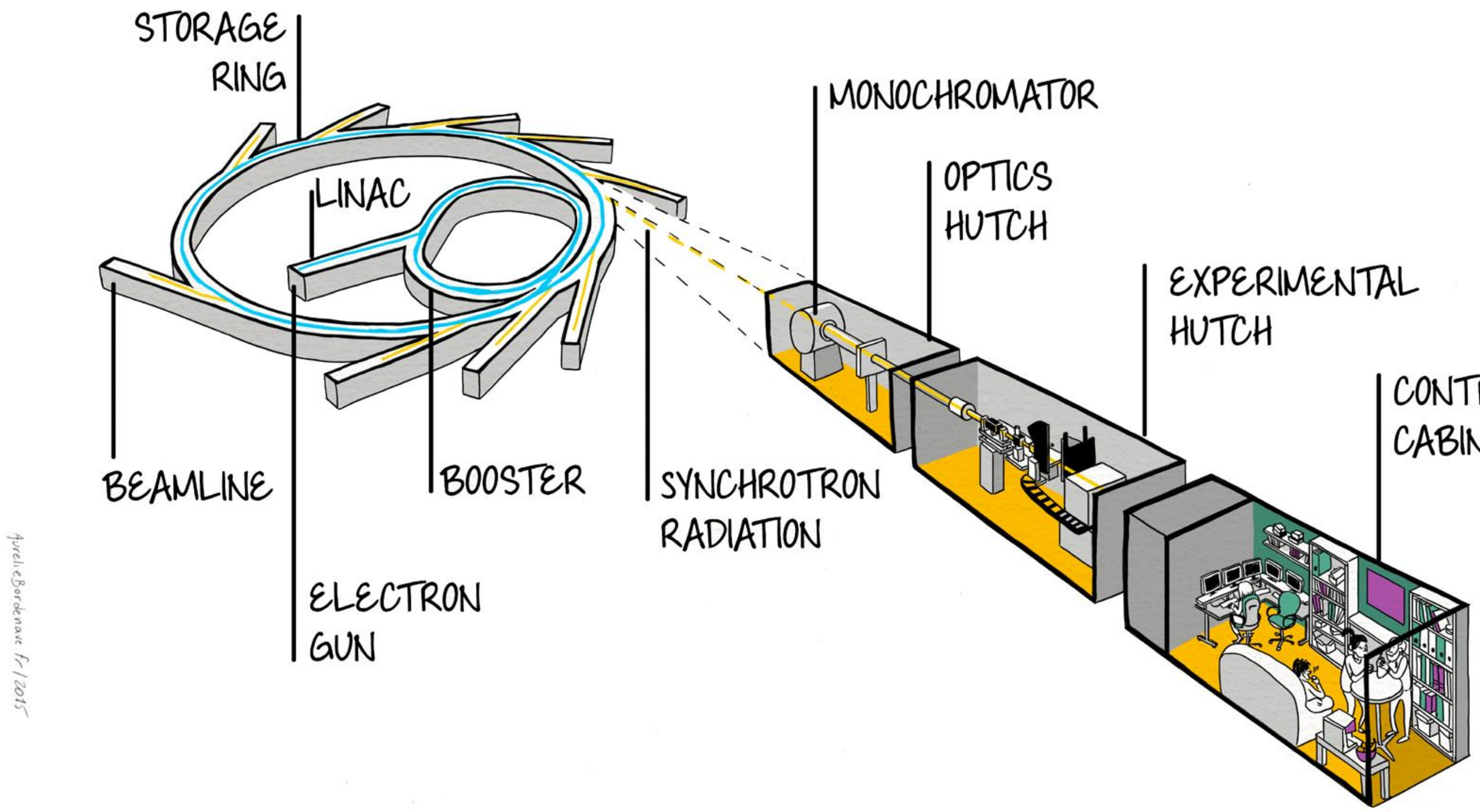
TUNTWIN's Workshop

Session A: Basics in Synchrotron Techniques for Environmental and Food from Basics to Application

Funded by the Horizon 2020 Framework Programme of the European Union under the GA N° 952306

Beam quality factors that affect the performance of a beamline (and your measurements).

Roberto Boada



http://www.synchrotron-soleil.fr/Presse/Videos/Lumieres-de-SOLEIL
Synchrotron SOLEIL (illustration by Aurelie Bordenave)

Overview of the factors that affect the quality of the measurements

Beam quality factors

Intrinsic beamline/SR facility parameters:

- Brilliance/photon flux
- Energy resolution
- Harmonics and mirrors
- Drifts and vibrations
- Beam homogeneity
- Set-up reproducibility

Polarization of the beam

Other factors

Not-beam related parameters:

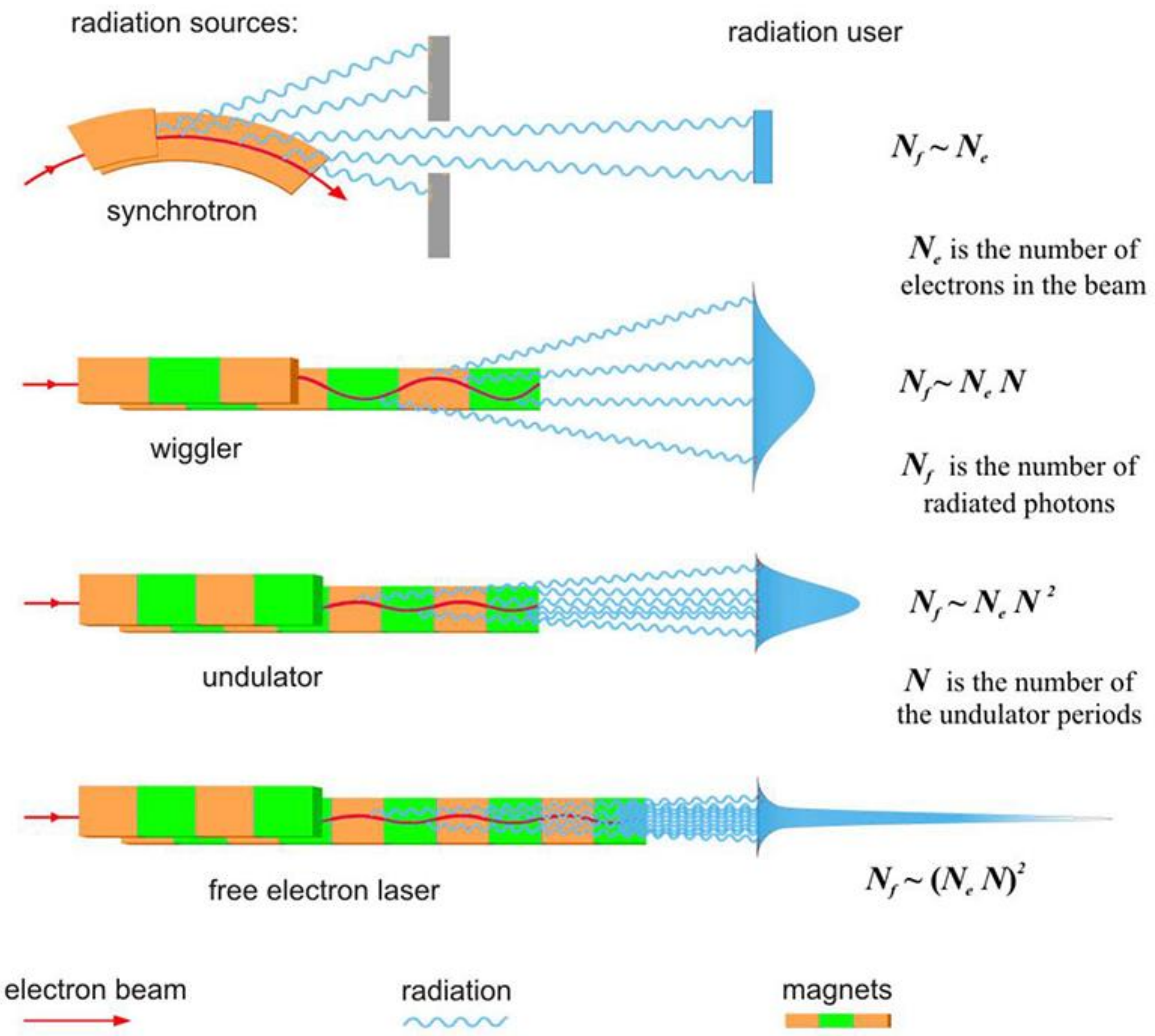
- linearity of the detectors

User defined parameters:

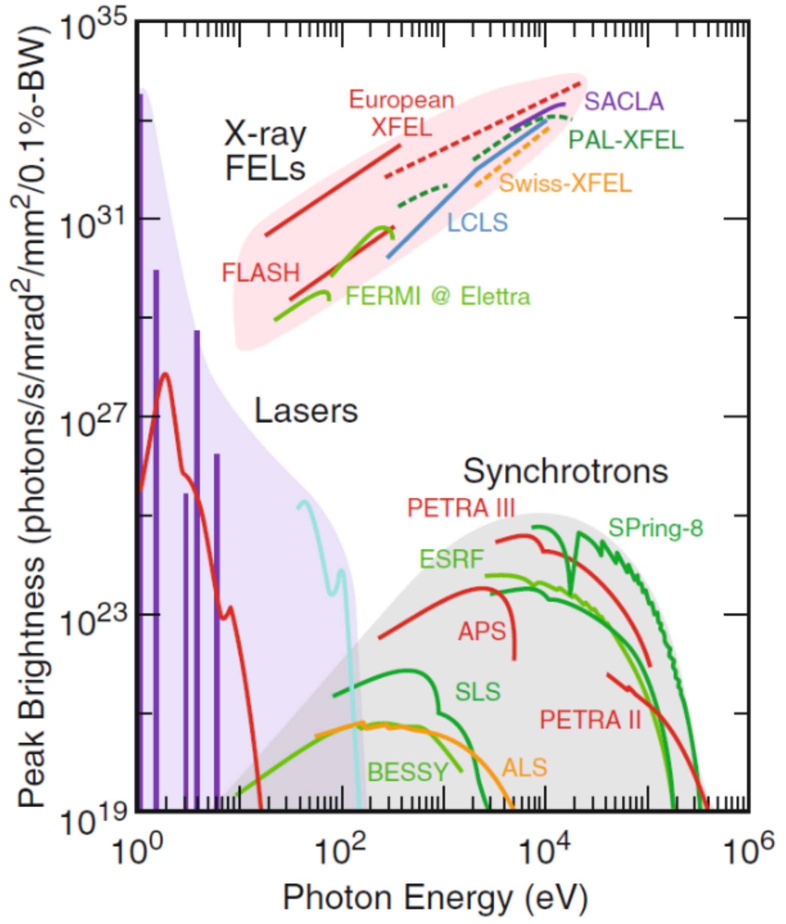
- Energy (fixed/range)
- beam-size
- collection time
- sample homogeneity/representativity

Radiation damage

Intrinsic parameters: brilliance/photon flux

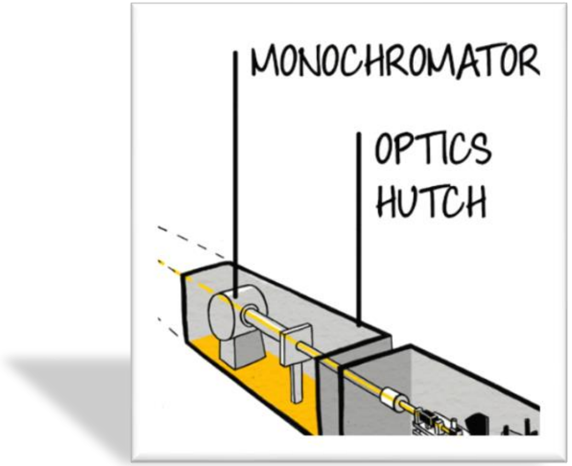


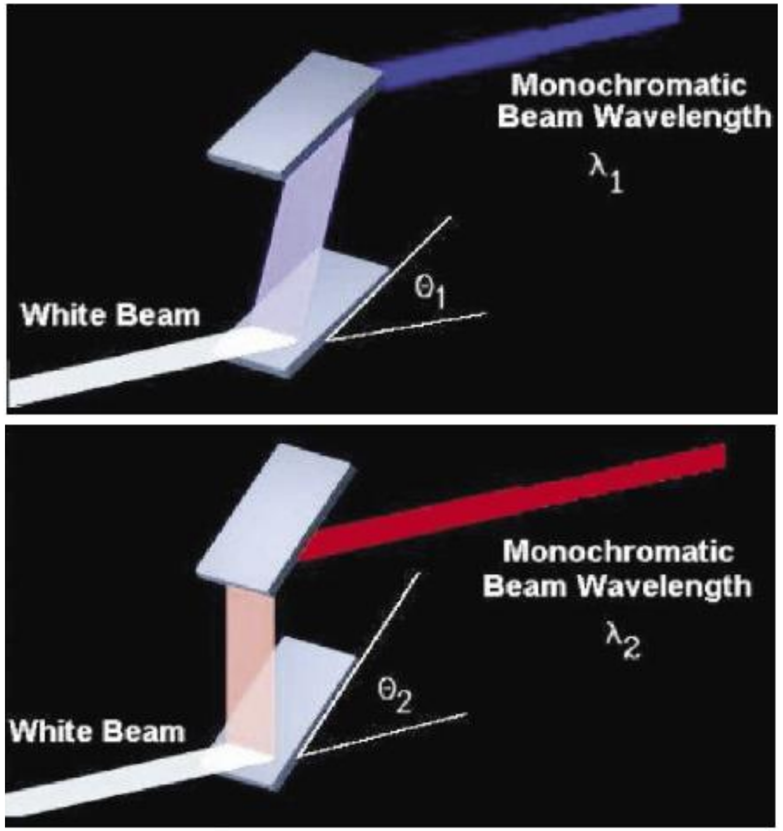
K. Zhukovsky, INTECHOPEN (2016), DOI: 10.5772/64439



X-ray Free Electron Lasers A Revolution in Structural Biology, Springer Nature (2018), DOI: 10.1007/978-3-030-00551-1

Intrinsic parameters: energy resolution



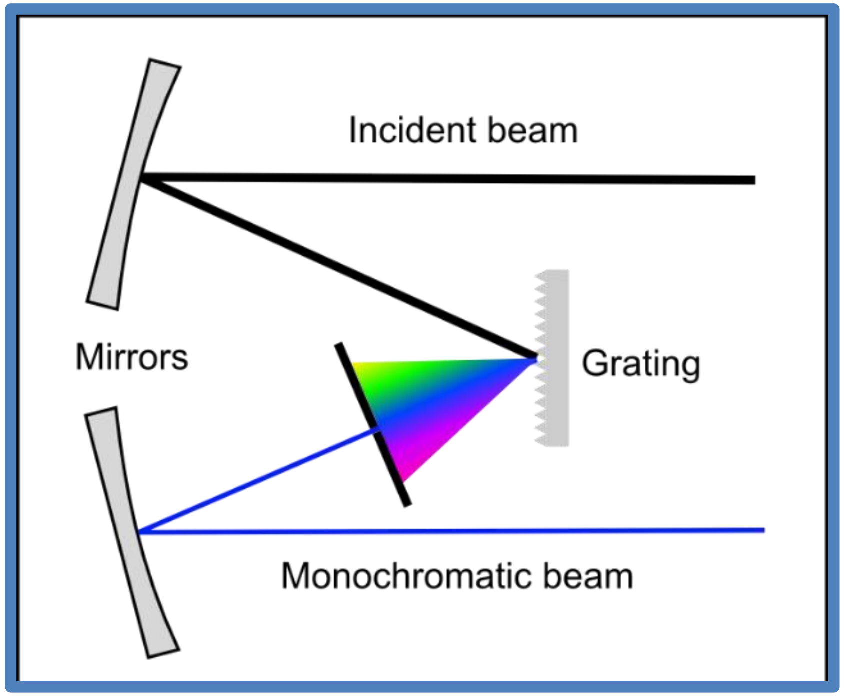


The **monochromator** selects "one" wavelength/energy from the broad band radiation generated by the SR source

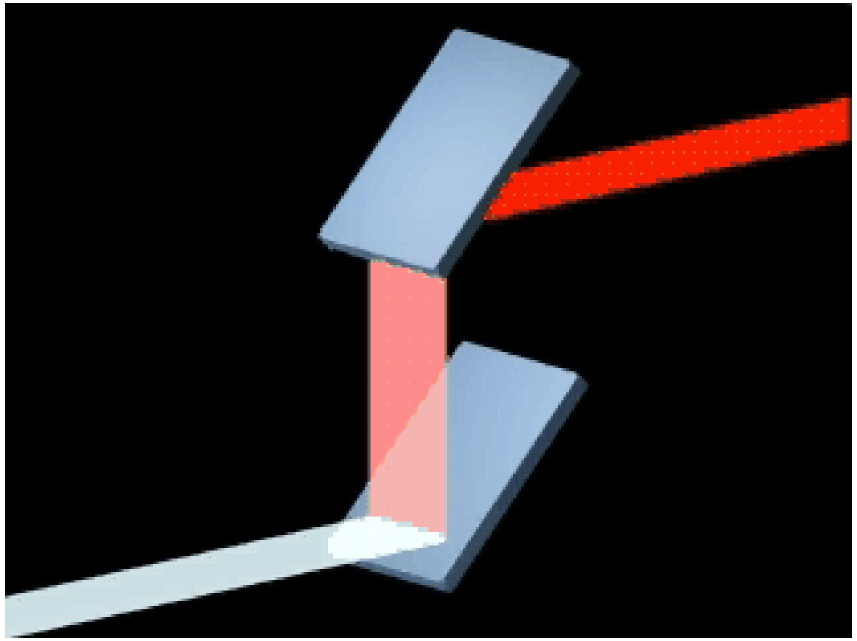
$n\lambda[Å] = 2d[Å]\sin\theta_B$	Bragg's Law: tells you what wavelength is passed at θ_B
$E[keV]= 12.39854/\lambda[Å]$	Conversion between x-ray energy and wavelength

Intrinsic parameters: energy resolution

grating monochromator
(soft X-rays)

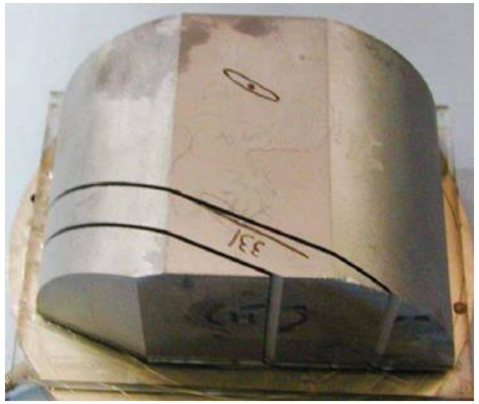


double-crystal monochromator
(hard X-rays)



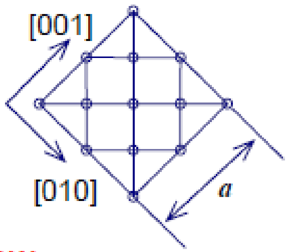
https://www.cells.es/en/beamlines/bl22-claess

Intrinsic parameters: energy resolution



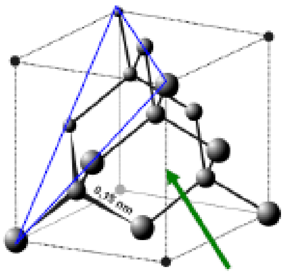
Lattice planes of silicon

Top view

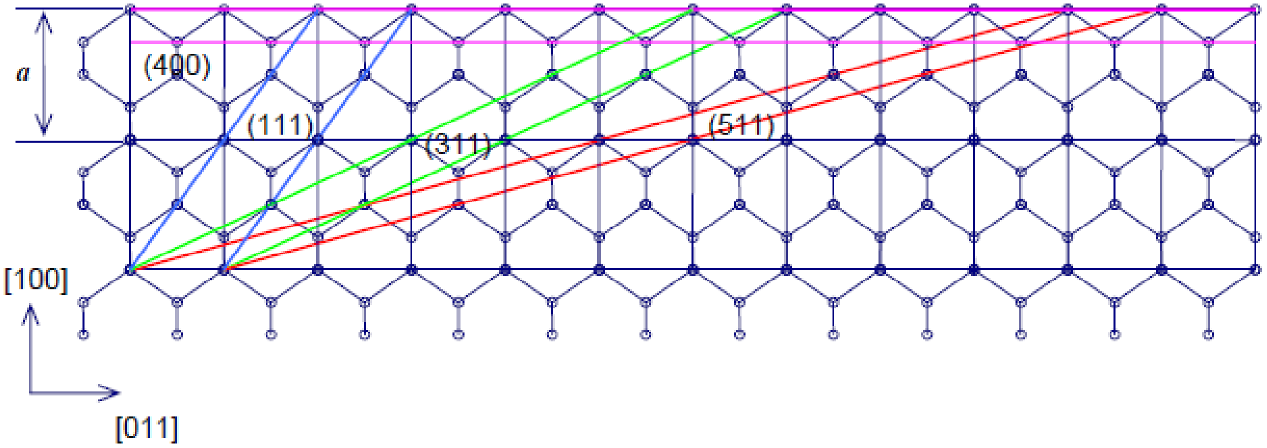


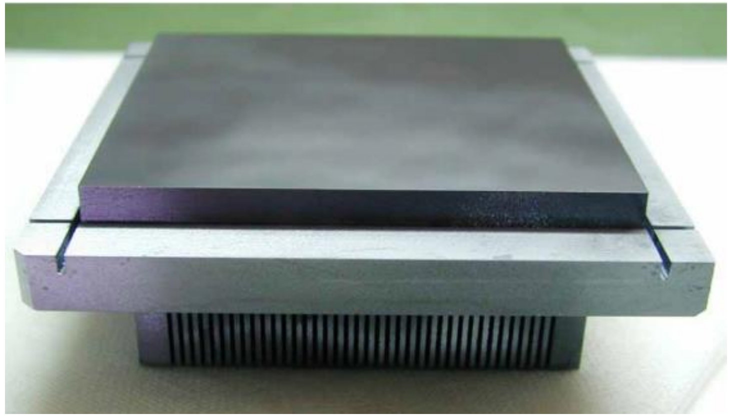
a_0 = 5.43095 Å

d-spacing	
	(400) : 1.3578 Å
	(111) : 3.1356 Å
	(311) : 1.6375 Å
	(511) : 1.0452 Å



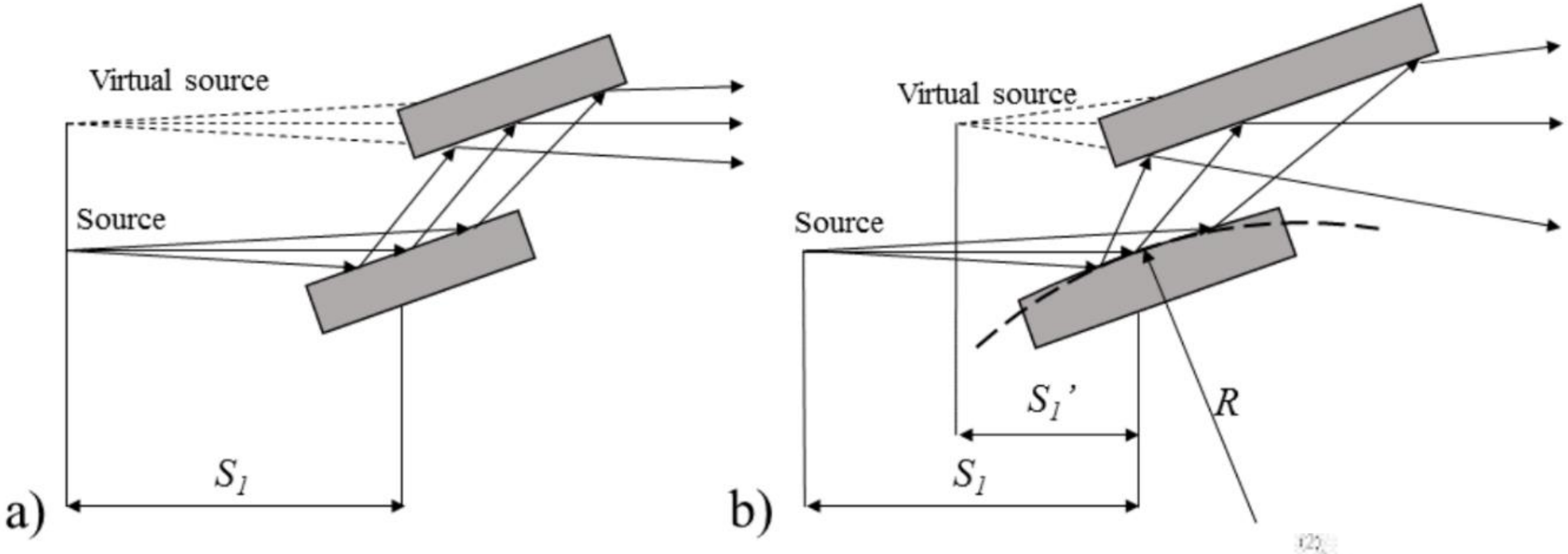
Side view



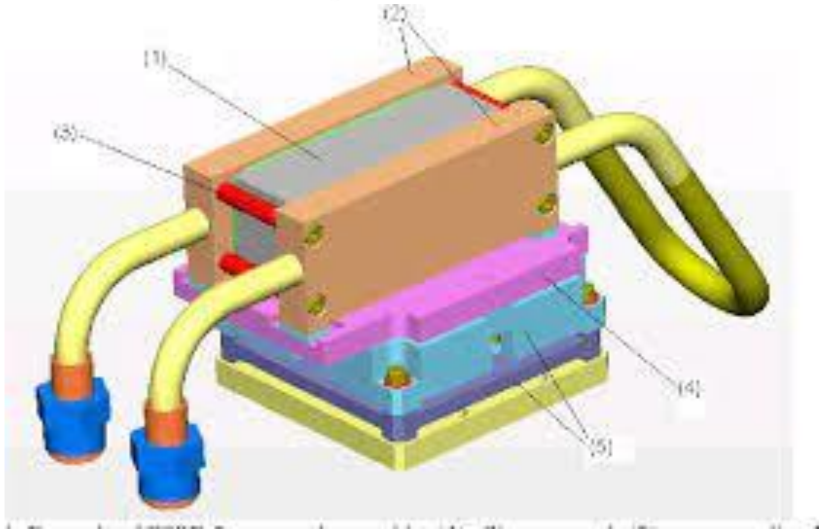


Intrinsic parameters: energy resolution

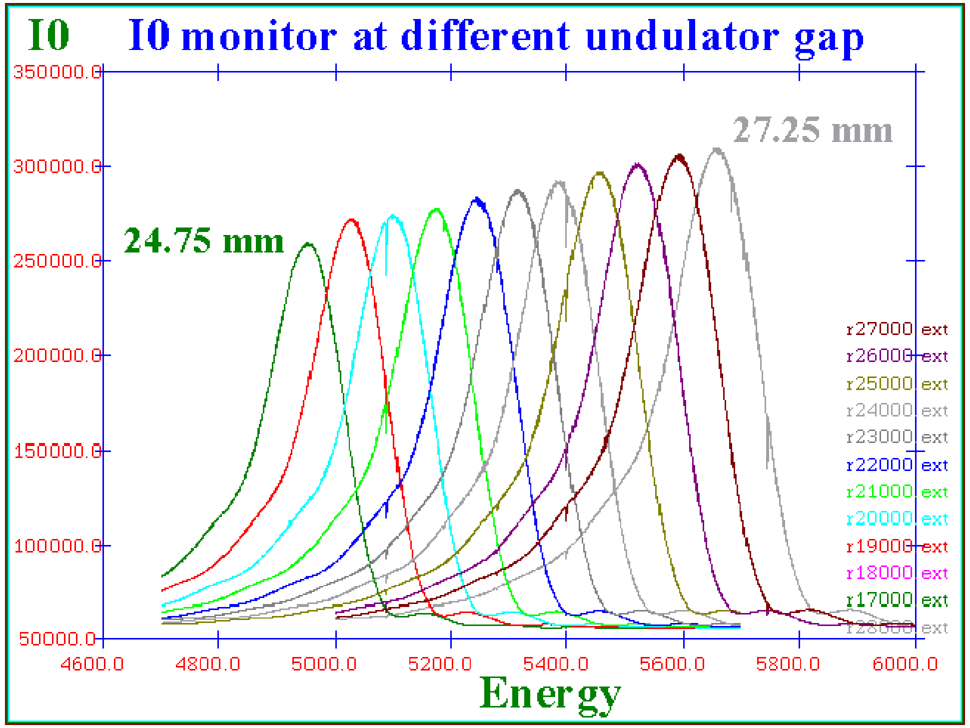
Heat bump (thermal deformation of the surface)



Liquid nitrogen or water cooled
monochromator crystals



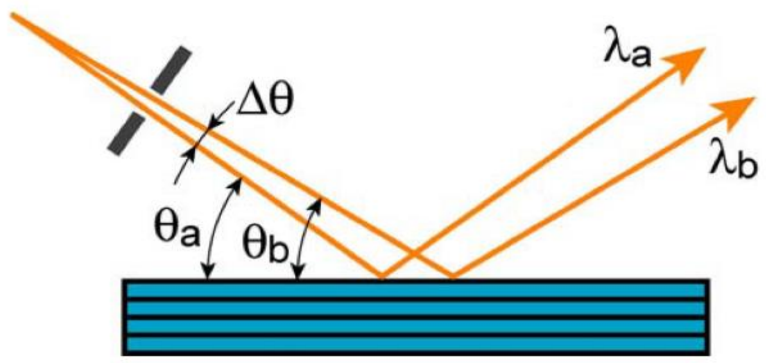
Intrinsic parameters: scanning energy

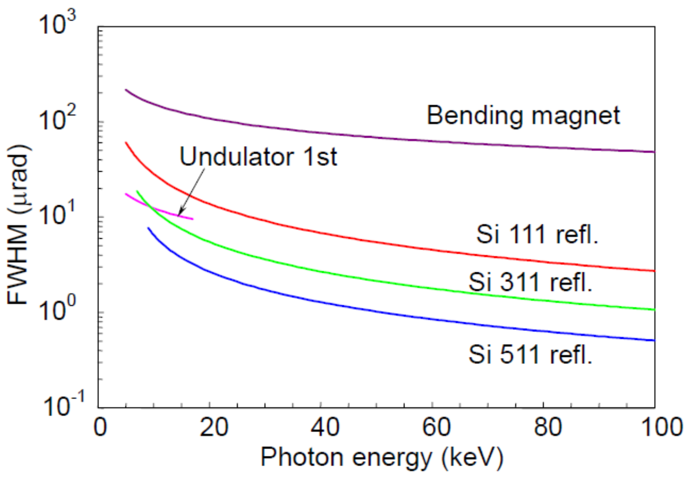


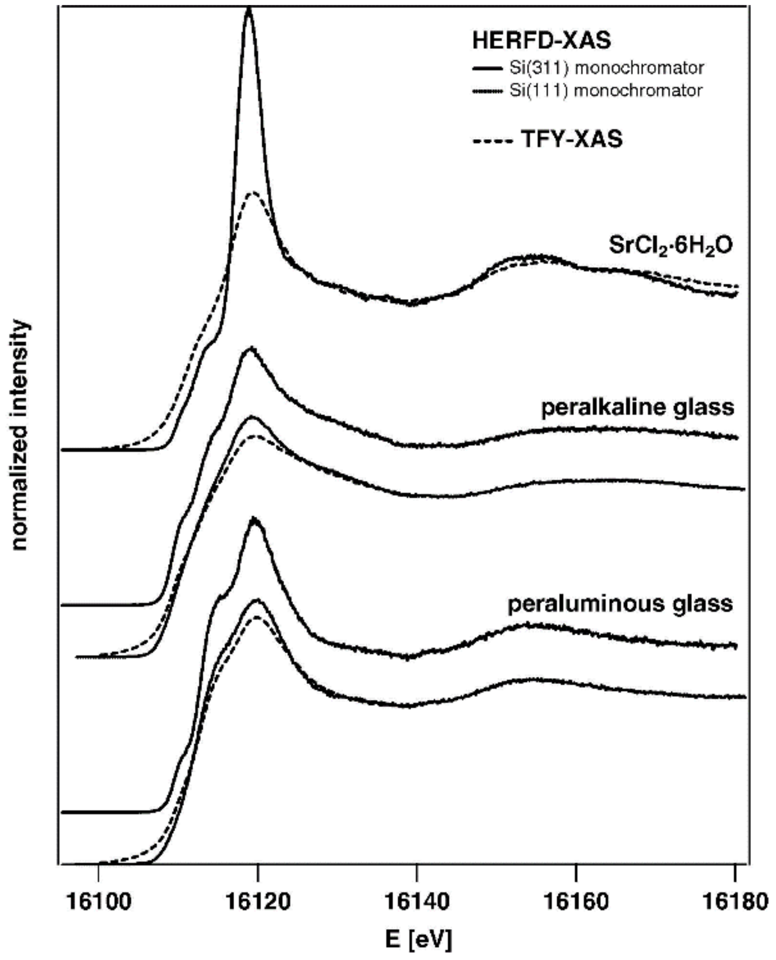
If the flux is to be maximized all while scanning the energy, the undulator gap must change as the energy is scanned.

https://www.esrf.fr/home/UsersAndScience/Experiments/EMD/ID26/Characteristics/ScanningModes.html

Intrinsic parameters: energy resolution







Geochimica et Cosmochimica Acta 142, (2014) 535-552

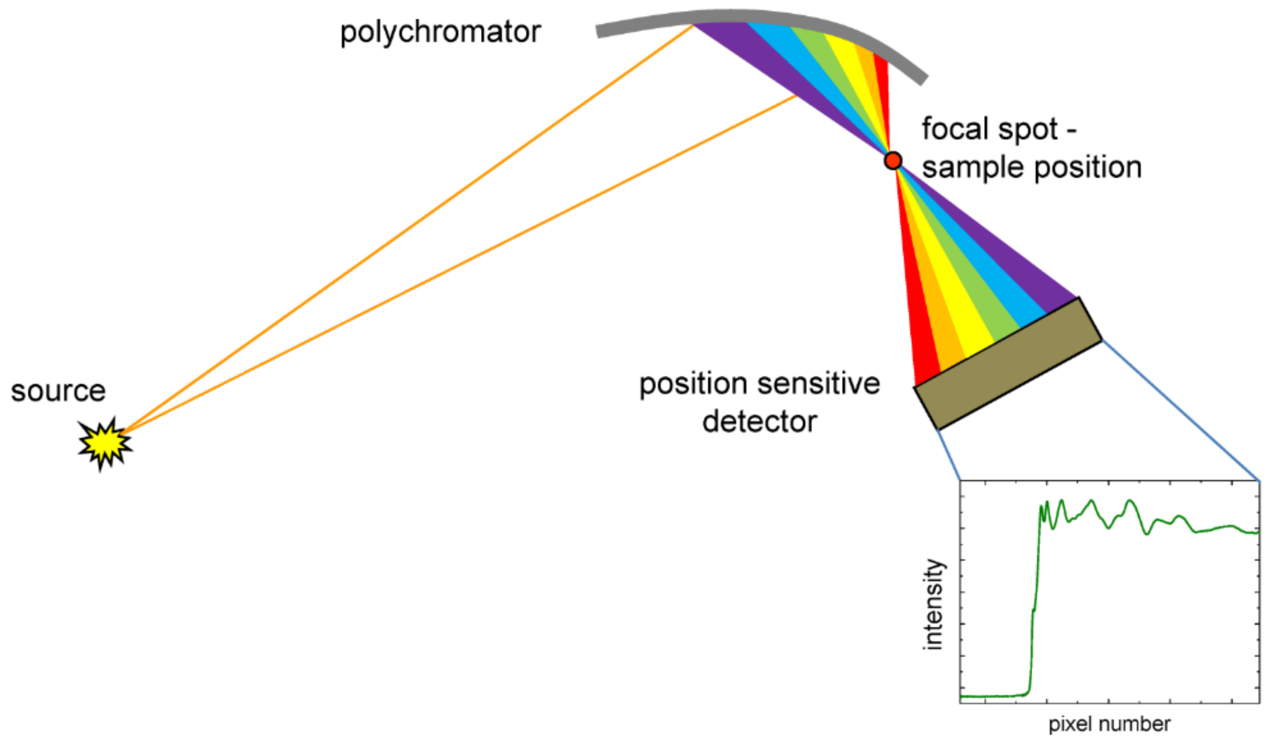
$$\frac{\Delta E}{E} = \cot\theta_B \sqrt{\omega^2 + \Delta\theta^2}$$

ω: intrinsic angular width

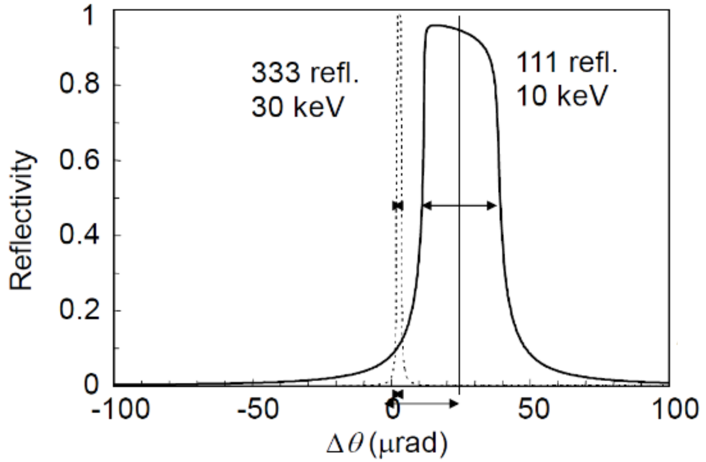
Δθ: X-ray angular divergence

T. Matsushita (2009) Cheiron School, Spring-8, Japan

Intrinsic parameters: energy resolution (polychromators)

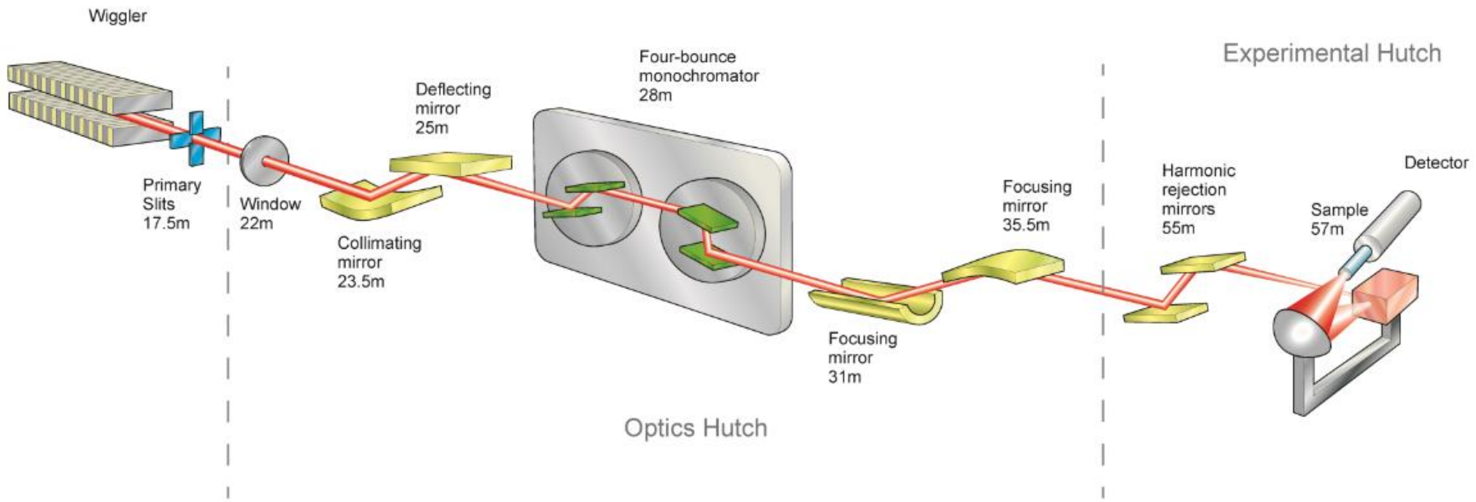


Intrinsic parameters: harmonics and mirrors



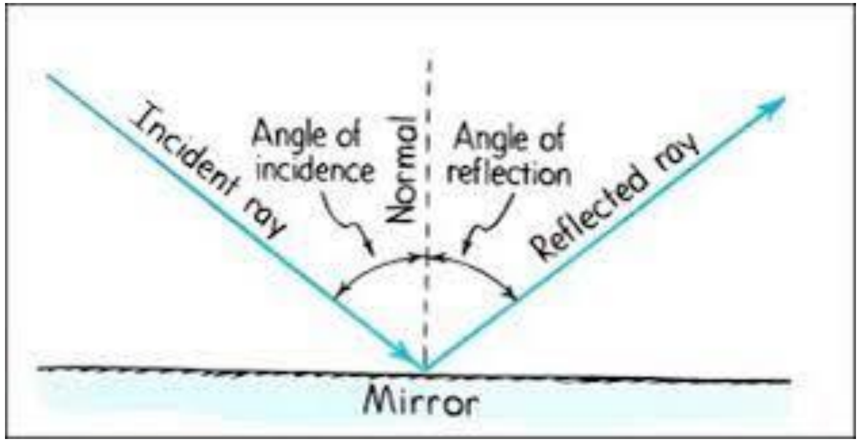
T. Matsushita (2009) Cheiron School, Spring-8, Japan

The sample will interact differently with high energy photons and the linearity might be compromised.



https://www.diamond.ac.uk/Instruments/SpectroscI20opy/I20/XAS_XES_Branchline/Specification.html

Intrinsic parameters: harmonics and mirrors



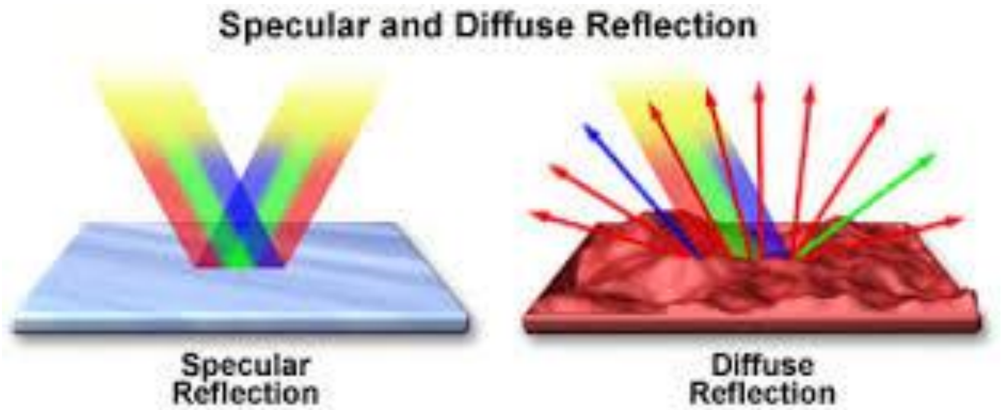
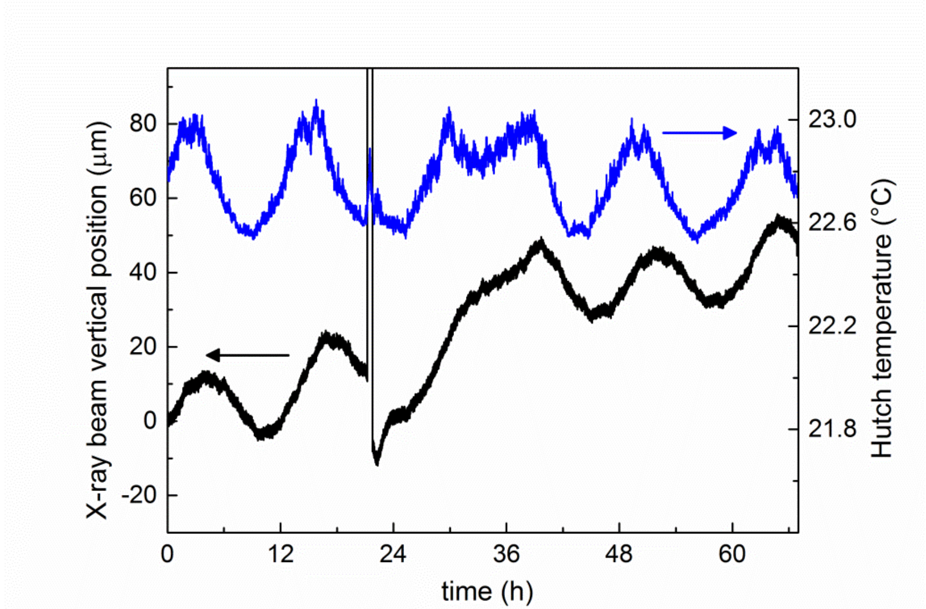


Figure 3

It is important to <u>reduce the **slope errors**</u> on the mirrors surface to get the appropriate beam collimation and beam focusing.

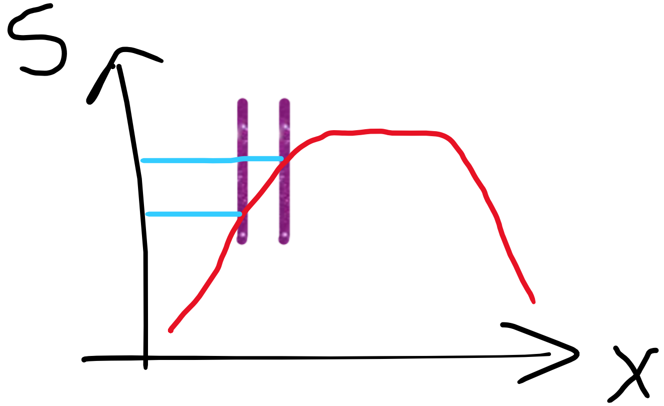
$\theta_c[°] = 1.6\ \lambda[Å]\ \sqrt{\rho}\ [g/cm^3]$	Critical angle for mirror reflection of x-rays
$R_m[m] = (2/\sin\theta)[F_1F_2 / (F_1 + F_2)]$	Bending radius for meridional focus
$R_s[m] = R_m \sin^2\theta$	Bending radius for sagittal focusing
$M = F_2/F_1$	Magnification (demagnification) factor definition

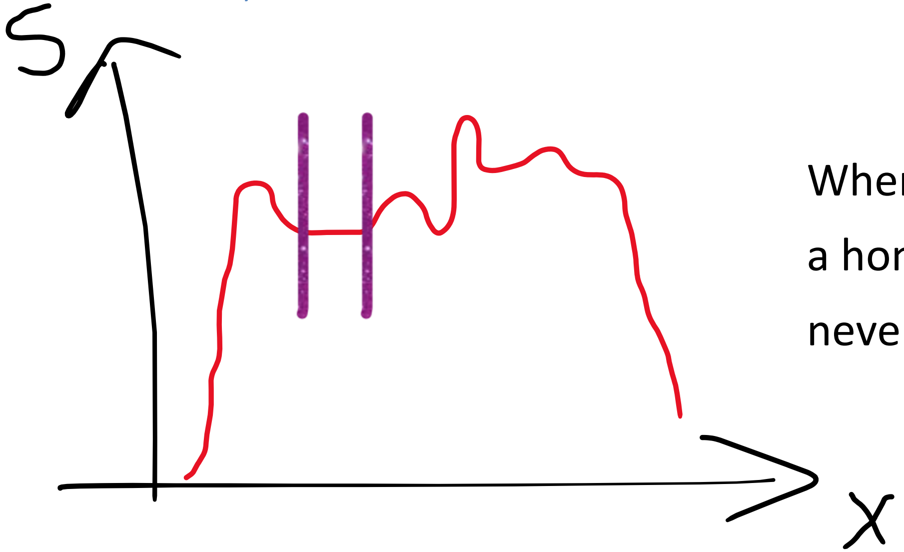
Intrinsic parameters: drifts and vibrations



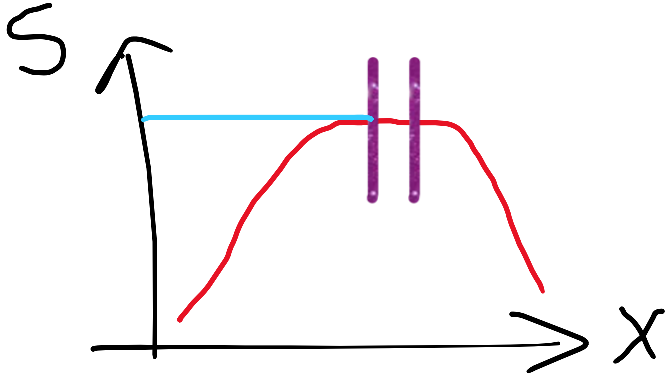
R. Boada & S. Diaz Moreno. Intern. Tables of Cryst.: (Vol. I). IUCr, 2020. DOI: 10.1107/97809553602060000116

Correlation between the temperature of the optics hutch and the vertical position of the X-ray beam at the sample point.

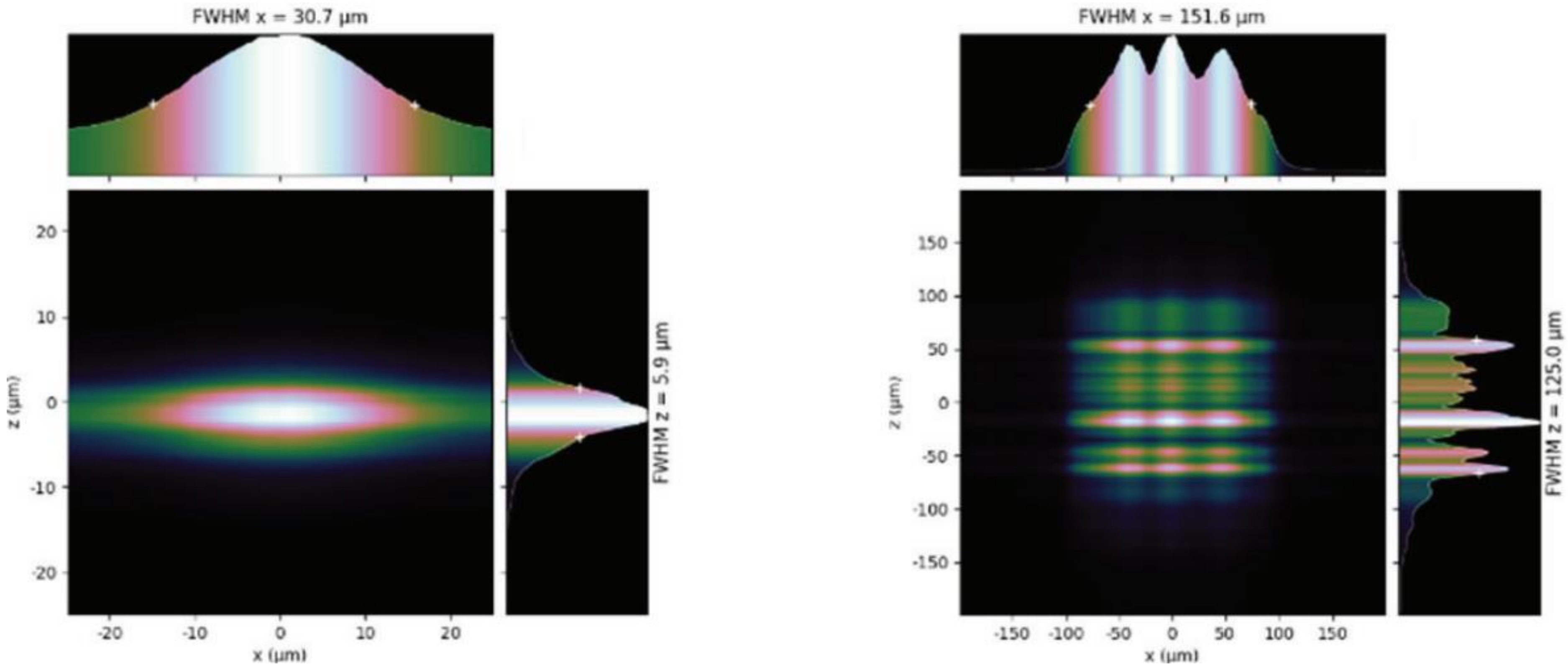




When scanning THE sample choose a homogeneous part of the sample, never measure on the edge.



Intrinsic parameters: beam homogeneity and beam focus



The beam might be focused or unfocused depending on the application, sample homogeneity (grain size, thickness and heterogeneity), radiation damage… however, a stripped or patterned beam is usually not the best choice…

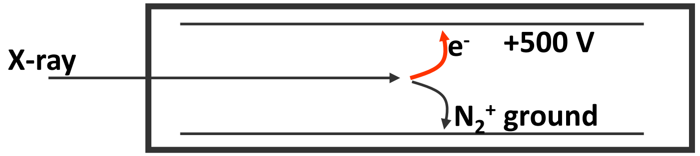
Intrinsic parameters: set-up reproducibility

Sources of uncertainty:

- Compensation mechanisms of mirrors (sagittal compensation, benders)
- Mirrors angular alignment
- Alignment of X-ray monochromator
- Harmonic content
- ID gap and monochromator synchronization

Not-beam related: linearity of the detectors

Gas ionization detectors ("ionization chambers")



$$\text{Flux [photons/sec]} = \frac{I_{absorbed}[\text{C/sec}] \times \text{Eloss}[\text{eV/e}^-]}{(1-e^{-\mu*\rho*L(cm)}) \times (1.602\times10^{-19}[\text{C/e}^-]) \times (\text{Energy[eV]})}$$

μ = absorption coefficient, ρ = density

Energy-loss values for various gasses:

N_2: 34.6 eV/e^-
Ar: 26.2 eV/e^-
Air: 22.7 eV/e^-
He: 41.5 eV/e_-

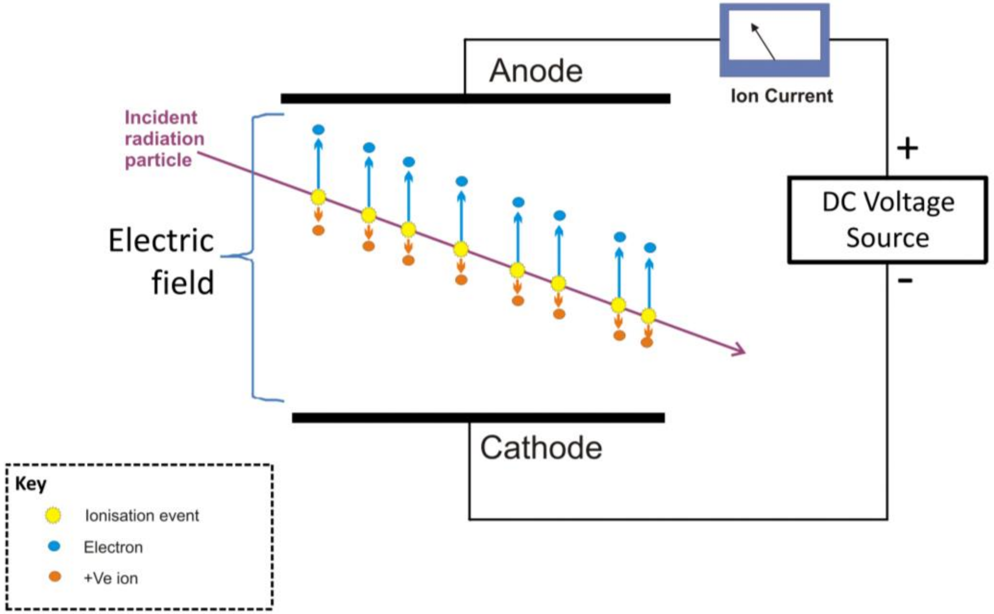
Gas selection:

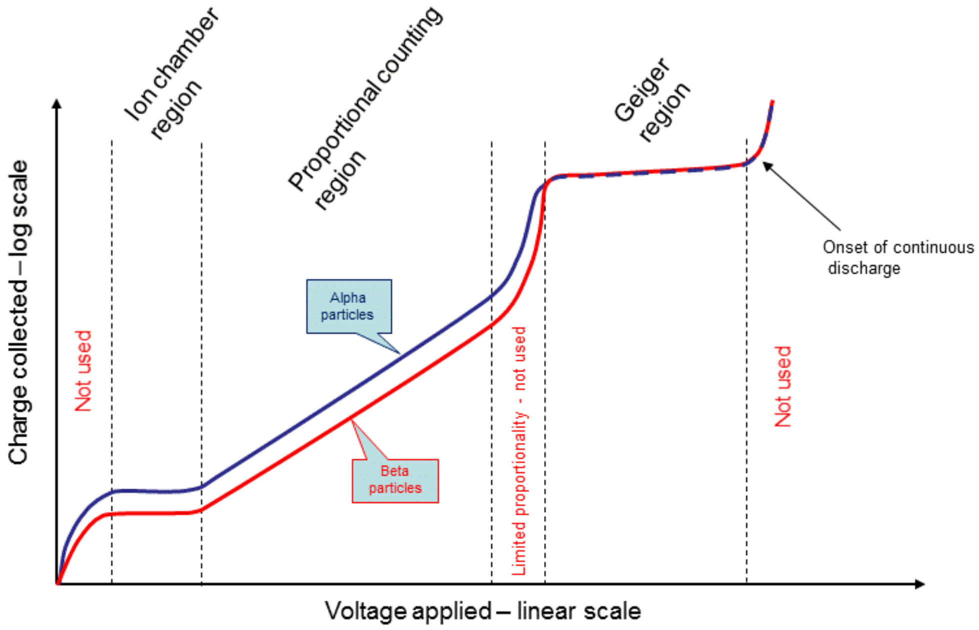
< 5 KeV: He
5 – 15 KeV: N2
> 15 keV: Ar

J.S. Lezama Pacheco and J. R. Bargar, Synchrotron X-Ray Absorption Spectroscopy Summer School (2011)

Not-beam related: linearity of the detectors

Gas ionization detectors ("ionization chambers")



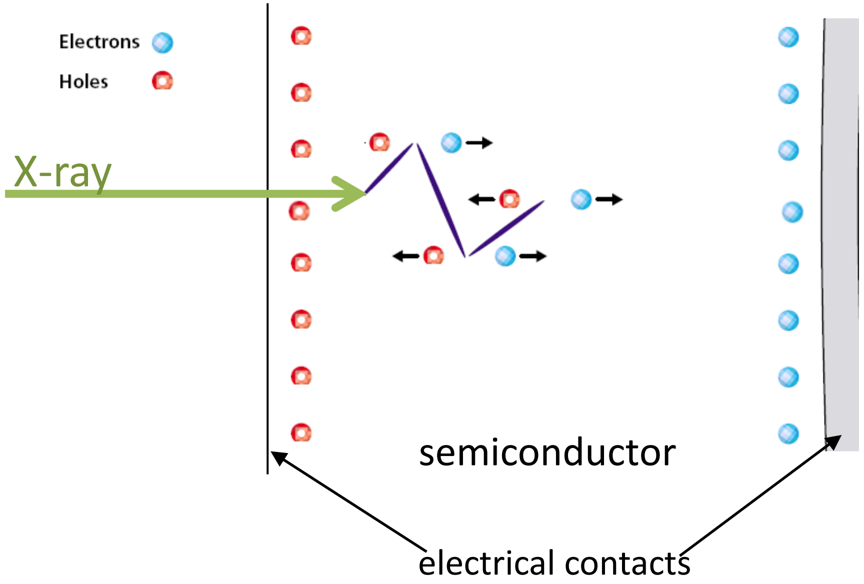


Doug Sim
https://en.wikipedia.org/wiki/Ionization_chamber

Not-beam related: linearity of the detectors

Solid State detectors (semiconductors)

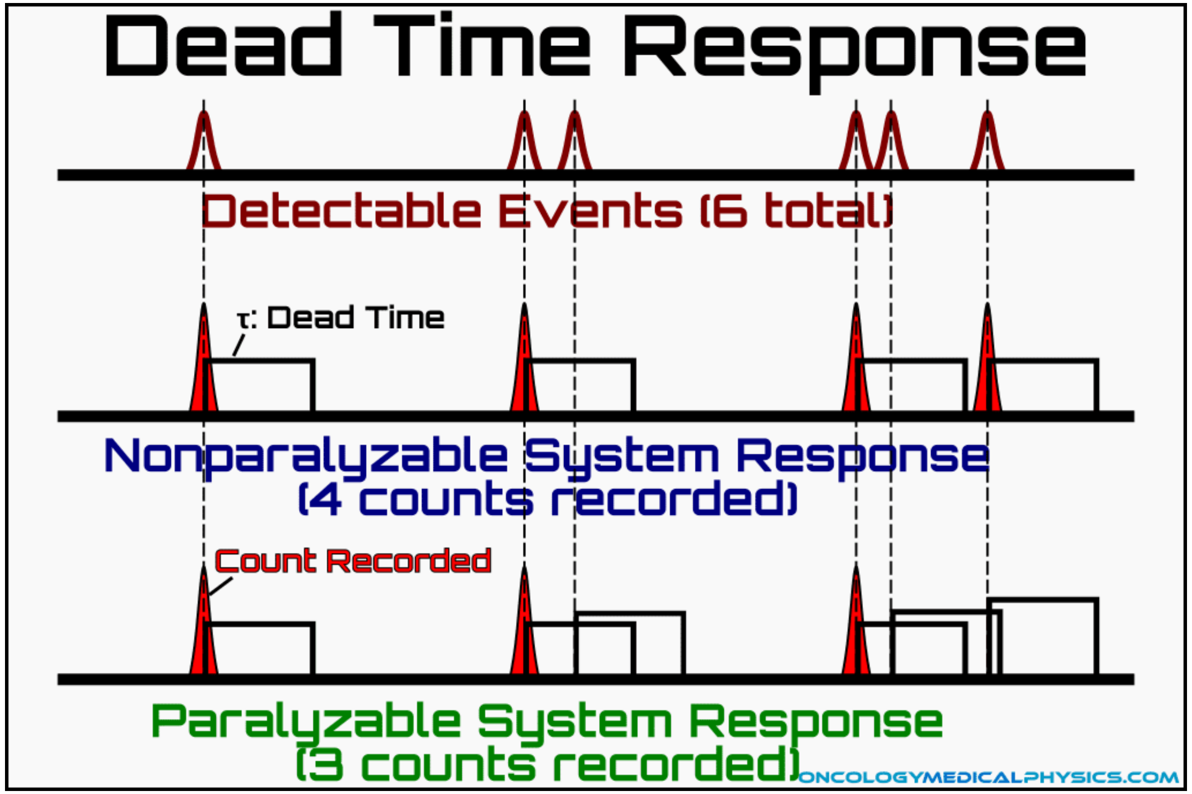
Semiconductor material (e.g. crystal of Si or Ge) with X-ray transparent contacts, applied electric field depletes bulk of thermally generated free charge.

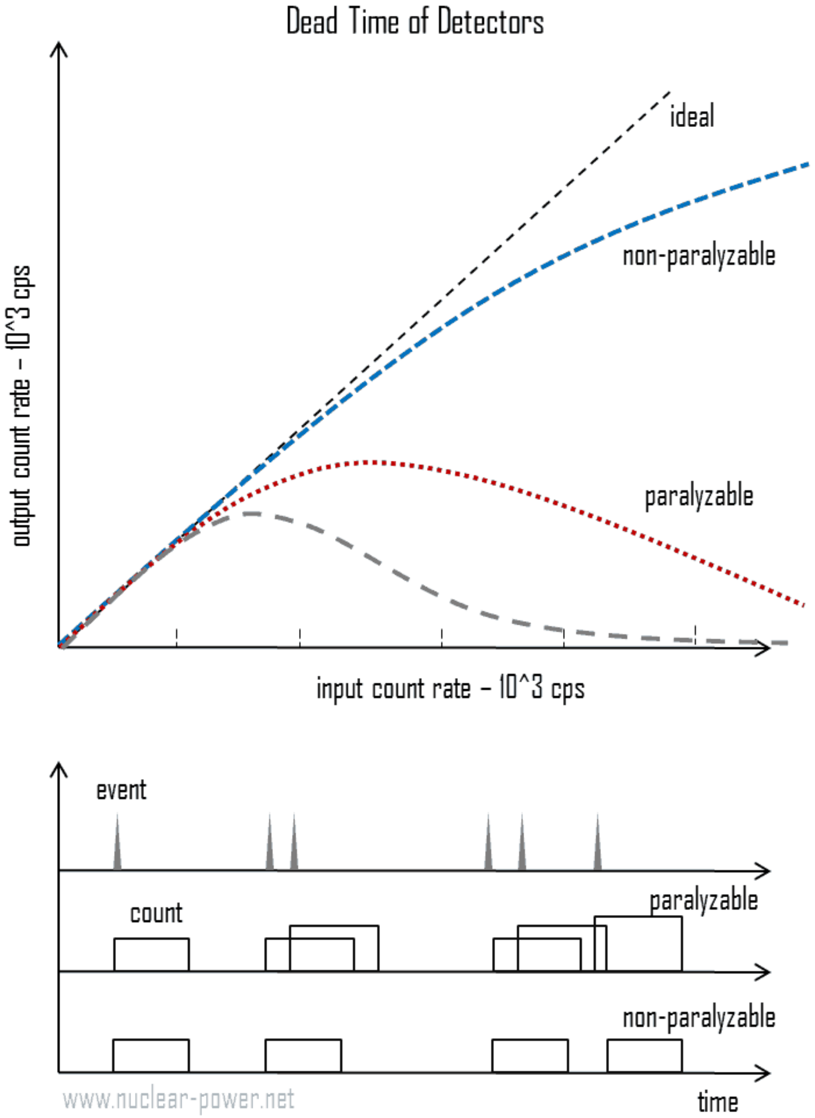


- photoelectric conversion of an X-ray creates 'hot' electrons which rapidly thermalize (~psec),
- hole, electron charges drift in applied field towards electrodes
- electrical signal develops *while the charge drifts in the bulk*

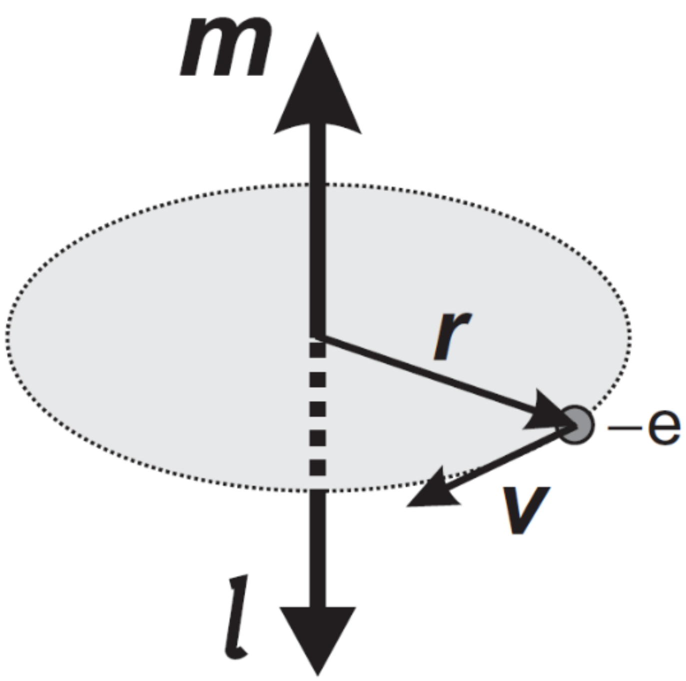
Not-beam related: linearity of the detectors

Solid State detectors (semiconductors)





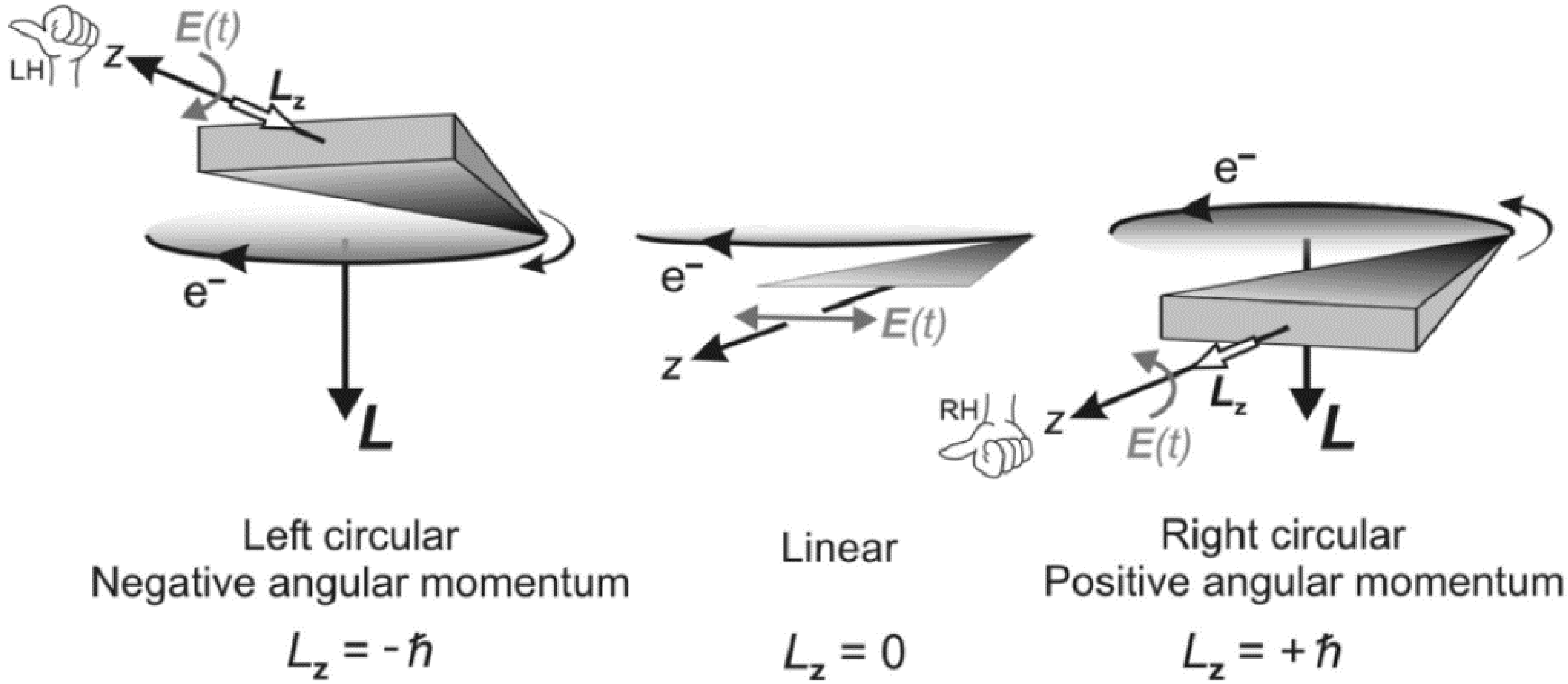
Polarization of the beam



the angular momentum of a circulating electron is defined according to the **right hand rule**

J. Stöhr and H.C. Siegmann, Magnetism: From Fundamentals to Nanoscale Dynamics (Springer , 2006)

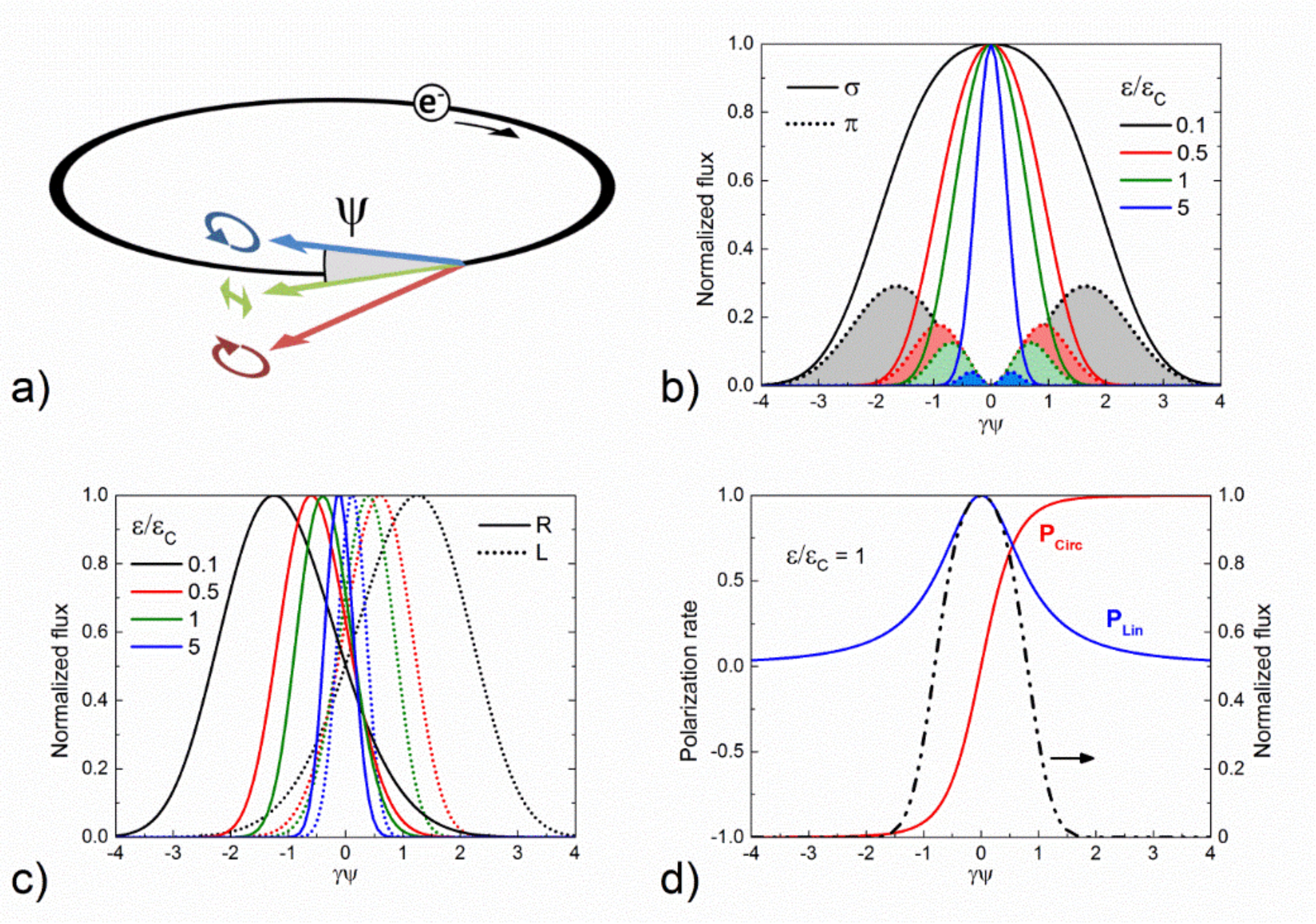
Polarization of the beam



J. Stöhr and H.C. Siegmann, Magnetism: From Fundamentals to Nanoscale Dynamics (Springer , 2006)

Polarization of the beam

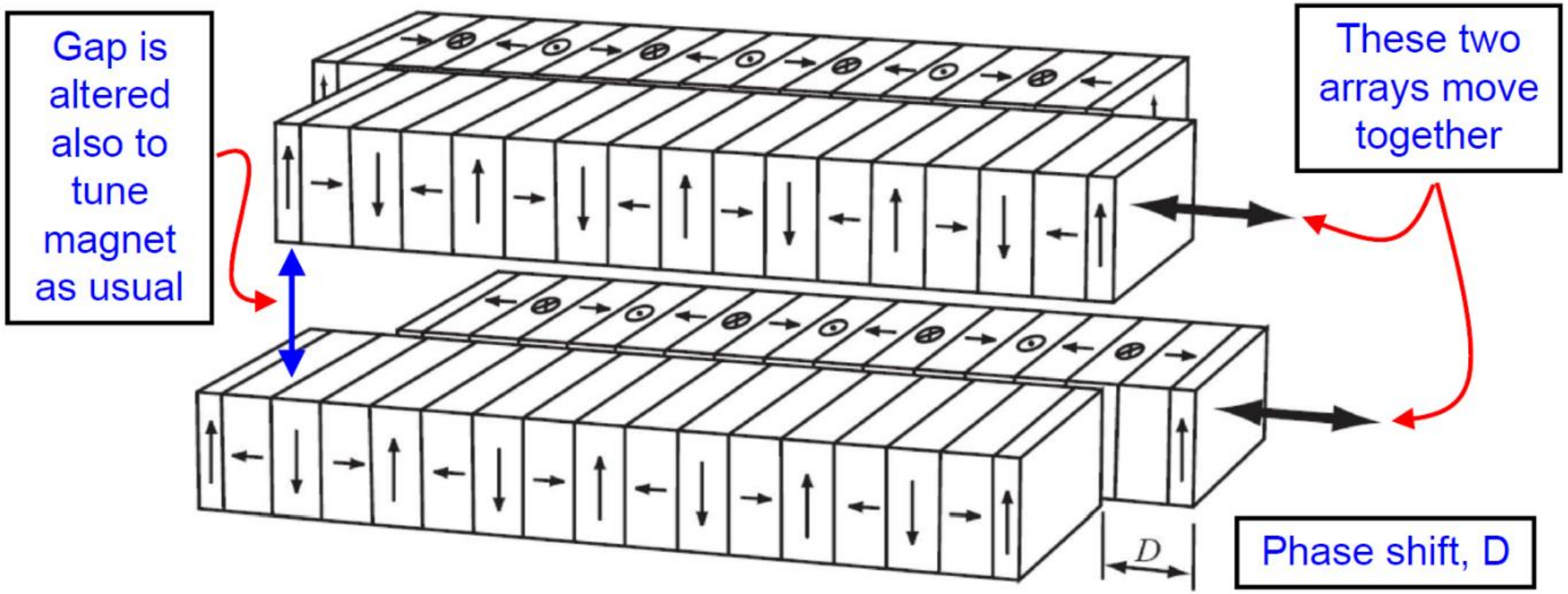
Circular and linear polarization rate: vertical angular distribution



R. Boada & S. Diaz Moreno. Intern. Tables of Cryst.: (Vol. I). IUCr, 2020. DOI: 10.1107/97809553602060000116

Polarization of the beam

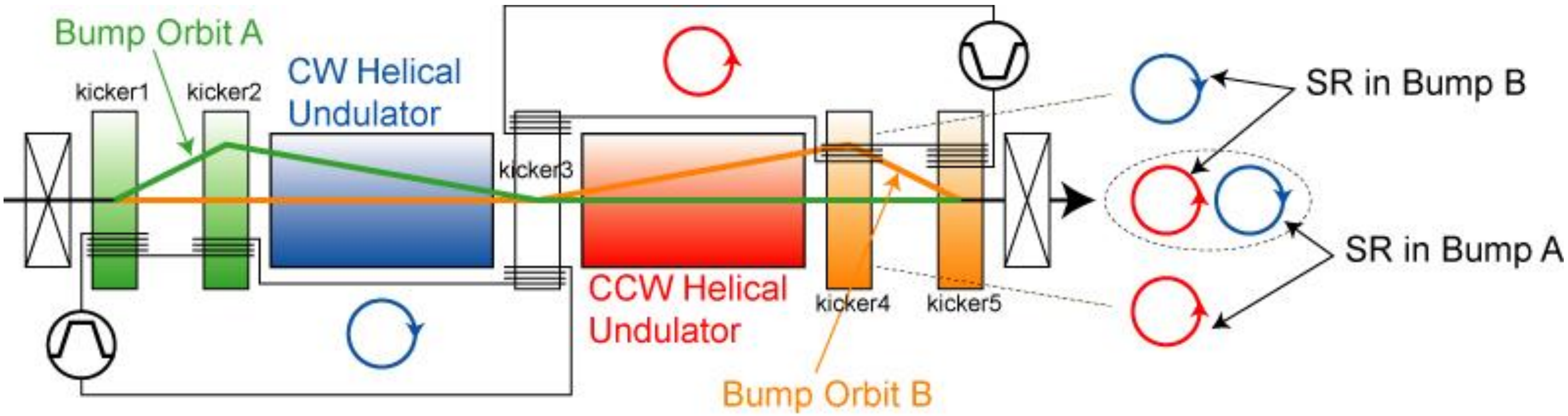
Helical undulator: permanent magnets (short periods)



J. Clarke, Undulator magnet designs, Lecture 4 at Cockcroft Institute (2014)

Polarization of the beam

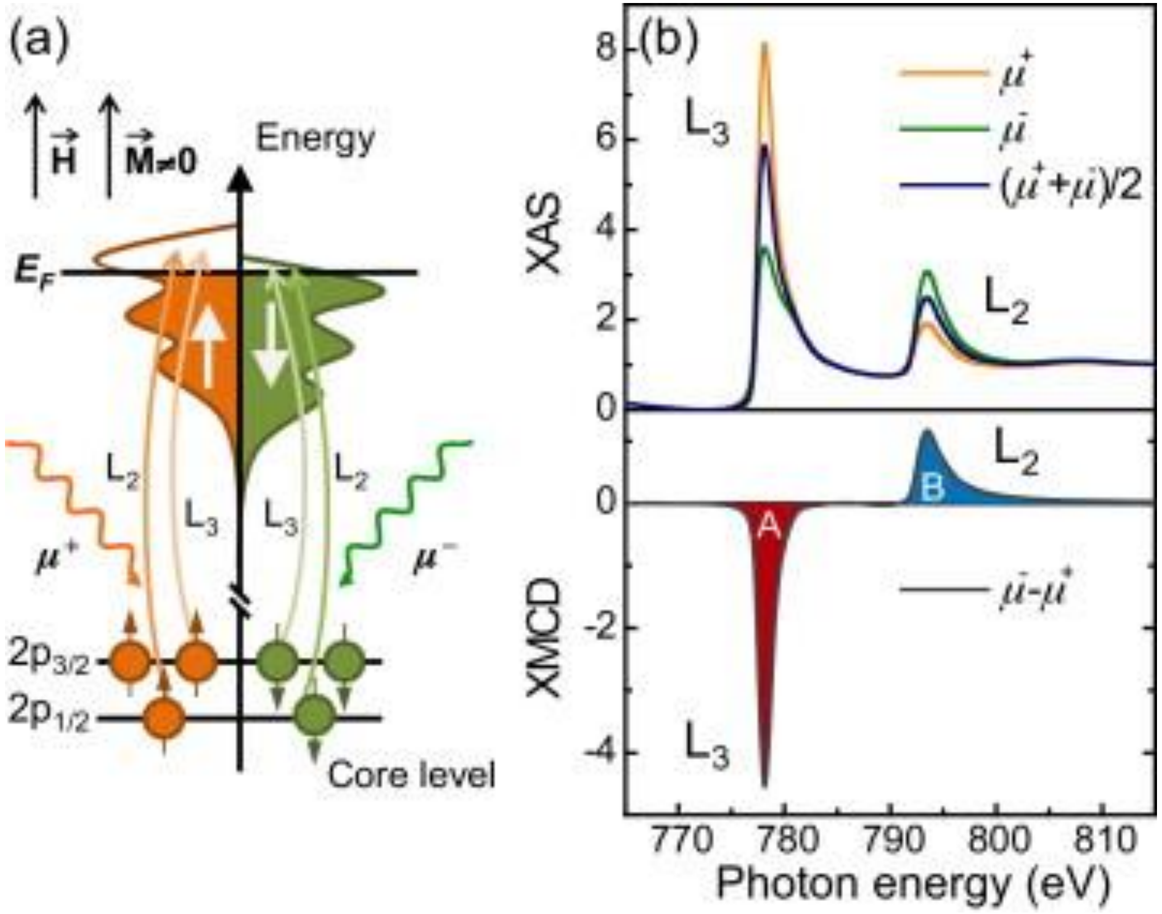
Helical undulator and kicker magnets



http://www.spring8.or.jp

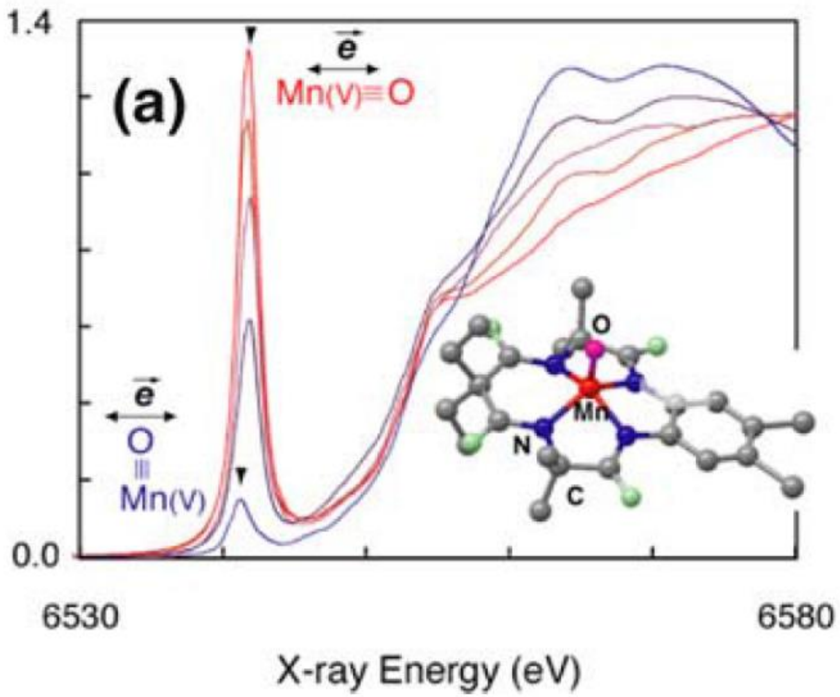
Uses of X-ray polarization…

X-ray magnetic circular dichroism (XMCD)



G. der Laan & A. I. Figueroa, Coord. Chem. Rev. 277–278 (2014) 95-129

Polarized XAS



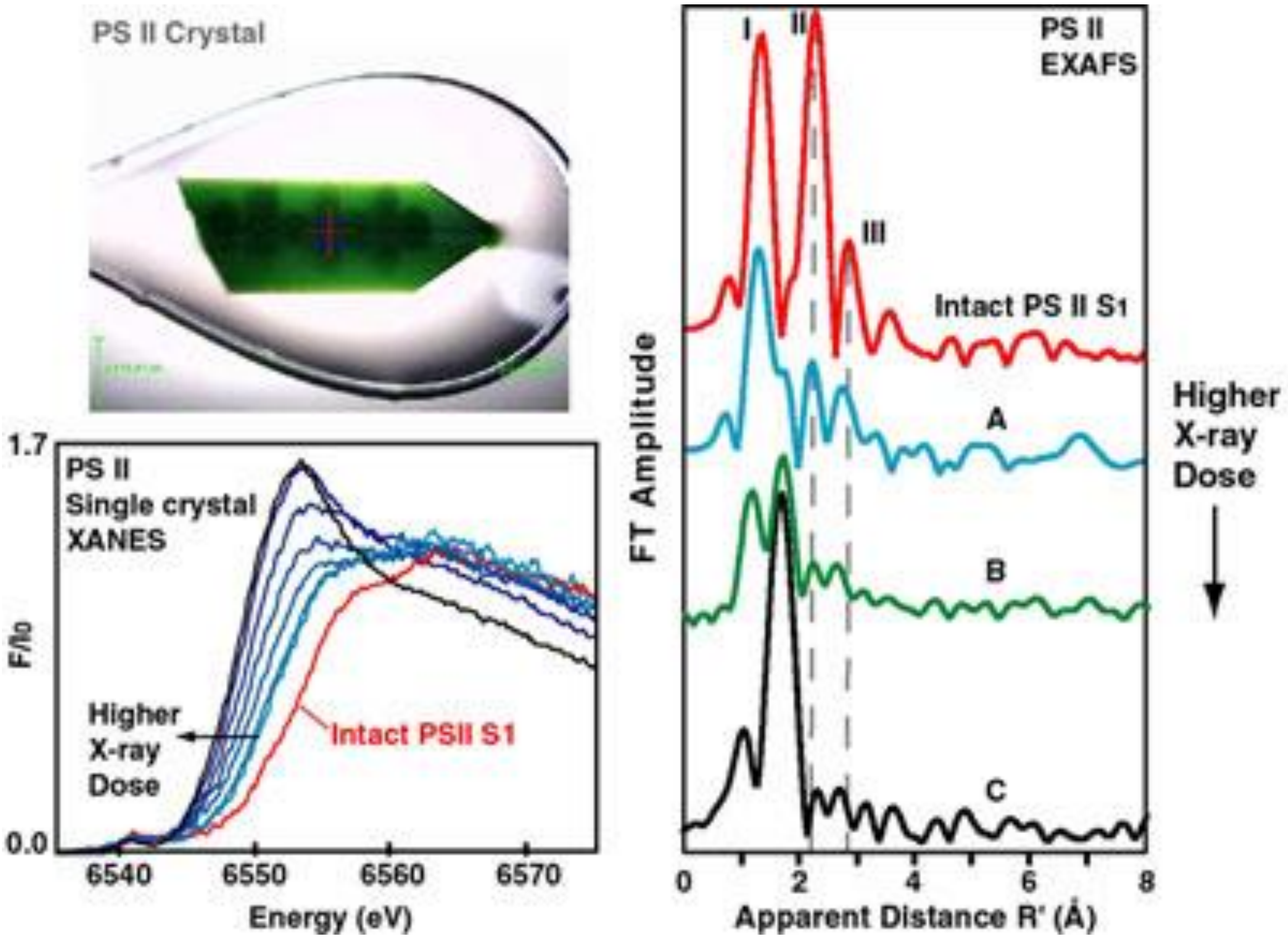
J. Yano et al., Photosynth Res (2009) 102:241–254

User defined parameters

- Energy (fixed/range)
- beam-size (µm – mm)
- collection time (µs – s)
- sample homogeneity/representativity

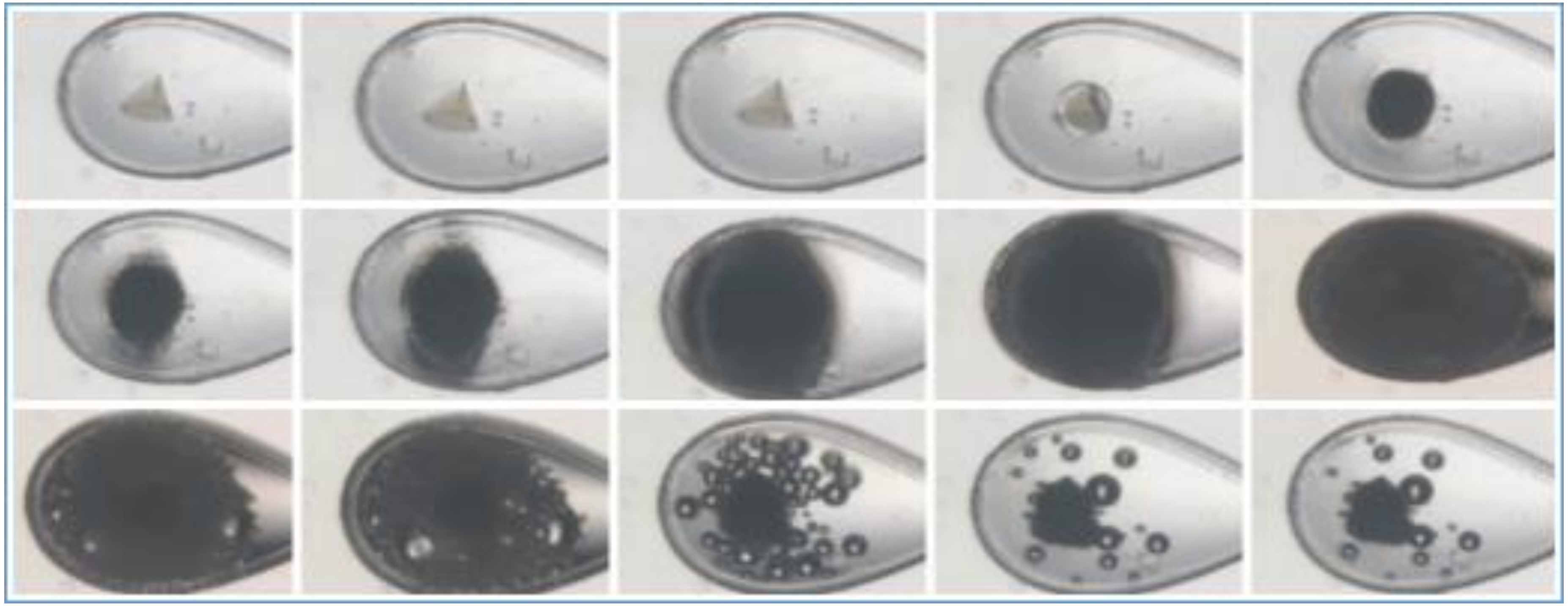
Radiation damage

Photoreduction



Radiation damage

Macromolecular crystallography (MX)



"Collection before destruction"

Radiation damage

Macromolecular crystallography (MX)

Cryogenic temperatures

Automatic loading

TUNTWIN's Workshop

شكرا

Merci!

Thank you!

¡Gracias!

CONTACT DETAILS:

Roberto Boada
Associate Professor
Chemistry Department, Science Faculty
Campus UAB, 08193, Bellaterra
Barcelona, Spain.

Tel.: 0034 93 581 4638
Email: roberto.boada@uab.cat

Commission européenne

Funded by the Horizon 2020 Framework Programme of the European Union under the GA N° 952306

TUNTWIN