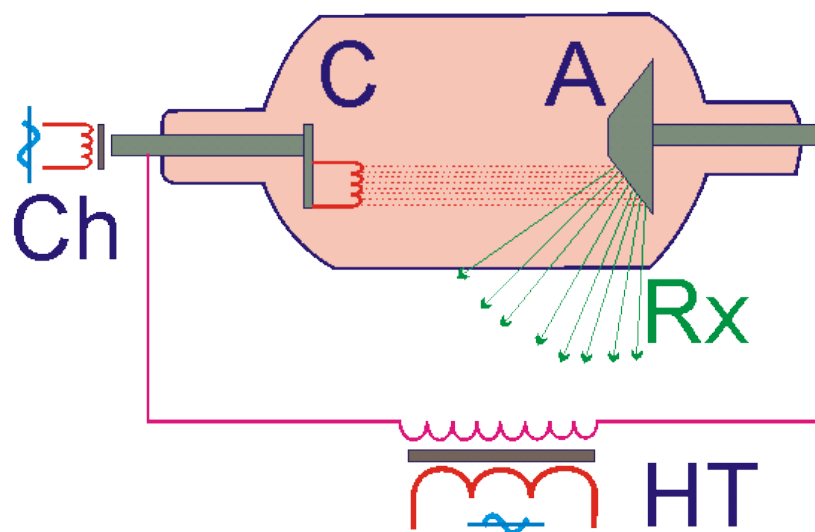
# X-Ray sources

## Sealed tubes and rotating anodes

- Sealed (fixed anode) tube



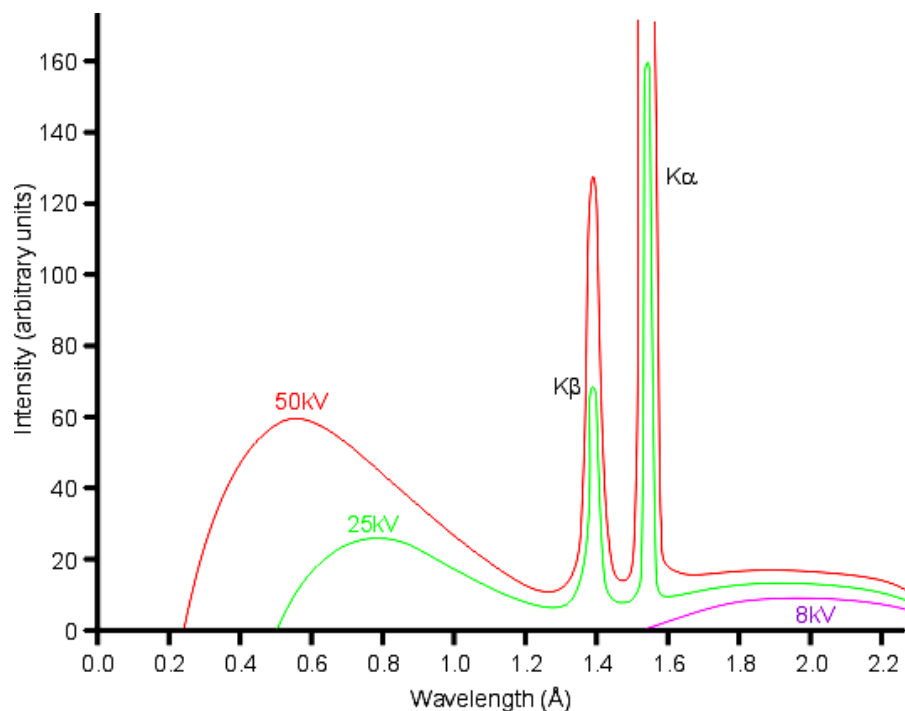
- Rotating anode



# X-Ray sources

## Energy spectrum

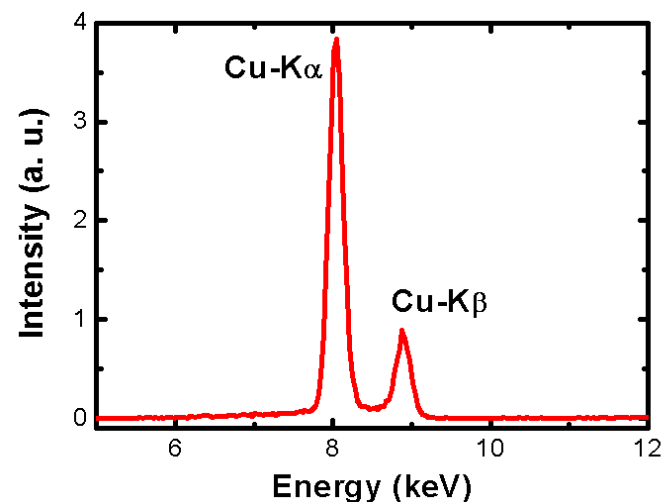
- Decelerating the e<sup>-</sup> from in a random way generate a continuous spectrum : "Bremstrahlung"
- Ionization of some outer edge electron from the target material: specific emission lines



$$E = h\nu = \frac{hc}{\lambda}$$

$$hc = 1239.842 \text{ eV} \cdot \text{nm}$$

$$= 12.39842 \text{ keV} \cdot \text{Å}$$

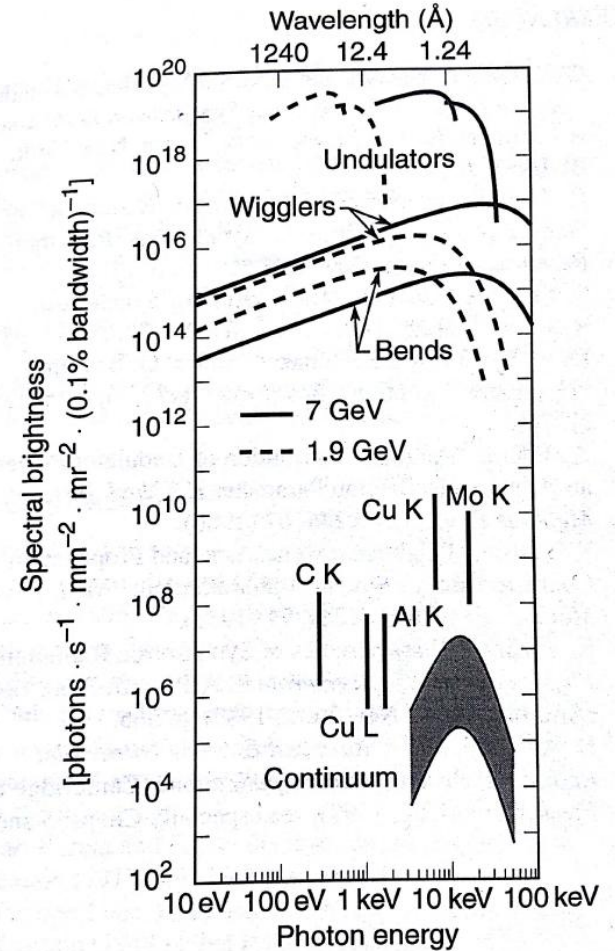


# X-Ray sources

## Synchrotron

Using macroscopic size magnetic field to periodically deflect the course of e- "synchrotrons"

Highly brilliant source (6 to 12 orders magnitude larger than laboratory sources)



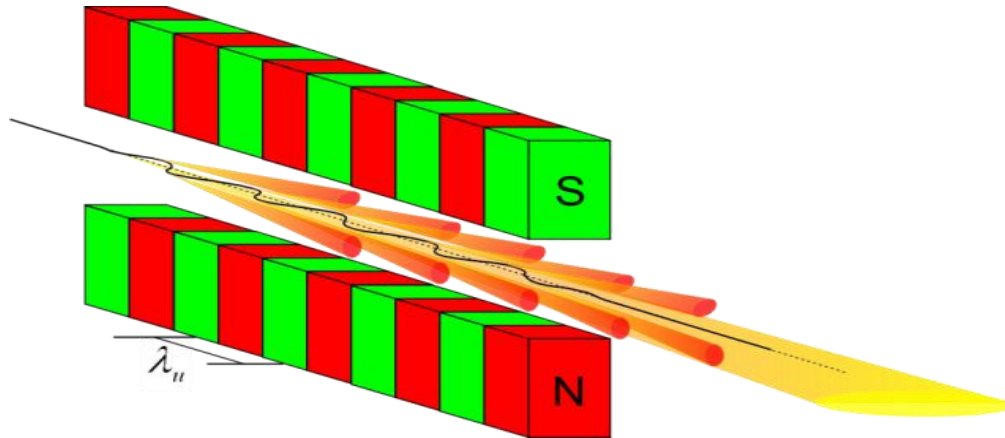
X-Ray Data Booklet. Center for X-Ray Optics, and Advanced Light Source. Lawrence Berkely National Laboratory.

# X-Ray sources

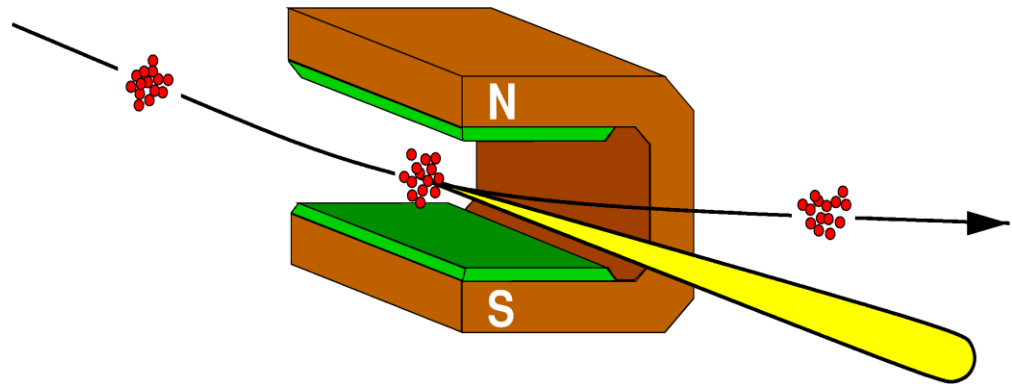
## Synchrotron

Insertion Device

- Wiggler
- Undulator



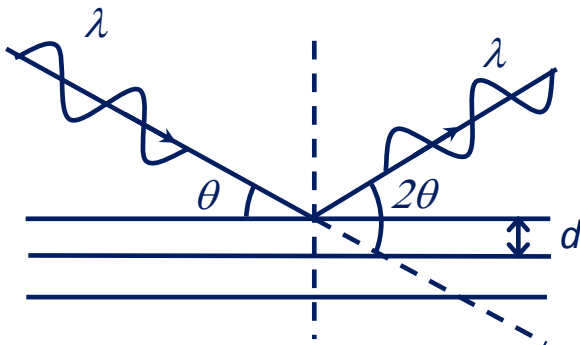
Bending magnet



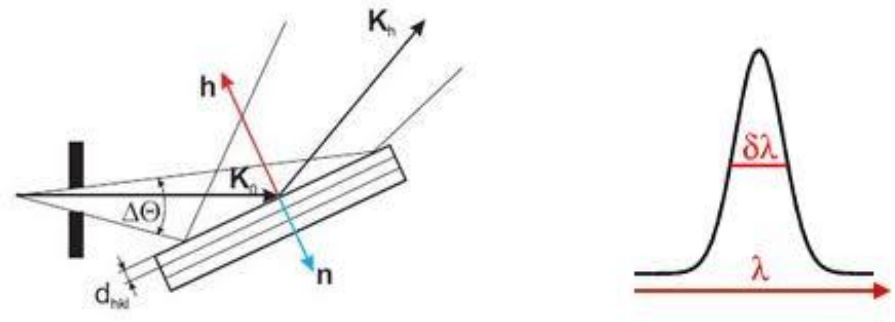
# Monochromators

## Perfect crystals

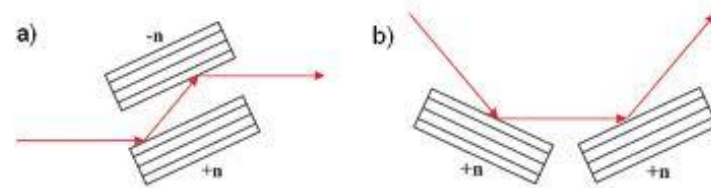
The Bragg's Law for diffraction



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



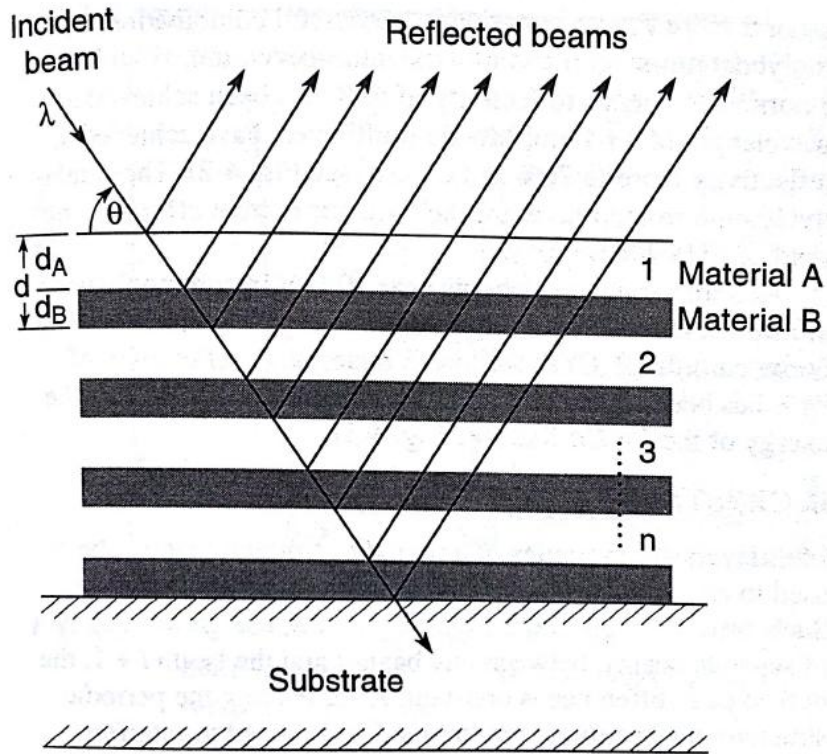
Effect of divergency in spectral purity



Monochromatization by double reflection

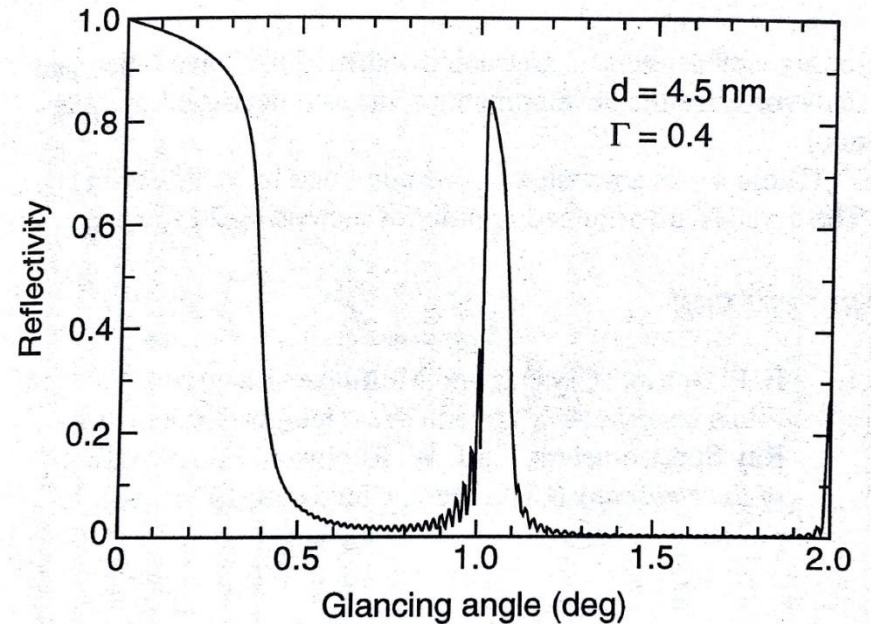
# Monochromators

## Multilayer mirrors



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

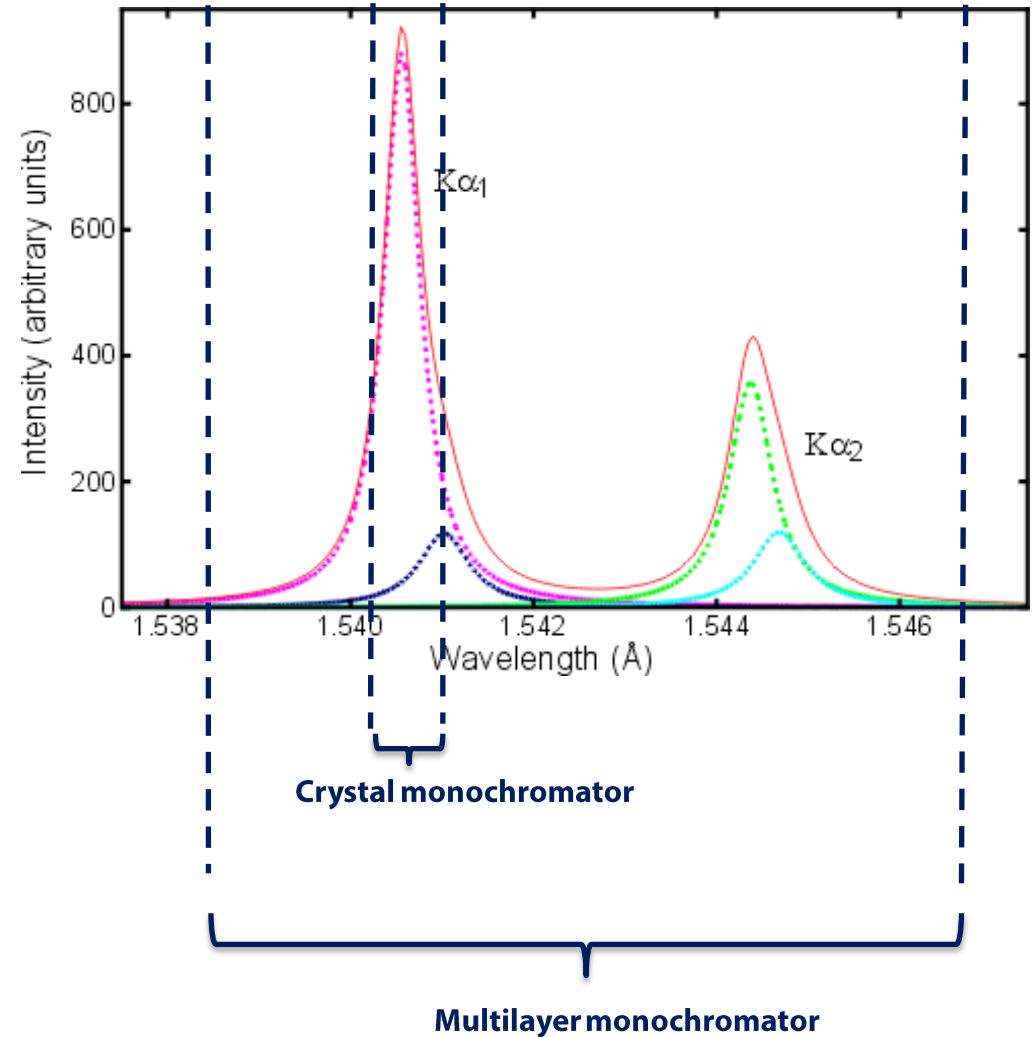
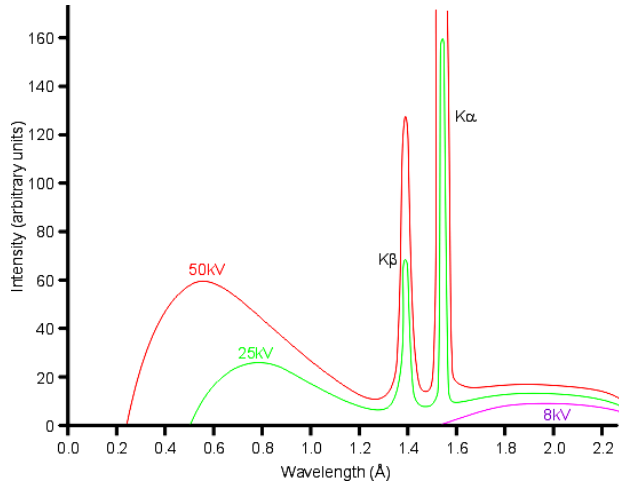
## Very small reflection angle



X-Ray Data Booklet. Center for X-Ray Optics, and Advanced Light Source. Lawrence Berkely National Laboratory.



# Wavelength range

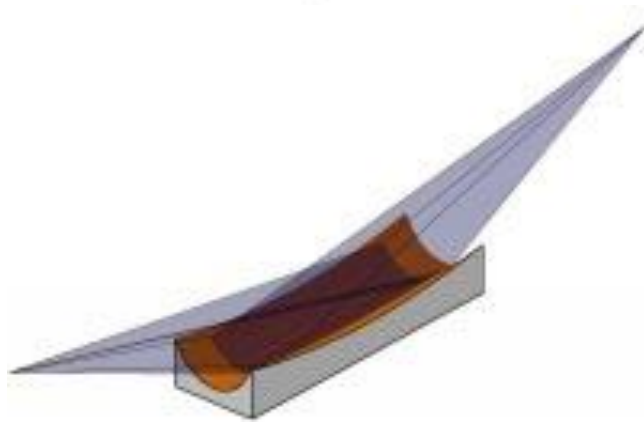
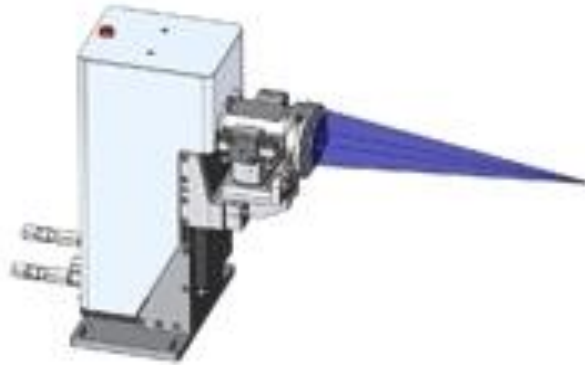


$$\lambda_{Cu} = 1.5489 \text{ \AA}$$

$$\lambda_{Mo} = 0.7108 \text{ \AA}$$

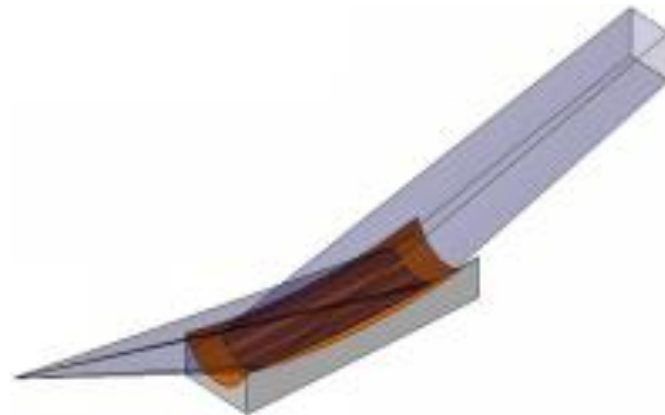
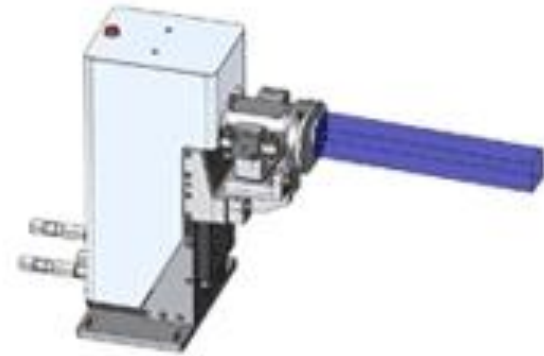
# Xenocs Optics

## Focalizing collimation



Ellipsoid of revolution

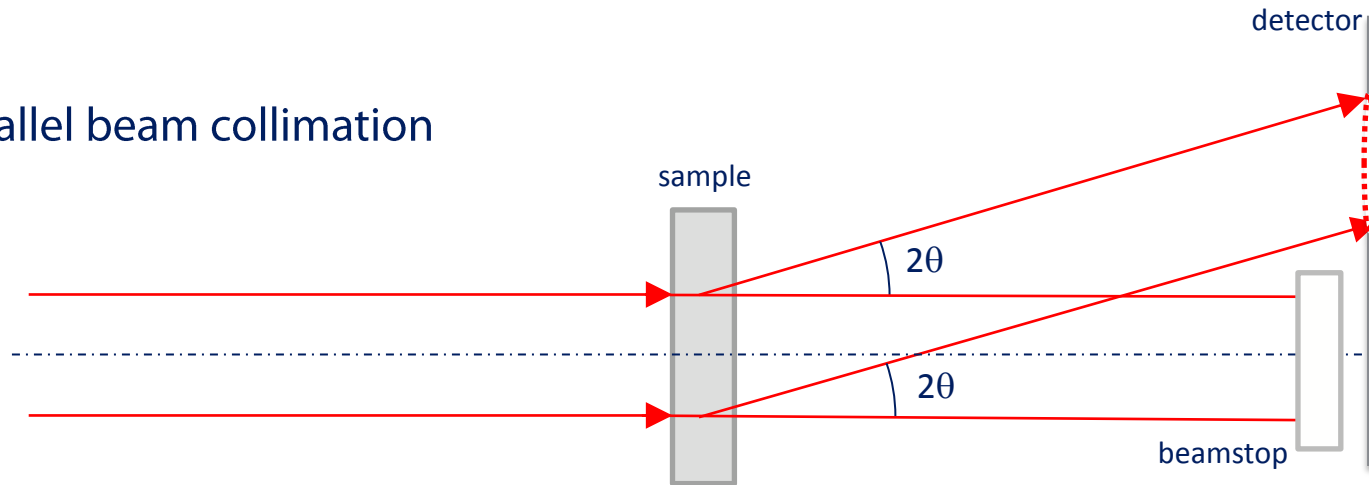
## Parallel beam collimation



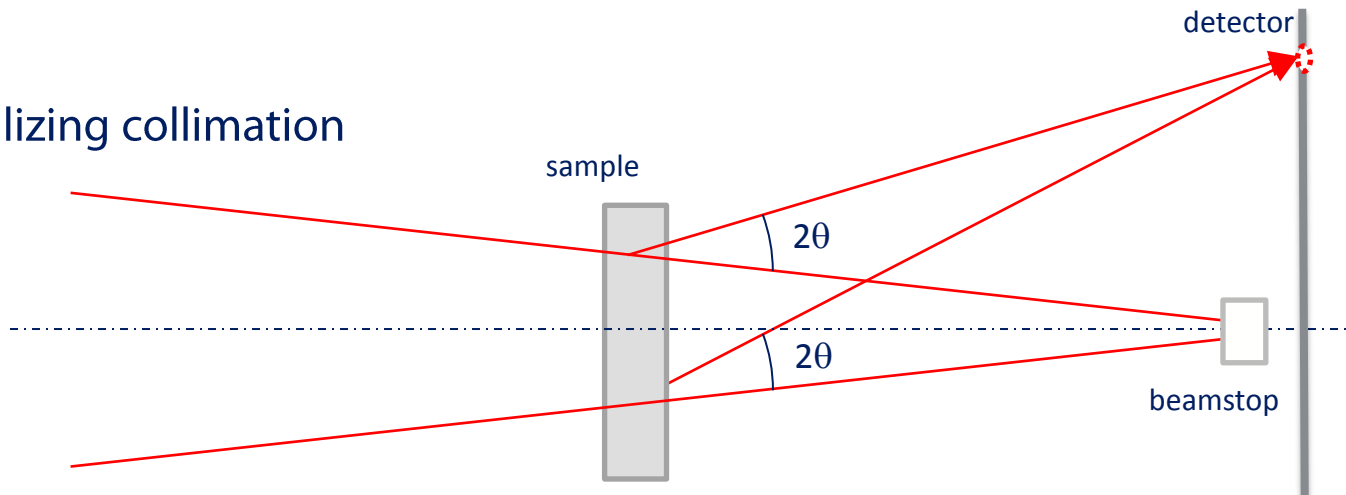
Paraboloid of revolution

# Collimation geometry

## Parallel beam collimation

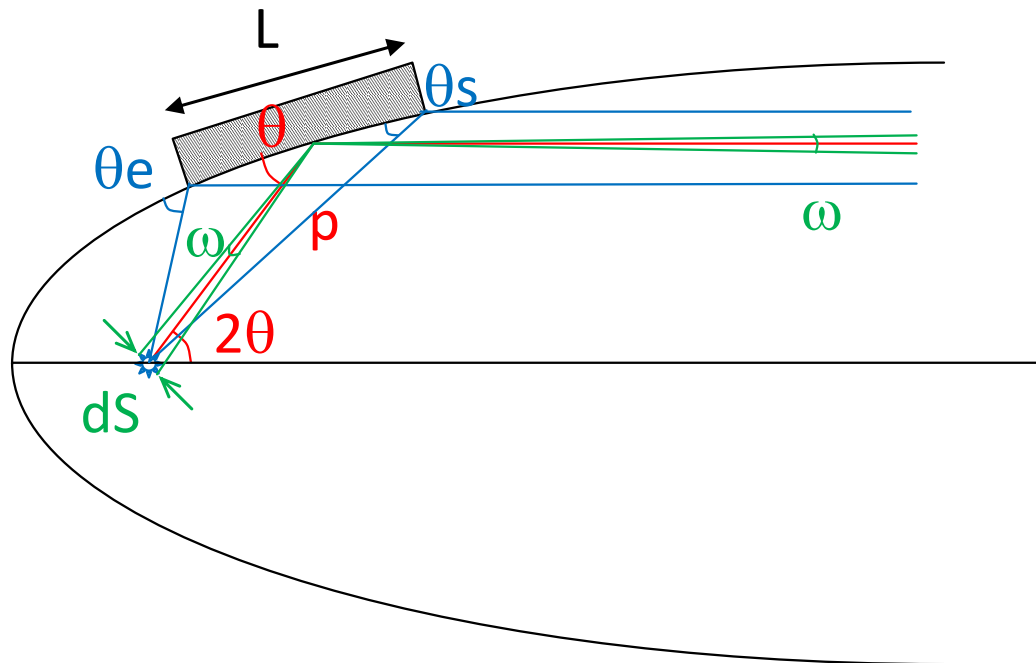


## Focalizing collimation



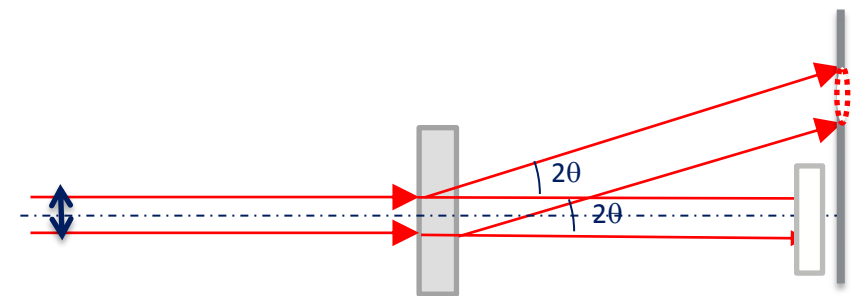
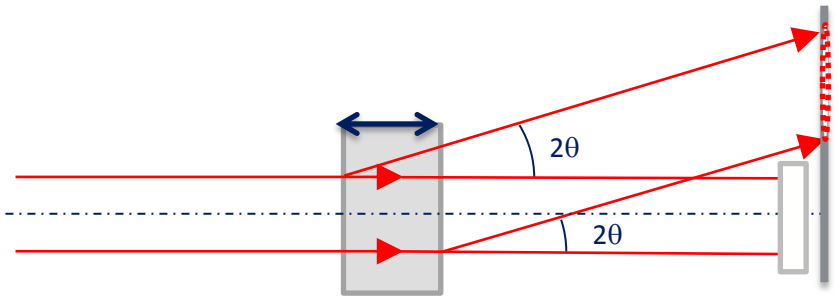
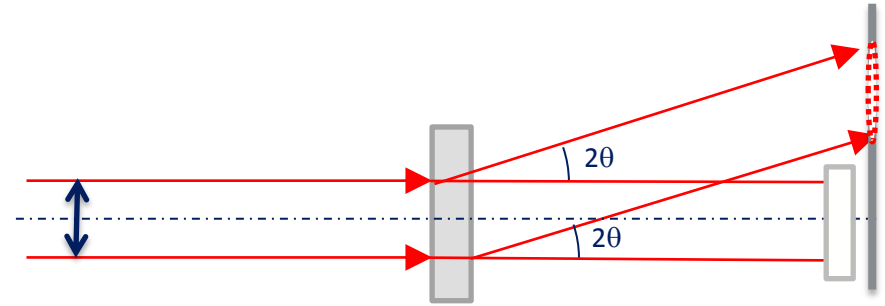
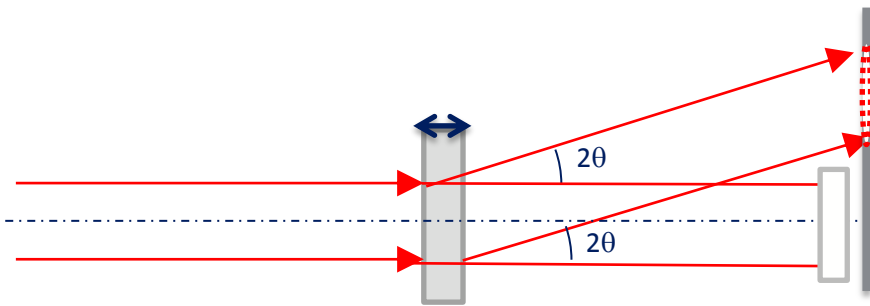
# Collimation – Parallel beam Optics

Paraboloid of revolution



# Instrumental Function

## Beam size and sample thickness

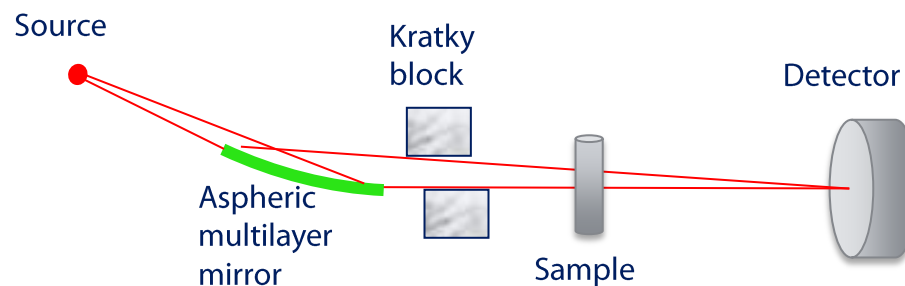


# Small-angle X-ray Scattering

## Lab source and Kratky camera

- Compact geometry

- Microfocused source
- Multilayer monochromator/focusing-collimating mirror
- Beam clean up by 2 down stream slits or Kratky block

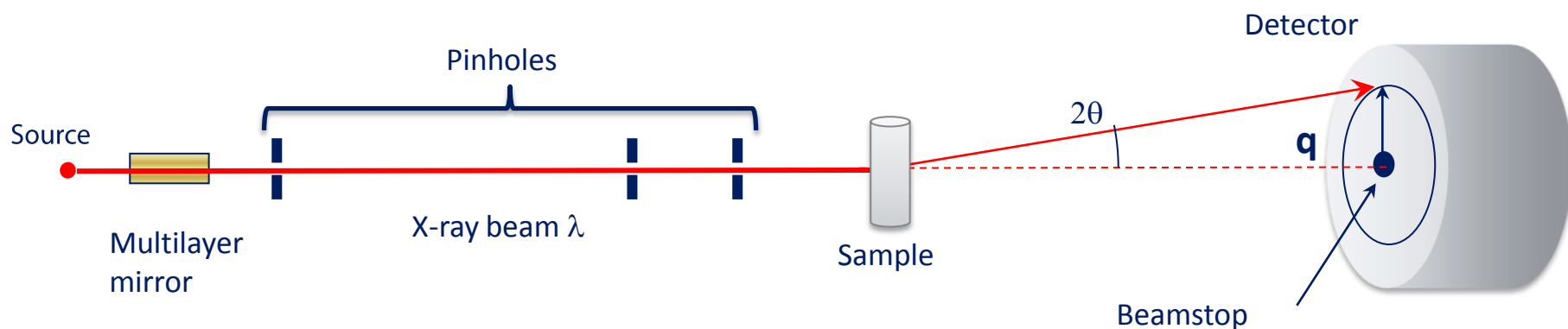


- Loss of 98% *only* of the power of the source:  
Over x1000 improvement of the flux

# Small angle x-ray scattering

## Lab source and pinhole camera

- Intense rotating anode/microfocus sealed tube
- Crystal monochromator/collector
- Collimation by 3 sets of crossed slits
- Beam clean up by 2 down stream slit

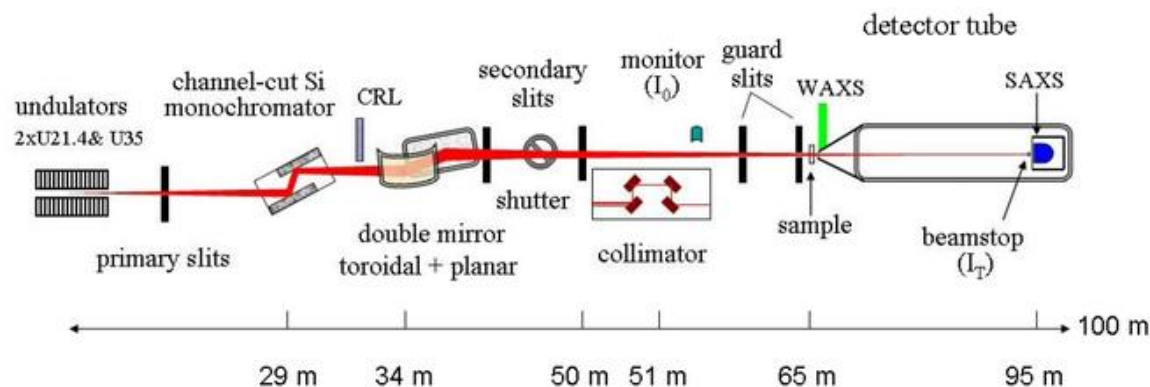


- Loss of 99.99% of the power of the source
- RECENT PROGRESS IN SLITTING leads to increase x5 in flux

# Small angle x-ray scattering

## Synchrotron : the E.S.R.F. ID02 beamline

- Beam conditioning
  - Choice of the source
    - ESRF: low divergence, high brilliance straight section: "High Beta section"
  - Monochromator "heat load" remover
  - High precision 1m long toroidal mirror, focal length 33m, image 1:1 of source, slope error  $< \text{qq } \text{\AA}$
  - Collimation by 3 sets of crossed slits
  - Beam clean up by 2 down stream slits



- Beamstop
  - Required with an adapted size, monitoring of transmitted intensity



# Scatterless Slits

S1

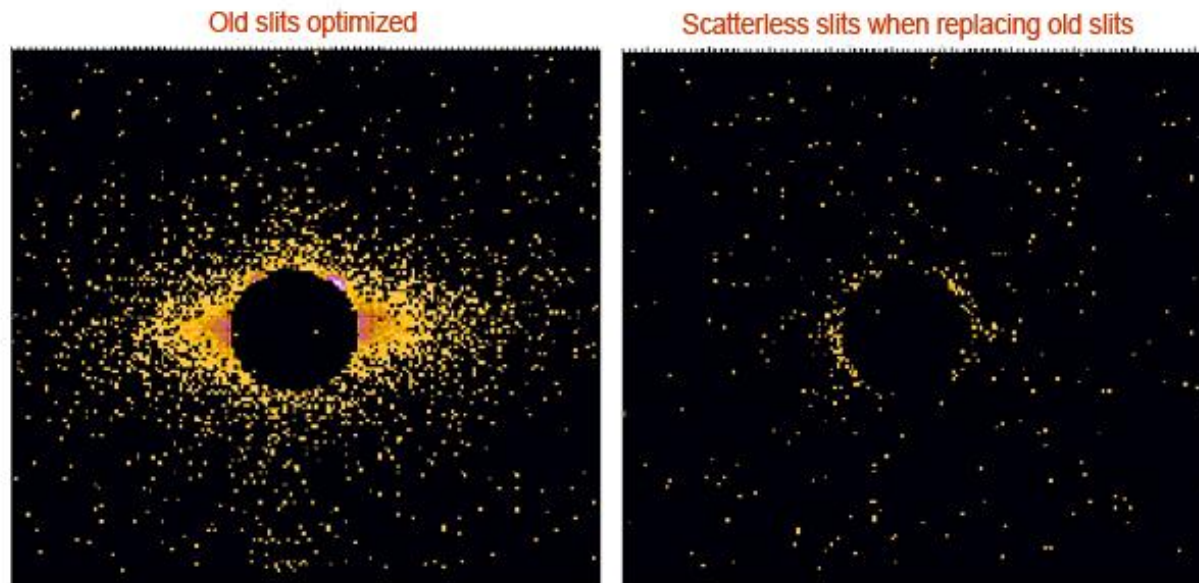
S2



Pinholes: fix  
Slits : adjustable

# Collimation

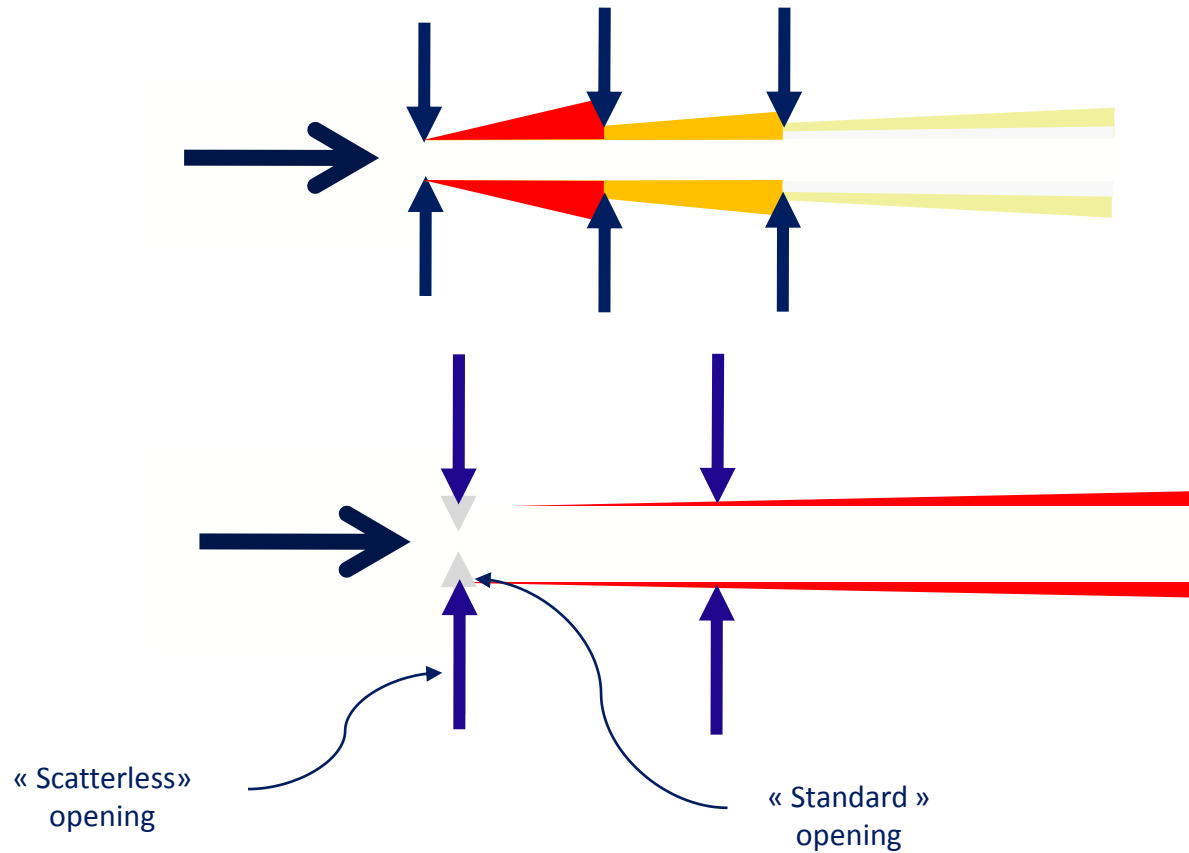
## Clean-beam technology



**Figure 1** : Comparison of the background scattering close to the beamstopper between our previous setup and the scatterless slits. The images are ROIs taken on a Pilatus 100k detector (Dectris)

# Collimation

## Scatterless Slits



# SAXS – WAXS – USAXS

Small- Wide- and Ultra-small- angle X-ray Scattering: Matter of distance

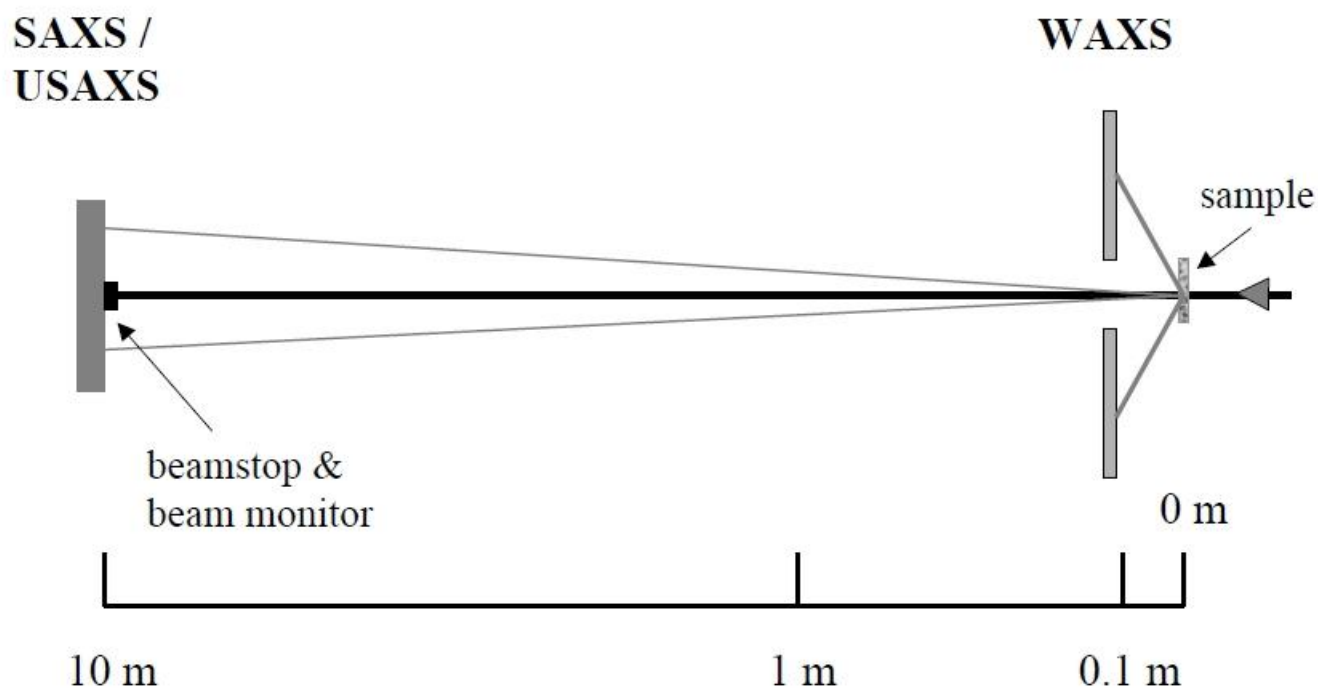
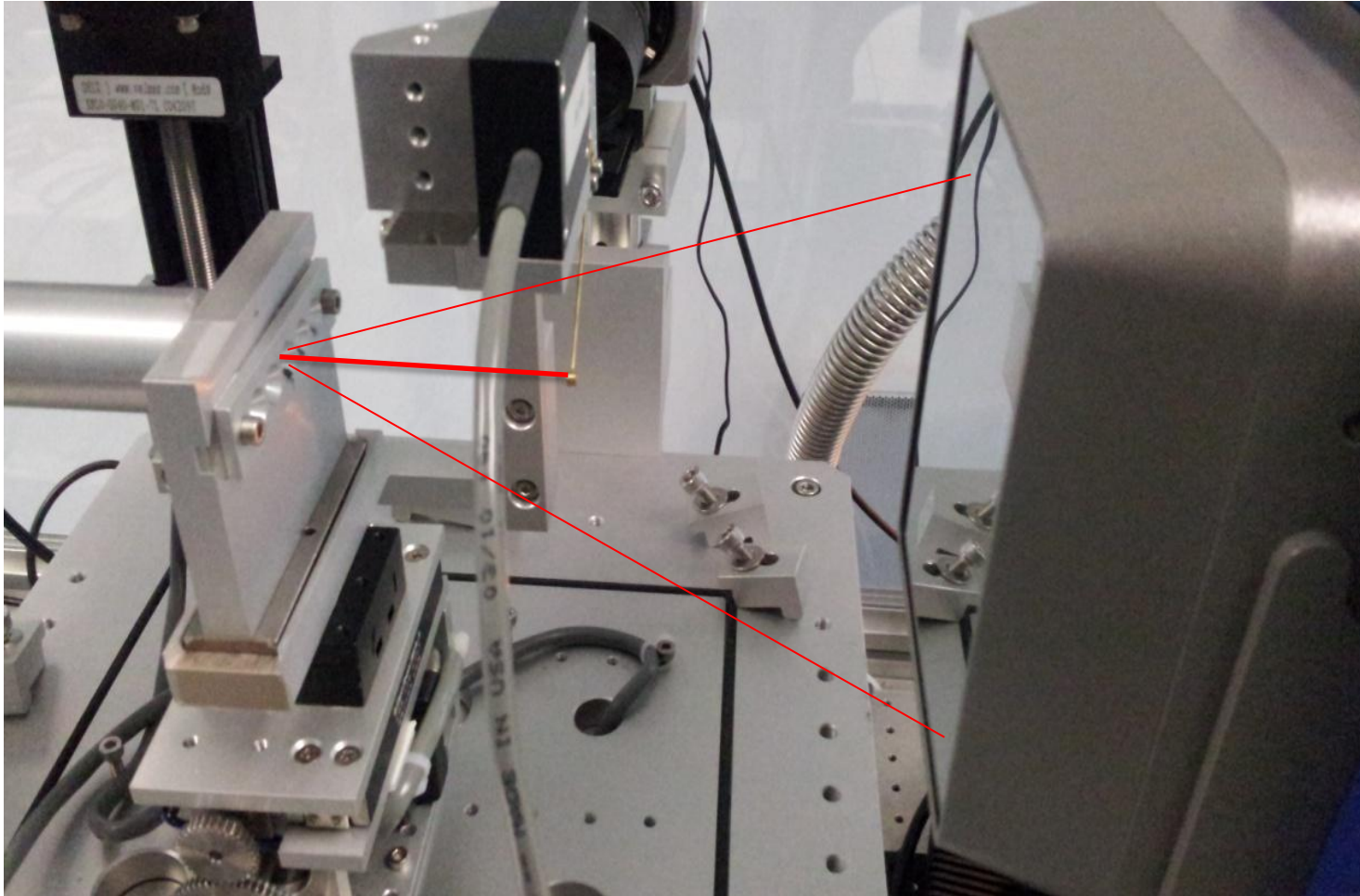


Figure 10: Schematic layout of a combined SAXS/WAXS setup. The SAXS configuration is the same as in Fig. 1 and to which an ideal WAXS detector is added.

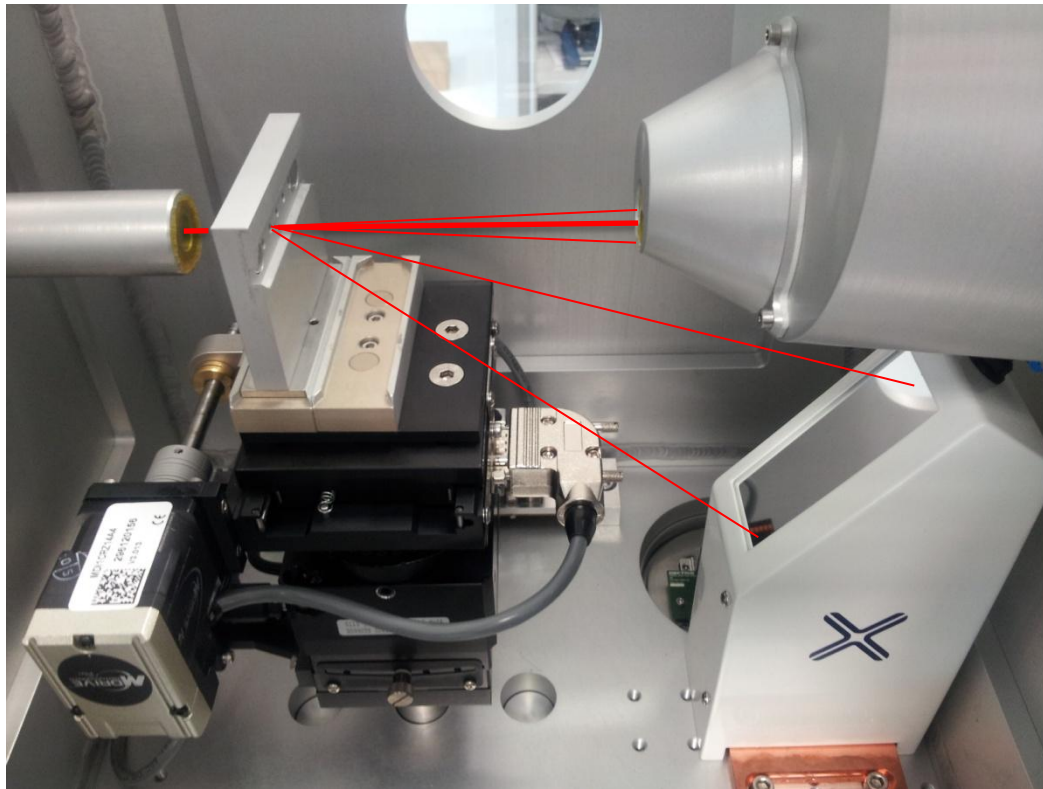
# SAXS/WAXS in a Xeuss 1.0 System

SAXS and WAXS non simultaneous

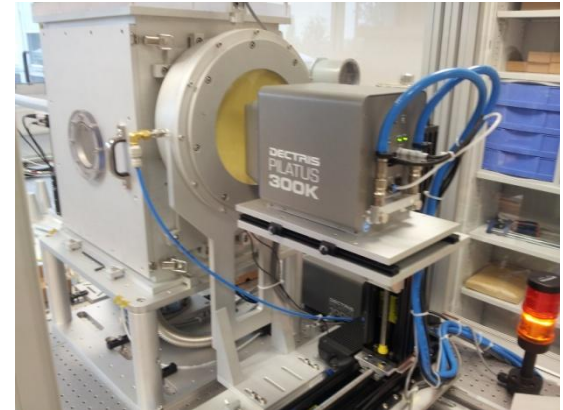


# SAXS/WAXS in a Xeuss 2.0 System

## Simultaneous SAXS/WAXS



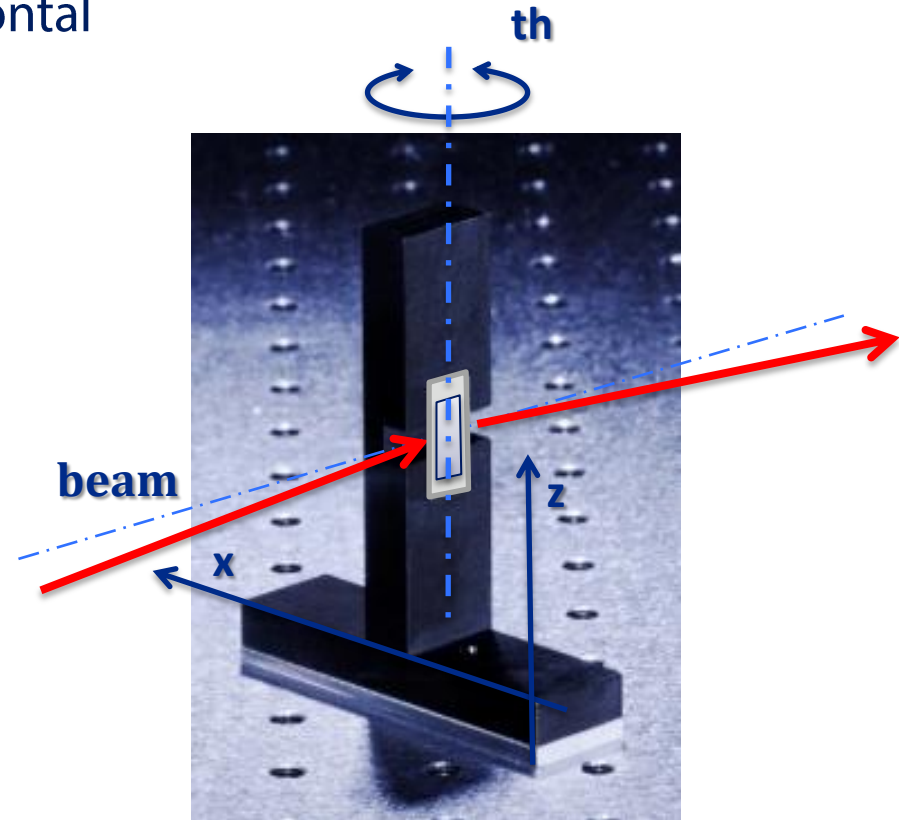
Pilatus 100k-Xenocs  
**WAXS**



Pilatus 100-200-300k or 1M  
**SAXS**

# GISAXS / Reflectometry

Simple Stage : reflection in horizontal

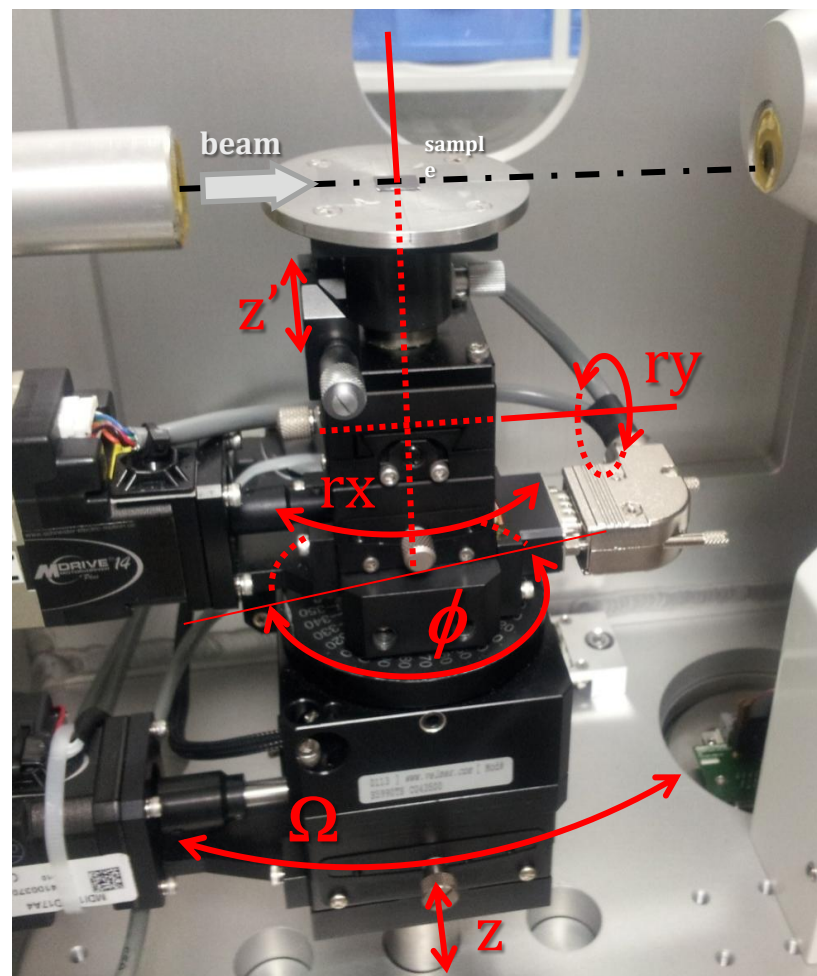


z : vertical translation  
x : lateral translation  
th : reflection tilt

# GISAXS / Reflectometry

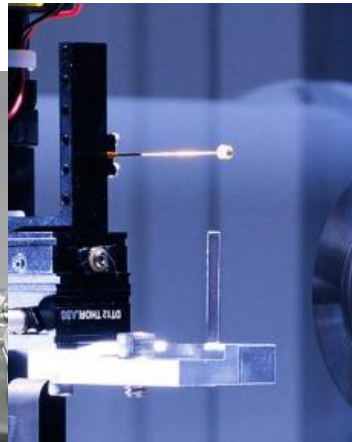
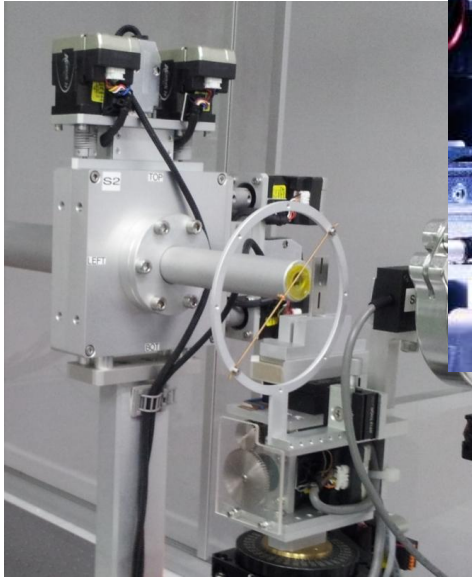
## Horizontal Stage Vertical reflection

- $z$  : vertical translation
- $\omega$  : omega, reflection tilt
- $\phi$  : vertical rotation
- $rx$  : fine tilt
- $ry$  : fine reflection tilt
- $z'$  : manual vertical translation

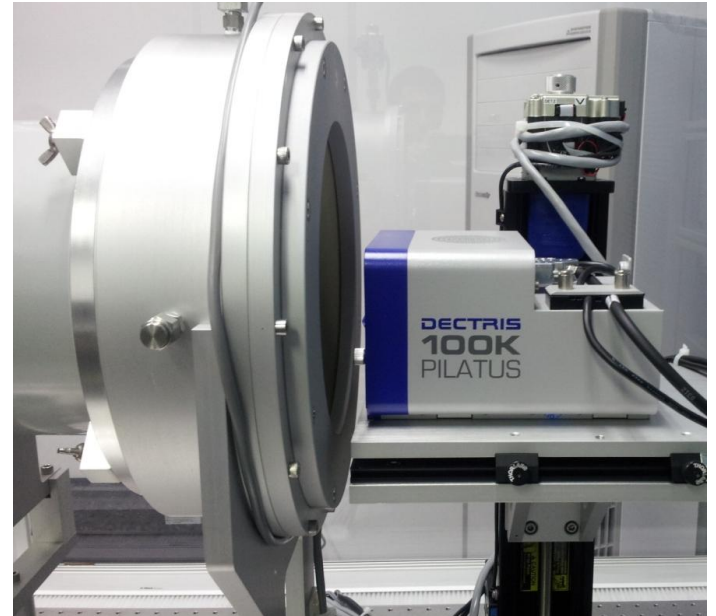




# Beamstops



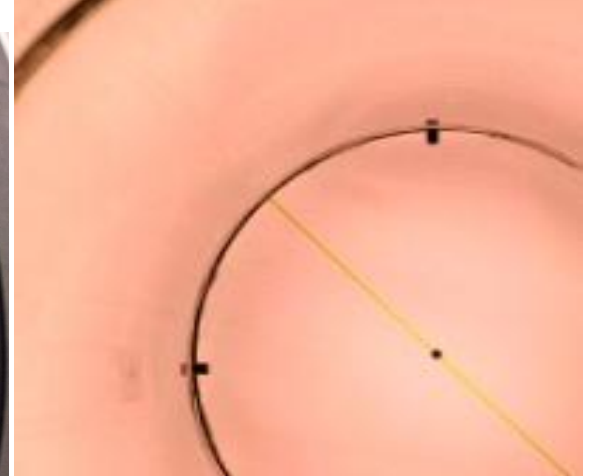
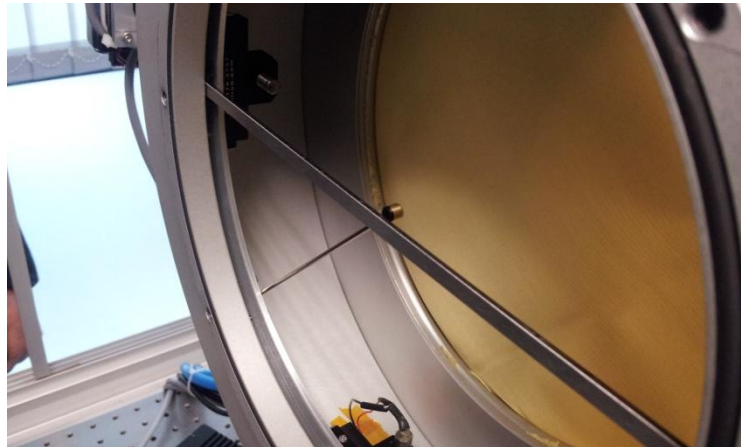
WAXS



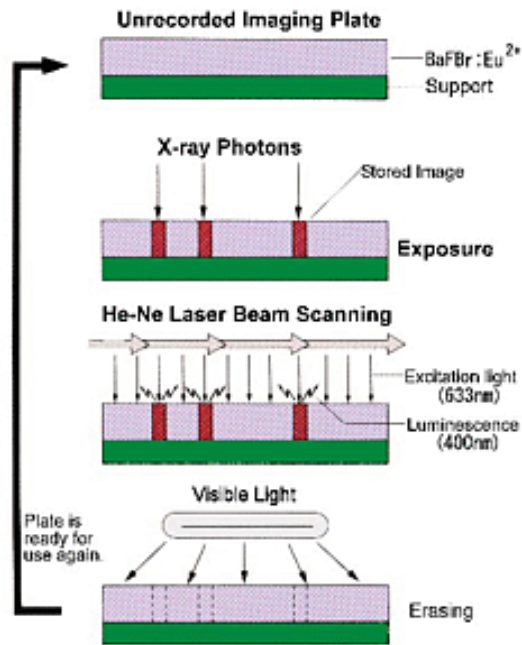
SAXS & GISAXS

WAXSIN  
WAXSOUT

bsx  
bsz  
take\_direct\_beam

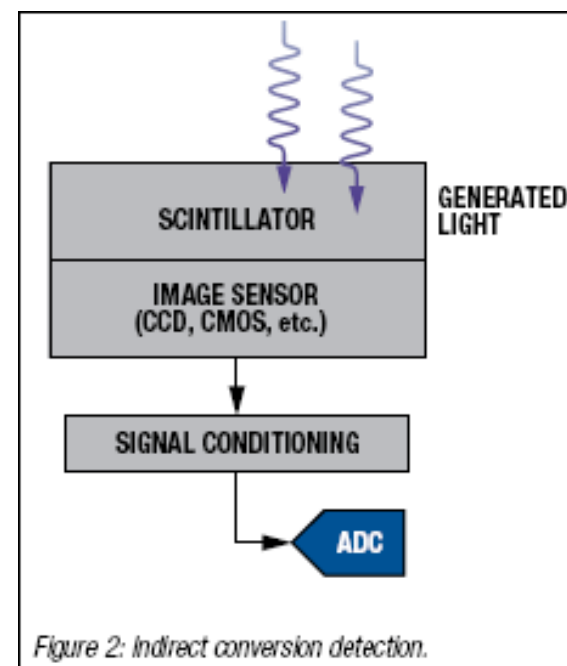
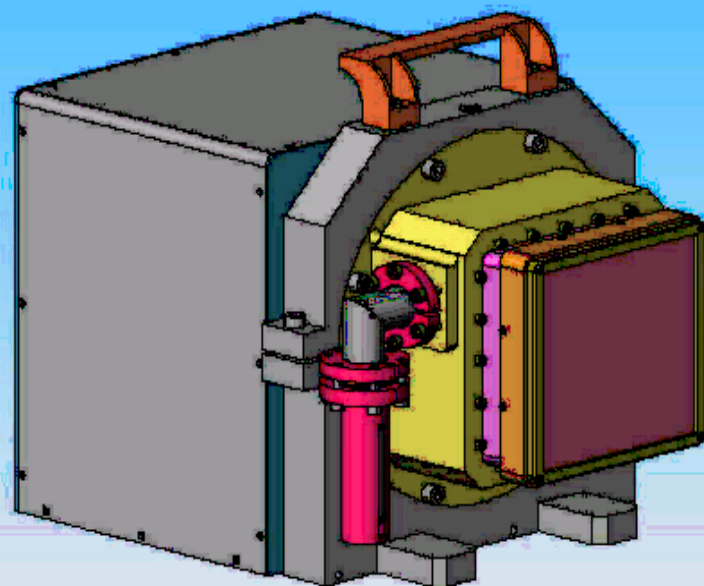


# On-line Image Plate

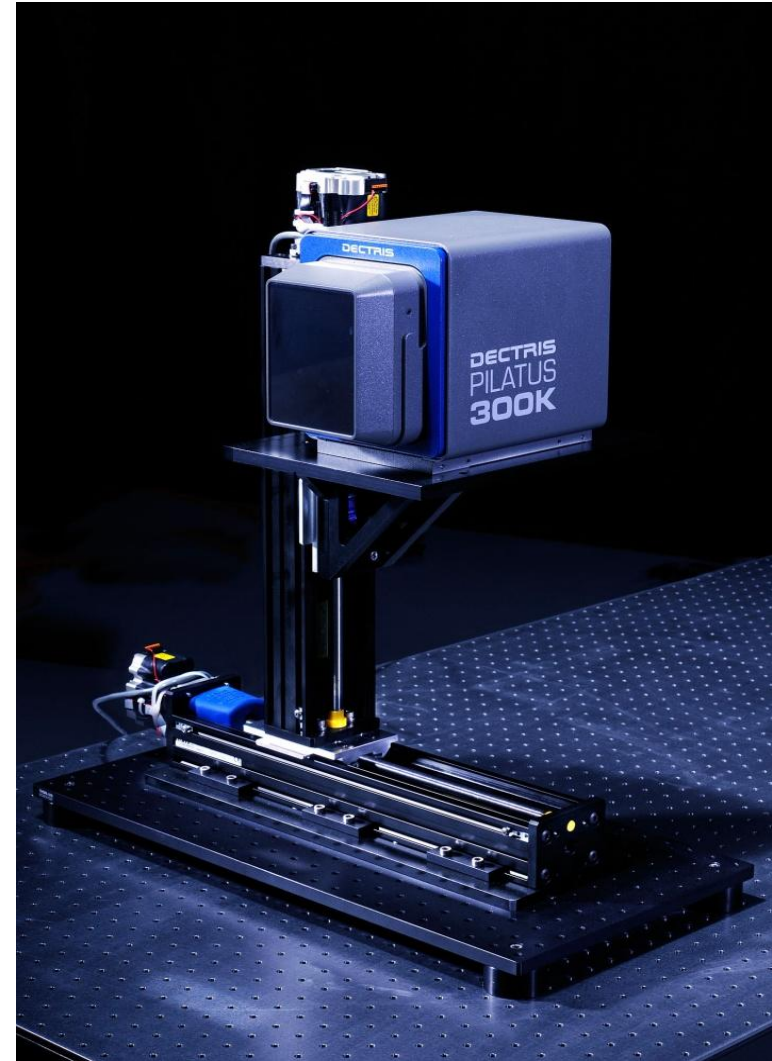
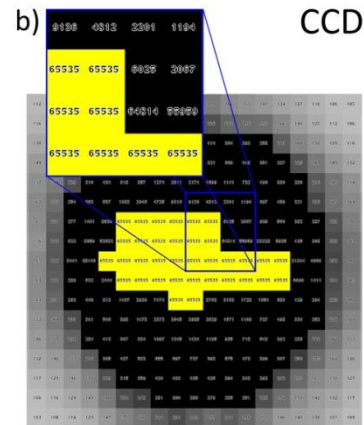
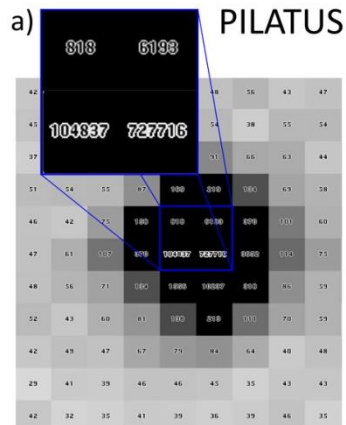
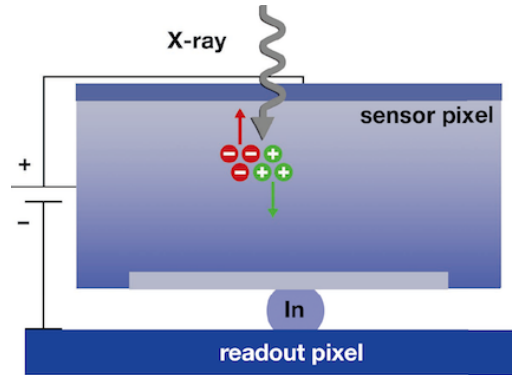


# CCD –Pixel Detector

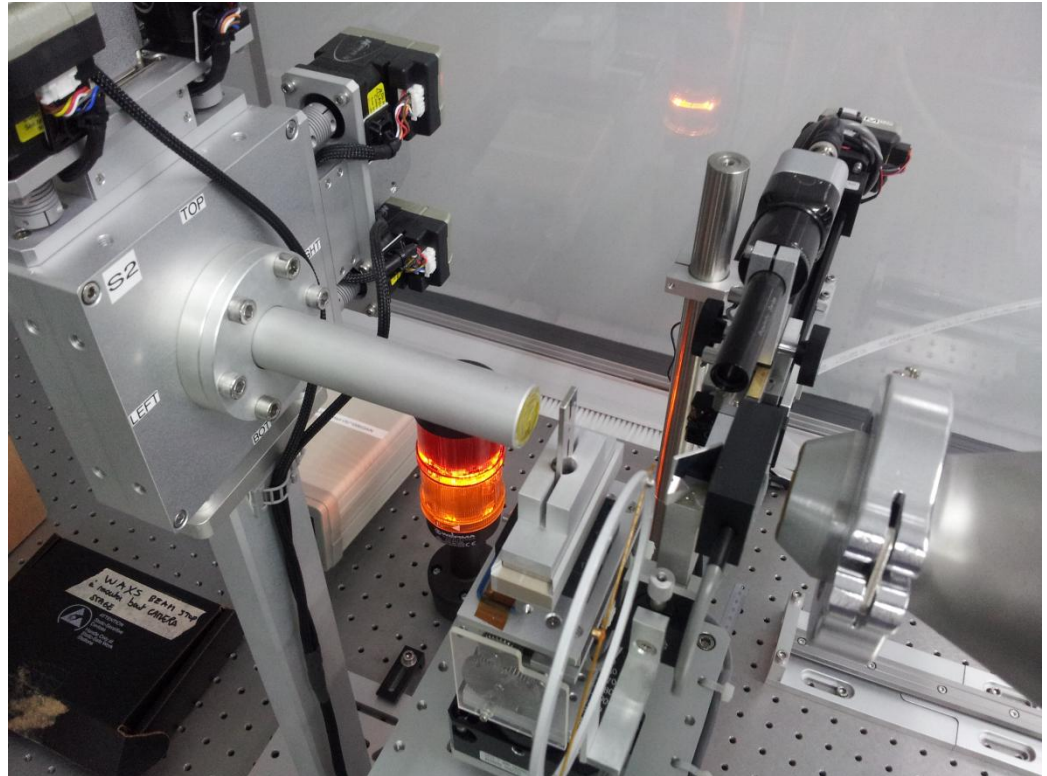
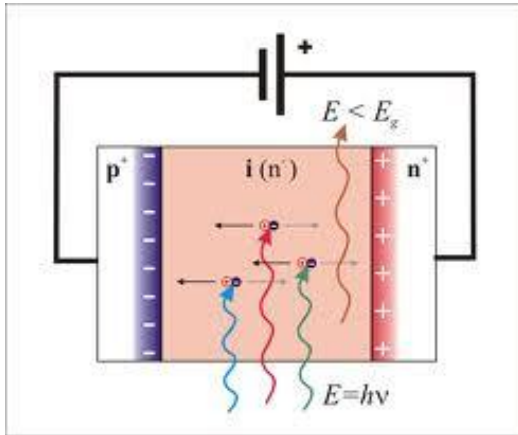
FReLoN – Kodak KAF- 4320E



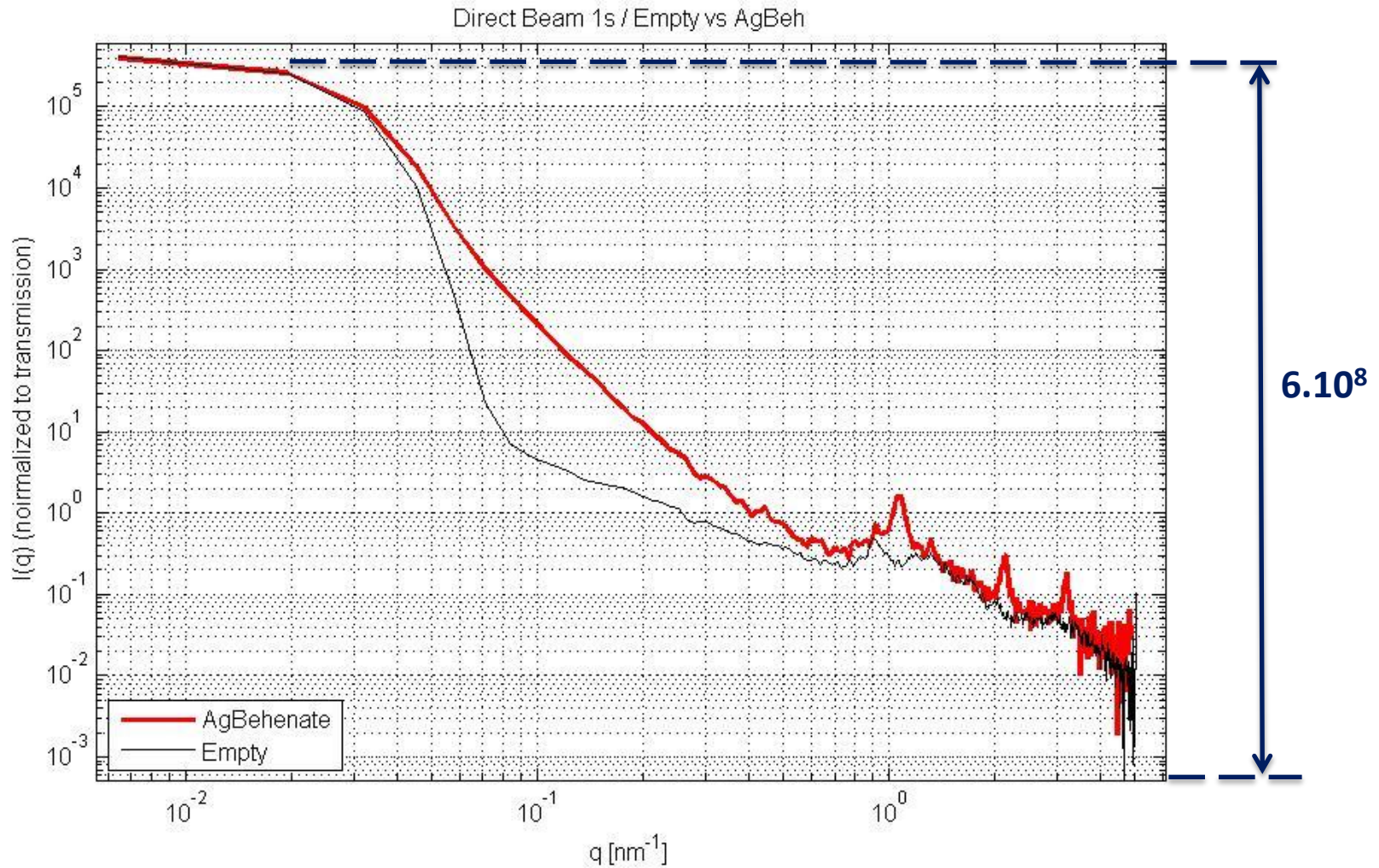
# Hybrid pixel detector



# PIN-diode



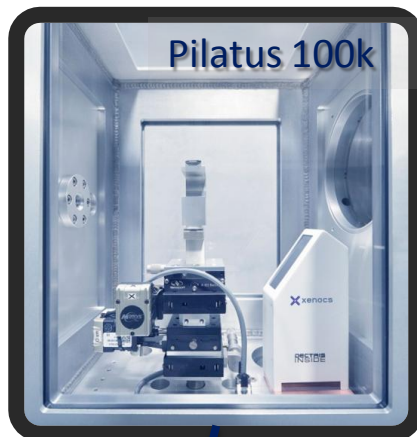
# Hybrid pixel Detector / Direct beam analysis



# System details / operation and safety



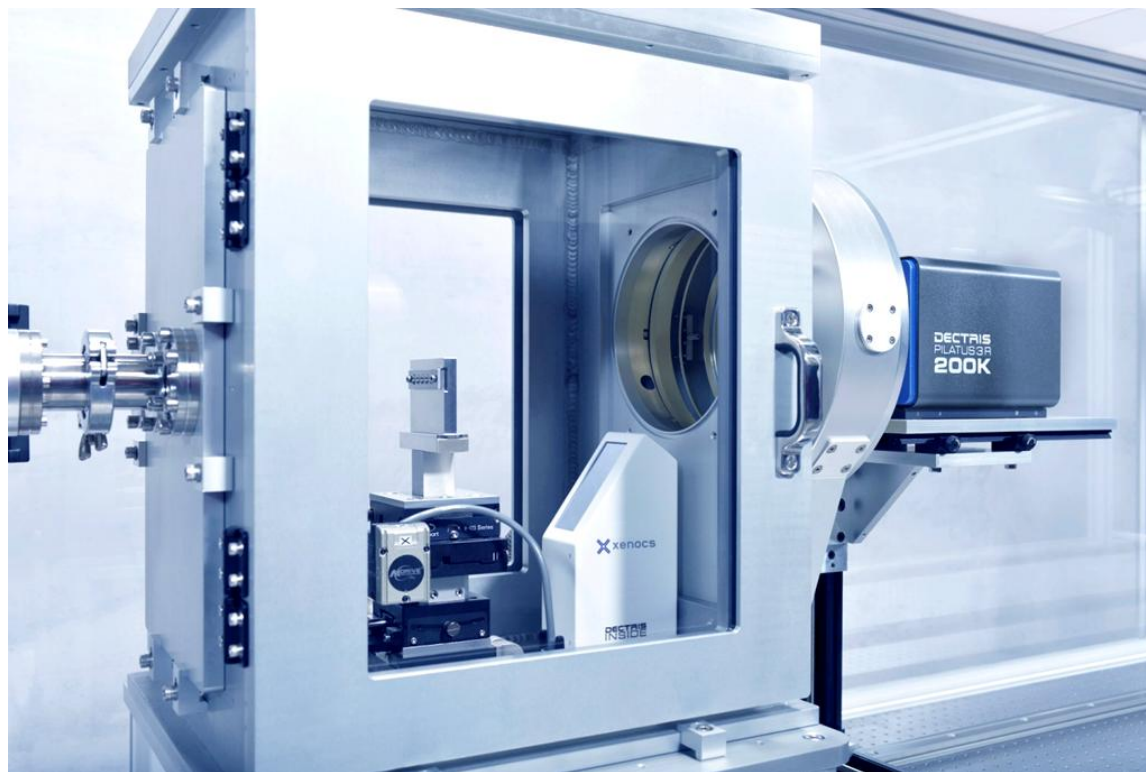
# System details / source and detectors





# System details

## Simultaneous SAXS/WAXS



Thank you  
for your attention!