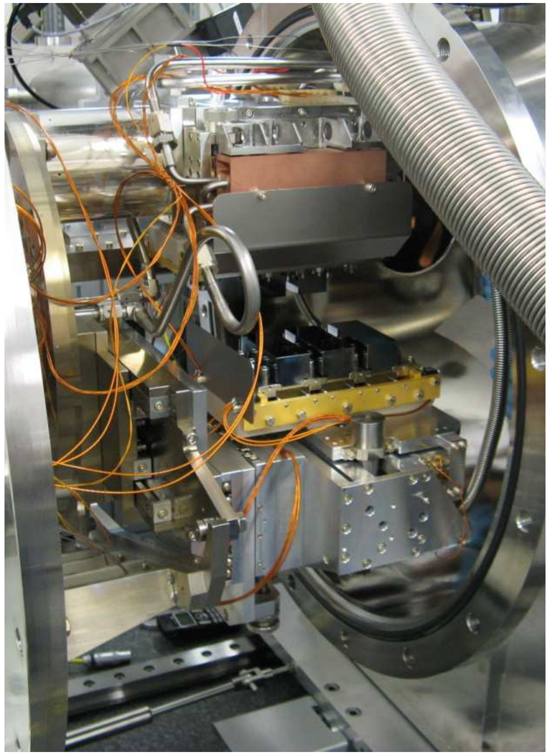
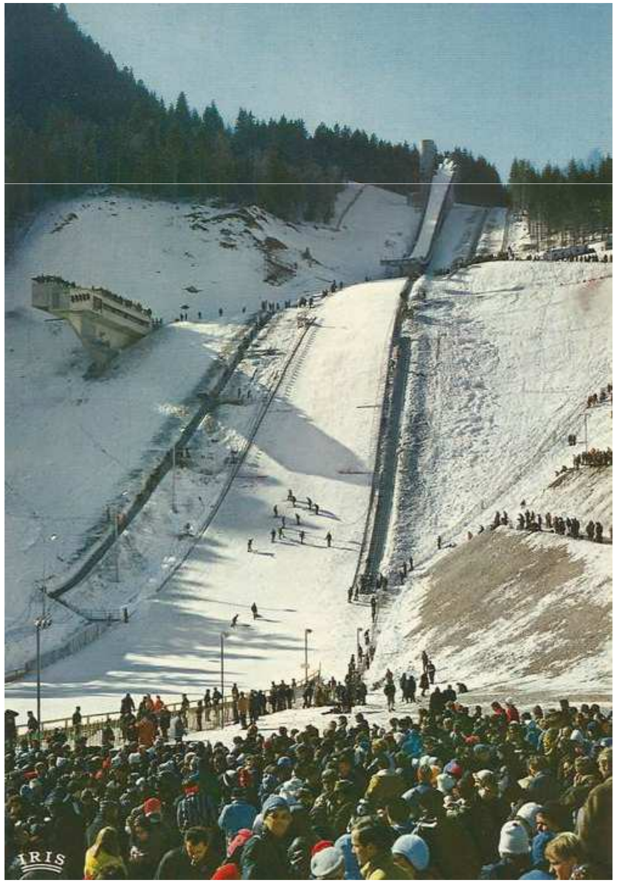
ESRF | The European Synchrotron



Overview and future needs for ESRF double crystal monochromators dedicated to spectroscopy

O. Mathon, P. Glatzel, M. Krisch, A. Rogalev, M. Salome,
R. Tucoulou and S. Pascarelli

Grenoble 1968, winter Olympic games



Workshop dinner, close to the Olympic springboard

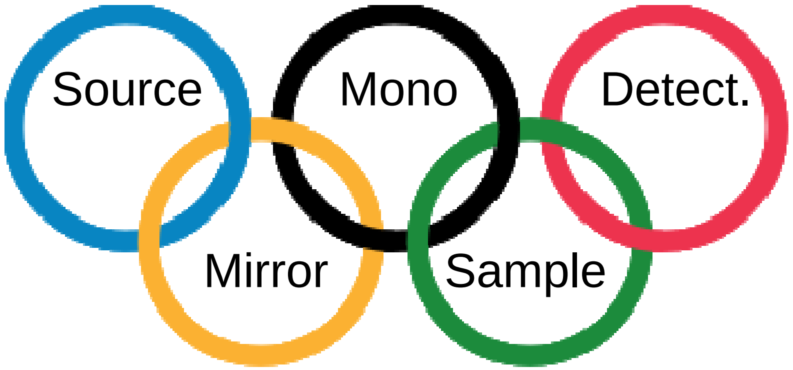
INTRODUCTION

… but also because we propose to adopt for the monochromator workshop
… the Olympic motto, proposed by Pierre de Coubertin on the creation of the International Olympic Committee in 1894...

Citius, Altius, Fortius

... which is Latin for "Faster, Higher, Stronger."

Also because the Olympic symbol, the rings could be a good illustration of the role of the mono …central … but inside a complex instrumentation chain



Double Crystal Monochromator for spectroscopy

- **Overview of ESRF double crystal monochromators dedicated to spectroscopy**

- **Present status and future needs**

- **Monochromator specifications for spectroscopy applications**

SPECTROSCOPY AT ESRF

At ESRF, 14 beamlines are performing spectroscopy activity
(+ 1 soft ID32 + 1 EDXAS ID24)

6 ESRF beamlines

ID12	Polarization dependent spectroscopy	Linear and circular dichroism, XANES
ID16B	NINA	Nano-XRF, nano-spectroscopy
ID20	IXS 1	Inelastic X-ray scattering
ID21	X-ray microscopy	Soft X-ray Nano-XRF, nano-spectroscopy
BM23	EXAFS	EXAFS, XANES, micro-XAS, XRF
ID26	XAS-XES	Emission spectroscopy, XANES, EXAFS

6 CRG beamlines

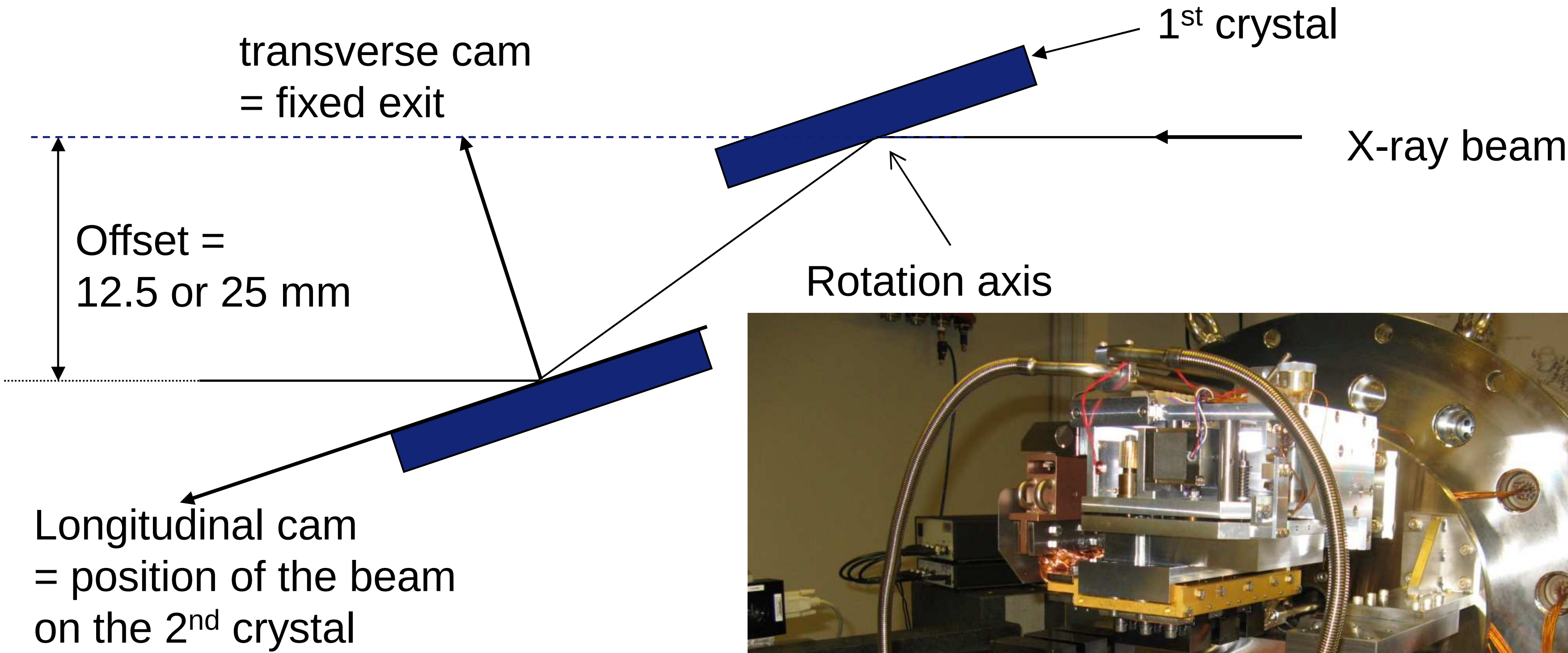
BM01B	SNBL	Combined XRD/XAFS/Raman measurements in operando conditions
BM08A	Gilda	EXAFS, XANES, Refl-XAS
BM20B	RoBL	Radiochemistry XAFS
BM25A	SpLine	EXAFS-XANES
BM26A	DUBBLE	EXAFS, XANES, catalysis infrastructure
BM30B	FAME	XAFS on highly diluted materials, XES, XRF and microXAS

All of them use a Double Crystal Monochromator

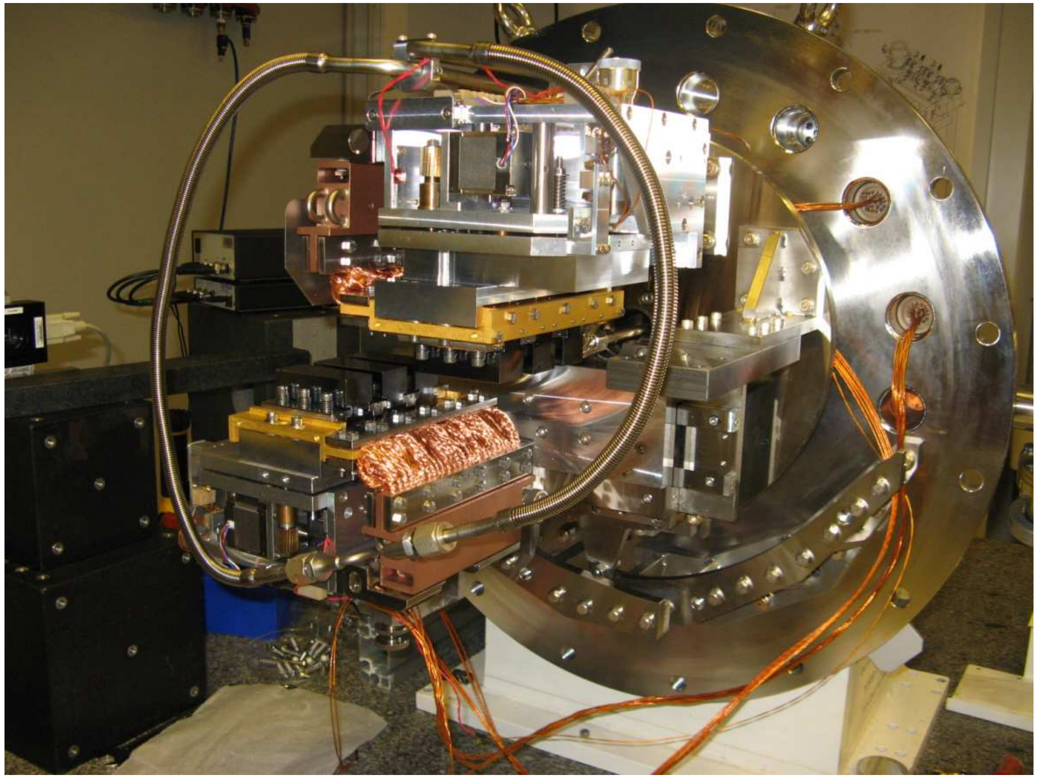
ESRF BEAMLINES – MONOCHROMATOR TECHNOLOGY

	ID12	ID16	ID20	ID21	BM23	ID26
Type	Fixed exit double cam	Fixed exit double cam	Fixed exit double cam	Fixed exit double cam	Fixed exit double cam	Fixed exit double cam
Manufact.	Kohzu	Kohzu	Kohzu	Kohzu	Kohzu	Kohzu
Crystals	111	111/311	111	111/220/ Multilayers	111/311/511	220/311
Angular stroke (°)	9 – 78	3 - 26	5 – 30	3 – 75	3 – 30	5 – 60
resolution	0.1 ”	0.2 ”	0.2 ”	0.2 ”	0.1 ”	0.1”
Offset (mm)	-12.5	-12.5	Variable	-12.5	+25	-25
Cooling	He gas at -190 °C, braids	LN2, side cooling	LN2, side cooling	N_2 at -4 °C	LN2, braids	LN2, side cooling
Upgrade/ modification	Cooling	Cooling, Support	Cooling, geometry, Suppression of horiz. cam	Cooling	Cooling, Support, crystal cage	Cooling, motorization of the horiz. cam
Optimized for…	Polarization, S/N	High energy with nano beam	Inelastic scattering	Low energy with micro beam	EXAFS, S/N, ΔE μXAS	RXES, High flux

KOHZU DOUBLE CAM FIXED EXIT MONOCHROMATOR PRINCIPLE



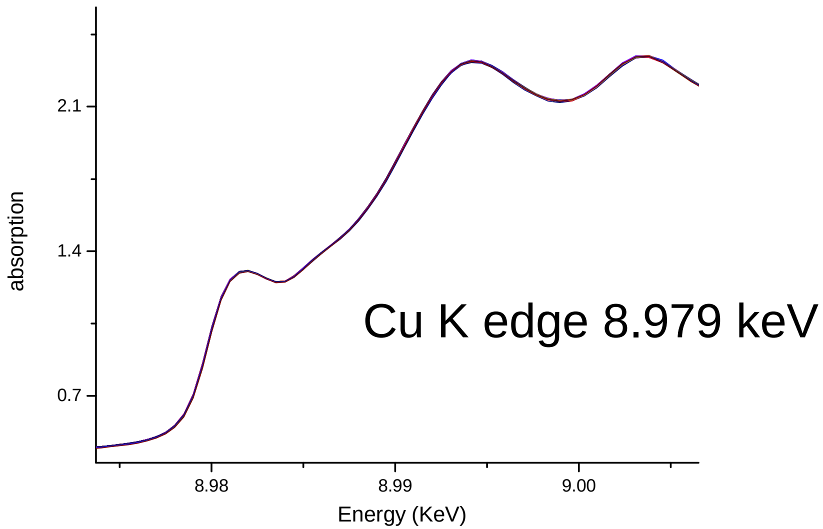
Kohzu has delivered only the mechanical parts.
- Crystals/crystals mounting
- Cooling

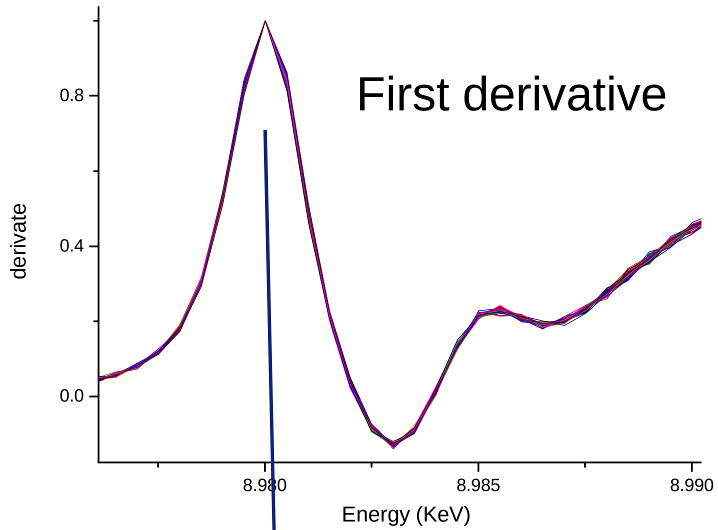


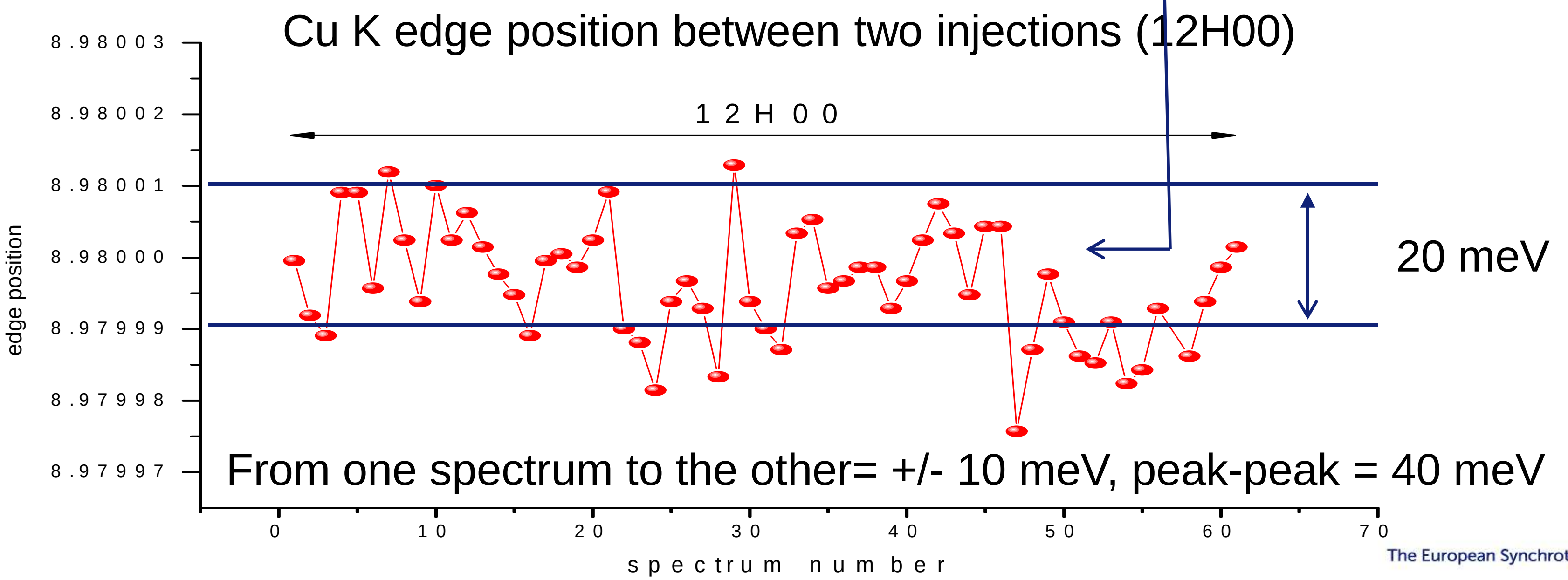
BM23 monochromator

PERFORMANCE : REPEATABILITY

For a XAS beamline, the repeatability of the energy scale is crucial

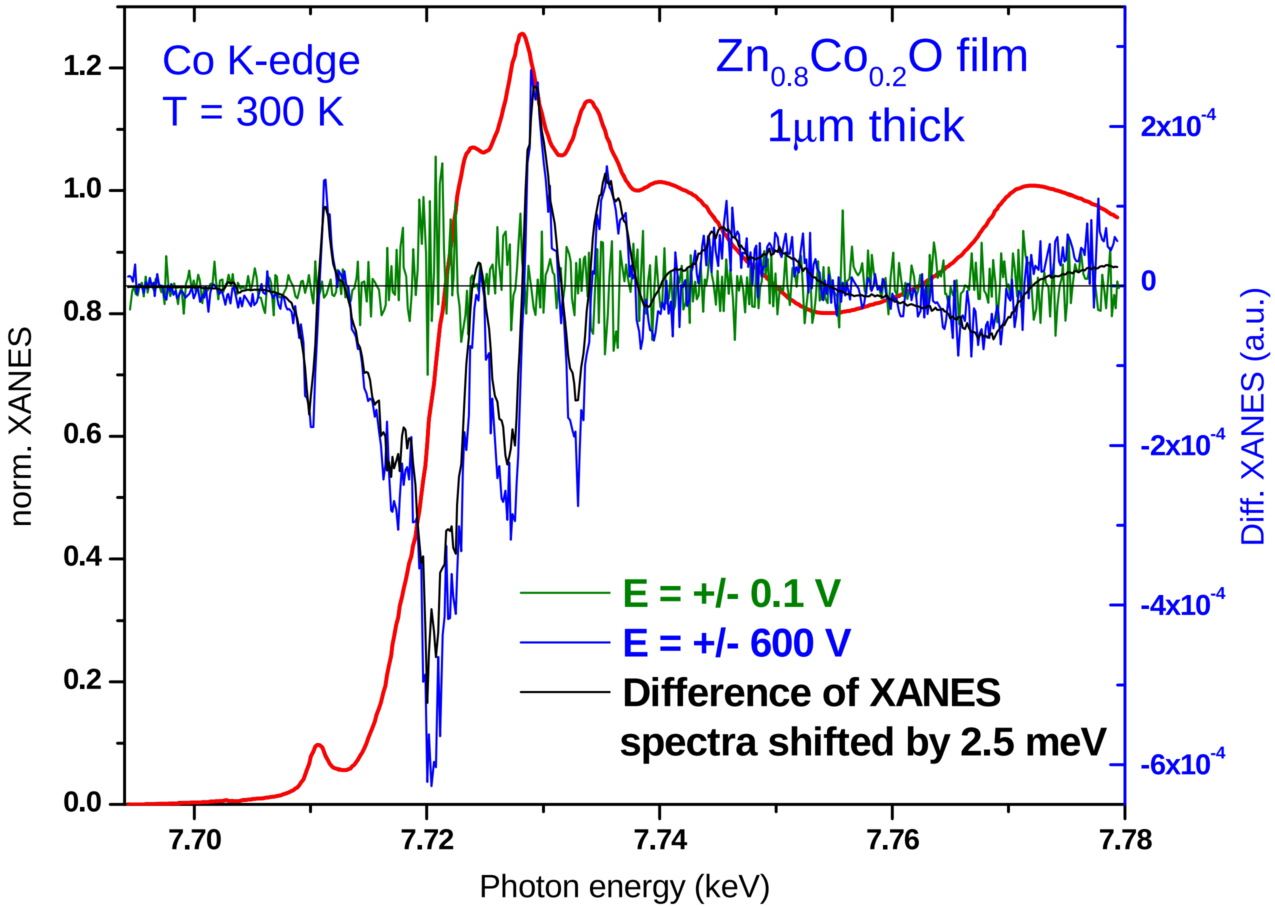






PERFORMANCE : REPEATABILITY

For a differential measurement (XMCD, linear dichroism), where the difference between two successive spectra is performed, the repeatability of the energy scale is fundamental for the S/N ratio.



A. Ney and V. Ney, Linz University, Austria

Slope of the Co K edge
→ 0.2 μ/ eV
A 2.5 meV repeatability error between two successive scans is equivalent to
→ Δμ = 0.0005

PERFORMANCE : PRECISION

$$\frac{\Delta E}{E} = \frac{\Delta\theta}{tg\theta}$$

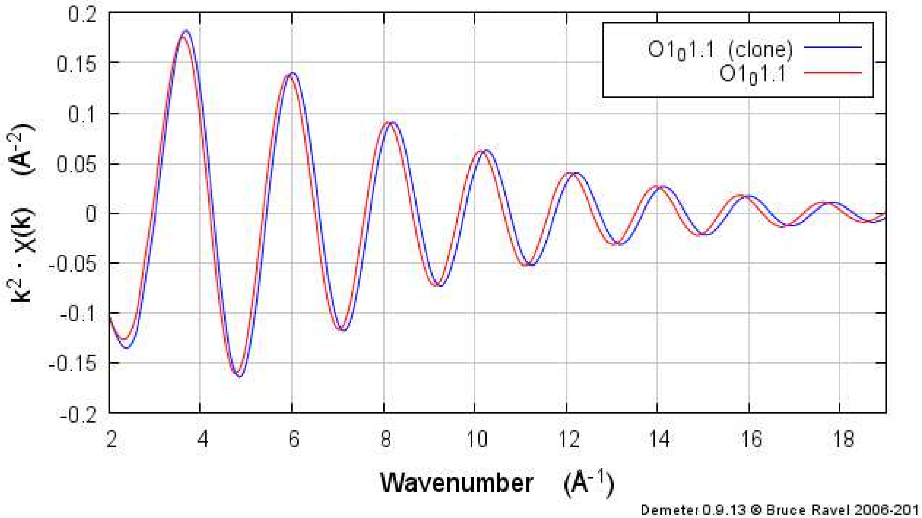
The energy at the edge can be calibrated
But a reasonable precision should be maintained during the EXAFS measurement

k = 20 Å^{-1}, 1500 eV after the edge

$$\Delta E_{\max} = 0.5\, eV$$

Error on photoelectron wave vector:

$$\frac{\Delta k}{k} = \frac{1}{2}\frac{\Delta E}{E_{kin}} = \frac{0.5}{1500} < 2\,10^{-4}$$



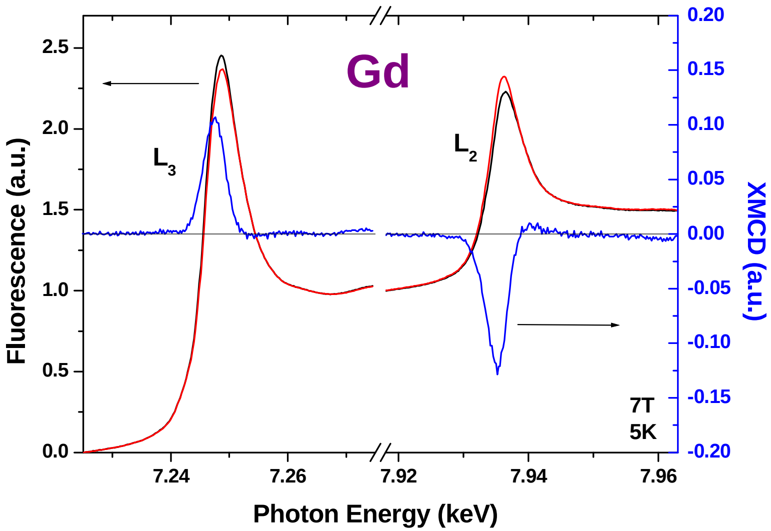
Error on distance:

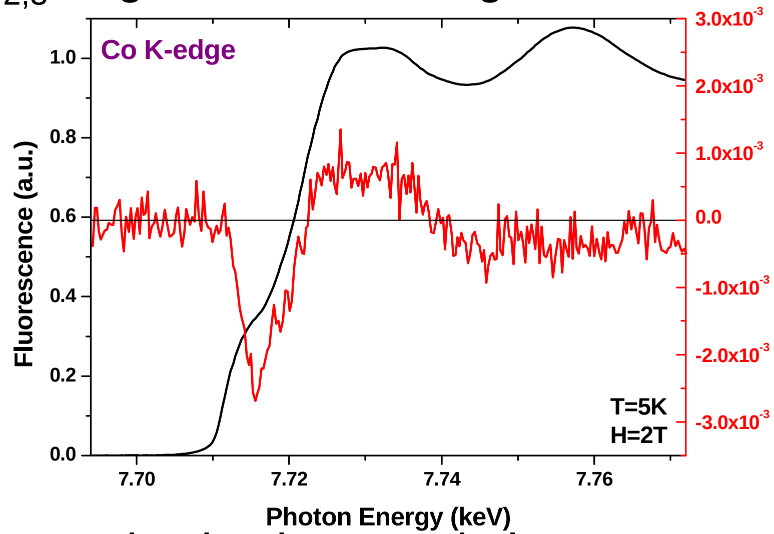
$$\Delta R = R\frac{\Delta k}{k} < 2.5 \times 2\,10^{-4} = 0.0005 \quad Å$$

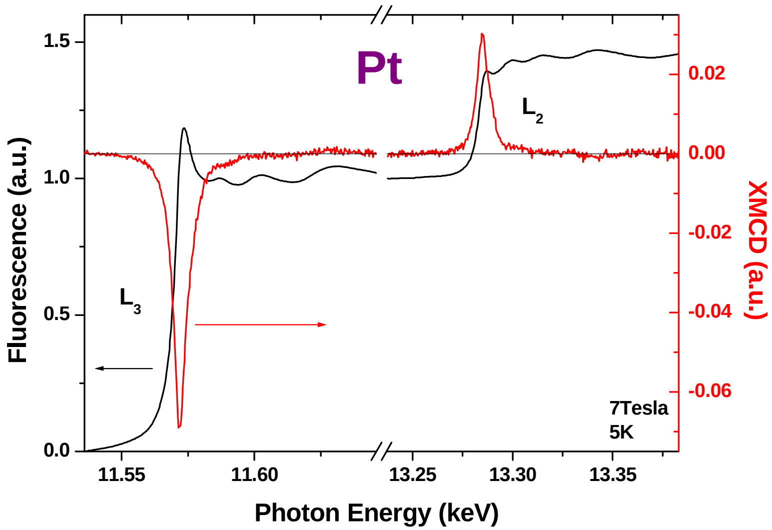
PERFORMANCE : PRECISION

For multi edge XANES measurements, high precision is also necessary.

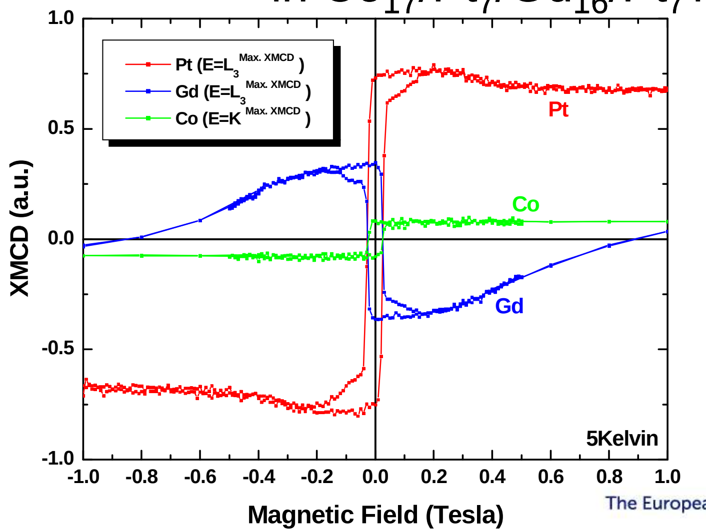
XMCD at Pt and Gd $L_{2,3}$-edges and Co K-edge







Element selective hysteresis-loops in $Co_{17}/Pt_7/Gd_{16}/Pt_7$ multilayer

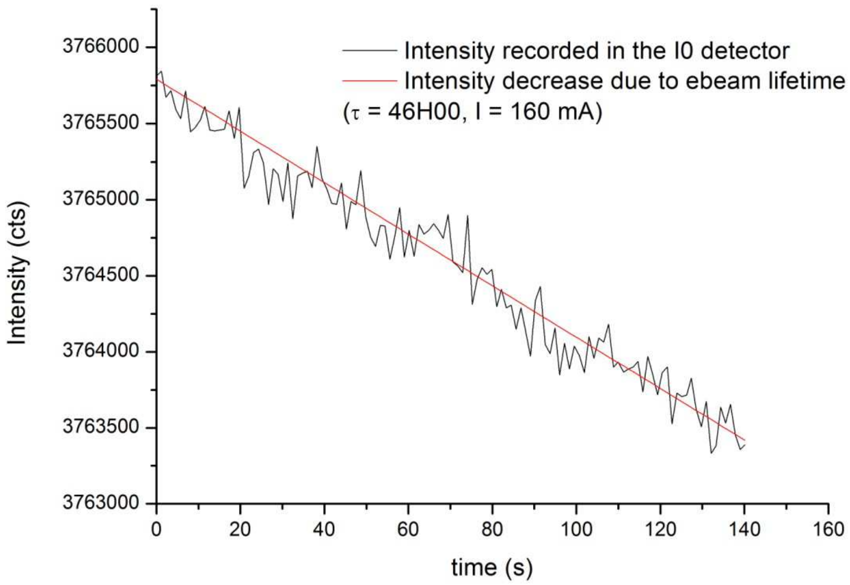


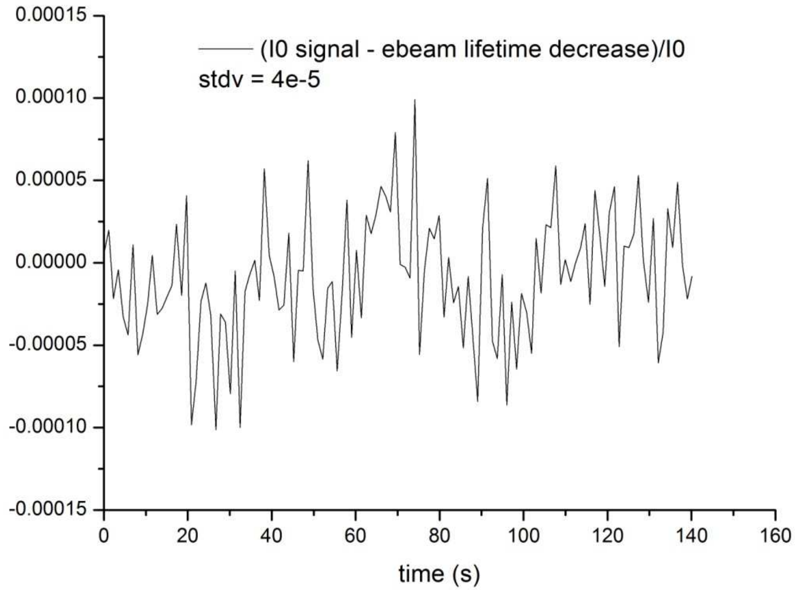
PERFORMANCE : STABILITY AT FIXED ENERGY

The stability of the beam intensity at fixed energy is very sensitive to any drift (thermal drifts or mechanical vibrations).

Important for XRF, combined diffraction and XAS in general !

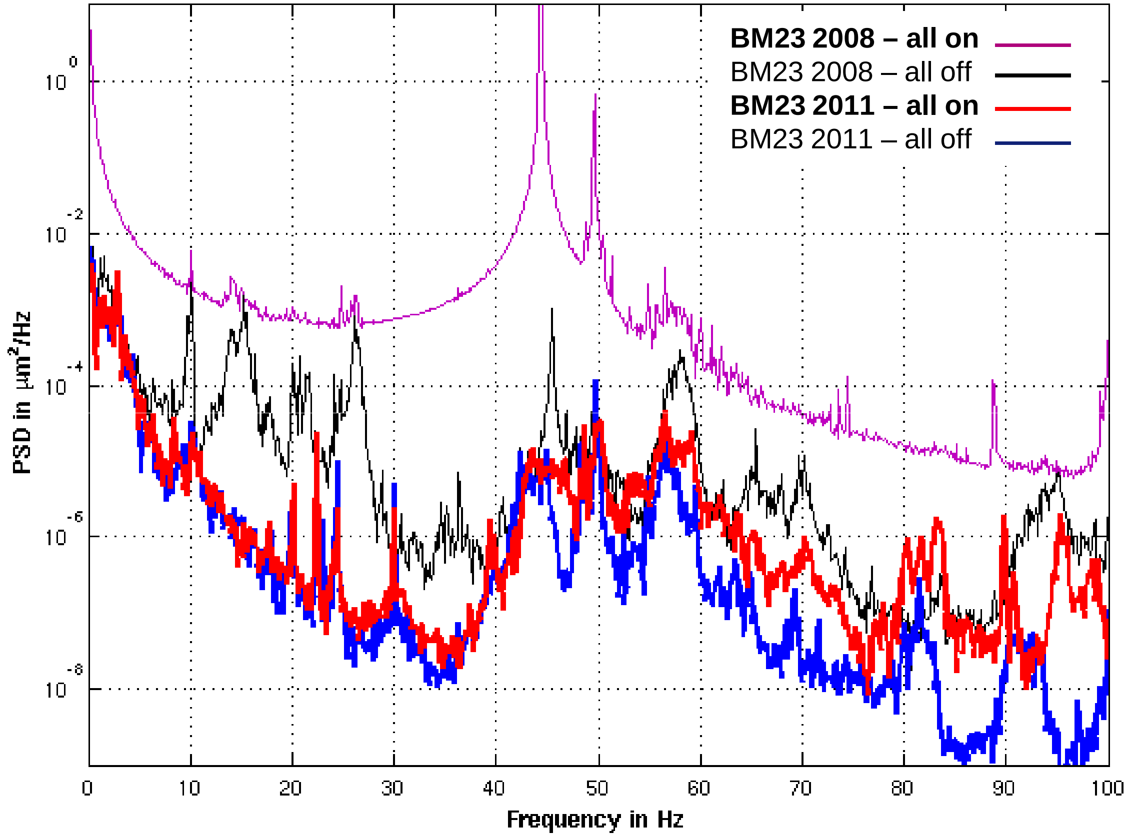
Crucial for RXES





PERFORMANCE : STABILITY AT FIXED ENERGY

Mechanical stability, **vibration**: crucial for new applications like hyperspectral mapping where the **continuous scan** acquisition scheme is mandatory.



Upgrade of the monochromator support

Kohzu metallic support → ESRF granite support

Upgrade of the cooling system

He gas close circuit → ESRF LN2 circuit

PERFORMANCE : CRYSTAL PARALLELISM AND FIXED EXIT DURING SCAN

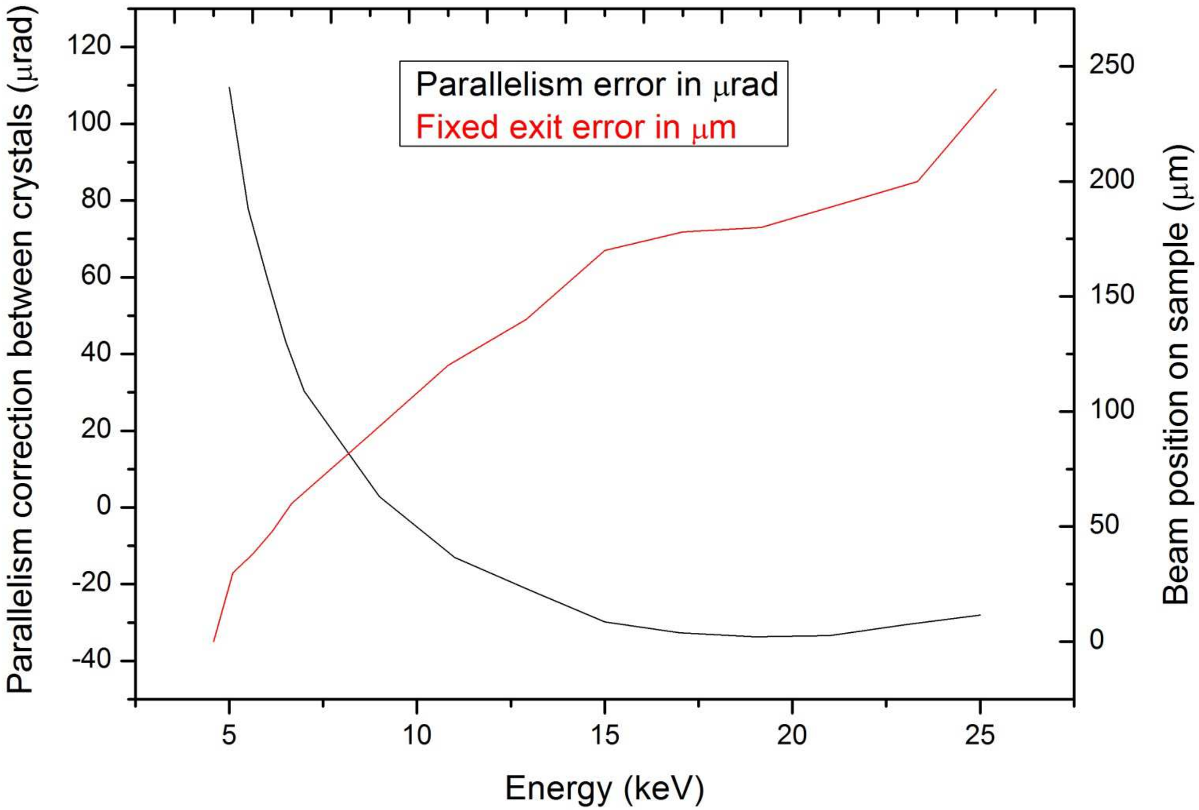
Main drawback of BM23 Kohzu monochromator

Crystal parallelism and “fixed” exit of the X-ray beam during scan

In average for BM23 :

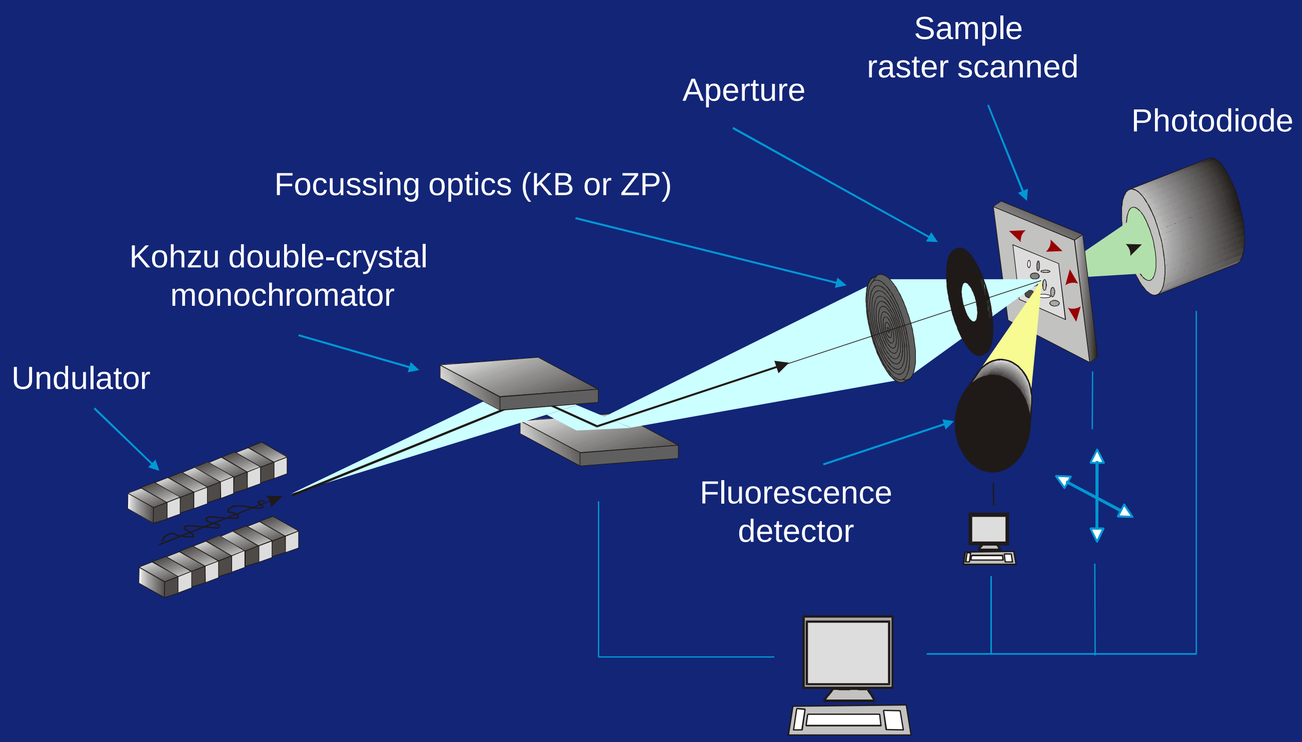
8 μrad/keV (8.5 μrad/deg.)

12 μm /keV (12.6 μm/deg.)

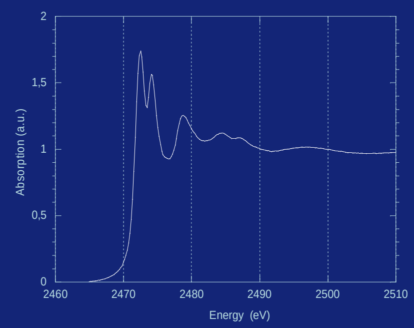


→ **feedback** on the piezo is **mandatory** to perform a XAS spectrum

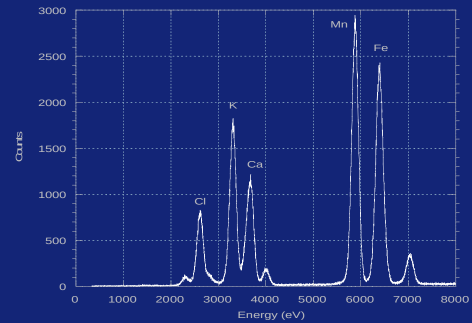
PERFORMANCE : CRYSTAL PARALLELISM AND FIXED EXIT DURING SCAN



μ-XANES



μ-XRF



- Energy range: 2 – 9 keV

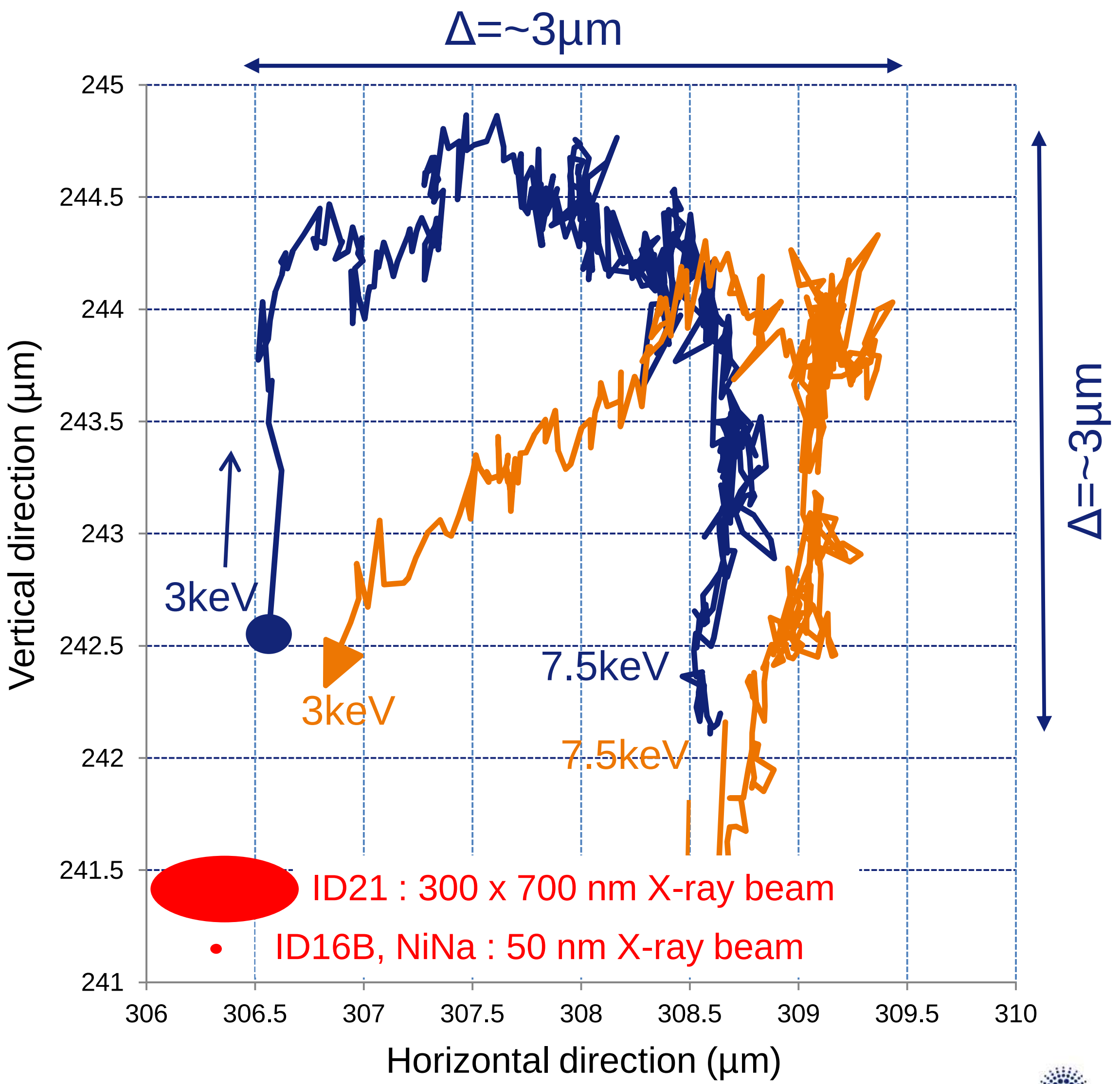
- Double crystal, fixed exit Kohzu monochromator
  Si 111, Si 220, NiB_4C multilayers

- Micro-beam 0.3 x 0.7 μm^2 ~10^{10} photons/s

PERFORMANCE : CRYSTAL PARALLELISM AND FIXED EXIT DURING SCAN

Micro-beam trajectory in sample plane during an energy scan

- Si (111) monochromator
- KB focused micro-beam
- Energy range : 3 keV to 7.5 keV and back,10 eV steps
- Angular range: 41.23 to 15.28 º
- Micro-beam position measured on fluorescence screen in KB focal plane with video-microscope in BPM mode
- Δ=3µm in focal plane corresponds to ΔRy=~10µrad



PERFORMANCE : CRYSTAL PARALLELISM AND FIXED EXIT DURING SCAN

Micro-beam trajectory in sample plane during an energy scan

1- Beam movement is very reproducible

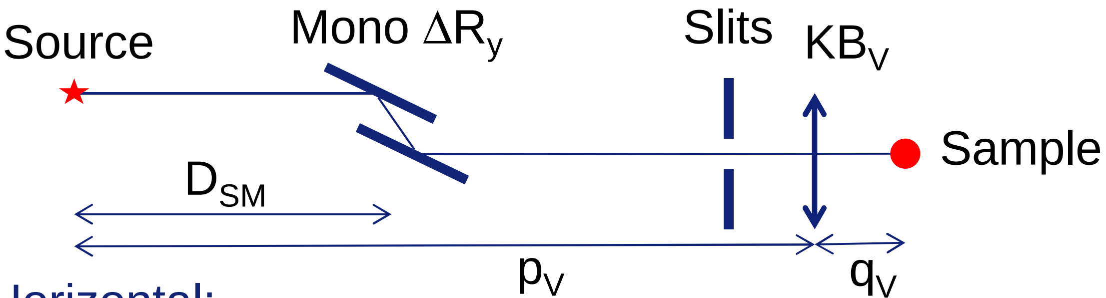
→ Present strategy : Compensation strategy “Spot tracking”

2 - Better monochromator performance

→ Stronger specifications on ΔR_y, ΔR_x and fixed exit

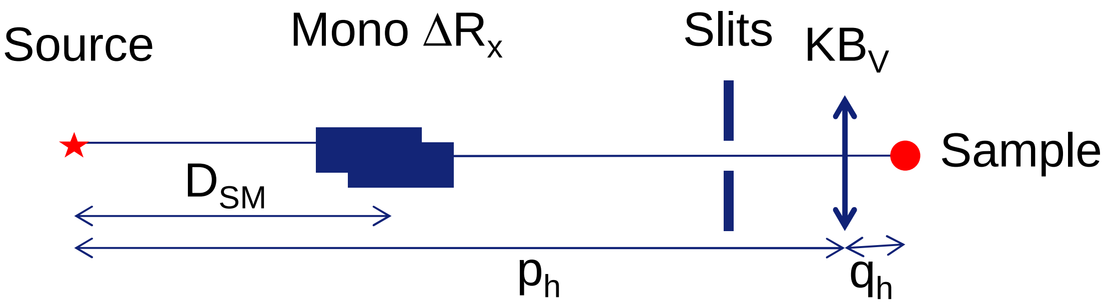
3 - Choice of the optical configuration to be less sensitive to monochromator imperfection

Vertical:



$$\Delta V = 2\frac{D_{SM}}{p_V} q_V \Delta R_y$$

Horizontal:



$$\Delta H = 2\frac{D_{SM}}{p_h} q_h \Delta R_x \sin\theta_B$$

PERFORMANCE : AGEING BEHAVIOR

	ID12	ID16	ID20	ID21	BM23	ID26
Delivery	1993	1995	1997	1997	1993	1995

Remarkable longevity :

The DCM are operational and daily used for 20 years (ID12/BM23) !

Modifications have been done

- Cooling (all)
- Crystals mounting (all)
- Geometry (ID20)
- Cam system (ID26/ID20)
- Motors

Maintenance performed regularly

- Crystals
- Mechanics (principal gear, reduction)
- Motors
- Setup of the cam/translations

4 generations later !

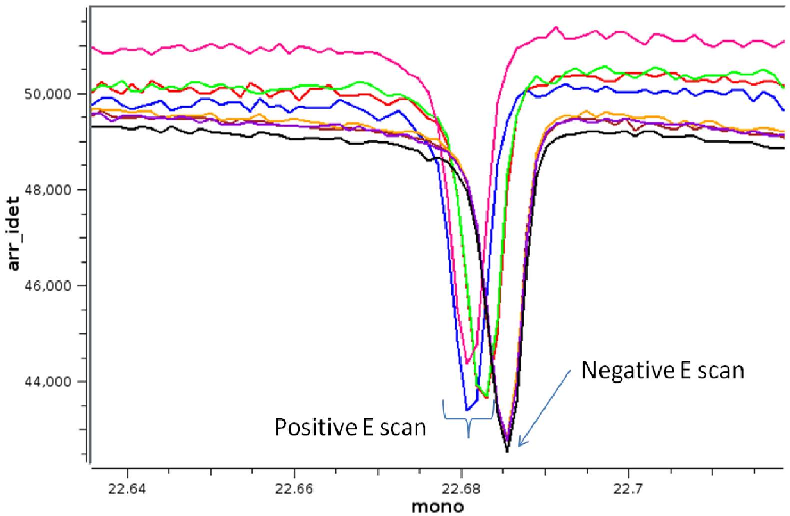
But certain parts have never been modified:

- Main axis
- Ferrofluidics seal

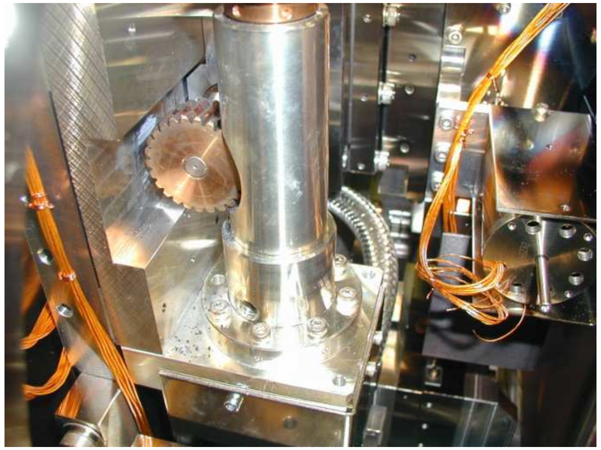
PERFORMANCE : AGEING BEHAVIOR

	ID12	ID16	ID20	ID21	BM23	ID26
Delivery	1993	1995	1997	1997	1993	1995

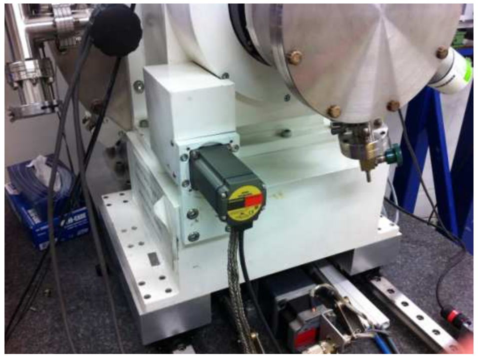
… but start to suffer from ageing



Degradation of the energy scale stability around certain very used edges



Mechanical wear on the worm gears damaged and replaced



Main rotation motor replaced

… And also a conception that is perhaps not adapted to new spectroscopy challenges (thermal, vibration, control, global conception)

Ageing : clear checks and maintenance procedures

FUTURE REQUIREMENT : SCIENTIFIC AND TECHNICAL GOALS

	ID12	ID16	ID20	ID21	BM23	ID26
EXAFS	Y	Y	N	Y	Y	Y
XANES	Y	Y	Y	Y	Y	Y
XRF	Y	Y	Y	Y	Y	Y
RXES/RIXS/NRIXS	Y	Y	Y	Y	Y	Y
Final state E scan	N	N	Y	N	N	Y
Combined diffraction	Y	Y	Y	Y	Y	Y
Micro/nano beam	Y	Y	N	Y	Y	Y
XRF Mapping	Y	Y	N	Y	Y	Y
Hyperspectral mapping	N	Y	N	Y	Y	Y
Step by step	Y	Y	Y	Y	Y	Y
Continuous scan	N	Y	N	Y	Y	Y

No request for asymmetric cuts, detuning mode, sagittal focusing or polarization transfer specifications.

FUTURE REQUIREMENTS : XAS

	ID12	ID16	ID20	ID21	BM23	ID26
EXAFS	Y	Y	N	Y	Y	Y
XANES	Y	Y	Y	Y	Y	Y

- Cover large number of elements edges → Accessible angular range of the mono
- Number and type of crystals mounted inside the monochromator
- Scan the angle (energy) → K = 20 $Å^{-1}$ = 1500 eV after the edge
  - At high energy = 0.3 deg.
  - At low energy … 20 deg.

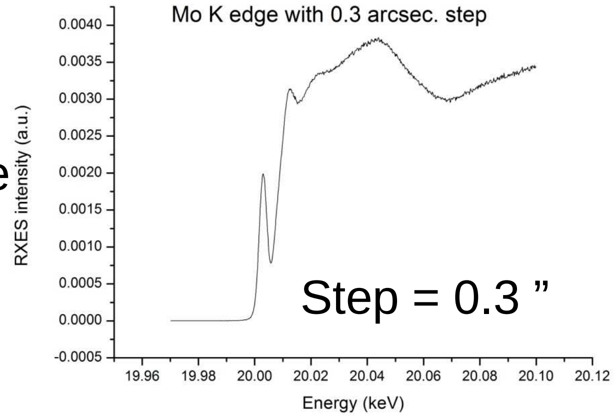
	ID12	ID16	ID20	ID21	BM23	ID26
Min. Bragg angle (deg.)	7	3	5	10	2	4
Max. Bragg angle (deg.)	80	20	30	81	35	70
Nb. of crystals pairs	2	2	1	2 to 3	2 to 3	>2
Crystal types	111/?	111/311	111	111/311/?	111/311 /511	111/220 311/411
Scan angle range (deg.)	0.1 - 4	0.2 - 4	0.1 - 1	0.5 - 20	0.2 - 8	0.2 - 8

FUTURE REQUIREMENTS : XAS

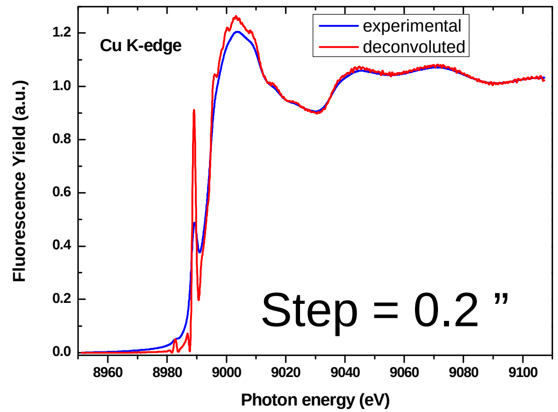
	ID12	ID16	ID20	ID21	BM23	ID26
XANES	Y	Y	Y	Y	Y	Y
RXES	Y	Y	Y	Y	Y	Y

- Minimum angle step :
  → Energy step needed linked to the core hole life time γ
  → γ/E (k edge) ≈ 2.10^{-4}
- A minimum step resolution of $\gamma/20$ is needed (≈ 0.1 eV step at Fe K edge, for example)
  $\Delta\theta$ = 3 µrad at Fe K edge … and $\Delta\theta$ = 0.5 µrad at W K edge
- Advanced spectroscopy : $\gamma/40$ could be needed

ID26 : RXES at Mo K edge



ID12 : Cu k deconvolution techniques



	ID12	ID16	ID20	ID21	BM23	ID26
Min. step angle (arcsec.)	0.1	0.1	0.2	0.2	0.1	0.1

FUTURE REQUIREMENTS : ENERGY RESOLUTION

	ID12	ID16	ID20	ID21	BM23	ID26
EXAFS	Y	Y	N	Y	Y	Y
XANES	Y	Y	Y	Y	Y	Y
RIXS	Y	Y	Y	Y	Y	Y

•Energy resolution : $\Delta E / E = (\omega_D^2 + \Psi^2)^{1/2} \cot \theta_B$

Darwin width ω_D is linked to the crystal

Ψ is a complex contribution that includes
The divergence of the X-ray beam
All deformations of the crystal that can affect the front wave
(crystal fixation, thermal deformation, surface polishing …)

$\Psi << \omega_D$

	ID12	ID16	ID20	ID21	BM23	ID26
Intrinsic broadening Ψ (µrad)	< 1	< 1	< 1	< 4	< 1	<1

FUTURE REQUIREMENTS : REPEATABILITY/STABILITY OF THE ENERGY SCALE

	ID12	ID16	ID20	ID21	BM23	ID26
XANES	Y	Y	Y	Y	Y	Y
Hyperspectral mapping	N	Y	N	Y	Y	Y

In static mode

For XRF, not so crucial → < 1 eV at 20 keV (1 arcsec.)

For combined XRD → < 1 eV at 25 keV (1 arcsec.)

In scanning mode

From one scan to the other : repeatability in the order of 10 meV at Fe k edge

→ 0.5 µrad (0.1 ”) on the main Bragg angle

Stability: Maintain the repeatability for 24H00

	ID12	ID16	ID20	ID21	BM23	ID26
θ_B repeatability (arcsec.) over 24H00	<< 0.1	0.1	0.1	0.5	0.1	< 0.1

FUTURE REQUIREMENTS : PRECISION OF THE ENERGY SCALE

	ID12	ID16	ID20	ID21	BM23	ID26
XANES (multi edges)	Y	Y	Y	Y	Y	Y
EXAFS	Y	Y	N	Y	Y	Y

EXAFS : the precision of the energy scale determines the precision on the distance of neighbors (over one EXAFS scan) → 0.5 eV : 5 arcsec.

XANES/XMCD : multiple edges measurements (over 10 deg.) < 100 meV → 1 arcsec.

	ID12	ID16	ID20	ID21	BM23	ID26
Angle precision (arcsec.)	< 0.5	1	1	1	1	< 0.5

FUTURE REQUIREMENTS : BEAM POSITION STABILITY (STATIC MODE)

	ID12	ID16	ID20	ID21	BM23	ID26
XRF	Y	Y	Y	Y	Y	Y
XRF maps	Y	Y	N	Y	Y	Y

Notion of stability in **static mode** is linked to the **duration of the experiment** (maps)

Constraints could be different between
macro beam operation
micro/nano beam operation

The stability requirements are defined **on the sample** and then interpreted in terms of thermal drifts, mechanical drifts and vibrations limits on the monochromator

	ID12	ID16	ID20	ID21	BM23	ID26
Δz, Δy on sample over 24H00, unfocused (μm)	1 by 1	0.005 by 0.005	0.5 by 0.5	0.03 by 0.03	1 by 1 0.2 by 0.2	1 by 1

FUTURE REQUIREMENTS : BEAM POSITION STABILITY (SCANNING MODE)

	ID12	ID16	ID20	ID21	BM23	ID26
XANES	Y	Y	Y	Y	Y	Y
EXAFS	Y	Y	N	Y	Y	Y

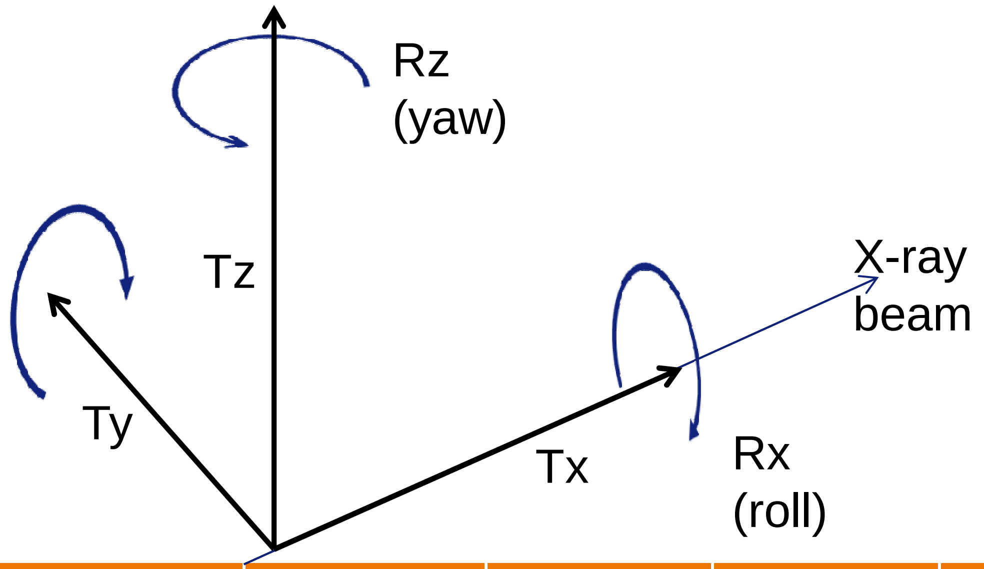
Notion of stability in **scanning mode** is linked to the **angular range of the scan**.

Again, constraints could be different between
macro beam operation
micro/nano beam operation

The monochromator should be intrinsically stable and errors should be reproducible (lookup tables corrections) as much as possible. Residuals errors could be optimized with active feedback : control/communication issue

	ID12	ID16	ID20	ID21	BM23	ID26
Δz, Δy on sample over 1 deg. (μm)	1 by 1	0.01 by 0.01	0.5 by 0.5	0.05 by 0.05	0.2 by 0.2	1 by 1
Δz, Δy on sample over 5 deg. (μm)	1 by 1	0.025 by 0.025	NA	0.15 by 0.15	0.5 by 0.5	1 by 1
Δz, Δy on sample over 20 deg. (μm)	NA	NA	NA	0.15 by 0.15	NA	NA

FUTURE REQUIREMENTS : BEAM POSITION STABILITY (SCANNING MODE)



	ID12	ID16	ID20	ID21	BM23	ID26
ΔRy over 1 deg. (μrad)	0.2	0.15	0.2	0.1	0.1	0.1
ΔRy over 5 deg. (μrad)	0.5	0.5	NA	0.2	0.25	0.25
ΔRy over 20 deg. (μrad)	NA	NA	NA	0.5	NA	NA
ΔRx over 1 deg. (μrad)	0.2	1.5	3	0.7	0.7	0.7
ΔRx over 5 deg. (μrad)	0.5	1.5	NA	1.4	1.4	1.4
ΔRx over 20 deg. (μrad)	NA	NA	NA	1	NA	NA
ΔRz (μrad)	10	10	10	10	10	10

FUTURE REQUIREMENTS : CONTINUOUS SCAN

	ID12	ID16	ID20	ID21	BM23	ID26
Continuous scan	N	Y	N	Y	Y	Y

Stability (vibration) issues are critical for continuous scan mode as the energy (angular) scale becomes also a time scale.

Bi-directional energy scan becomes important for rapid continuous scan.

Control issues:

The monochromator should communicate with the detection and with the source.

Complex trajectory of the Bragg angle could be envisaged

Accurate measurement of the Bragg angle at full speed

	ID12	ID16	ID20	ID21	BM23	ID26
EXAFS (s/scan)	NA	3	NA	10	1	1
XANES (s/scan)	NA	1	NA	3	0.2	0.2

CONCLUSIONS

Vertical and Horizontal homogeneity of the requests

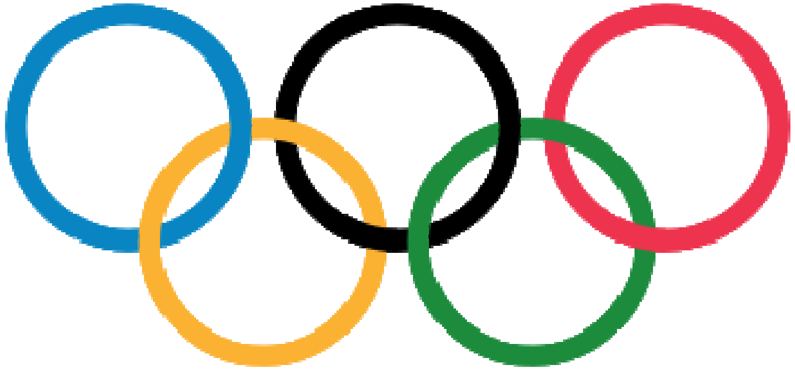
Angle range: Low energy / high energy monochromator compatible ?

We have presented a list of requirements for a future double crystal monochromator dedicated to spectroscopy….

…. That are in general a factor 10 more stringent than the present performance announced by the main industrial suppliers

Chapter and Topic	unit	data f[illegible] spectroscopy beam lines						Comments
		ID16	ID20	B[illegible]23	ID21	ID26	ID12	
Bragg angle configuration								
Max Bragg	deg.	20	30	[illegible]5	81	70	80	Energy range
Min Bragg	deg.	3	5	[illegible]	10	4	7	
Min angle step	change to	0.1	0.2	[illegible]1	0.2	0.1	0.1	arcsec (0.1 '' = 0.5 microrad) for doing scienctific experiments
Angular resolution								should be a fraction of the line above: 1/4 about (engineering feature)
~~angle total repeat~~								
total angle precision	definition			0.[illegible] ?				? Absolute angles (deg) : precision de EXAFS
Bragg angle scans								
angle range EXAFS	deg	4 - 0.1	NA	8 -[illegible].3	40-2	8.0 - 1	15 - 0.5	deg
angle range XANES	deg	0.6 - 0.1	1-0.2	1 -[illegible].2	20 - 0.8	1-0.2	2 - 0.2	deg
Continuous scan velocity EXAFS	s	3	NA			1	NA	s
Continuous scan velocity EXAFS	ms/pt	1	NA		100	1	NA	ms/pt
Continuous scan velocity XANES	s	1	NA	0[illegible]3		0.1	NA	s
Continuous scan velocity XANES	ms/pt	1	NA		100	1	NA	ms/pt
step by step motor velocity	s/deg	3(no spec)	3				NA	s/deg vitesse moteur
Continuous Scan frequency EXAFS	Hz	1	NA			0.1	NA	Hz
Continuous Scan frequency XANES	Hz	1	NA			1	NA	Hz
Triangular scan time based								
Triangular scan Energy based								
continuous scan		Y	N	[illegible]	y	y	N	
step by step		Y	Y	[illegible]	y	y	Y	
Fixed Energy		Y	Y	[illegible]	y	y	Y	
Fixed energy stability in time	meV/day	23.8	50	1[illegible]	100 over 12 H	100	<10	10 meV/day at 7 keV = 0.5 microrad
Fixed energy stability in time		1						microrad/day at 7 keV = 0.5 microrad
angle repeatability forward		10	10	1[illegible]	50	10	<10	meV at 7 keV
angle repeatability backward		10	10	1[illegible]	50	10	<10	
angle precision forward		100	100	10[illegible]?	100?	10/100	< 50	meV depends on the stroke
angle precision backward		100	100	10[illegible]?	100?	10/100	< 50	
Crystal setup Thermal Stability								
stabilisation time constant to energy change	mn	10	10	[illegible]	10	NA(10)	~ 2	mn (for a large angle change >3 deg)
Energy thermal settling time during scanning	s	0.3	0.1	0[illegible]1	0.1	0.1	0.1	s (Time range to get new stable energy set point, step angle)
Temperature first crystal	°K	130	130	1[illegible]0	130	130	130	K
Temperature second crystal	°K	130	130	1[illegible]0	130	130	130	similar to the 1st crystal temperature
Temperature of the mechanics							> 5	°C
Crystal parallelism (without feedback correction)								
ΔRy static	µrd							
ΔRx static	µrd							
ΔRy during scan	µrd	0.15	1	[illegible]		0.5	0.5	microrad (Parallelism error over 1 degree)
ΔRx during scan	µrd	3	3	[illegible]		10 micron a 30 m + KI	1	microrad (Parallelism error over 1 degree)
ΔRz during scan	µrd	NA(10)	30	1[illegible]	NA	NA(10)	1	microrad (Parallelism error over 1 degree)
DRy Parallelism over 10 degree	µrd	1.5	5	[illegible]	0.2	2.5	2	
DRx Parallelism 10 degree	µrd	5	15	[illegible]	0.2	0 micron a 30 m + KI	2	
DRz Parallelism 10 degree	µrd	NA(10)	30	[illegible]	NA	NA(10)	2	
DRy Over 45	µrd	NA	NA	N[illegible]	3	NA		
DRx over 45	µrd	NA	NA	N[illegible]	3	NA		
Crystal setup Mechanical stability								
Dz-Dy								
Dz,Dy at sample position-typically	µm	0.005 x 0.005	0.5 by 0.5	1 b[illegible]1		1 by 1	2.5x2.5 over v	E Static not focused
Dz,Dy at sample position-typically	µm	0.005 x 0.005	0.5 by 0.5	1 b[illegible]1		1 by 1	2.5x2.5 over v	E Static focused
Dz,Dy at sample position-typically	µm	NA	NA	5 b[illegible]5		5 by 5	2.5x2.5 over v	not focused operation ?
	µm	0.025 x 0.025	0.5 by 0.5	0.2 b[illegible]0.2		5 by 5	<0.05x0.05	focused operation
Crystal setup								
Number of crystal		2	1	2 t[illegible]3	2 to 3	>2	2	
crystal types		111/311	111	111/[illegible]11	111/311/KTP	111/220/ 311/ 400	Si 111 / Beryl	
crystal size	mm	25 x 50	?	25 x[illegible]50		25x50(for the momen	25x50/25x25	active size: width - length
Asymmetry		0	0	0	0	0	0	
Crystal parallelism with piezo correction								
Crystal parallelism, Energy	µrd	0.15	0.2	0.[illegible]	0	0.1	0.2	microrad with piezo correction
Crystal parallelism, position	µrd	3 and 0.15	3	0.2 an[illegible] 0.12	0.2 and 0.2	0 micron a 30 m + KI	0.2	H and V in microrad - This value corresponds to a acceptable beam movement on the sample (spatial requirement) range ?
Intrinsic broadining of the reflexion	µrd	<1?	<1	<[illegible]	<4	0.7	<1	microrad with beam
Beam Thermal load								
Beam size		1 x 1	1X1	15 x[illegible]1		1x1	1x1	mm (HxV) on mono FWHM?
Max Power density	w/mm2			1[illegible]			500	W/mm2
Max total power	w			10[illegible]				W-Check consequences of new lattice

CONCLUSIONS



And for the double crystal monochromator dedicated to spectroscopy

… the winners are….

Repeatability

Stability

Precision

Thank you for your attention