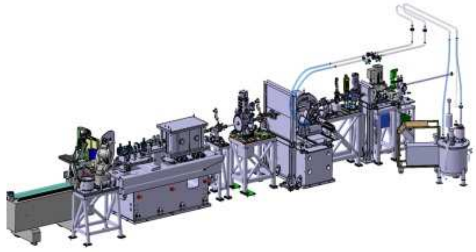
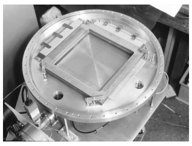
SRS – 1978-1991

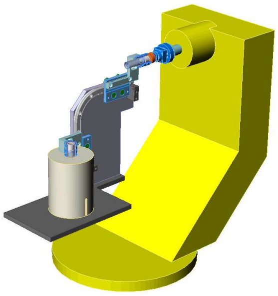
The SRS at Daresbury was the first dedicated SR source which came on line in 1981.

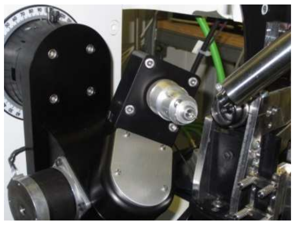
EMBL Grenoble, 1991-2002

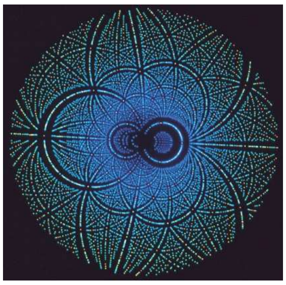
SOLEIL 2002-present

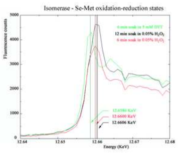


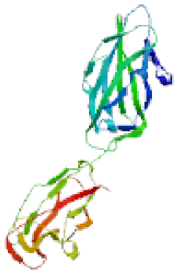


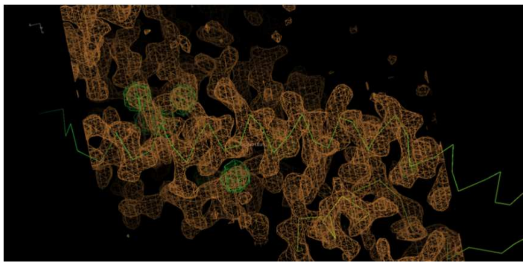




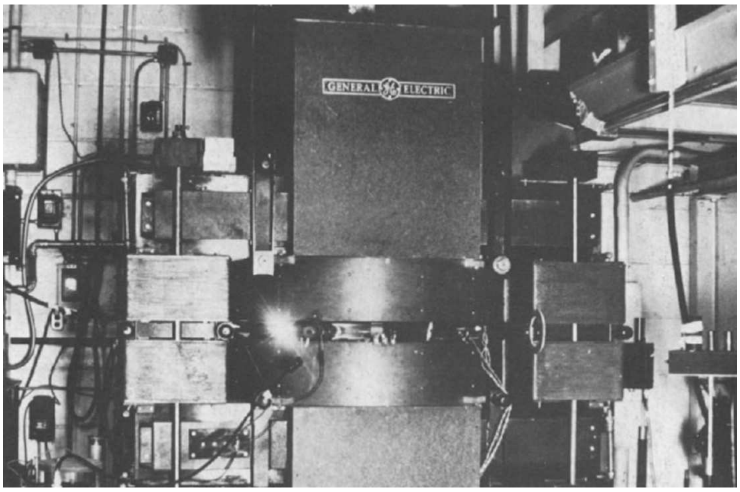








Synchrotron Radiation for Integrative Structural Biology



ReNaFoBis Summer School, Ile d'Oleron, June 1- 6 June 2014.

L.G.Parratt, Rev. Sci. Inst (1959) 30, 297-299
THE REVIEW OF SCIENTIFIC INSTRUMENTS
VOLUME 30, NEW SERIES JANUARY-DECEMBER, 1959
A publication of the
AMERICAN INSTITUTE OF PHYSICS
335 East 45 Street, New York 17, New York

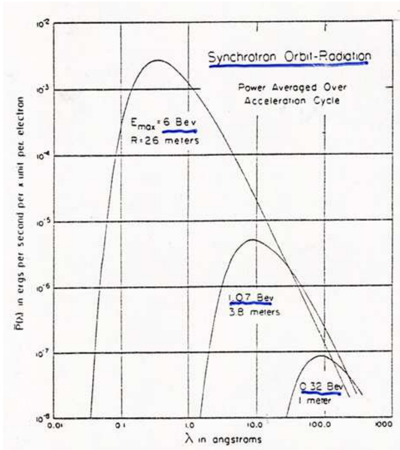
Use of Synchrotron Orbit-Radiation in X-ray Physics*

L. G. PARRATT
Cornell University, Ithaca, New York
(Received December 1, 1958)

It is well known by now that intense x-radiation is emitted by the centripetally accelerated electrons in the orbit of a high-energy synchrotron.[1] Comparison is made here of the prospective usefulness of this radiation in the range of wavelengths 0.1 to 20 Å with the x-rays obtainable from a conventional x-ray tube.

much greater intensity would be produced by applying a sudden high magnetic field pulse in a small length of the path of the high-energy electron. This, for instance, could be done as a "kink" in the synchrotron orbit, **or** at the output of a linear accelerator.

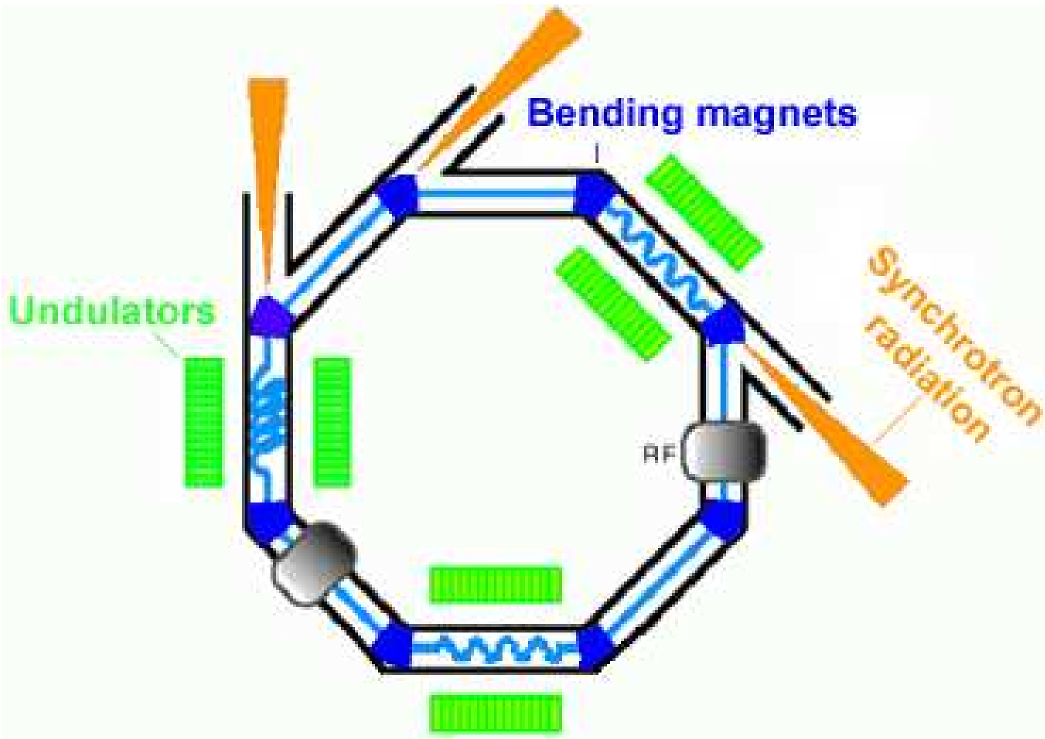
If one uses the radiating time of one "bunch" of electrons as it passes in orbit, the maximum rate for useful irradiation of material with the 6-Bev beam is about 10^{20} photons per second at 1 Å;



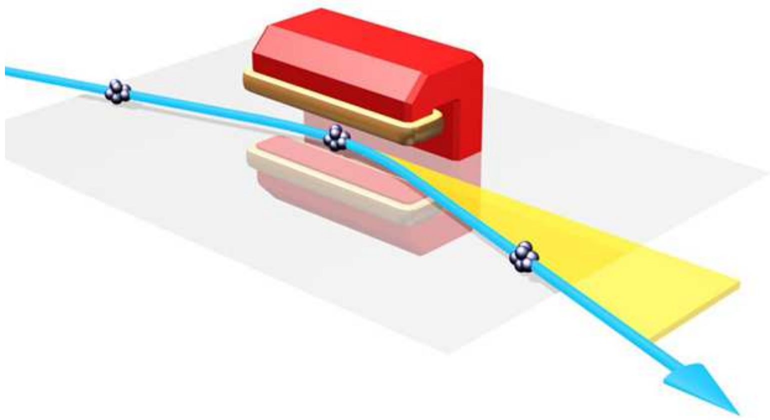
Synchrotron Orbit-Radiation Power Averaged Over Acceleration Cycle E_{max}=6 Bev R=26 meters 1.07 Bev 3.8 meters 0.32 Bev 1 meter λ in angstroms 6 BeV 1.07 BeV 0.32 BeV

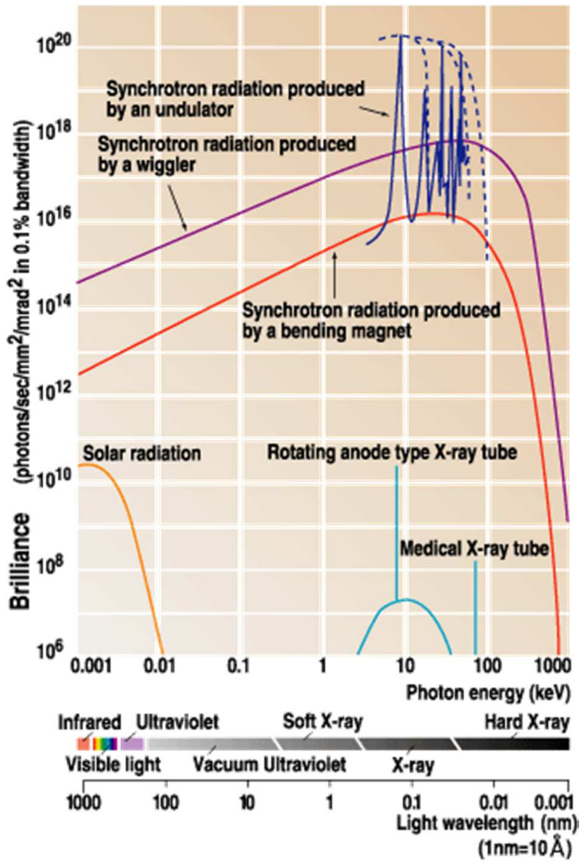
FIG. 1. The electromagnetic radiation emitted from centripetally accelerated electrons in synchrotrons in the wavelength range of ordinary x-rays. (The calculations for this figure were made by Tomboulian and Bedo[2].)

$$\varepsilon_x = \sigma_x \sigma_x' \qquad \varepsilon_y = \sigma_y \sigma_y'$$



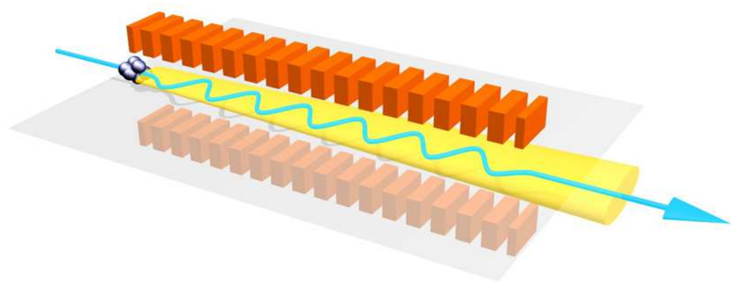
Synchrotron Radiation Source Points.

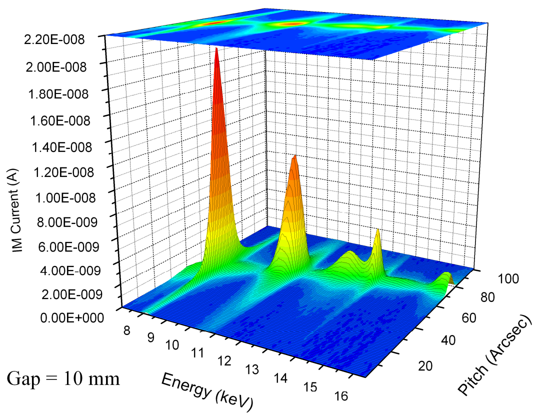




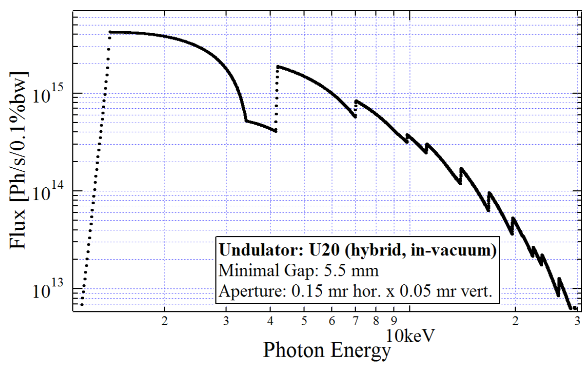
Bending Magnet.

$$\lambda_c = \frac{4\pi\rho}{3\gamma^3} = \frac{5.59\rho}{E^3} = \frac{18.6}{BE^2} \qquad \gamma = \frac{E}{m_e c^2}$$





Wiggler or Undulator.



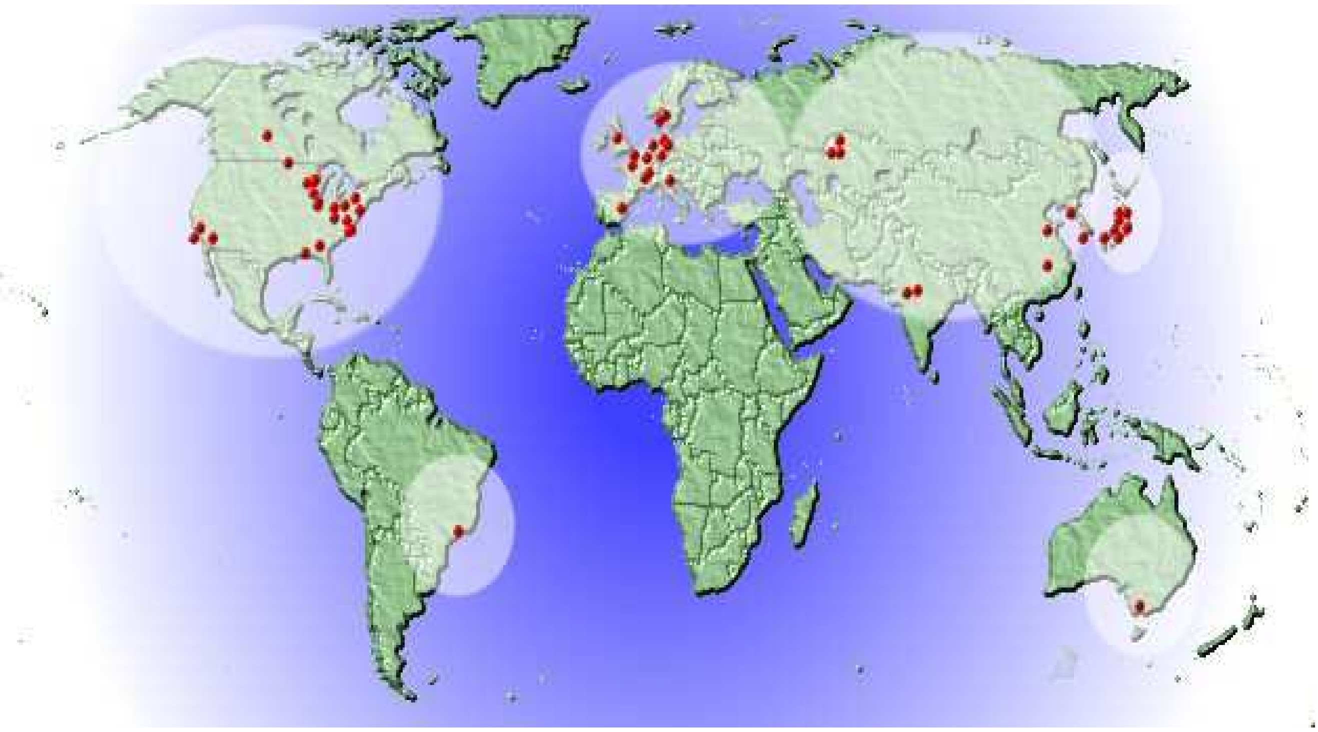
Visual model of SR

- Program due to Shintake (RIKEN, Hyogo, Japan) gives visual simulation of field lines from moving charges and different trajectories.

Properties of Synchrotron Radiation.

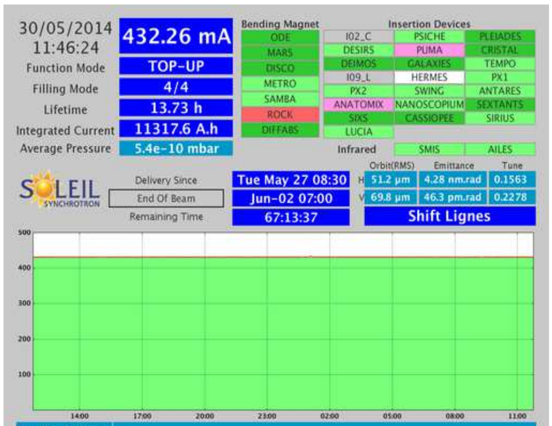
- Very high intensity.
- Very broad energy spectrum (X ray – IR).
- Naturally highly collimated
- Small source size.
- High degree of polarisation.
- Pulsed time structure.
- High brilliance machines give partly coherent beam, next generation of sources will give almost fully coherent beams.

http://srs.dl.ac.uk/srworld/world_sr.html

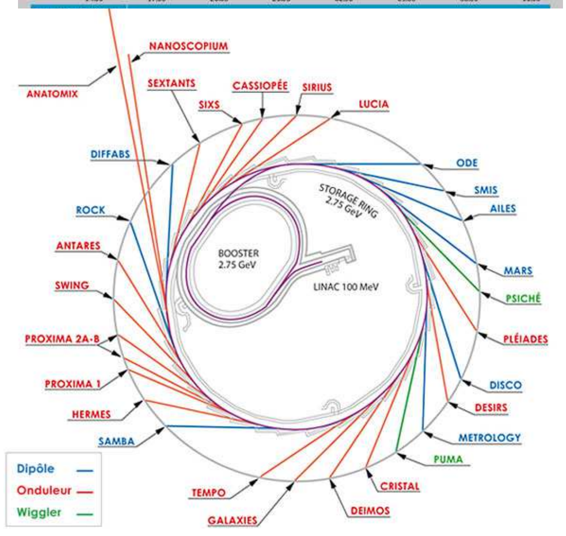


SOLEIL

Saint Aubin, near Gif/Orsay/Saclay, close to Paris.

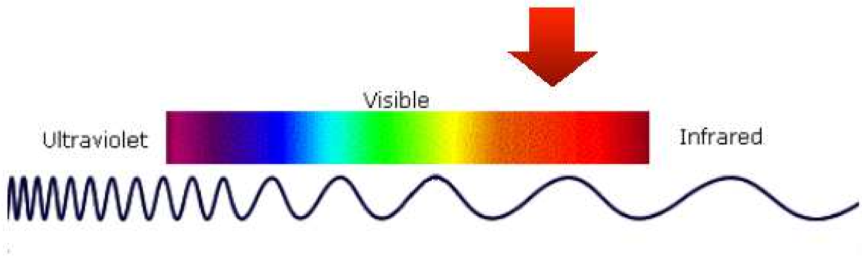


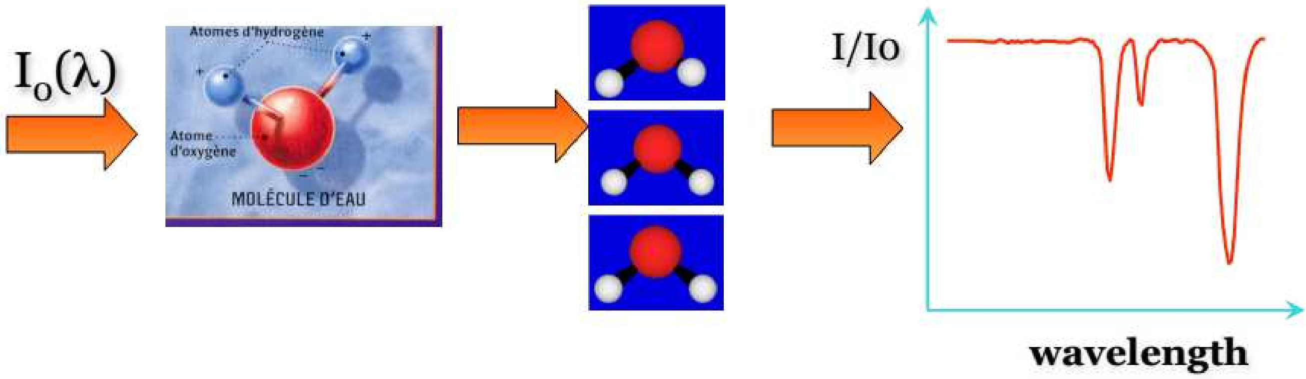
2.75 GeV electron storage ring, opened 2008.
21 straight sections available for insertion devices
26 beamlines open to users, 3 under construction
Broad energy coverage: from far IR to hard X-rays.
Operation mode : 425 mA, « top up injection »



INFRARED SPECTROSCOPY : REMINDER

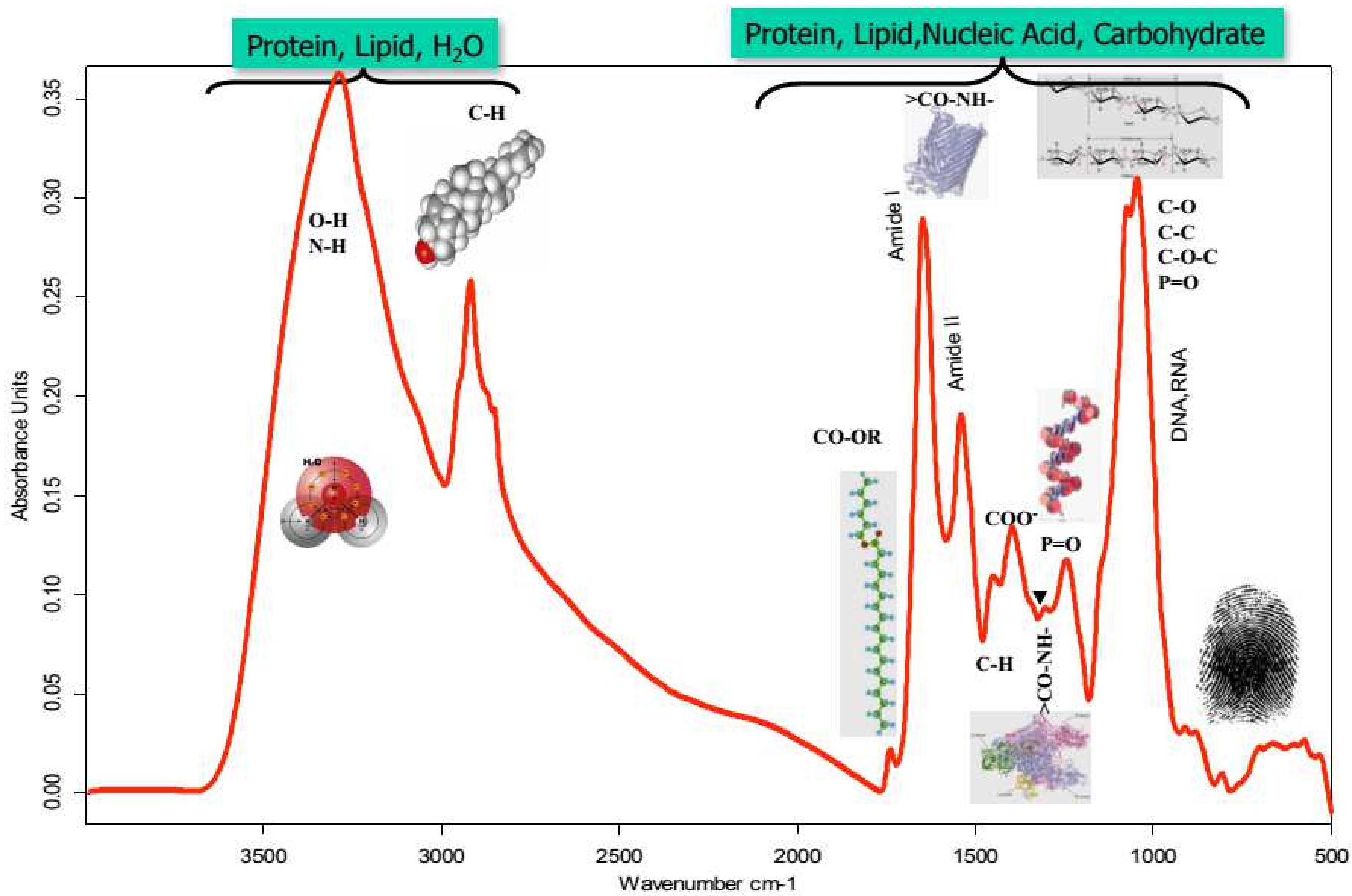
An infrared spectrum:





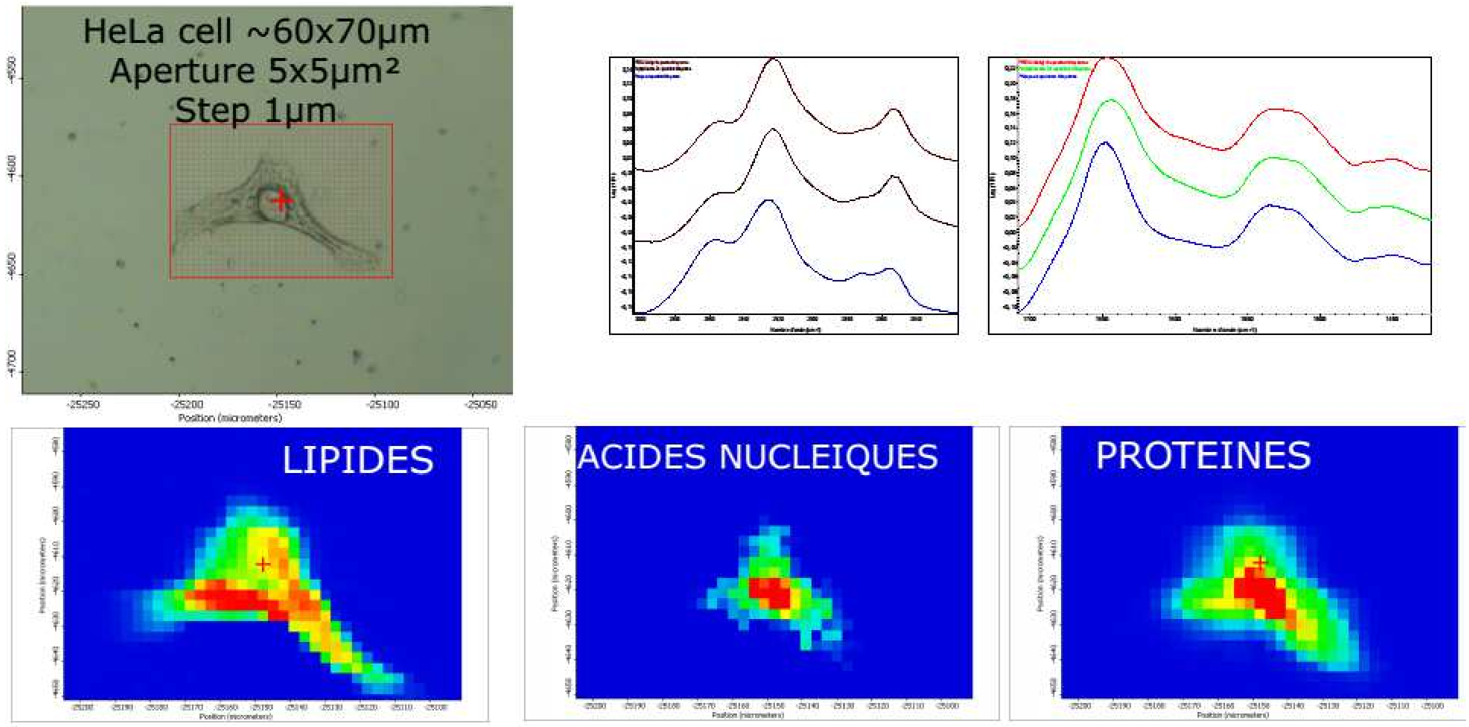
It's an absorption process involving vibrational motions of molecular groups

BIOLOGICAL SIGNATURES ON IR SPECTRA



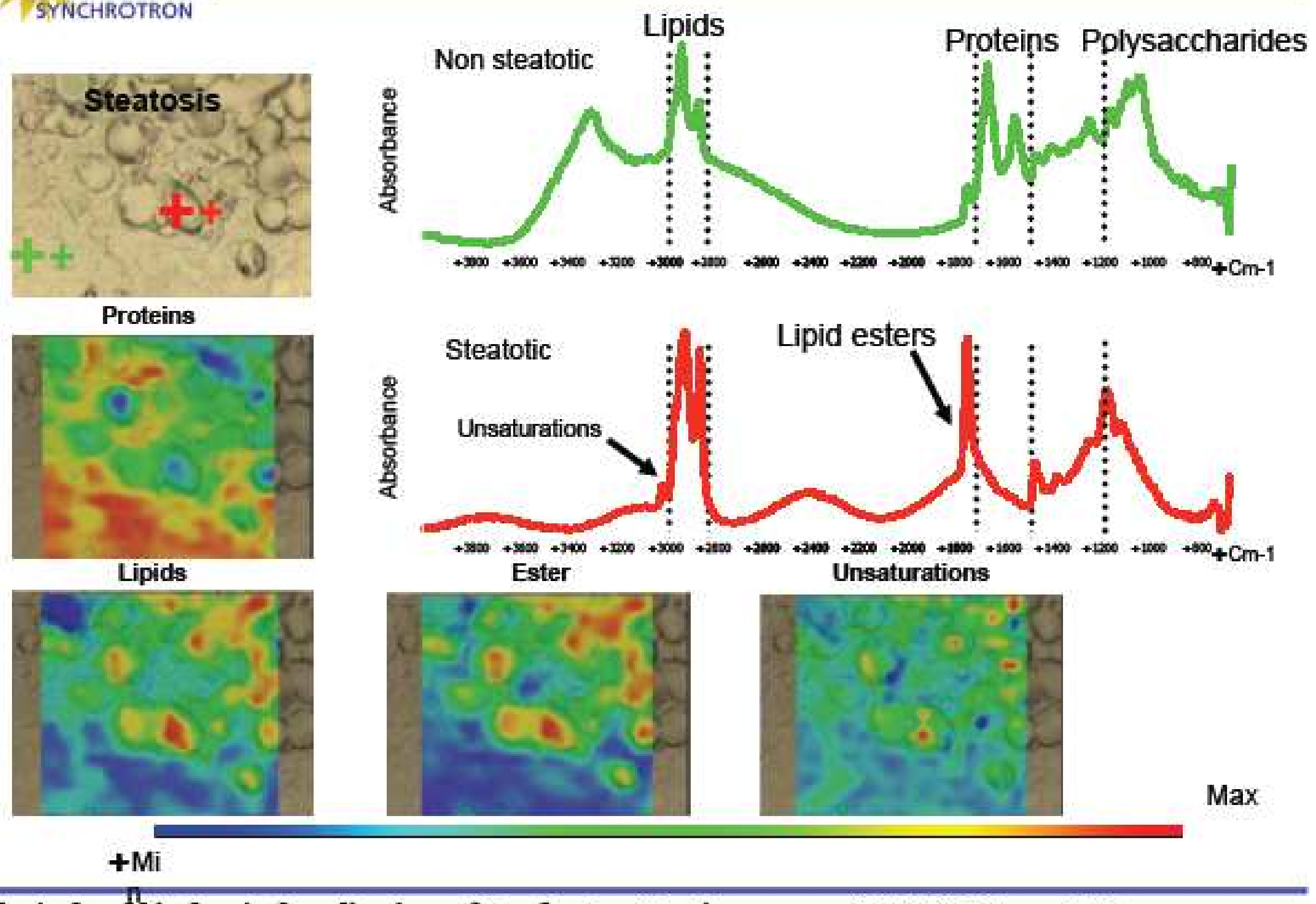
IMAGING WITH SUB CELLULAR RESOLUTION

Max



Min

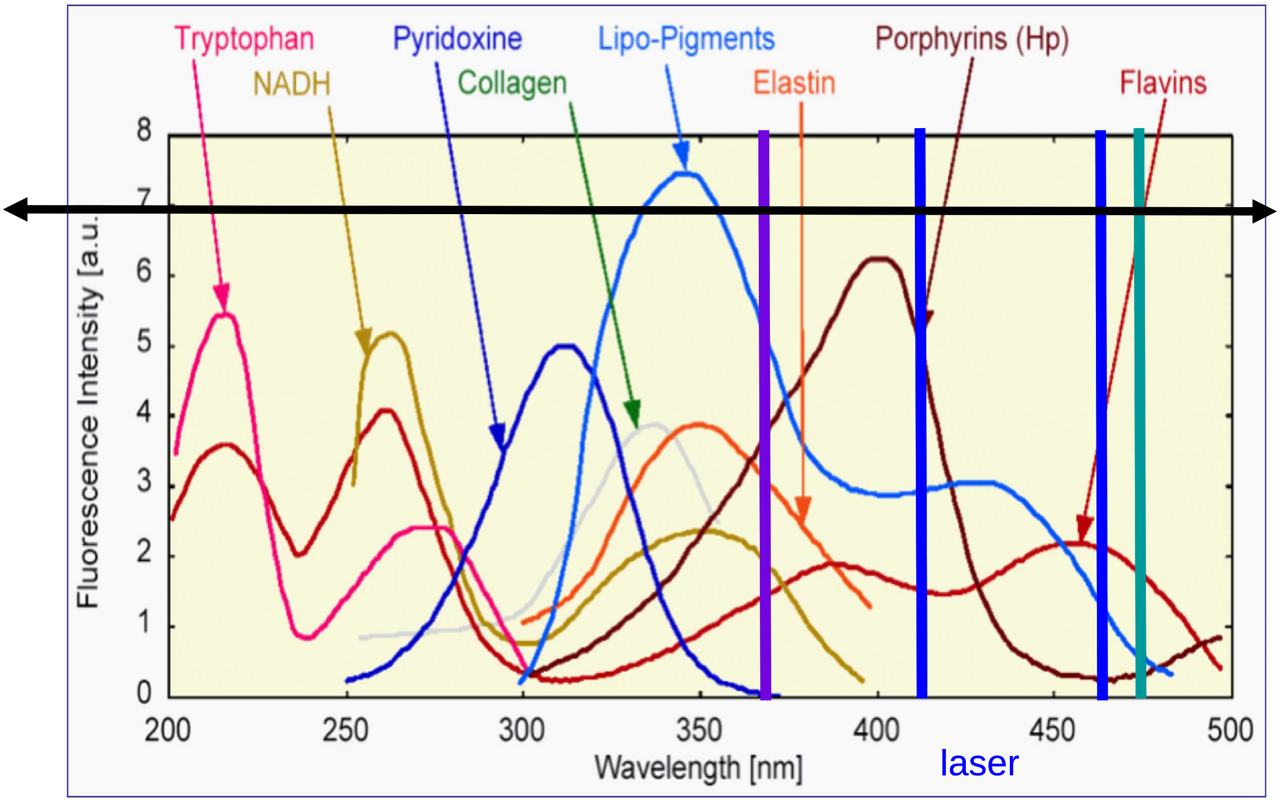
LIVER STUDY: STEATOSIS



imagerie

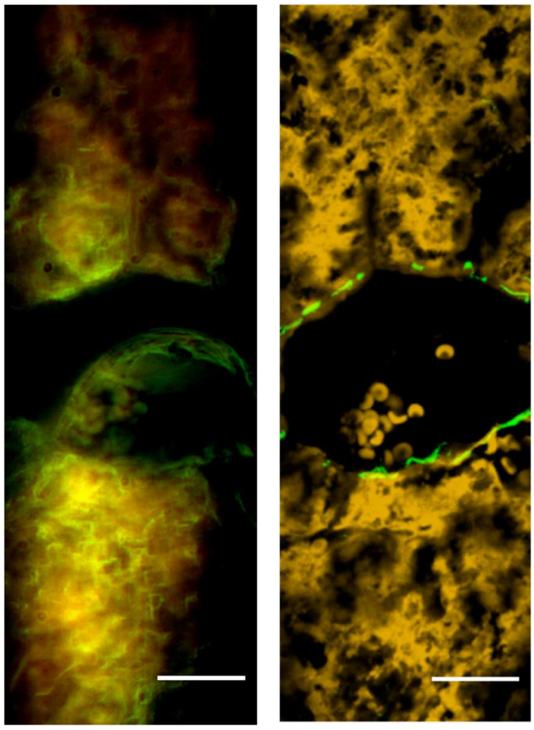
fluorescence excitation spectra of natural occuring fluorochromes

sr



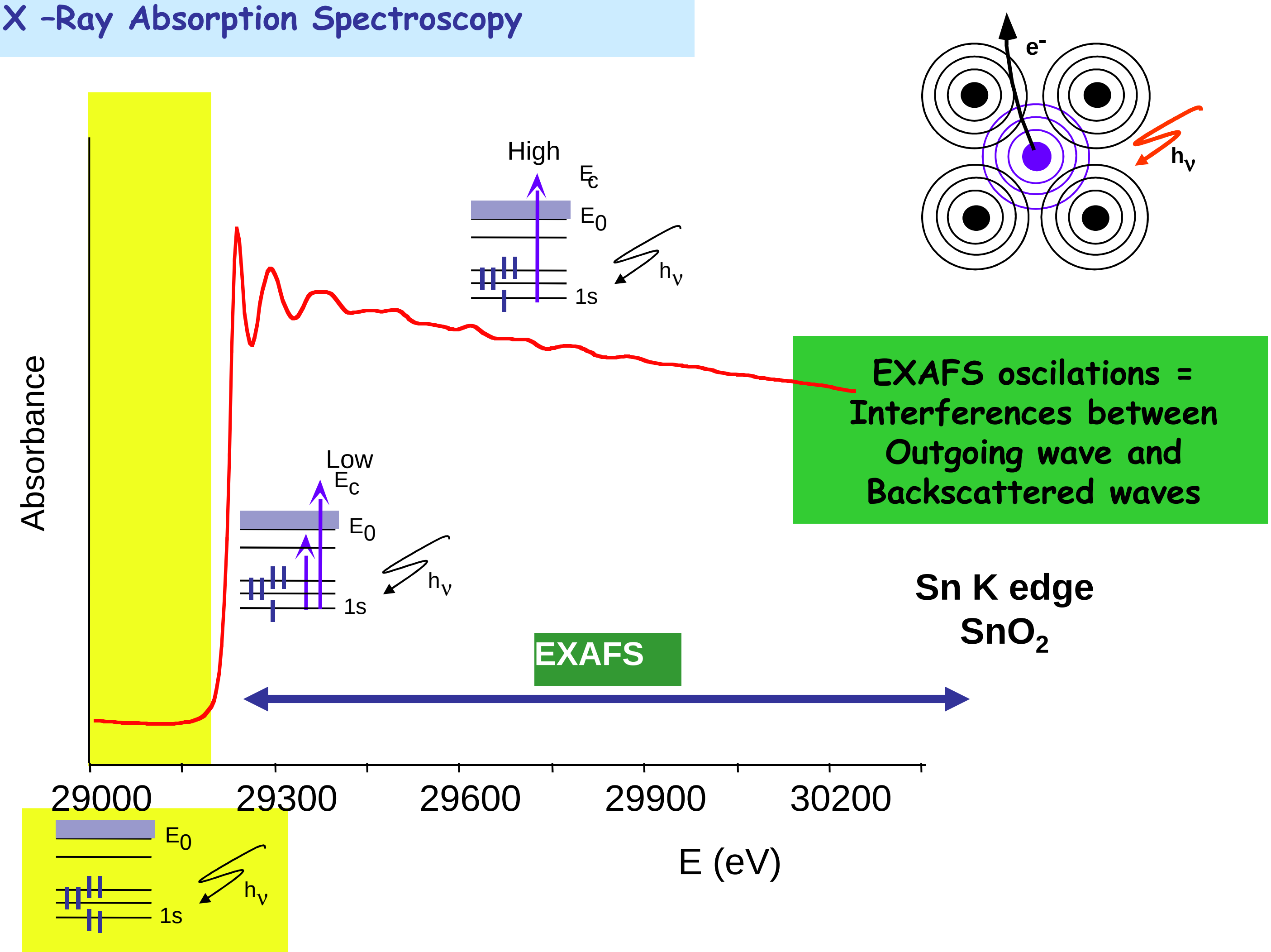
First reported combination of SHG and SR light excitation auto-fluorescence for biological tissue

- Left :“Single photon” UV Fluorescence image (different bandpass filters, collagen green, a.a’s red) of mouse liver : sensitive to all types of collagen. Right : SHG (green, only sensitive to types I and II) and TPEF (ochre).
- Scale bar is 20 µm, section thickness 20 µm



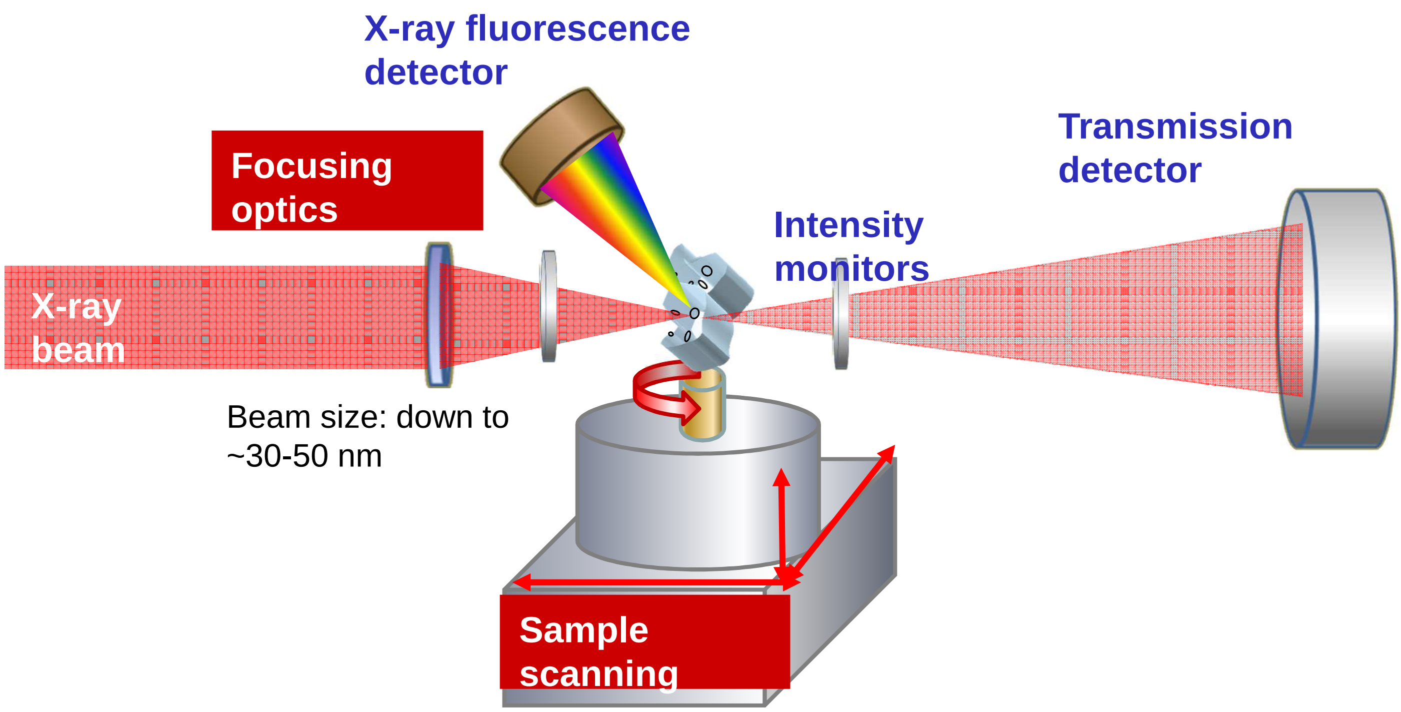
Zubkovs et al., Analyst (2014)

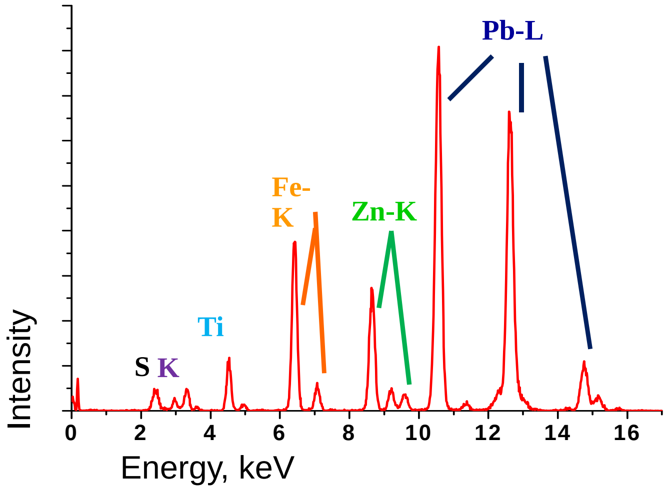
X -Ray Absorption Spectroscopy

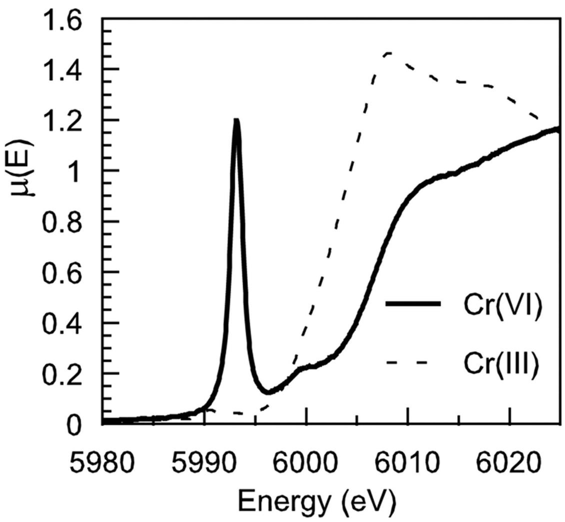


Scanning hard X-ray imaging techniques

Raster scanning of the sample
3D information (tomography): by sample rotation
Simultaneous multiply detections

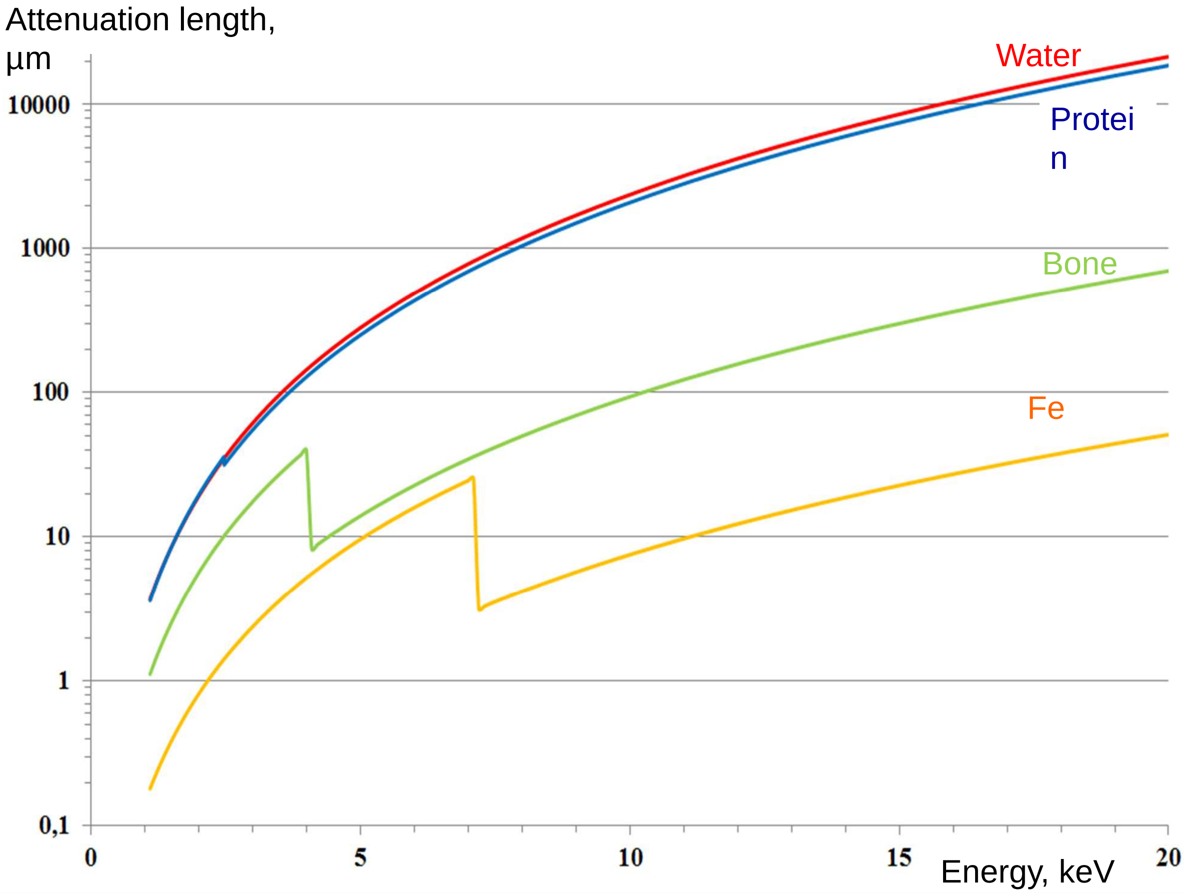




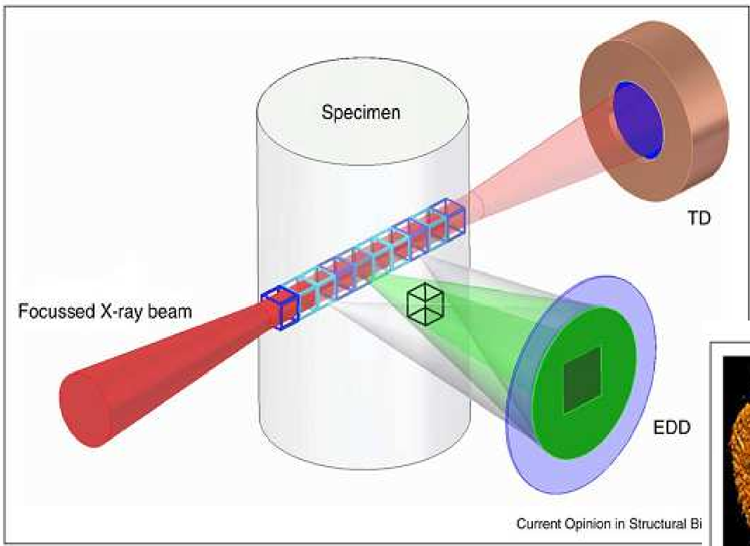


Hard X-rays
High penetration depth
No vacuum is necessary

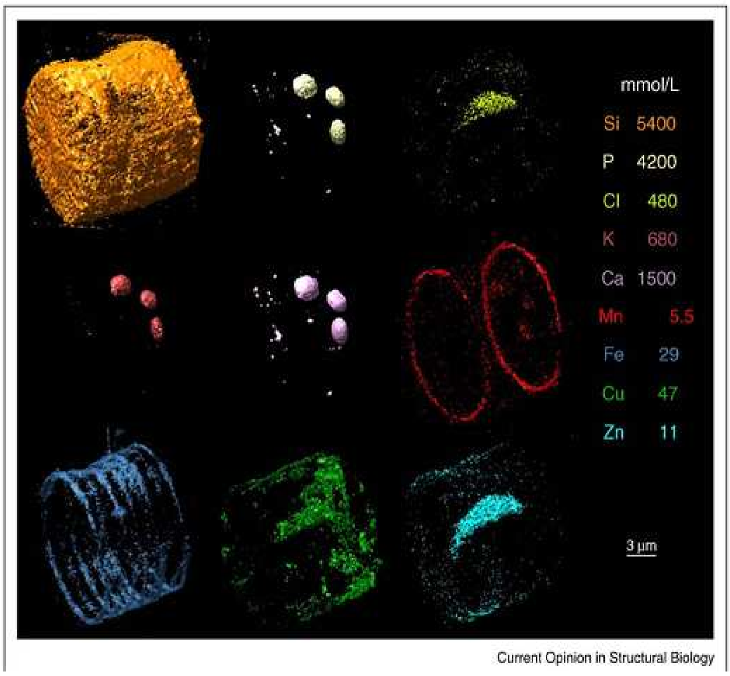
Non-destructive 3D imaging



XRF tomography: quantitative 3D elemental distribution,

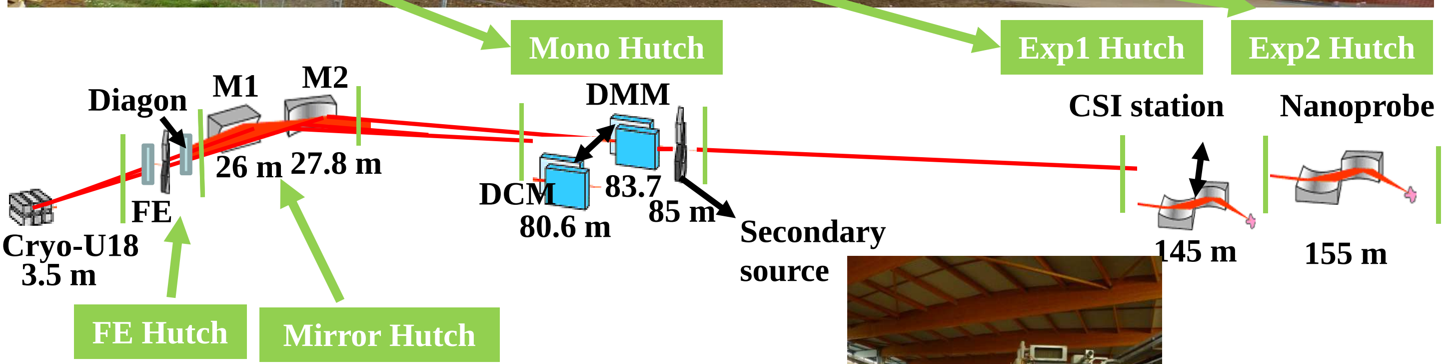


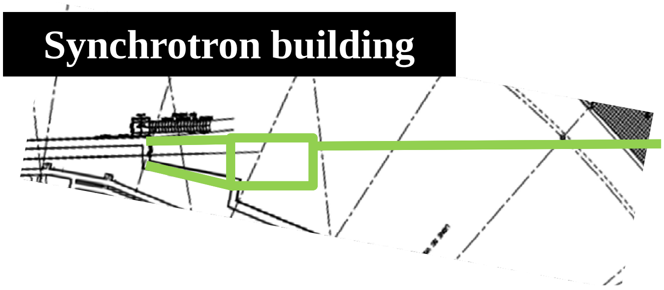
Three-dimensional rendering of the volumetric multi-element distribution within freshwater diatom

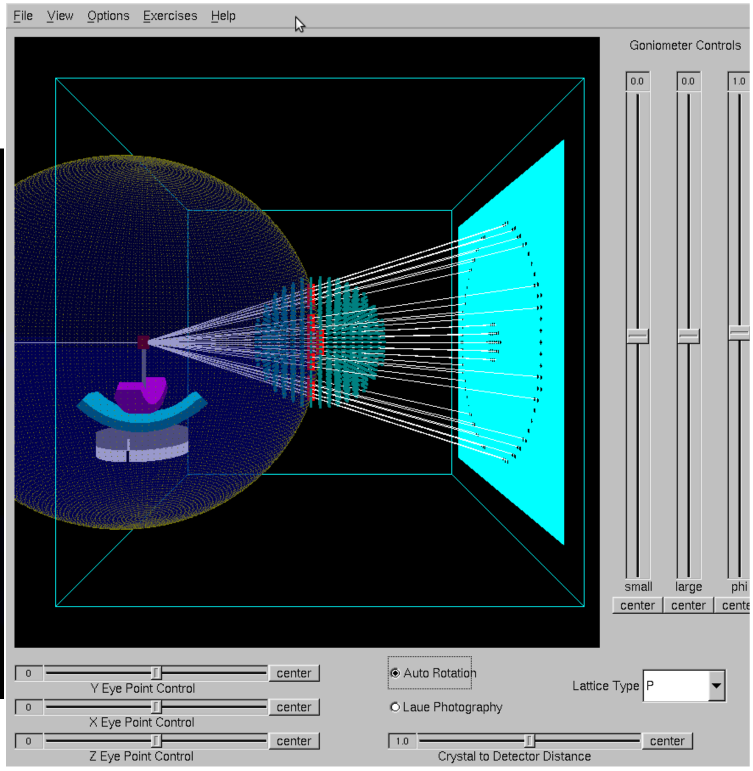


MD de Jonge et al., PNAS 2010, 107:15676-156

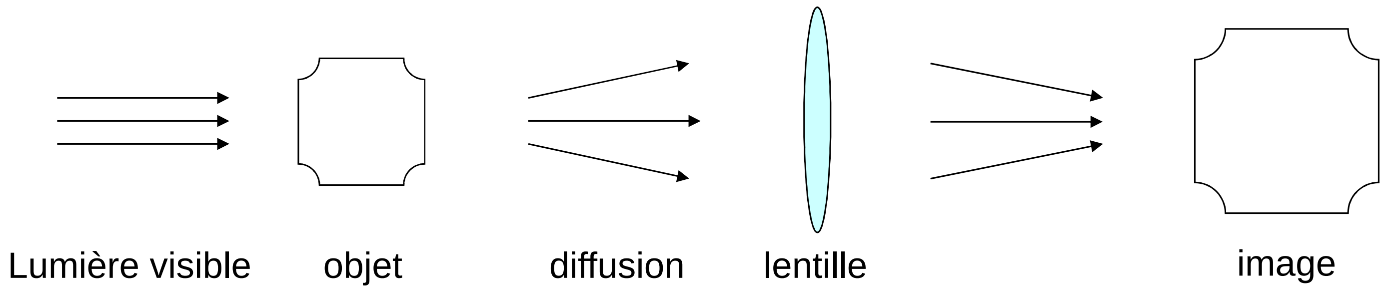
Optical scheme of Nanoscopium



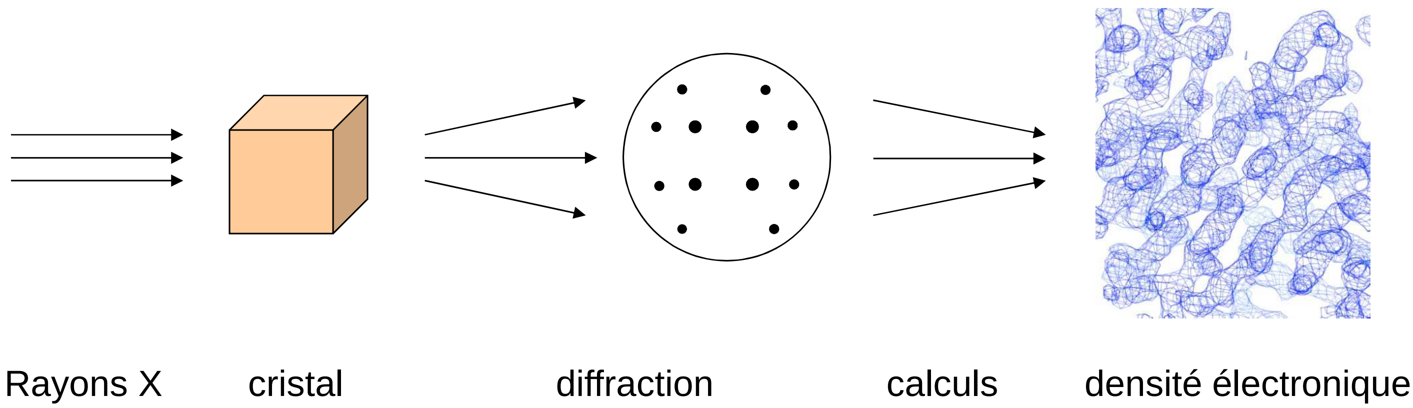




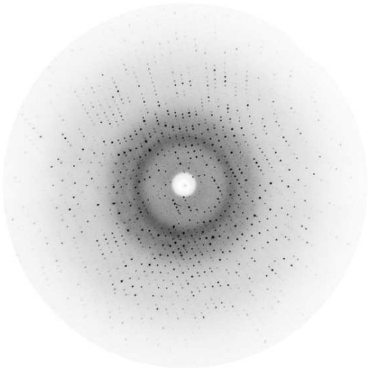
Microscopie optique

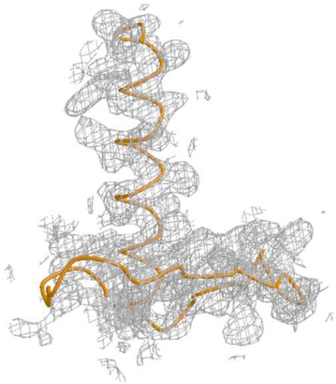


Cristallographie de rayons X



The phase problem





$$I \propto |F(\mathbf{h})|^2$$

Fourier Transform

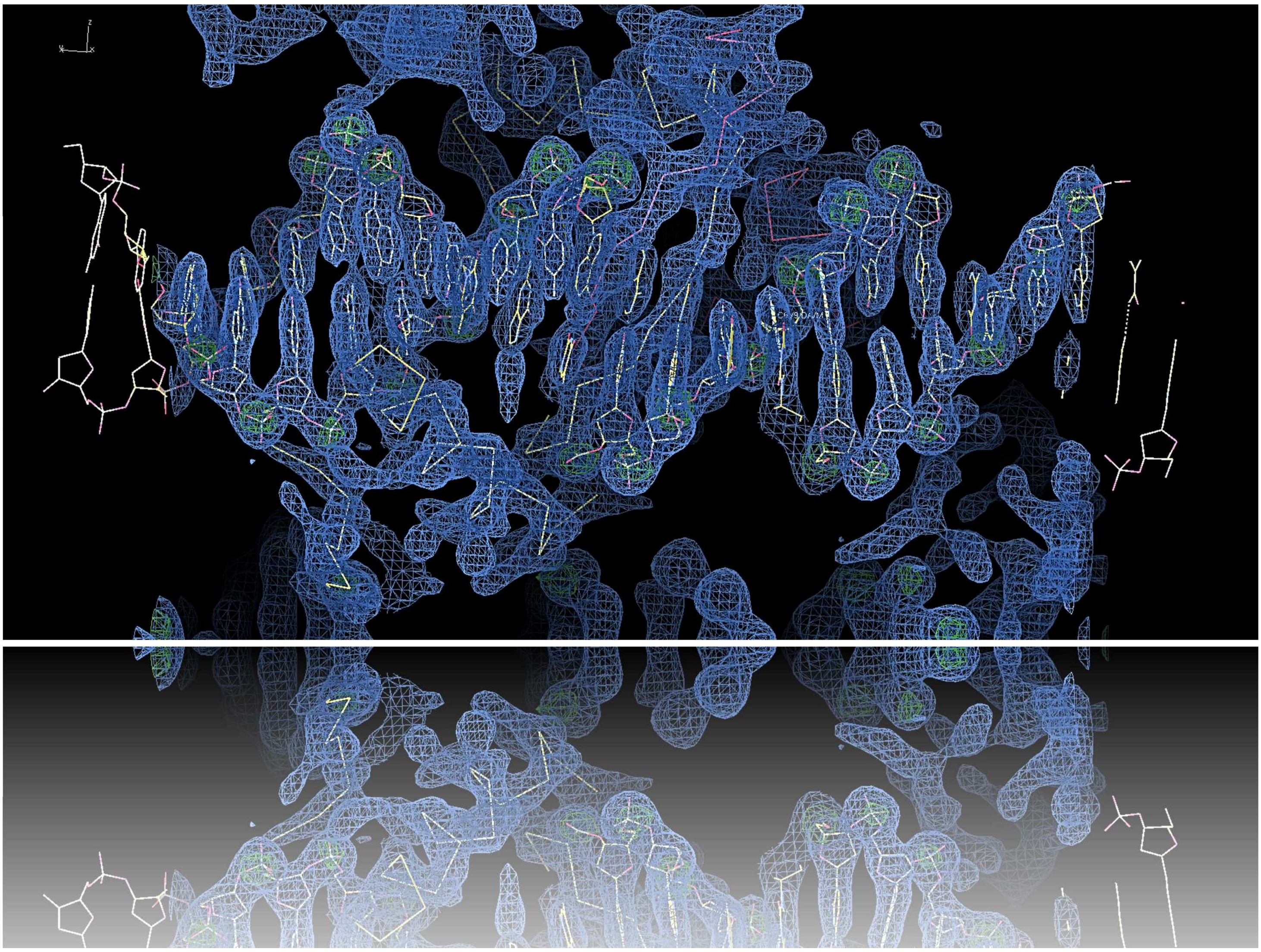
$$\rho(\mathbf{r}) = \frac{1}{V}\sum_{\mathbf{h}} F(\mathbf{h})\exp(-i2\pi\mathbf{h}.\mathbf{x})$$

with $F(\mathbf{h}) = |F(\mathbf{h})|\exp(i\Phi(\mathbf{h}))$

$$= \frac{1}{V}\sum_{\mathbf{h}} |F(\mathbf{h})|\exp[i\Phi(\mathbf{h})]\exp(-i2\pi\mathbf{h}.\mathbf{x})$$

known unknown

The phase problem

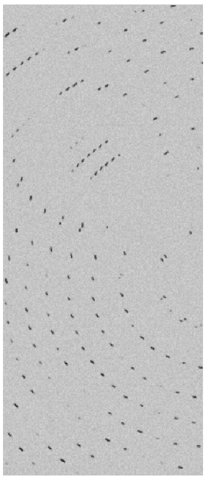


2D X-ray detector characterization

Detectors Group – Proxima 1

Evaluation of the performances to reproduce the photons image

Perfect image



- Nb of photons
- Poisson noise

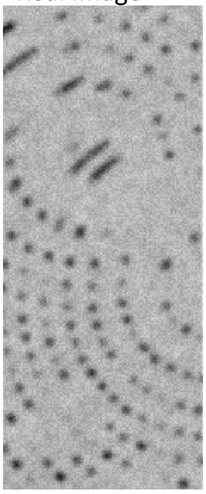
Detector

- Efficiency
- Dynamic
- Linearity
- Point spread function
- spatial sampling
- noise

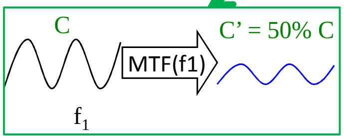
- Contrast reduced
- noise increased

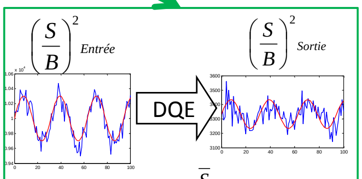
→Signal/noise reduced

Real image



Quantify the degradation of input image with the MTF and DQE





$$DQE(\nu) = g \times \frac{\bar{S}}{NPS(\nu)} \times MTF(\nu)^2$$

Quantitative evaluation of the performances of a 2-D X-Ray detector

S. Hustache, P. Legrand, A. Thompson and K. Medjoubi.

Imaging performance of 2D X-ray detectors can be described by the linear system transfer theory. In this approach, input-output relationships of an imaging system are expressed in terms of three transfer functions: the modulation transfer function (MTF), the noise power spectrum (NPS) and the detective quantum efficiency (DQE). These functions quantify the propagation quality of the spatial distribution of the incident photons, including statistical fluctuations. More familiar parameters [such as X-ray sensitivity, dynamic range (which includes linearity), spatial homogeneity, spatial linearity, stability, lag effects and, if available, energy resolution] are implicit in these transfer functions. Quantified separately, these parameters improve the understanding of the physical consequences of specific aspects of the MTF, NPS and DQE.In this document, these functions and parameters are defined and methods of measuring them are established.
In the following document only monochromatic beam exposure is considered and measurements are to be performed at different energies. All the relevant quantities will be determined after the usual image corrections (energy dependant flat field corrections, dark current subtraction, spatial distortion correction etc …).
These measurements will be done with different configurations of the detector (Binning, Gain, shaping time, threshold…) potentially used during scientific experiments, on different parts of the detector (including interfaces between modules to examine boundary effects) and at different energies.

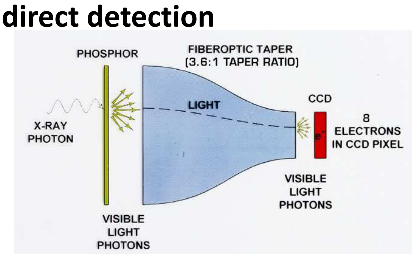
- MTF, NPS and DQE
- Linearity and dynamic range
- Spatial Homogeneity
- Spatial Linearity
- Noisy or dead pixels
- Imaging stability
- Frame rate
- Lag effects
- Energy resolution

K. Medjoubi, Detector Group, Synchrotron Soleil

From the ADSC Q315 to the Pilatus Detector

The ADSC Q315

Indirect detection



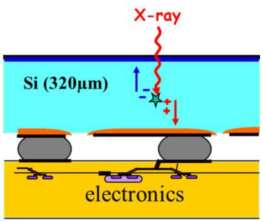
Active area	315 mm x 315 mm
scintillator	gadox
Number of pixels	6140 x 6140
Eff. pixel size	51 µm x 51 µm
CCD pixel size	14 µm x 14 µm
Readout time	Andy
Operating temp.	-45°C
Dark current	0.015 e-/pixel/second
Readout noise	11 e- / 2.7 ADU
Dynamic	14 bits
PSF, MTF(3lp/mm)	90µm, 40%
DQE(>100 photons) @ 10keV	80%
DQE(<50 photons) @ 10keV	<40%

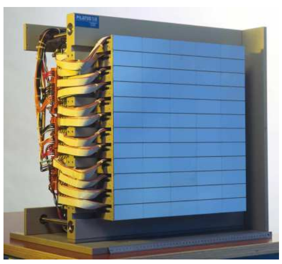
Drawbacks:
readout time, limited dynamic, signal/noise limited due to readout noise, use of a shutter, spread at the bottom of the PSF

Large Area Photon Counting X-ray Detector (Medipix, XPAD, Pilatus)

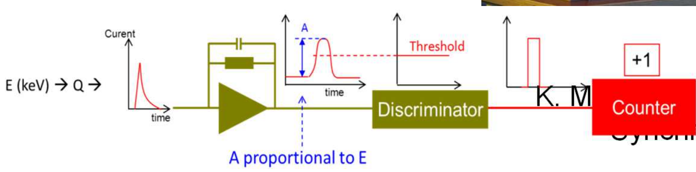
Pilatus 6M

Direct detection





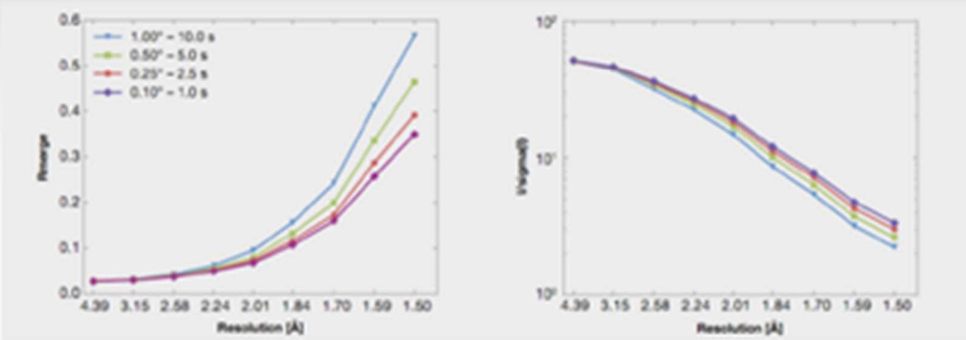
Active area	424 mm x 435 mm
sensor	320µm Si
Number of pixels	2463 x 2527
Pixel size	172 µm x 172 µm
Readout time	80 ms
No Dark current and no readout noise	
Shutterless operation	
Energy resolution	500eV
Adjustable threshold	2 – 20 keV
Dynamic	20 bits
PSF, MTF(3lp/mm)	170 µm, 60%
DQE @10 keV (1 up to 10^6 ph)	90%



K. M... Detectors Group, Synchrotron Soleil

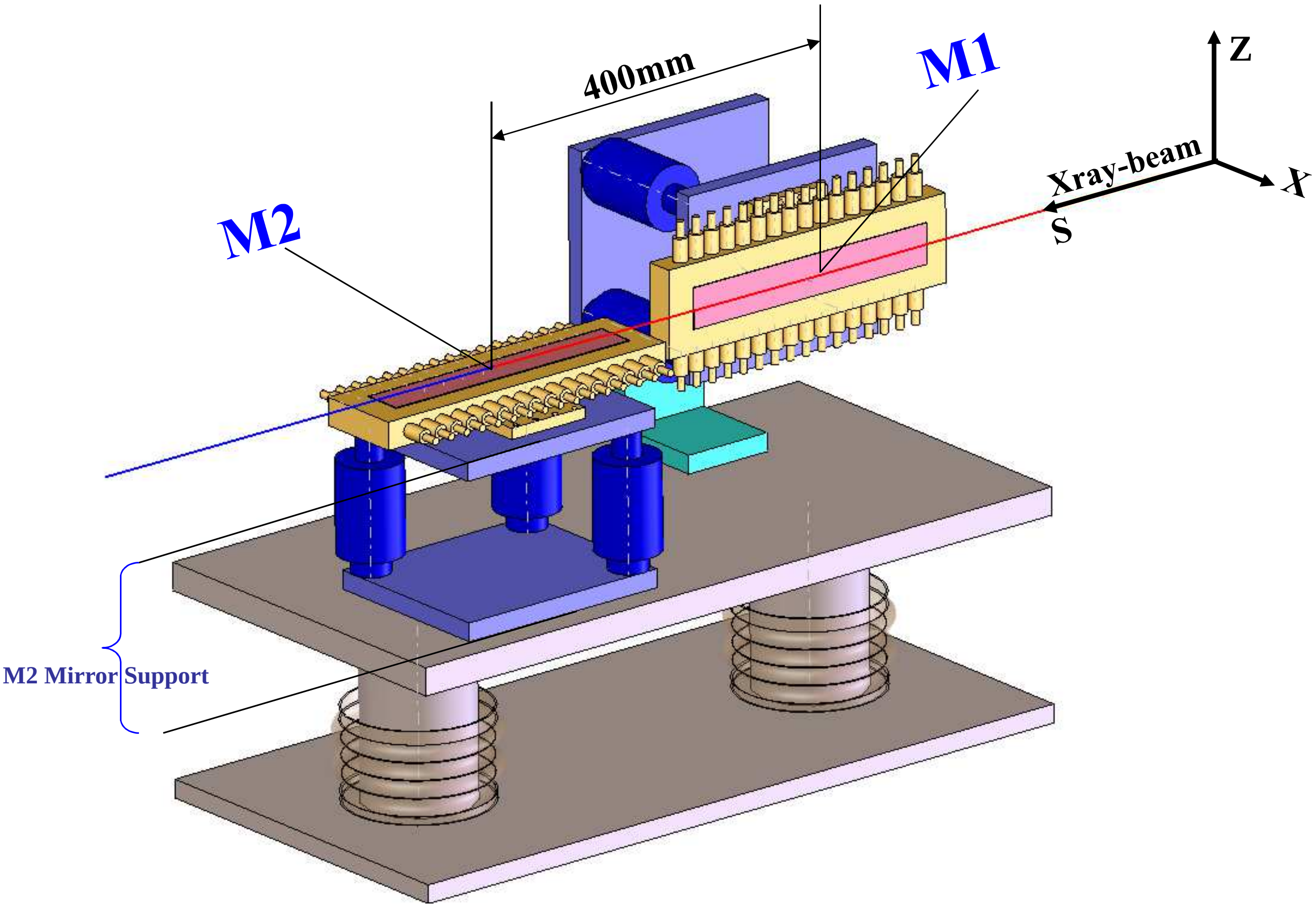
Effect of fine slicing / shutterless data collection using PILATUS 6M

3. Data quality improvement at high resolution

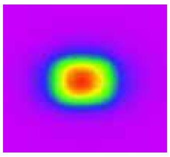


Insulin data sets covering 180° of rotation, all collected with the same angular speed.

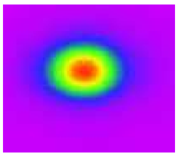
--> Gain in speed & data quality



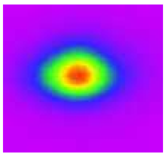
PROXIMA 1 operating conditions – Beam almost parallel.



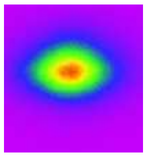
91 x 67 µm, d=200mm



94 x 66 µm, d=400 mm



104 x 70 µm, d=600 mm



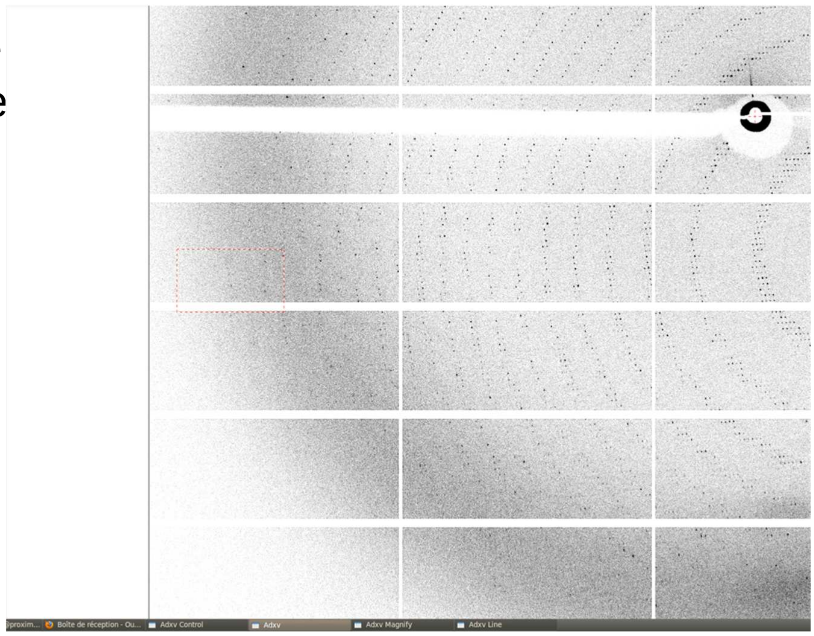
130 x 83 µm, d=900 mm

Bimorph mirrors take approximately 10 minutes to stabilise after beam focus change. Centroid of the focal spot remains stable (no additional re-alignment required). Horizontal divergence 45 µrad, vertical 31 µrad

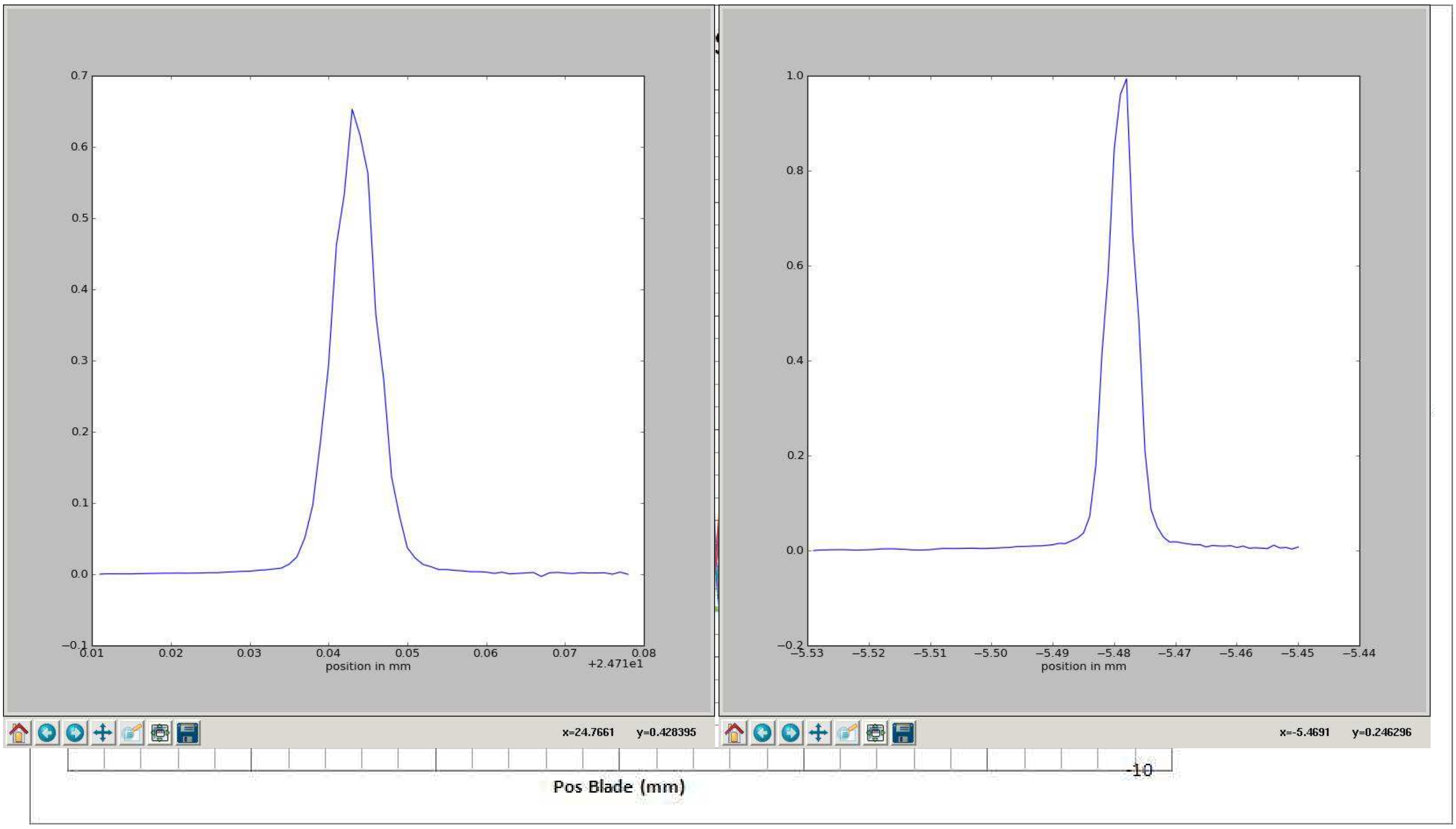
Images taken prior to PX1 optics upgrade (full focus 125 x 65 microns, now 80 x 30 microns)

Good s:n from low dose - “gentle” data collections

- SLS : Small beam, translate crystal (up to 6x) - low dose wedge - merge data from many crystals, PILATUS detector.
- Optimisation of background (slits, aperture), “slow” data collection with highly attenuated beam.
- Eukaryotic ribosome (Strasbourg).
- $P2_1$ 303 x 286 x 435 Å, β=99°.

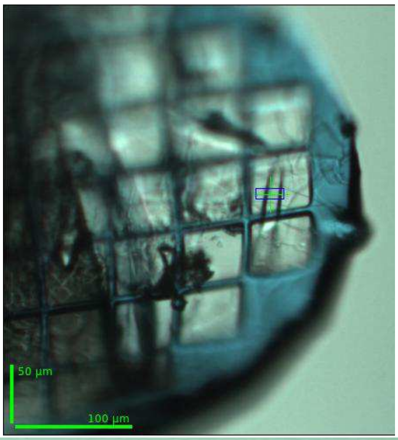


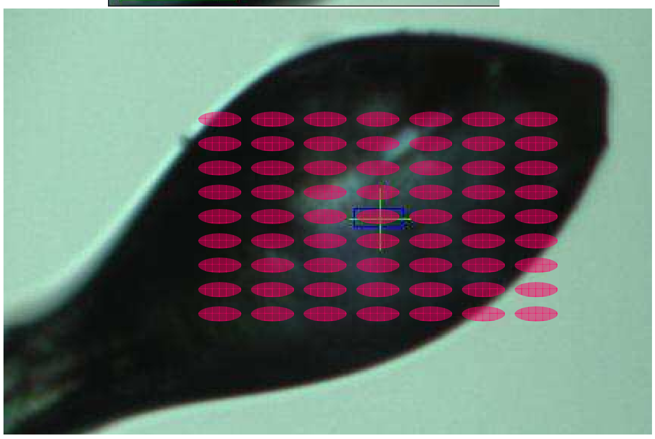
Focal Spot Optimisation of PROXIMA 2a Using Bimorphs



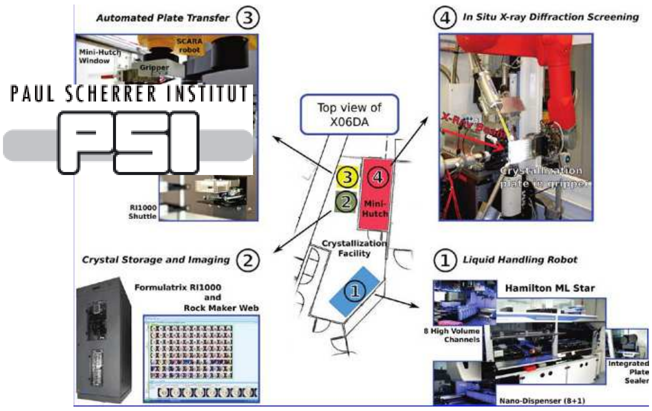
PX2-A Future Directions

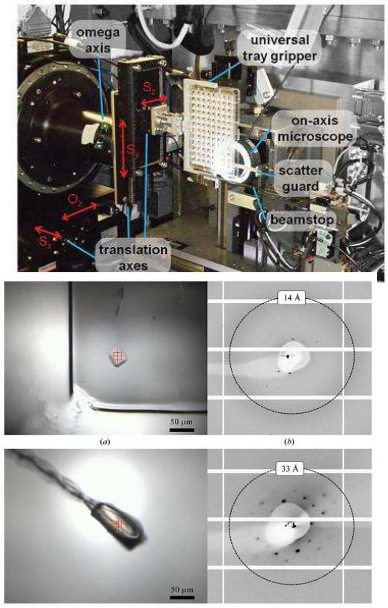
- Cartography
  - Finding the best diffracting part of a crystal
  - Low X-ray dose scans (raster / helical)
- Merging XRD data
  - Cluster analysis





In situ data collection : current state-of-the-art





Site	Current beamline capabilities		
	Routine plate-screening	Regular with sample env reconfigurations	Infrequent ad hoc set-ups
ESRF	FIP		BM16
SLS	PX III	PX II	
Diamond	I04	I24	
Bessy		14.1	
ALS		8.3.1	
APS			GMCA-CAT IMCA-CAT NE-CAT

New BLs proposing in situ modes : XALLOC (ALBA), Proxima2A/1 (SOLEIL), VM (DIAMOND).

Gavin Fox, PROXIMA 2a

From Boutet et al, Science May 2012.

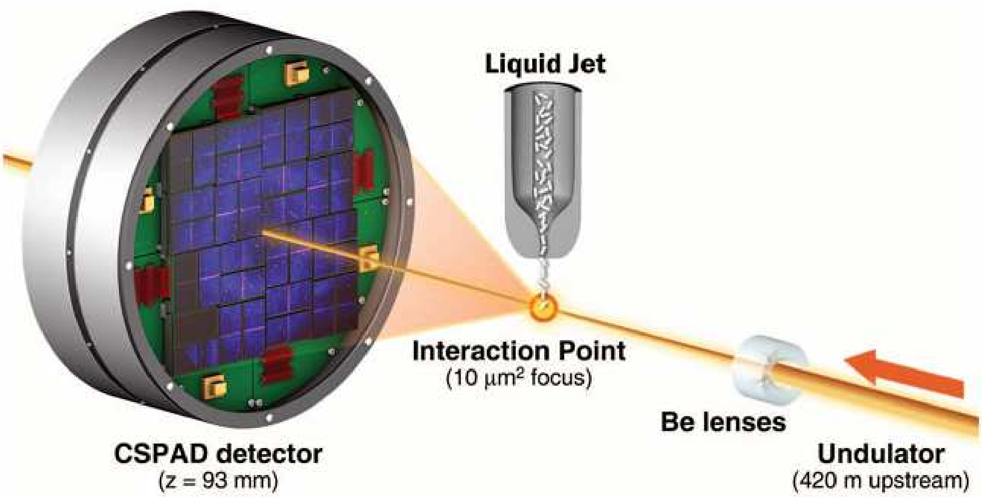
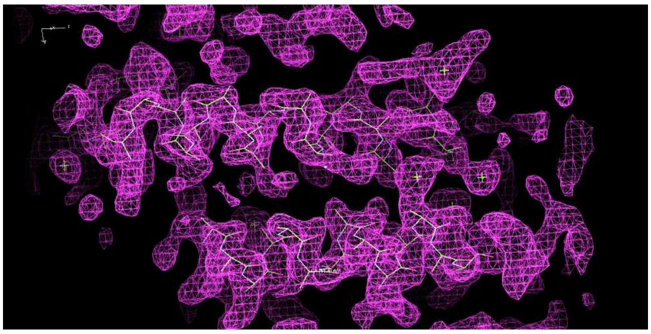
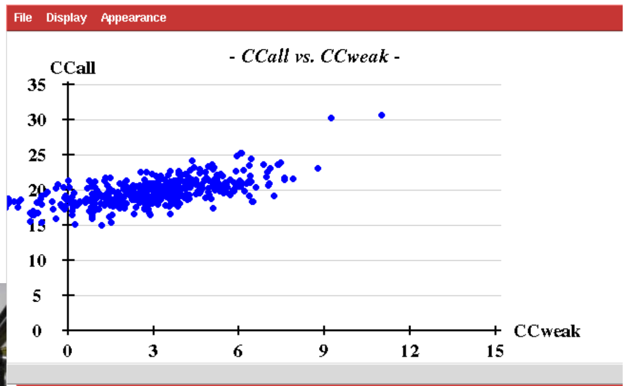


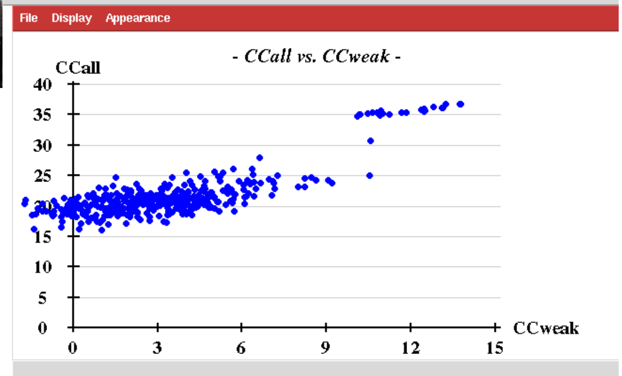
Fig. 1. Experimental geometry for SFX at the CXI instrument. Single-pulse diffraction patterns from single crystals flowing in a liquid jet are recorded on a CSPAD at the 120-Hz repetition rate of LCLS. Each pulse was focused at the interaction point by using 9.4-keV x-rays. The sample-to-detector distance (z) was 93 mm.

Phasing with anomalous signal of S at wavelengths around 1.8 – 1.9 Å.



Sites found with “identical dose data collection” – top = random geometry, lower = multiple orientations combined.

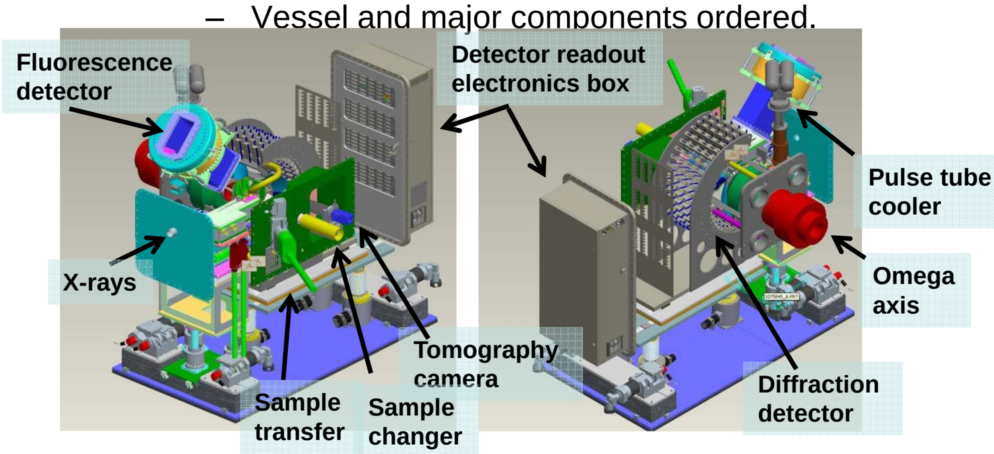




Data collection around multiple axes with kappa goniostat or multiple crystals (Hendrickson).
Use of “zero noise” pixel array detector, which permits collection at lower dose.
7 structures solved like this on PX1 in last 12 months. (3 in previous 3 years!) in various different space groups (lowest symmetry $P2_1$)

Current Status

- In-vacuum End Station
  - Vessel and major components ordered.



Detector
- In-vacuum Pilatus 12M.
- Cylindrical detector
- 2θ = ± 90º
- Mechanics built.

- Vacuum vessel
  - In manufacture.

- Experimental Hutch
  - Installation ongoing.

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- DISCO – M. Refregiers
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- SAMBA – V. Briois
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- Storage ring performance and operation – A. Nadji
- DIAMOND soft X-ray beamline – A. Wagner
- All the many users of synchrotron facilities that have brought exciting projects and discussed with me over the last 35 years …..
- Roger Fourme and R. Kahn, to whom French MX owes a huge debt of gratitude.