

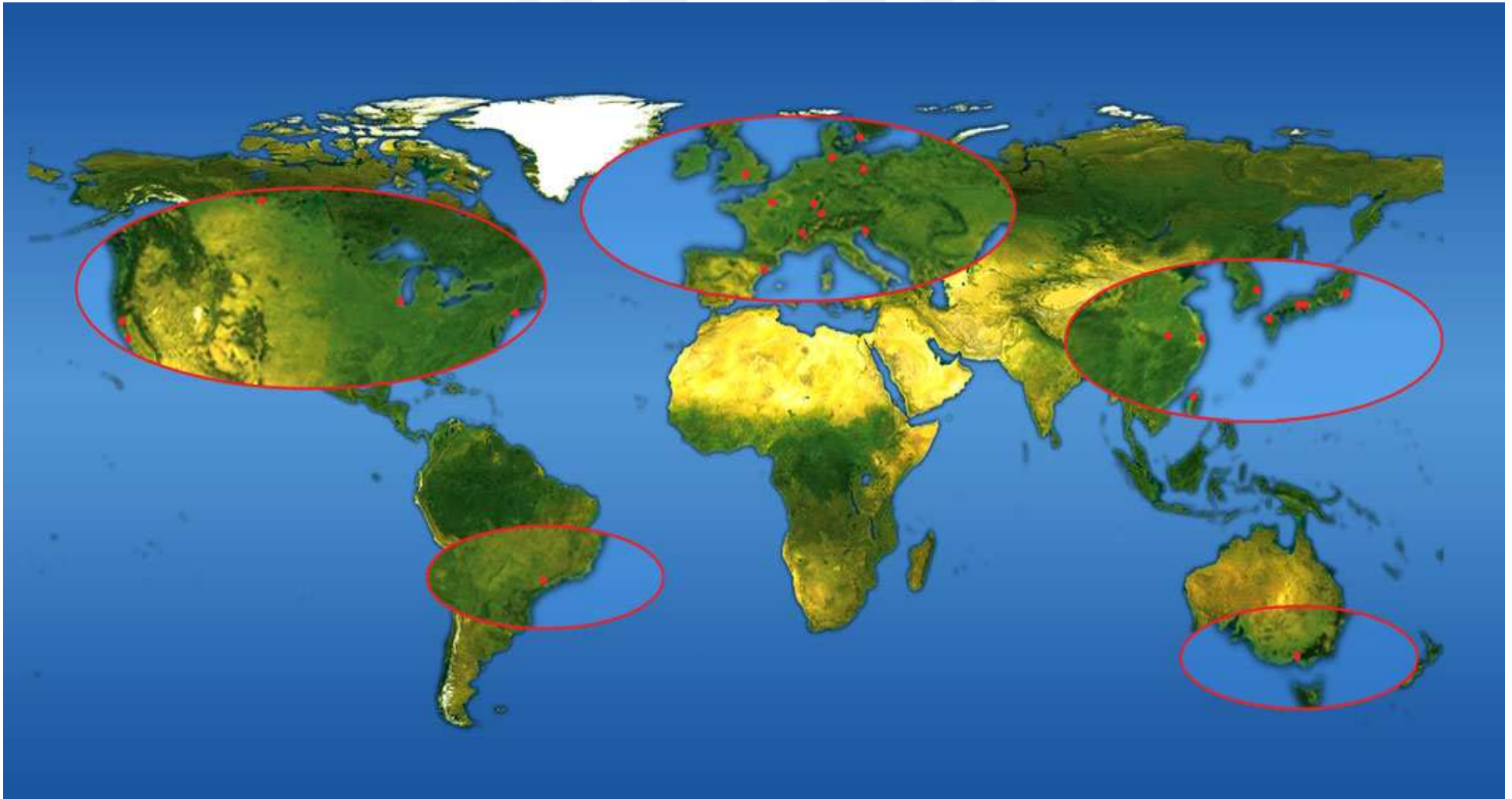


European Synchrotron Radiation Facility



David Flot
groupe de biologie structurale, ESRF

Plus de 20 nouveaux synchrotrons construits depuis 20 ans.





Société Civile de droit français



Etats membres

France	27.5 %
Allemagne	25.5 %
Italie	15 %
Royaume Uni	14 %
Espagne	4 %
Suisse	4 %
Benesync (Belgium, Netherlands)	6 %
Nordsync (Denmark, Finland, Norway, Sweden)	4 %

Etats associés

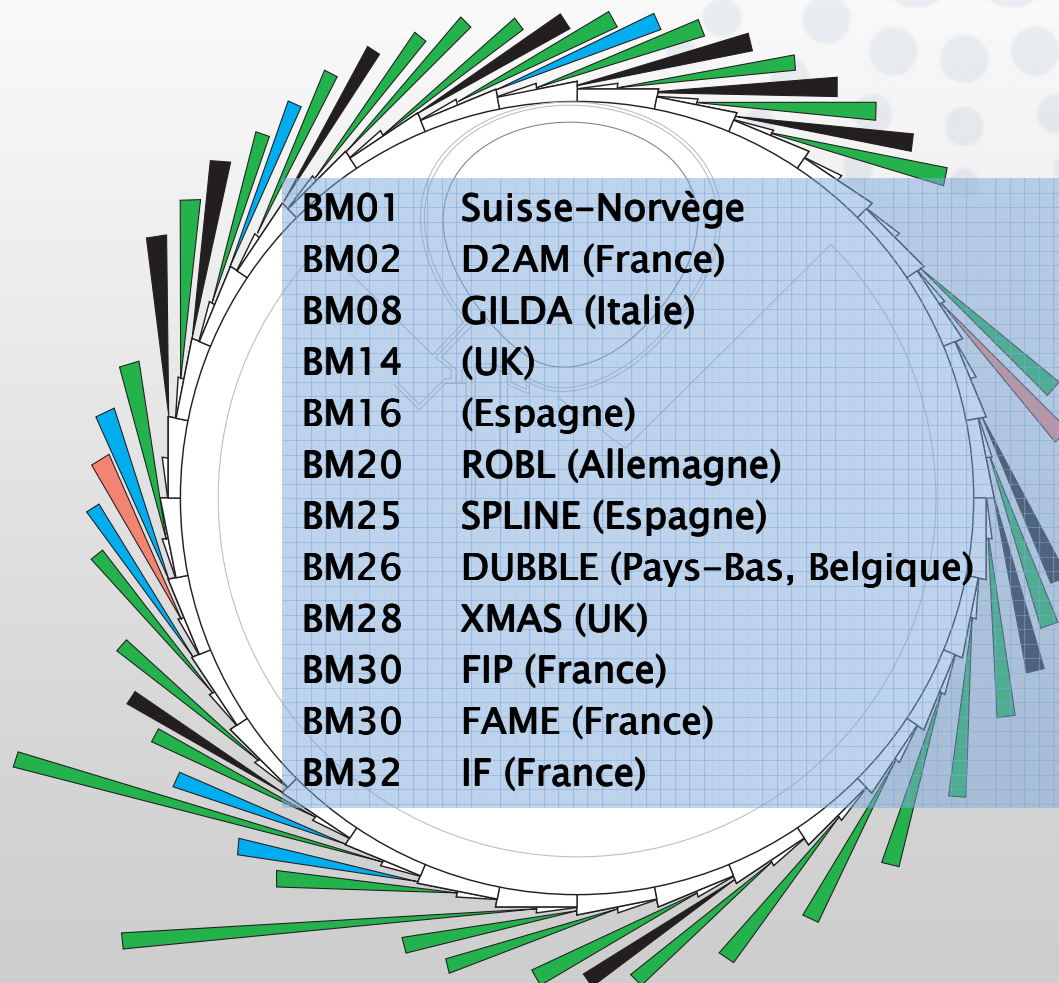
Portugal	1 %
Israel	1 %
Autriche	1 %
Pologne	0.6 %
CentralSync (CZ, H, SK)	1.05%

Quelques chiffres – 2008

- ~ 2000 propositions
- ~ 600 employés
- ~ 6300 utilisateurs (visites)
- ~ 90 M€ budget
- ~ 1500 sessions expérimentales
- ~ 1500 publications



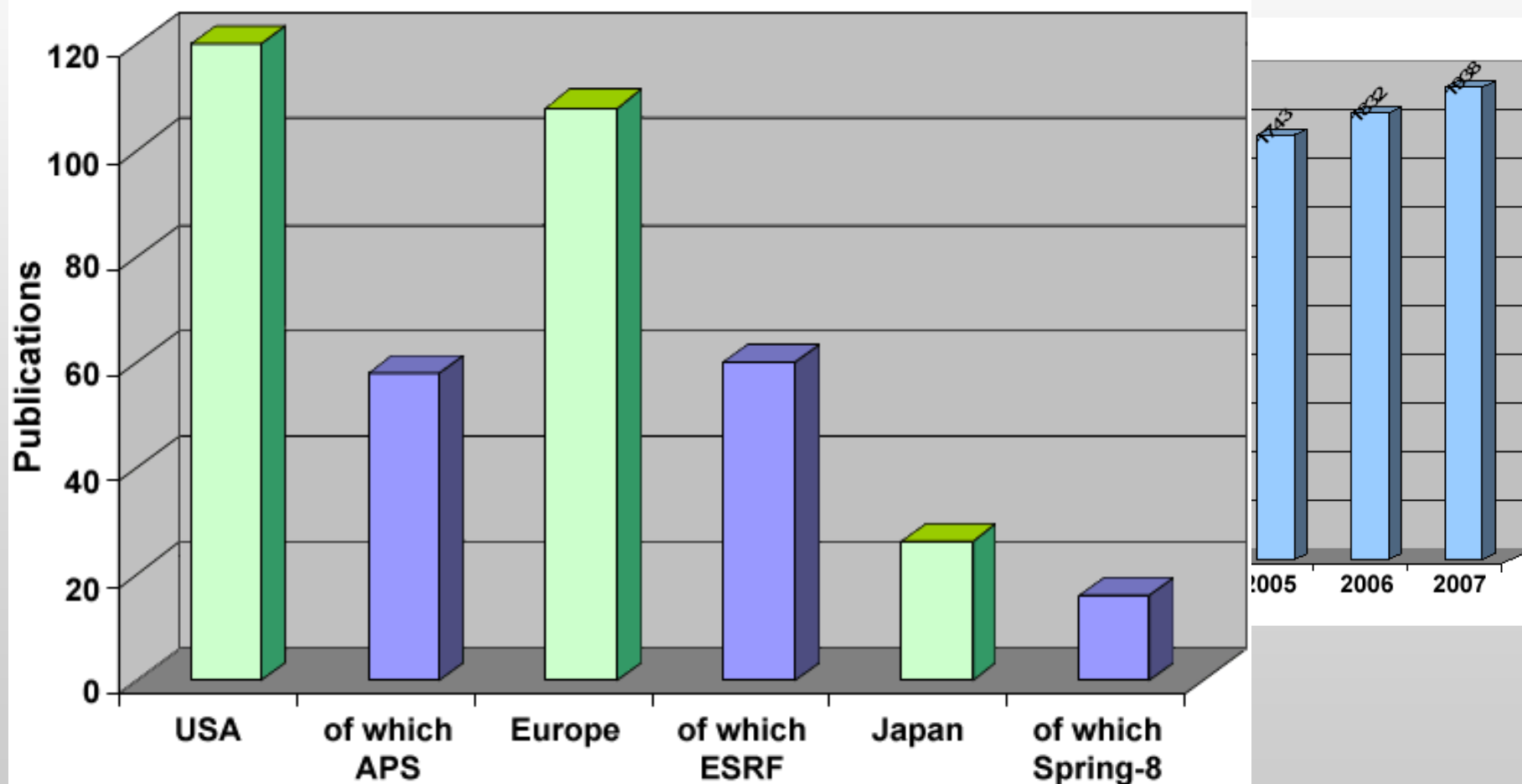
Beamline Topics



ID18 Diffusion nucléaire
 ID19 Microtomographie – Topographie
 Absorption et diffraction (diffraction)
 Science des matériaux
 Absorption et diffraction
 MX (MAD)
 Biologie structurale (MAD)
 Radiochimie
 Absorption et diffraction
 Multipurpose
 Magnetic scattering
 Biologie structurale (MAD)
 EXAFS
 Surfaces et interfaces
 BM29 Absorption

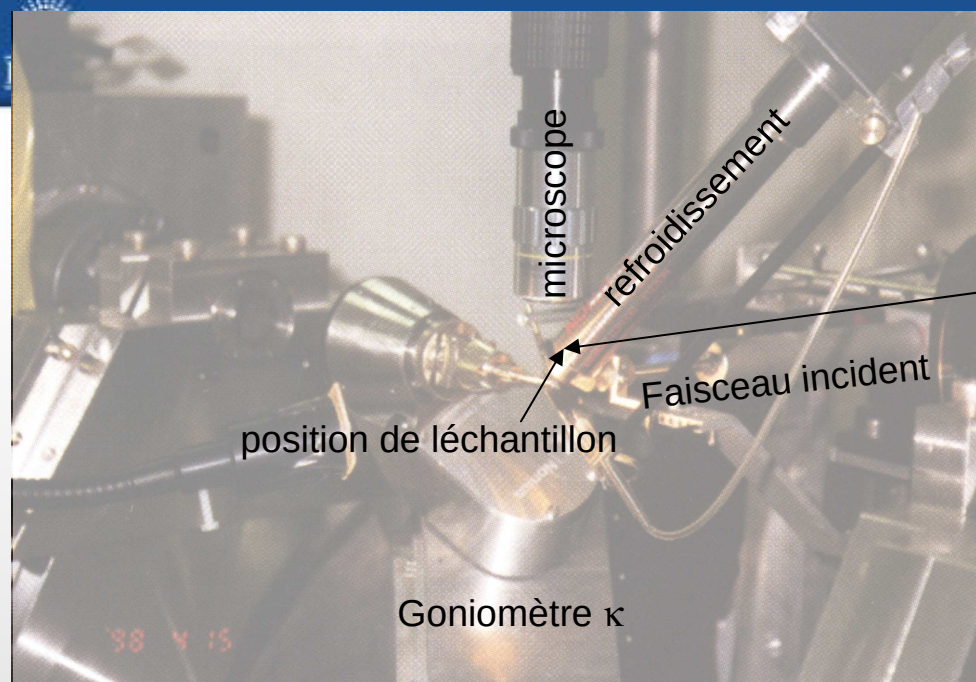
A World Class Laboratory

Publications in Nature and Science 2006/2007



Diffraction de monocristaux avec des petits faisceaux (ID13, ID23-2)

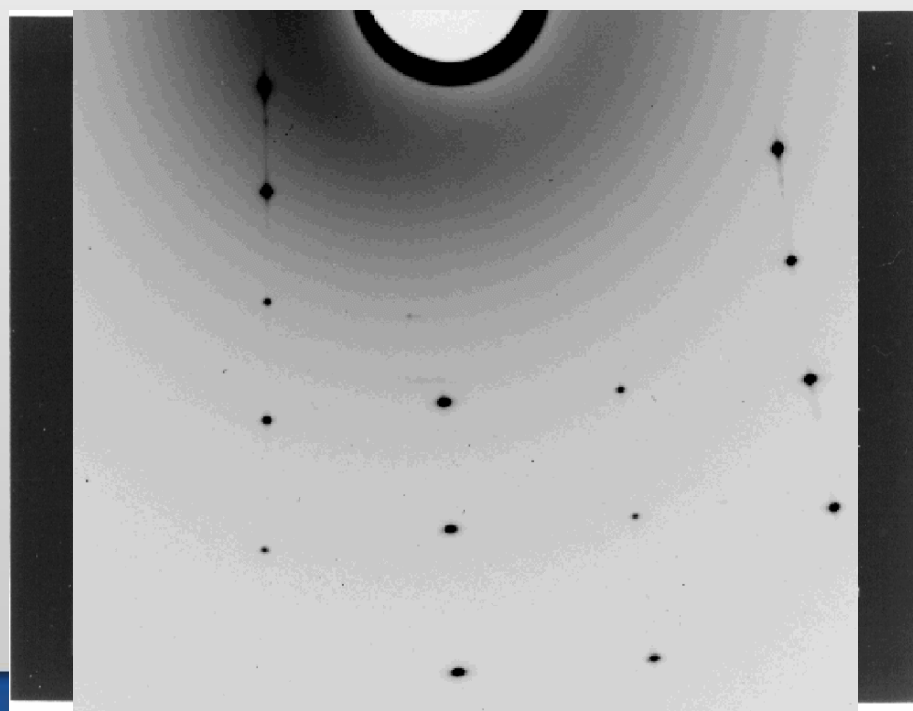
ID13 κ -goniomètre



- 4-cercles (MACH3 CAD4, ENRAF-NONIUS)
- sphère de confusion $5\mu\text{m}$
- collimateurs (5, 10, $30\mu\text{m}$)
- Détecteur 2-D : MarCCD - 130 mm
- sous-vide

Applications:

- monocristaux, fibres, diffusion diffuse etc... puis ... protéines.



Monocristal de kaolinite
Très performant...

$\sim 2 \times 0.5 \times 0.2 \mu\text{m}^3$
mais compliqué à utiliser

Neder, et al. ; Z. Kristallogr. (1996) **211(11)** 763-765
(centrage du cristal, alignement, etc...)



« l'échantillon »

Polymère

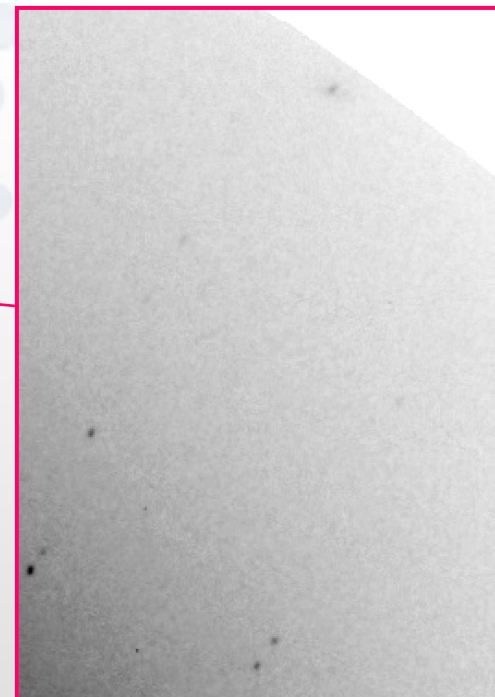
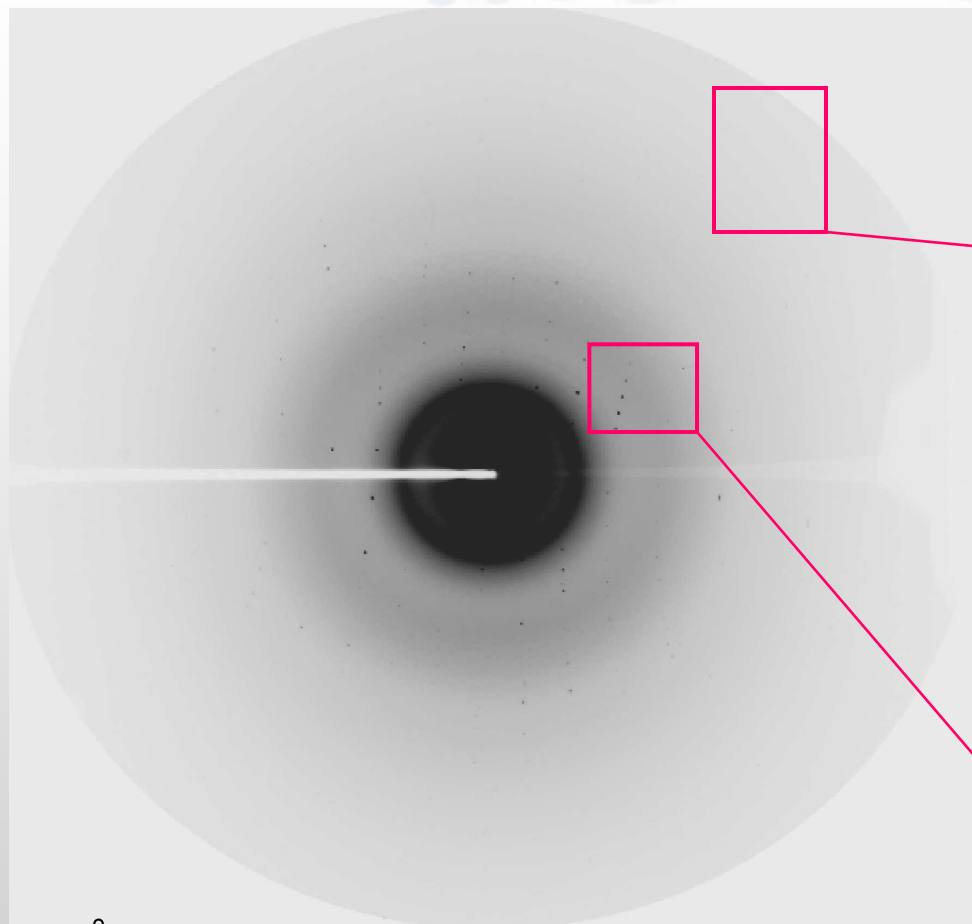
aiguilles

100 μm

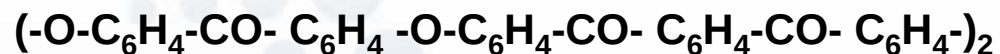
dimensions:

$\sim 26 \times 3 \times 1 \mu\text{m}^3$

50 μm



$-\lambda = 0.775 \text{ \AA}$
 $-T = 100\text{K}$
 $-\text{oscillation} : 6^\circ$
 $-\text{resolution} \sim 0.85 \text{ \AA} \quad 2\Theta = 56.2$



(EKKEK)₂

Space Group: P21/c (14)

unit cell: $a = 7.031(1)\text{\AA}$, $b = 16.875(1)\text{\AA}$, $c = 20.352(2)\text{\AA}$, $\beta = 94.391(2)^\circ$

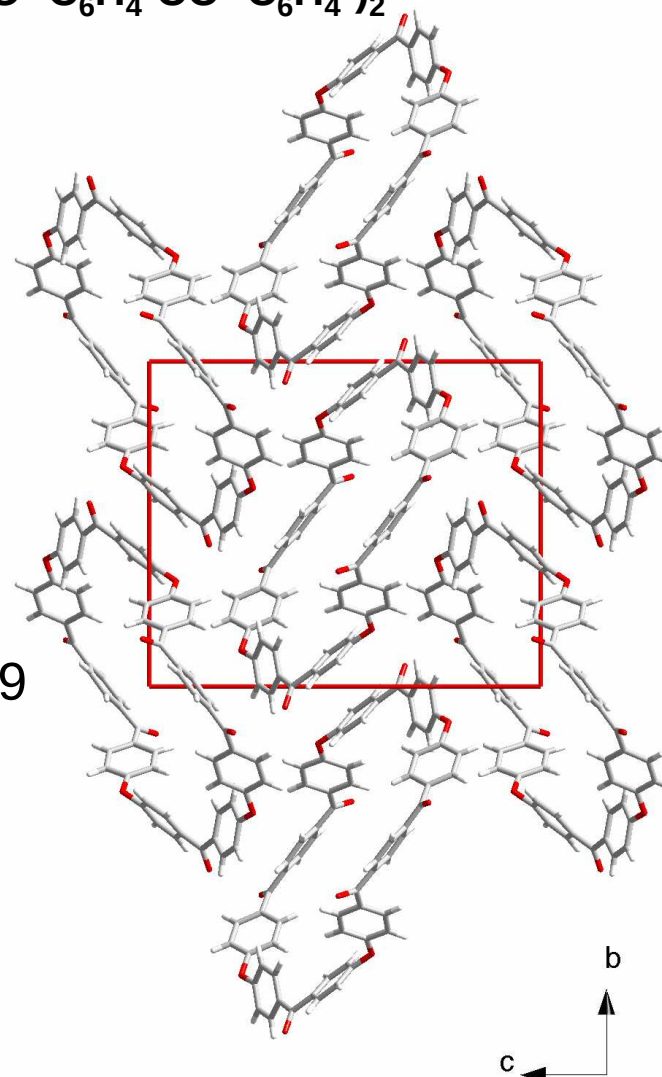
R1= 0.0522, $wR2 = 0.1184$, $GooF = S = 1.039$

for 3356 $F_o > 4\sigma(F_o)$

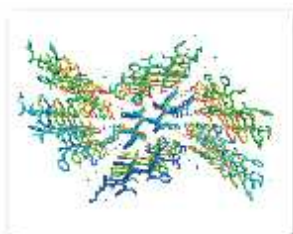
$R1 = 0.0745$, $wR2 = 0.1289$, for all 4344

data

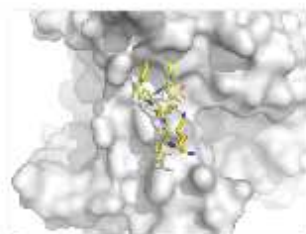
number of l.s. parameters = 343



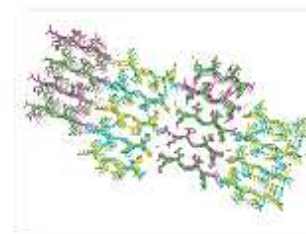
Structures of various Ab fragments :



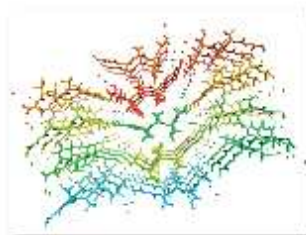
Ab¹⁶⁻²¹ - beta-sheet structure



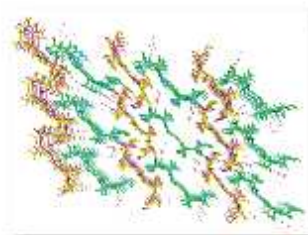
Ab¹⁶⁻²¹ - random coil structure



Ab³⁰⁻³⁵ - beta-sheet structure



Ab³⁵⁻⁴⁰ - beta-sheet structures



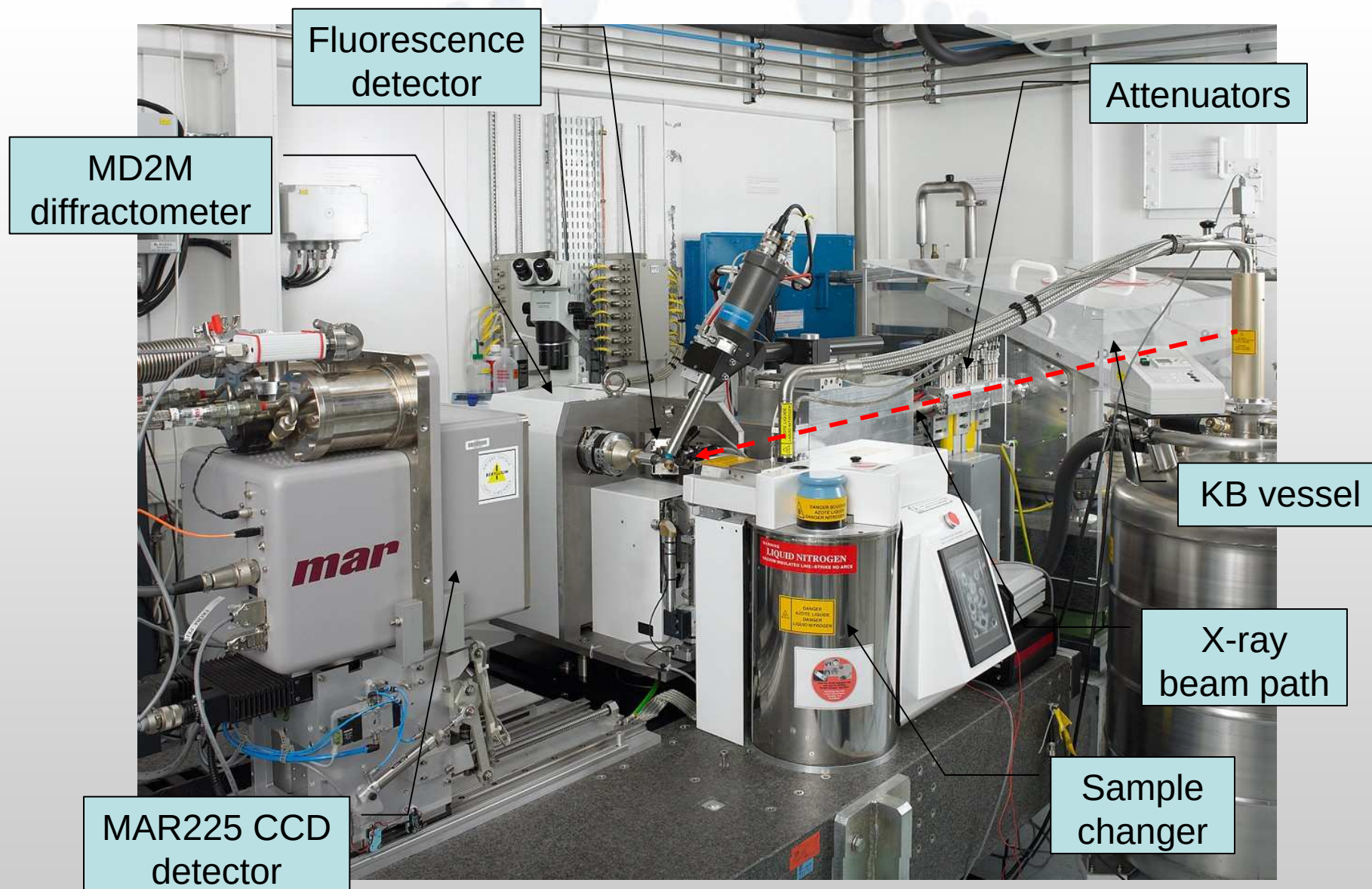
Ab³⁷⁻⁴² - beta-sheet structure

Sawaya *et al.*, (2007) *Nature*, 447, 453.

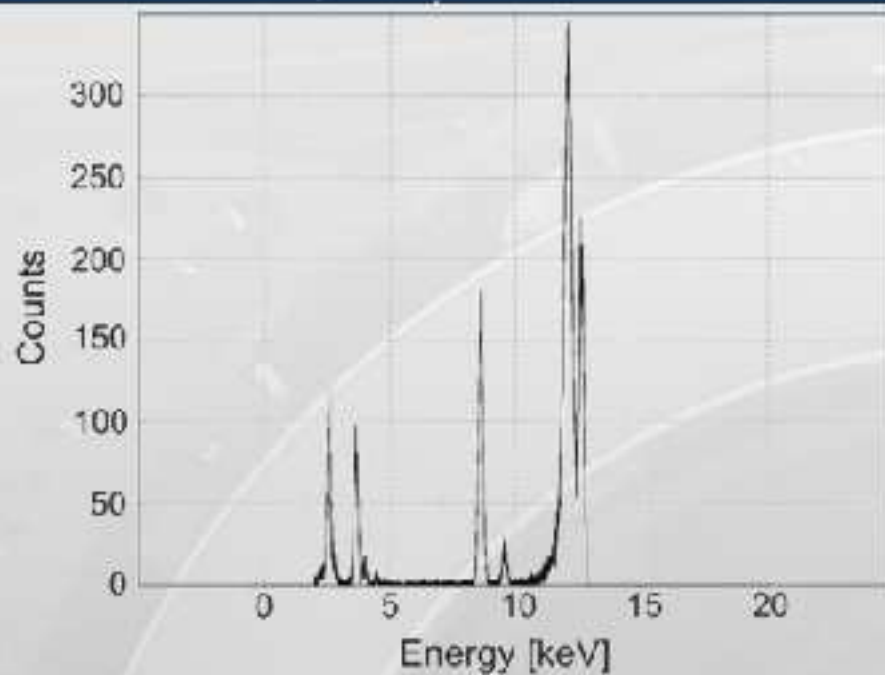


APP_{Chem}²¹⁻²⁶ - beta-sheet structure (intracellular)

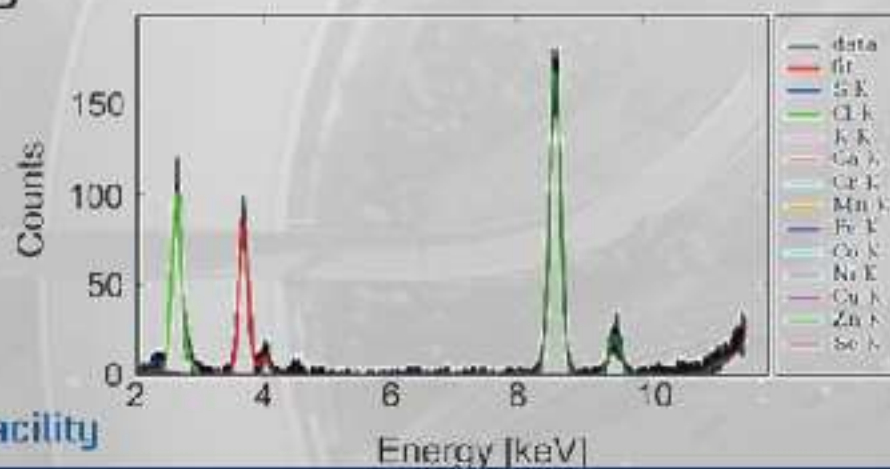




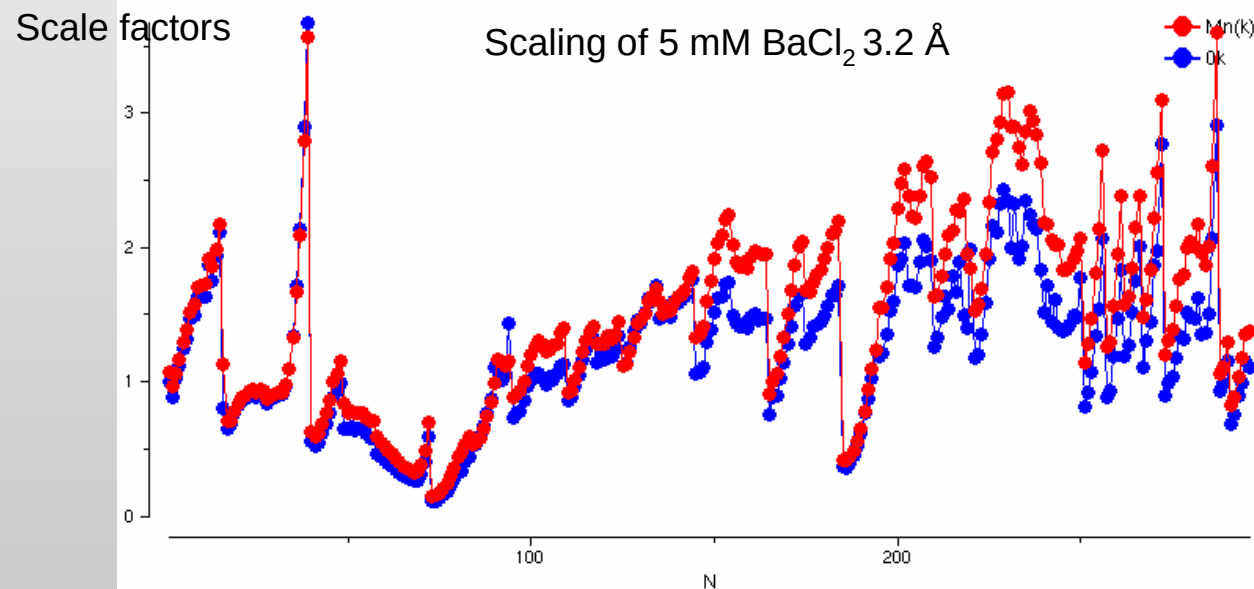
Fluorescence



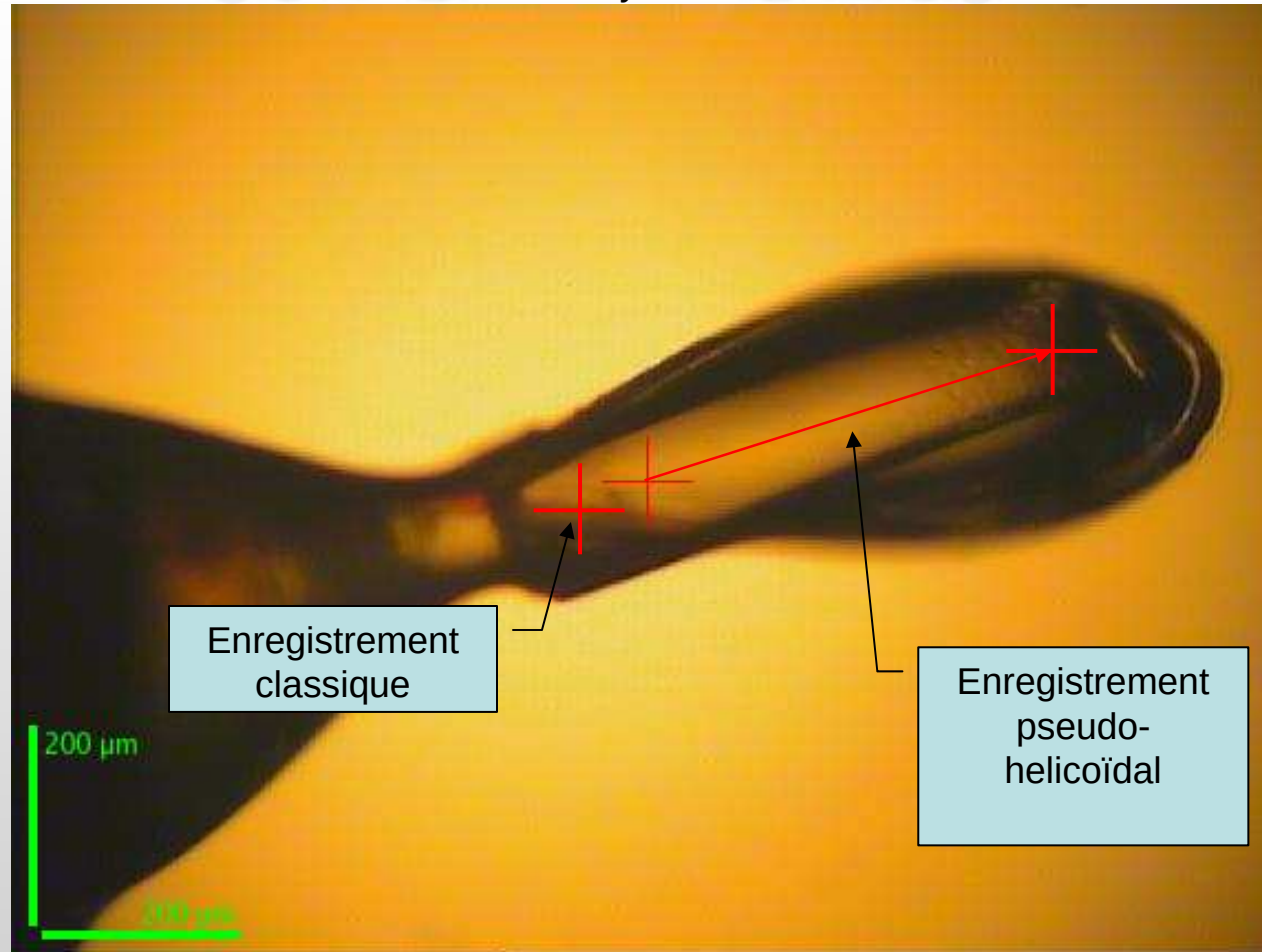
B



Structure of Epstein-Barr virus exonuclease : scaling of microfocus data (SCALA)



F AE crystal



Jeu 1: enregistrement classique , 270 degrees, 1 s 1 degree par image
 Jeu 2 : enregistrement pseudo helicoidal, 1.5 s exposure time per image

Statistiques (XDS)

SUBSET OF INTENSITY DATA WITH SIGNAL/NOISE ≥ -3.0 AS FUNCTION OF RESOLUTION

RESOLUTION LIMIT	NUMBER OF REFLECTIONS			COMPLETENESS OF DATA	R-FACTOR observed	R-FACTOR expected	COMPARED	I/SIGMA	R-meas	Rmrgd-F	Anomal Corr	SigAno	Nano
	OBSERVED	UNIQUE	POSSIBLE										
5.07	36929	6490	6526	99.4%	2.9%	3.5%	36928	43.82	3.2%	1.6%	90%	3.133	2853
3.61	67454	11654	11654	100.0%	3.1%	3.6%	67454	42.72	3.4%	1.7%	78%	2.172	5425
2.95	87304	15019	15019	100.0%	3.7%	4.1%	87304	36.28	4.1%	2.2%	73%	2.067	7113
2.56	103798	17801	17802	100.0%	4.9%	5.1%	103798	28.66	5.3%	3.1%	69%	1.926	8502
2.29	117267	20173	20173	100.0%	6.1%	6.3%	117267	24.02	6.7%	4.1%	59%	1.637	9687
2.09	128809	22249	22249	100.0%	7.7%	7.7%	128809	19.93	8.5%	5.4%	49%	1.418	10722
1.94	139178	24169	24169	100.0%	10.8%	10.7%	139178	15.15	11.9%	7.8%	38%	1.245	11689
1.81	149092	26055	26055	100.0%	16.0%	16.0%	149092	10.73	17.6%	11.8%	26%	1.075	12622
1.71	106265	25577	27669	92.4%	23.2%	23.4%	103797	6.12	26.3%	22.6%	14%	0.927	10900
total	936096	169187	171316	98.8%	5.6%	5.9%	933627	21.09	6.1%	5.6%	53%	1.523	79513

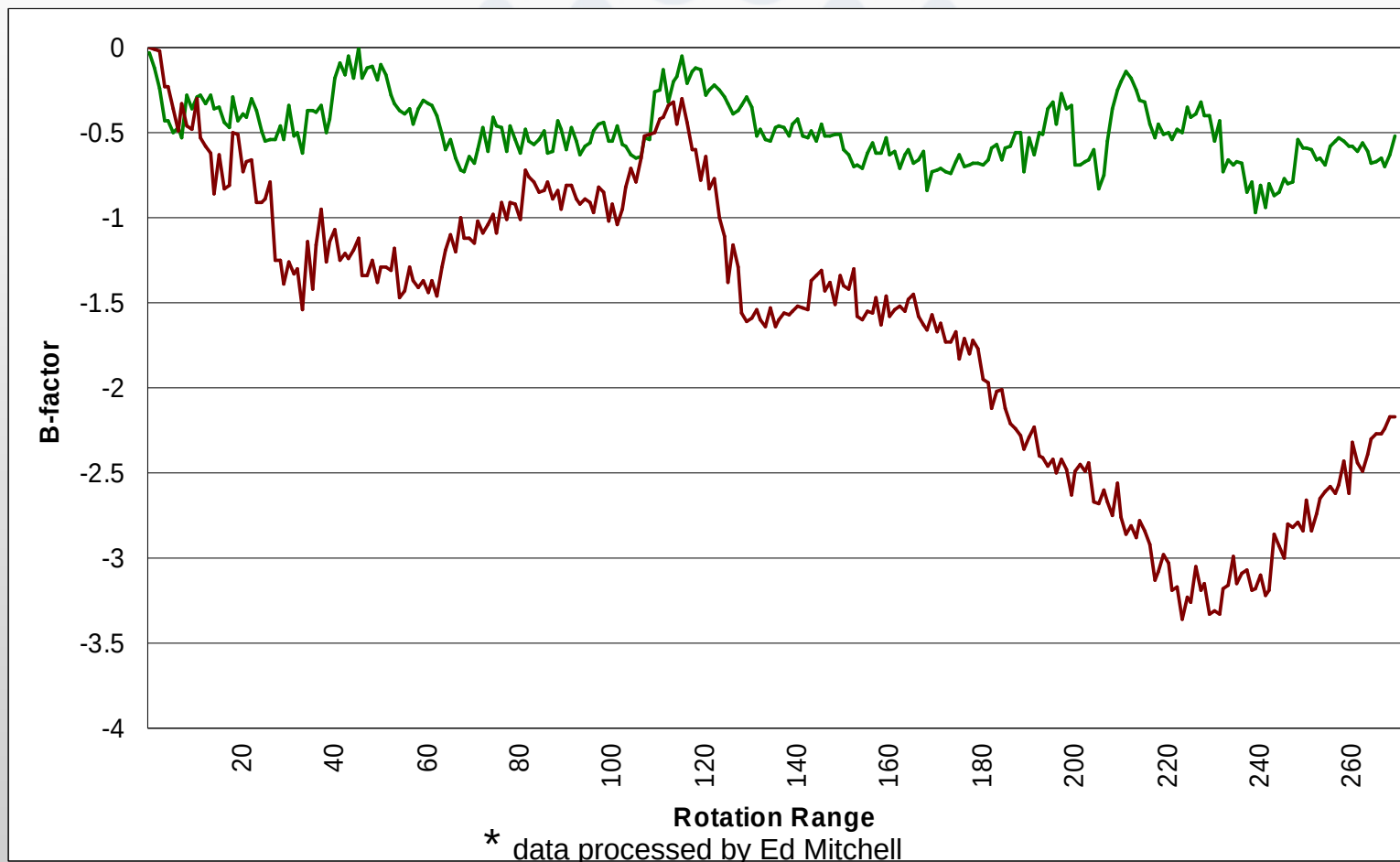
SUBSET OF INTENSITY DATA WITH SIGNAL/NOISE ≥ -3.0 AS FUNCTION OF RESOLUTION

RESOLUTION LIMIT	NUMBER OF REFLECTIONS			COMPLETENESS OF DATA	R-FACTOR observed	R-FACTOR COMPARED expected	I/SIGMA	R-meas	Rmrgd-F	Anomal Corr	SigAno	Nano	
	OBSERVED	UNIQUE	POSSIBLE										
5.09	48730	6401	6439	99.4%	2.0%	2.5%	48727	74.62	2.2%	0.9%	97%	5.649	2814
3.61	88981	11607	11607	100.0%	2.2%	2.6%	88981	72.69	2.4%	1.0%	93%	3.836	5403
2.95	115505	15025	15025	100.0%	2.6%	2.8%	115505	63.40	2.8%	1.2%	91%	3.700	7114
2.56	137168	17774	17774	100.0%	3.1%	3.3%	137168	52.18	3.4%	1.6%	89%	3.498	8489
2.29	155443	20164	20164	100.0%	3.7%	3.8%	155443	45.10	4.0%	2.0%	84%	2.963	9680
2.09	171089	22225	22225	100.0%	4.5%	4.5%	171089	38.57	4.8%	2.5%	79%	2.534	10710
1.93	185579	24196	24196	100.0%	5.8%	5.9%	185579	30.71	6.3%	3.4%	72%	2.139	11703
1.81	198774	26041	26041	100.0%	7.9%	8.0%	198774	23.18	8.5%	4.7%	63%	1.762	12616
1.71	141547	25989	27679	93.9%	10.6%	11.0%	139734	13.94	11.7%	8.6%	47%	1.334	11683
total	1242816	169422	171150	99.0%	3.5%	3.7%	1241000	39.41	3.7%	2.6%	79%	2.634	10212

Jeu 1: enregistrement classique , 270 degrees, 1 s 1 degree par image

Jeu 2 : enregistrement pseudo helicoidal, 1.5 s exposure time per image

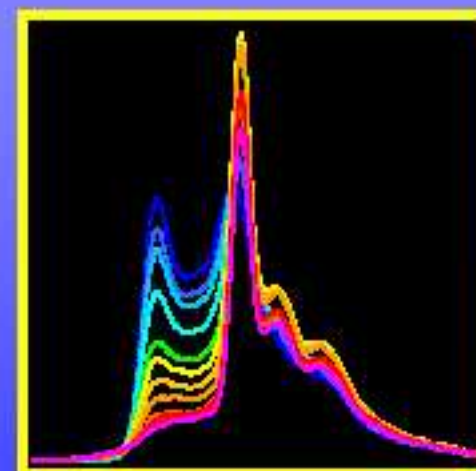
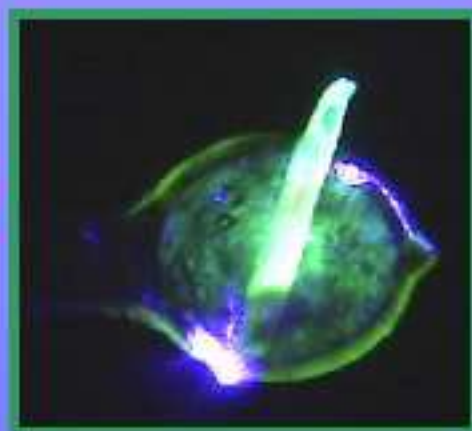
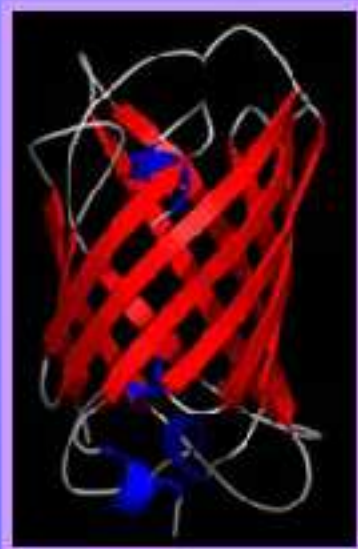
B factor from mosfilm/scala *





Online

John McGeehan, Florent Cipriani and Raimond Ravelli, EMBL



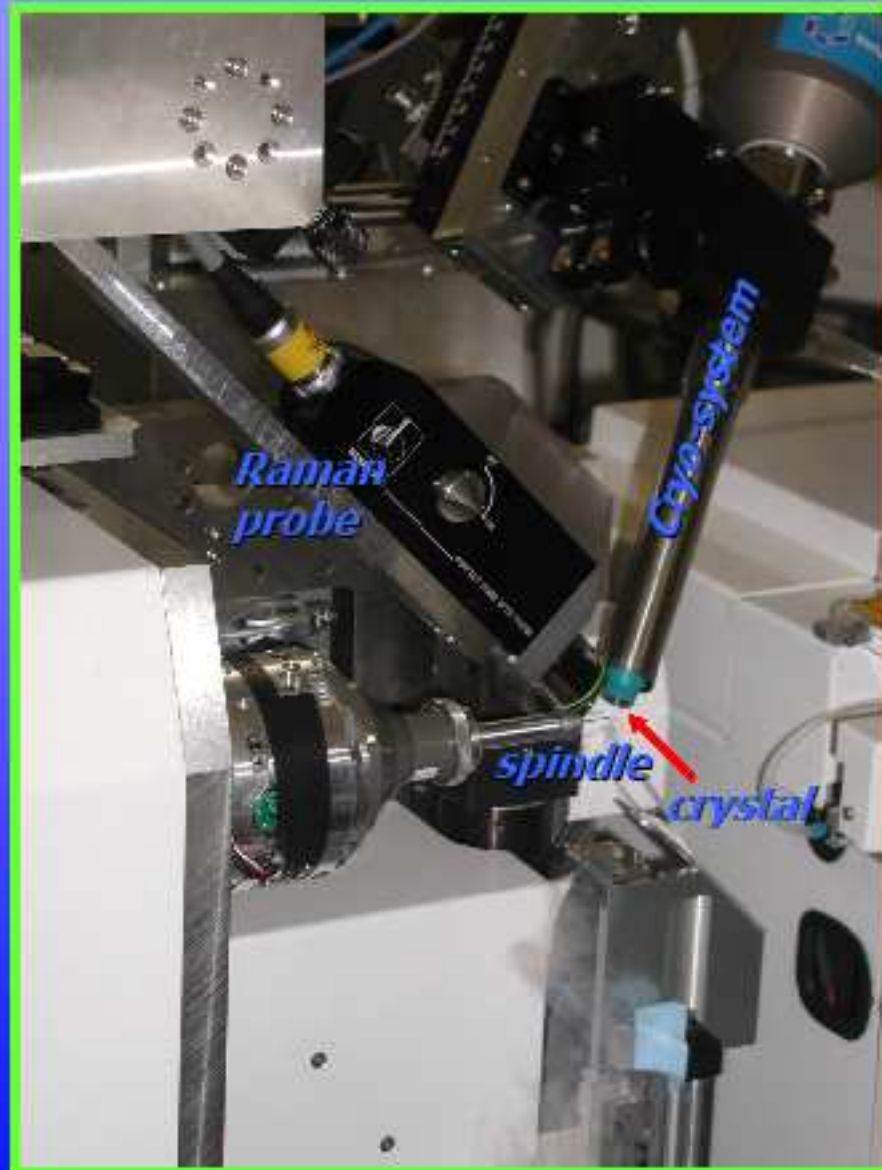
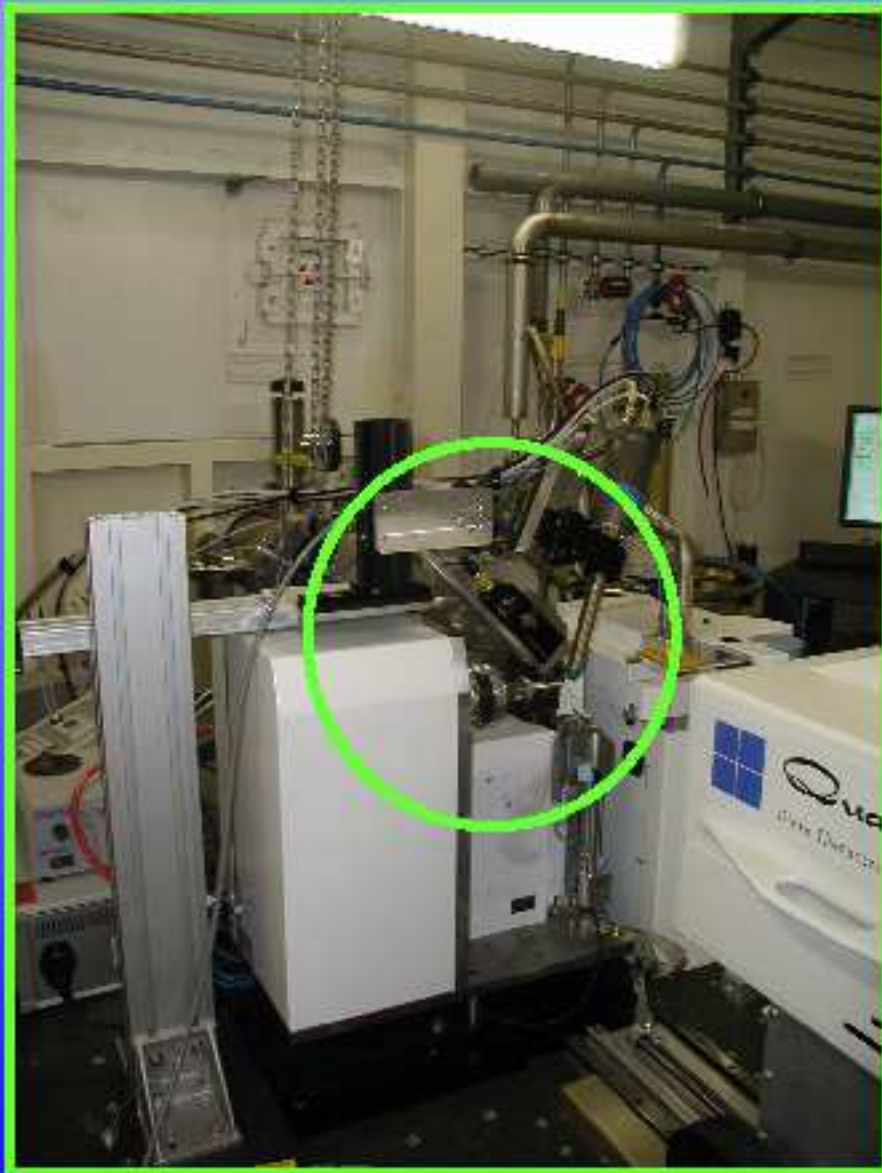
Identification of
intermediate state



Structure of
the
intermediate

a tool to follow reactions in crystals

On-Line Raman experiment



Accès distant

Enregistrement des données est contrôlé à distance par l'utilisateur.

Principe

- Une application maître (sur la ligne de lumière)
- Une application esclave (utilisateur)

Photo: "beta testeur", Johan Turkenburg , York.



Scanning micro-diffractometer

1 μm beam set up on ID13

Guard aperture support

Mirror chamber

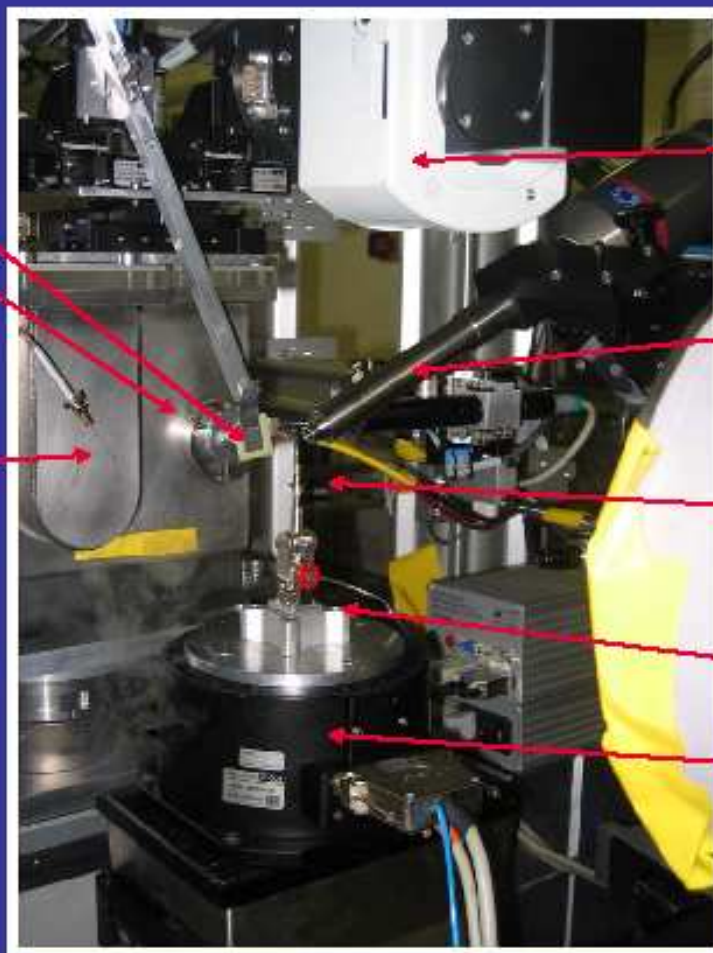
Beamstop

Microscope

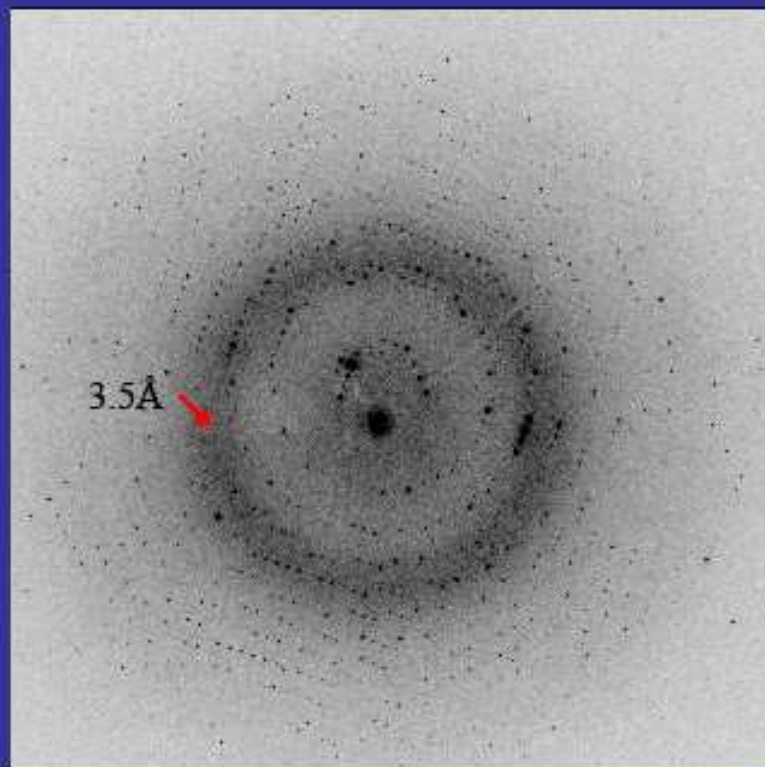
Cryostream

Sample

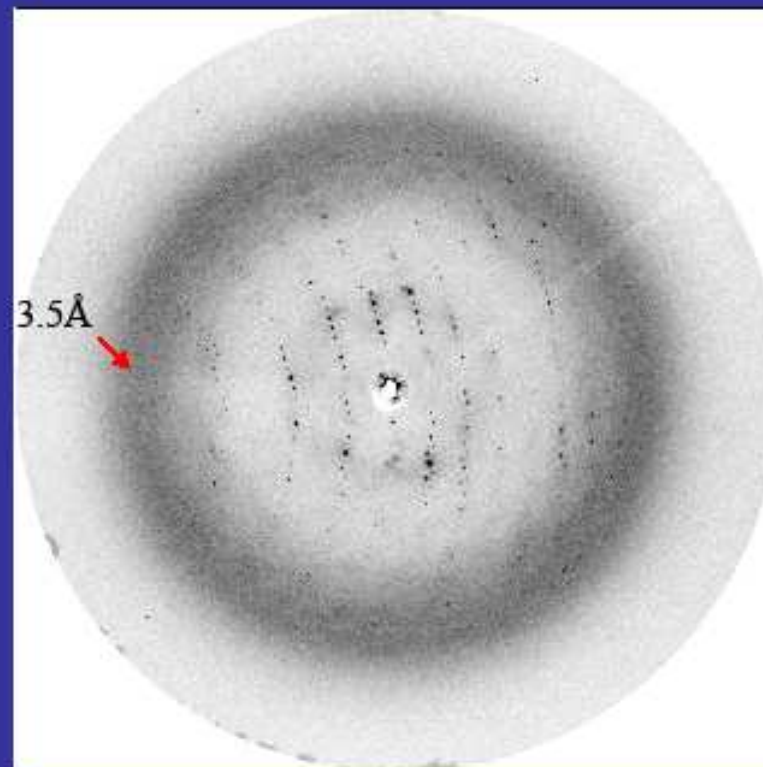
Piezo micromanipulator
with air bearing rotation axis



1 μ m patterns at ID13



Lysozyme



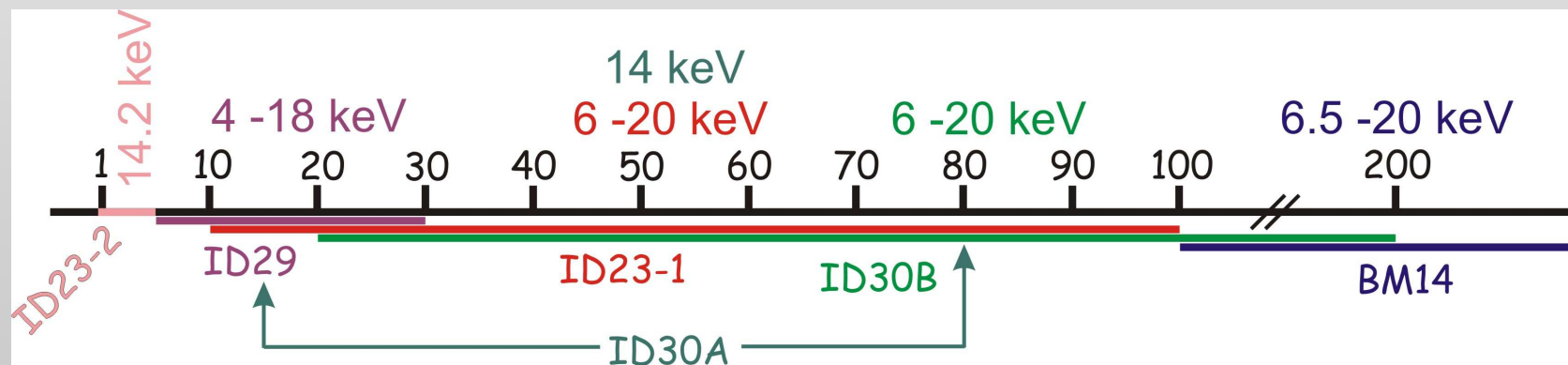
Beta1 adrenergic receptor

ESRF Upgrade Programme 2009–2018



Upgraded MX beam line properties

	<i>Wavelength</i>	<i>Beam size</i>	<i>Anticipated specialty</i>
ID23-1	0.62 - 2.5 Å	1 - 150 µm	Variable beam size
ID23-2	0.873 Å	1 - 5 µm	Microfocus
ID29	0.62 - 2.5 Å	5 - 30 µm	Long wavelength microbeam AD
BM29		700 µm	bio SAXS
ID30A	0.873 Å	2 x 80 µm 1 x 15 µm	Sample evaluation Sample evaluation
ID30B	0.62 - 2.5 Å	10 - 200 µm	Ultra low resolution data collection Flexible beam size



Sciences des matériaux (ID11)

Aujourd'hui:

- hautes énergies, petits faisceaux (140 nm @ 45keV).
- monocristaux (structures cristallographiques, structures électroniques, etc...),
- diffraction de poudre (suivie de réaction "in-situ").

Demain:

- plus de flux à haute énergie (nouvel onduleur, "transfocator")
- extraire informations type "monocristaux" à partir d'échantillons polycrystallins,
- étude de réactions irréversibles (~ 1 ms) par diffraction de poudres ou isolation de l'information provenant d'un seul grain.

Sciences des matériaux (ID31)

Aujourd'hui:

- Diffraction de poudre (6-60 keV, 3-1873 K, contribution instrumentale à la largeur de raie: $2 - 3 \times 10^{-3}^\circ$ 2θ),
- Etudes structurales (robot avec 50 capillaires), études "in-situ" (T, temps, tension, diffusion anormale, analyse quantitative (polymorphisme), microstructures, etc...

Demain:

- 6-90 keV, plus de flux ($E > 30$ keV),
- pouvoir étudier des processus plus rapides (nouveau détecteur, nouveau diffractomètre, etc...)

Et aussi...

ID09 TR (Laue, cinétique)

ID27 (haute pression, haute température)

ID15 (hautes energies, imagerie)

A decorative graphic consisting of numerous light blue and white circles of varying sizes, arranged in a pattern that suggests a beam of light or a diffraction pattern, located in the upper right quadrant of the slide.

**Merci
pour
votre
attention**