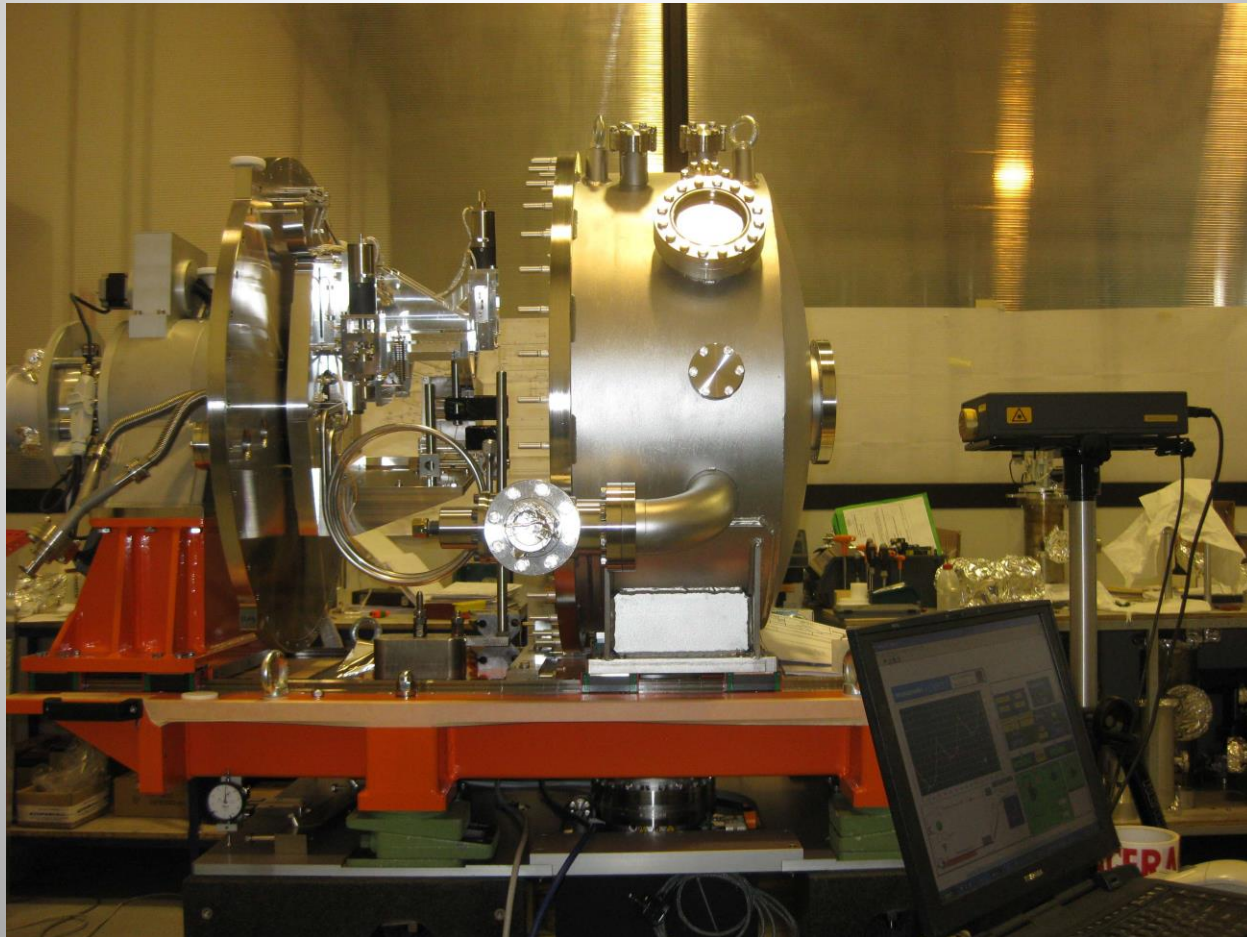


Double-crystal CINEL mono at the GALAXIES inelastic/photoemission (undulator) beamline

James M. Ablett

Scientist, Synchrotron SOLEIL (GALAXIES Beamline)

Beamline Staff: J.-P. Rueff (Beamline Responsible), D. Céolin (Scientist), D. Prieur (Technician), T. Moreno (Optician)



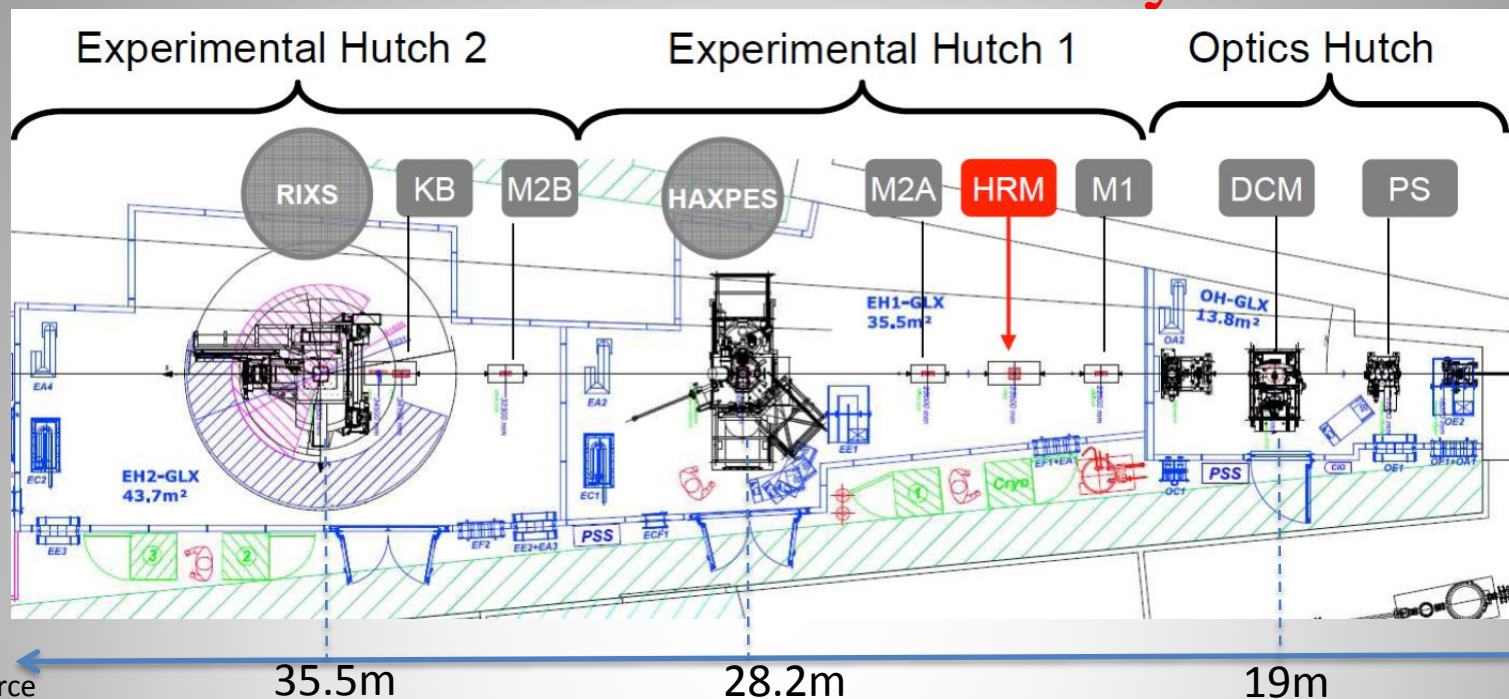
Outline

- **Brief Introduction to the GALAXIES Beamline**
- **Technical Specifications and Design of the GALAXIES DCM**
- **Performance Tests and Experimental Data**
- **Conclusion**

Timeline for the GALAXIES DCM

- Design started: **September 2008**
(2 DCMs in parallel: GALAXIES and SIXS)
- Finished: **November 2009** ✓ (on time)
- Delivered: **February 2010**
- Cost: **210,000 €** (on ordering 2 DCMs)
 - Si 111 crystals included (Crystal Scientific)
 - not including crycooler

GALAXIES Beamline Layout



Distance from undulator source ←

Resonant Inelastic Scattering Endstation



Techniques: Resonant and non-resonant IXS, X-ray emission spectroscopy, HERFD XANES

Topics: Strongly Correlated Materials, Magnetism, Superconductivity High-Pressure Physics.

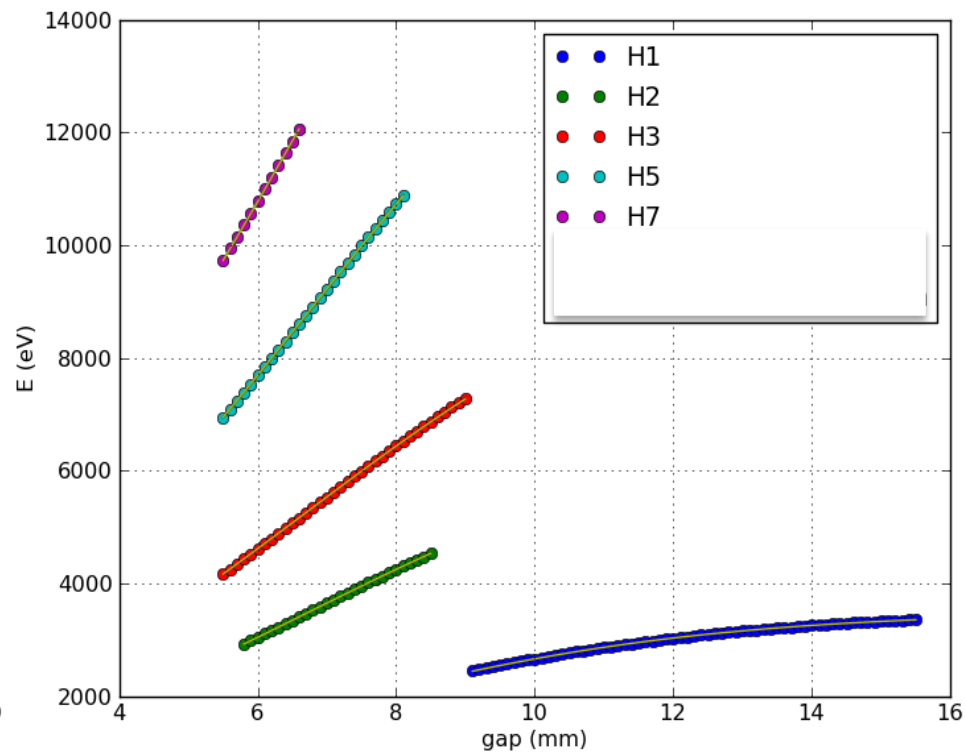
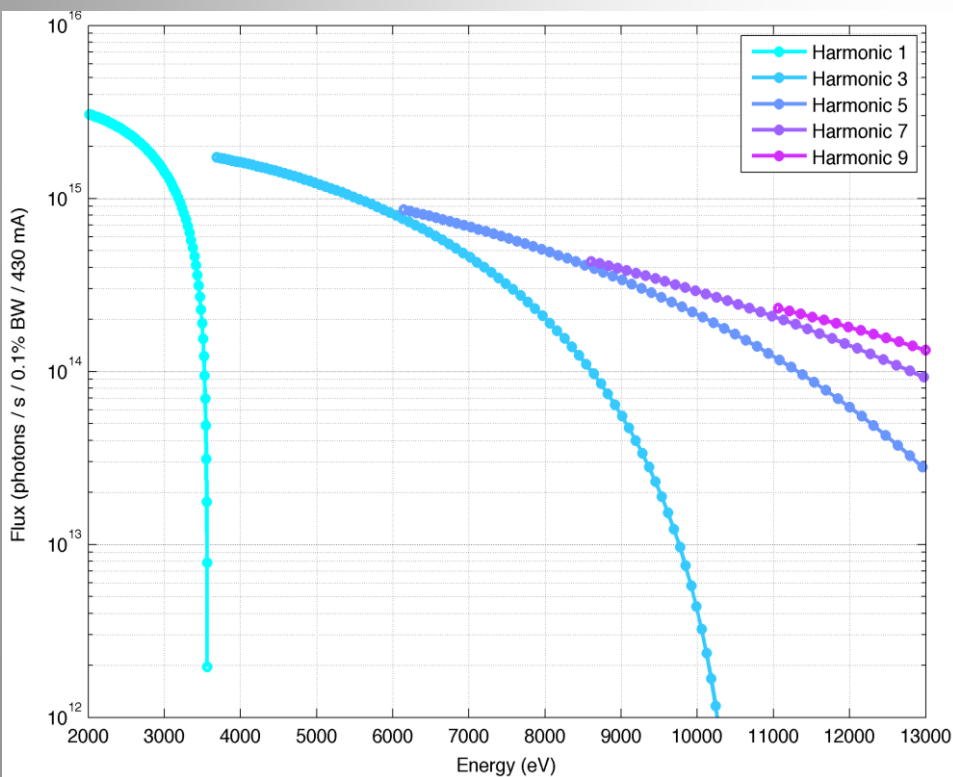
Hard X-ray Photoelectron Spectroscopy Endstation



Topics: Buried Interfaces, semi-conductors, thin-films, angle-resolved HAXPES, Gas-Phase

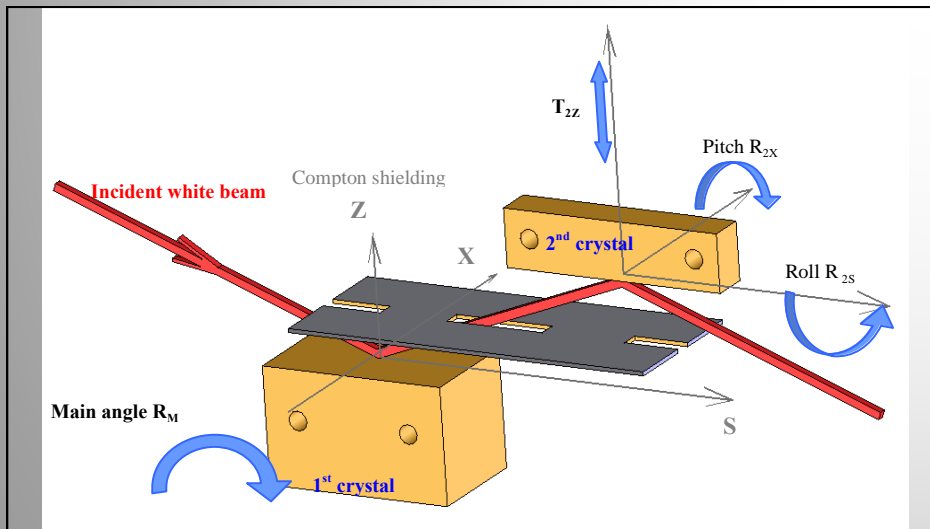
GALAXIES U20 Power and Spectral Characteristics

- Source (SOLEIL:2.75 GeV, 430mA (to increase to 500mA this year): U20 in-vacuum undulator ($\text{Nd}_2\text{Fe}_{14}\text{B}$, 20mm period, 98 periods, min gap 5.5mm)
- Energy Range: 2.3 -> 12keV (θ_{bragg} : $59.3 \rightarrow 9.5^\circ$ Si(111))
- DCM 19 m from source, beam height 1.4m, beam-off set 20mm
- Max. Beam size: $\approx 3\text{mm}$ (hor.) x 2mm (ver.)
- 1st xtal: Max. Power load: 200 Watts , Max power density: 33 W/mm^2

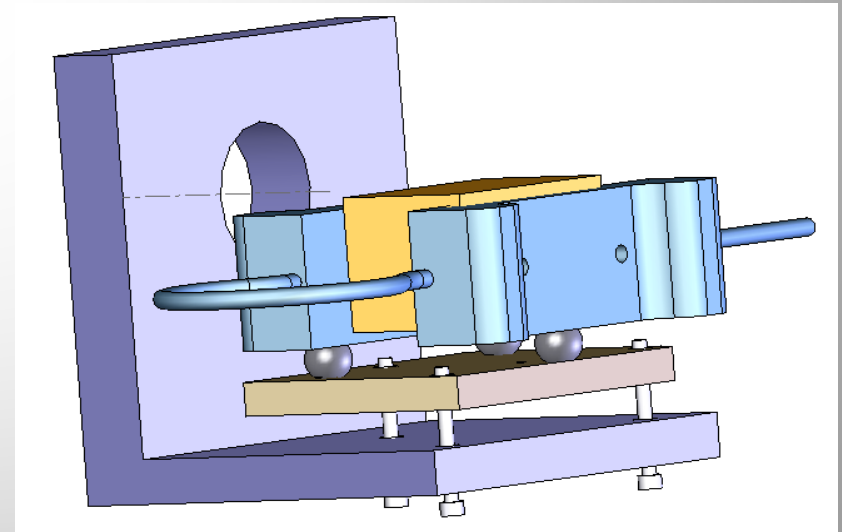


Technical Specifications

- Energy Range: 2.3 -> 12keV , θ_{bragg} : 59.3 ->9.5° Si(111).
- Fixed Exit (independent crystals).
- Flat crystals : no focusing.
- Pressure in chamber $<5 \times 10^{-8}$ bar.
- Centre of rotation on 1st crystal (incident beam).
- 1st crystal clamped between heat absorbers.
- 2nd crystal cooled via copper braids linked to 1st xtal mechanism.
- Hard limits on all motions.



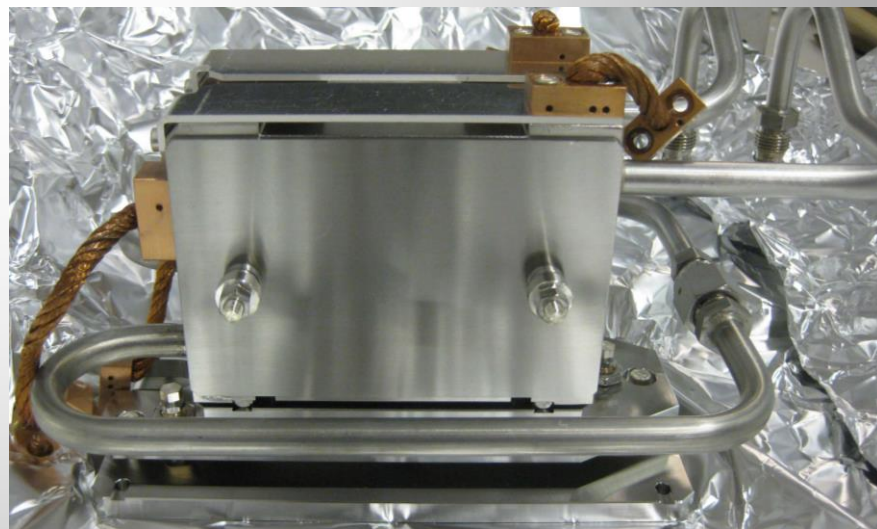
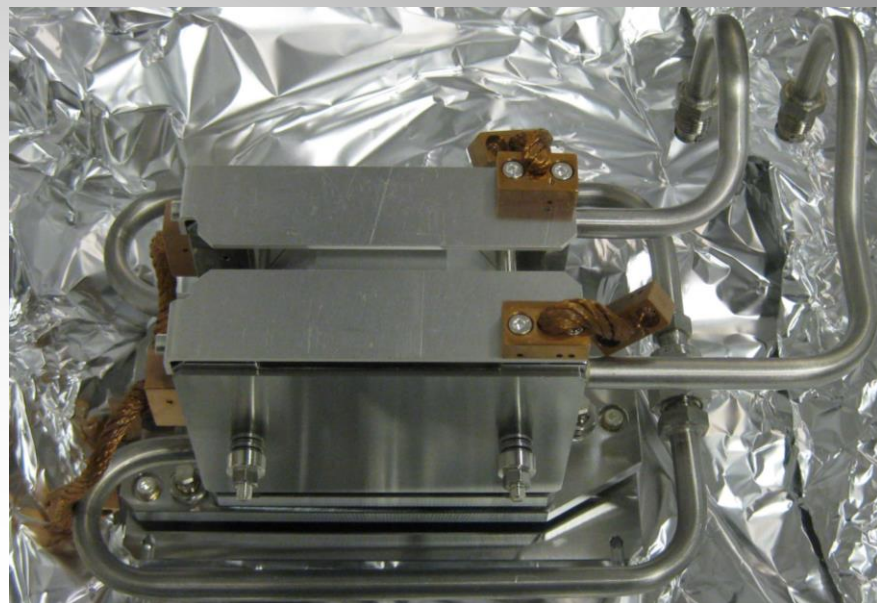
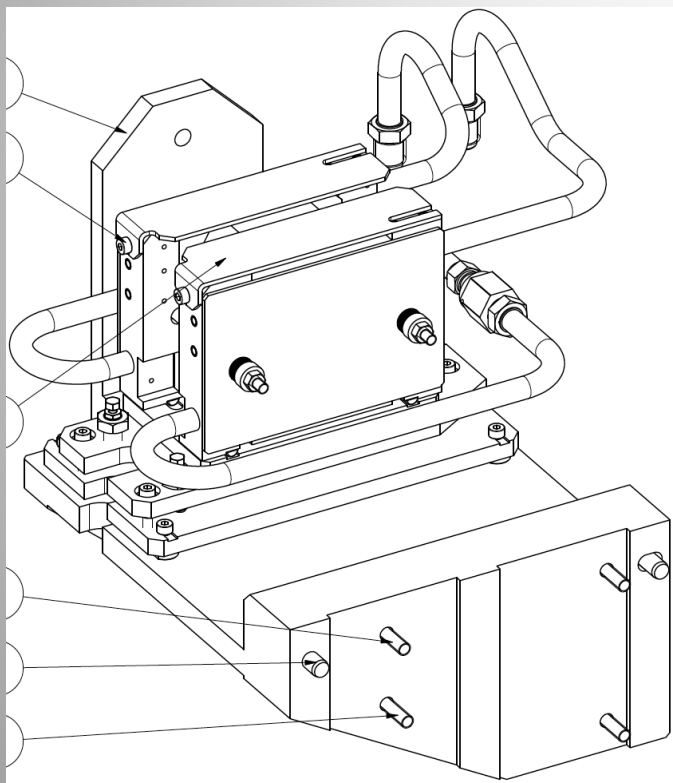
Galaxies initial proposed design. Added in later piezo.

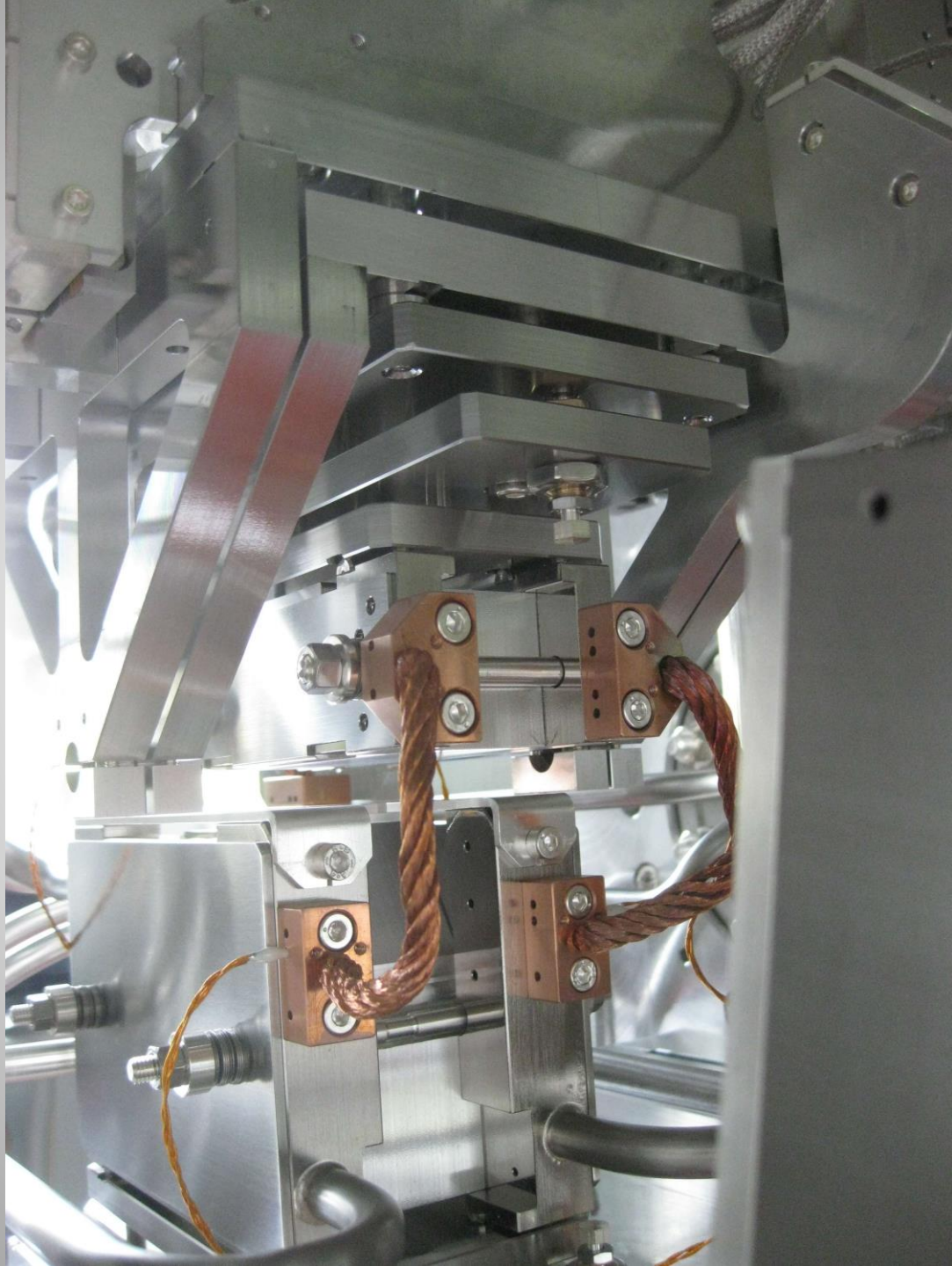


Galaxies proposed ,first xtal mounting and cooling arrangement.

GALAXIES 1st Crystal Mount Design

- Nickel plated heat absorbers
- 0.5mm thick Indium foil between absorbers and xtal
- In-Ga eutectic paste
- Copper braids: Cooling of 2nd xtal and Anti-Compton Shield





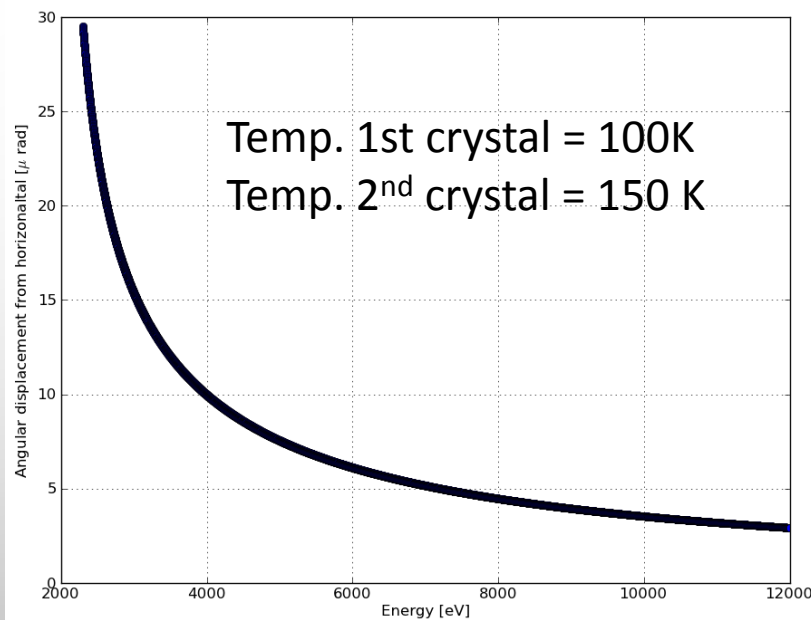
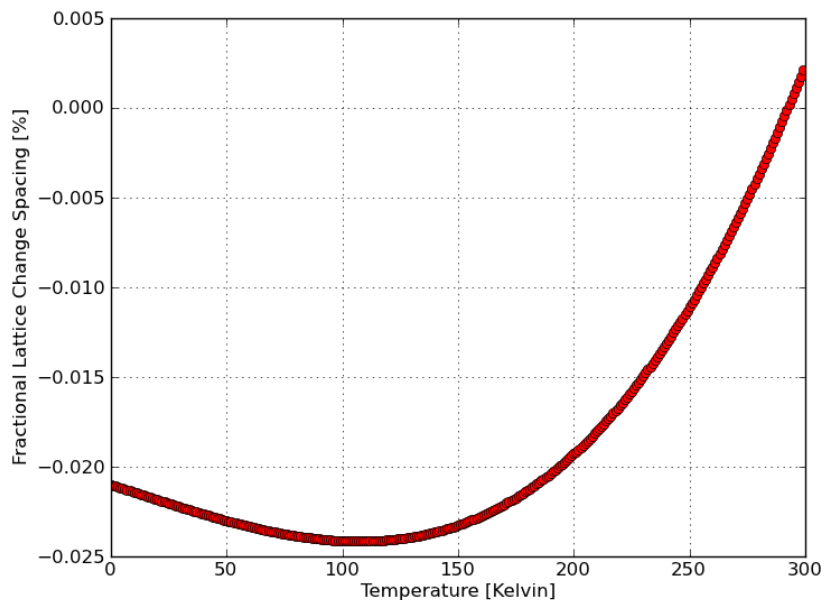
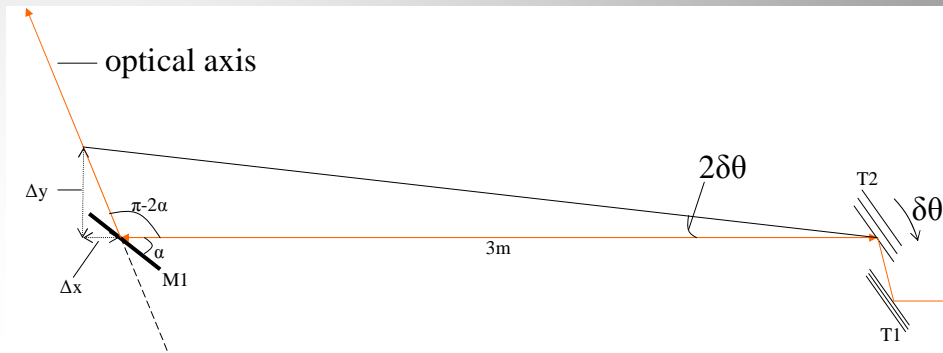
GALAXIES U20 Power and Spectral Characteristics

Total Power Emitted by U20 Undulator = 4.2 kW ($P[\text{kW}] = 0.633E^2[\text{GeV}] * B^2[\text{T}] * L[\text{cm}] * I[\text{A}]$)

Power absorbed by DCM 1st crystal = $83.4 \sin \theta_{\text{bragg}}$ W/mm²

Element	Power Transmitted (W)	Power absorbed (W)	Power Density W/mm2 ---check
Absorber (2.5x2.5mm ²) @ 10m	1325	2915	290 (centre)
Diaphragm (1.8x0.6mm ²) @ 11.7m	222	1103	206 (centre)
Primary Slits (2.0 x 0.7mm ²) @ 17m	139	83	100 (centre)
Si 111 First Crystal (23.3°, 5keV) @ 19m	8×10^{-3}	139	33 (at surface)

$$\frac{Dd}{d_{293K}} = 0.01 \left(-0.021 - 4.149 \times 10^{-5} T - 4.61 \times 10^{-8} T^2 + 1.4826 \times 10^{-9} T^3 \right)$$



Bragg Angle Technical Specifications

Item	Required (GALAXIES)	Obtainable (CINEL)	Note
Resolution	0.9 μrad	0.25 μrad	
Repeatability	1.8 μrad over 2°	3 μrad / < 1 μrad	Open/closed loop
Accuracy	1.8 μrad over 2°	$\pm 2 \mu\text{rad}$	Encoder limited
Wobble	<9 μrad	<7.5 μrad	
Encoder Resolution	<0.9 μrad	0.11 μrad	36000 lines, 400 fold interpolator, readout in $\frac{1}{4}$ of wave
Maximum Speed	$\geq 0.5^\circ/\text{s}$	0.5 $^\circ/\text{s}$	Speeds up to $1^\circ/\text{sec}$ have been obtained with DC motors. The optimum speed with stepper motor is $0.35^\circ/\text{sec}$

2 harmonic Drives
3 Dynamic Seals
Backlash free



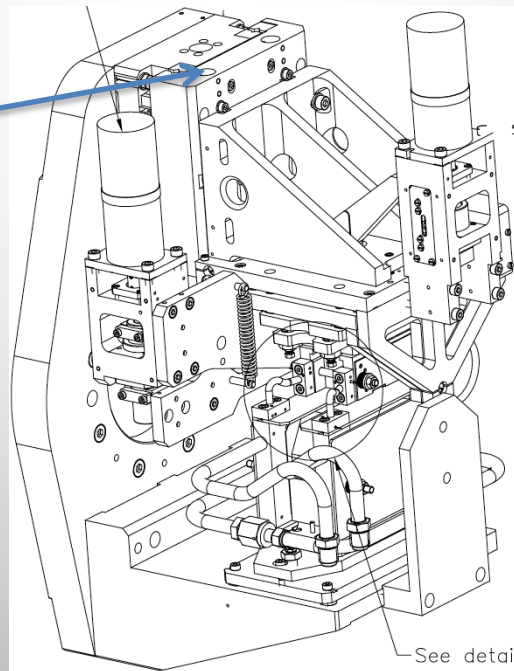
Galaxies DCM installed on the beamline

2nd Crystal Gap Technical Specifications

Item	Required (GALAXIES)	Obtainable (CINEL)	Note
Resolution	1 μm	0.05 μm	
Repeatability	2 μm	1 / 0.1 μm	Open/closed loop
Wobble	0.9 μrad	1 $\mu\text{rad}/\text{mm}$	

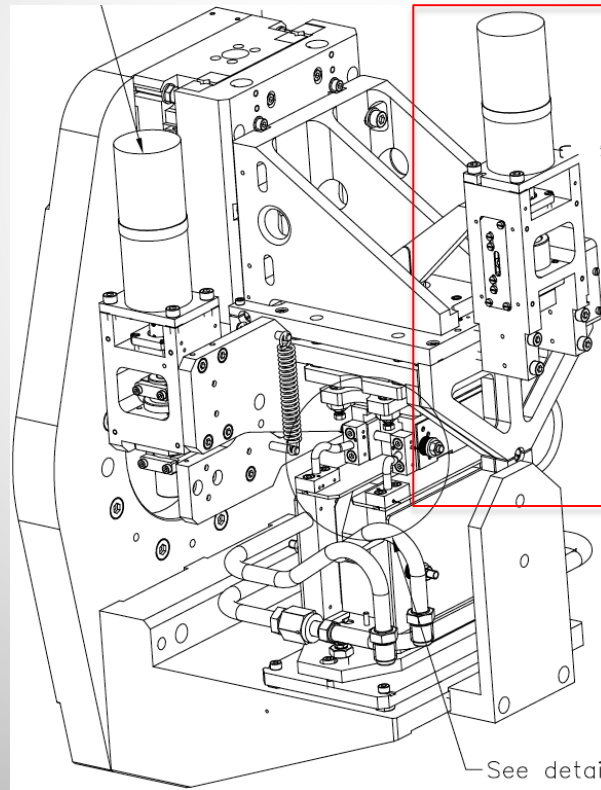
« The translation of the 2nd xtal must not induce perturbations to the pitch and roll larger than their specified repeatability »

Gap Slide



2nd Crystal Pitch Technical Specifications

Item	Required (GALAXIES)	Obtainable (CINEL)	Note
Resolution	0.45 μ rad	0.25 μ rad	
Repeatability	0.9 μ rad	3 / 0.9 μ rad	Open/closed loop



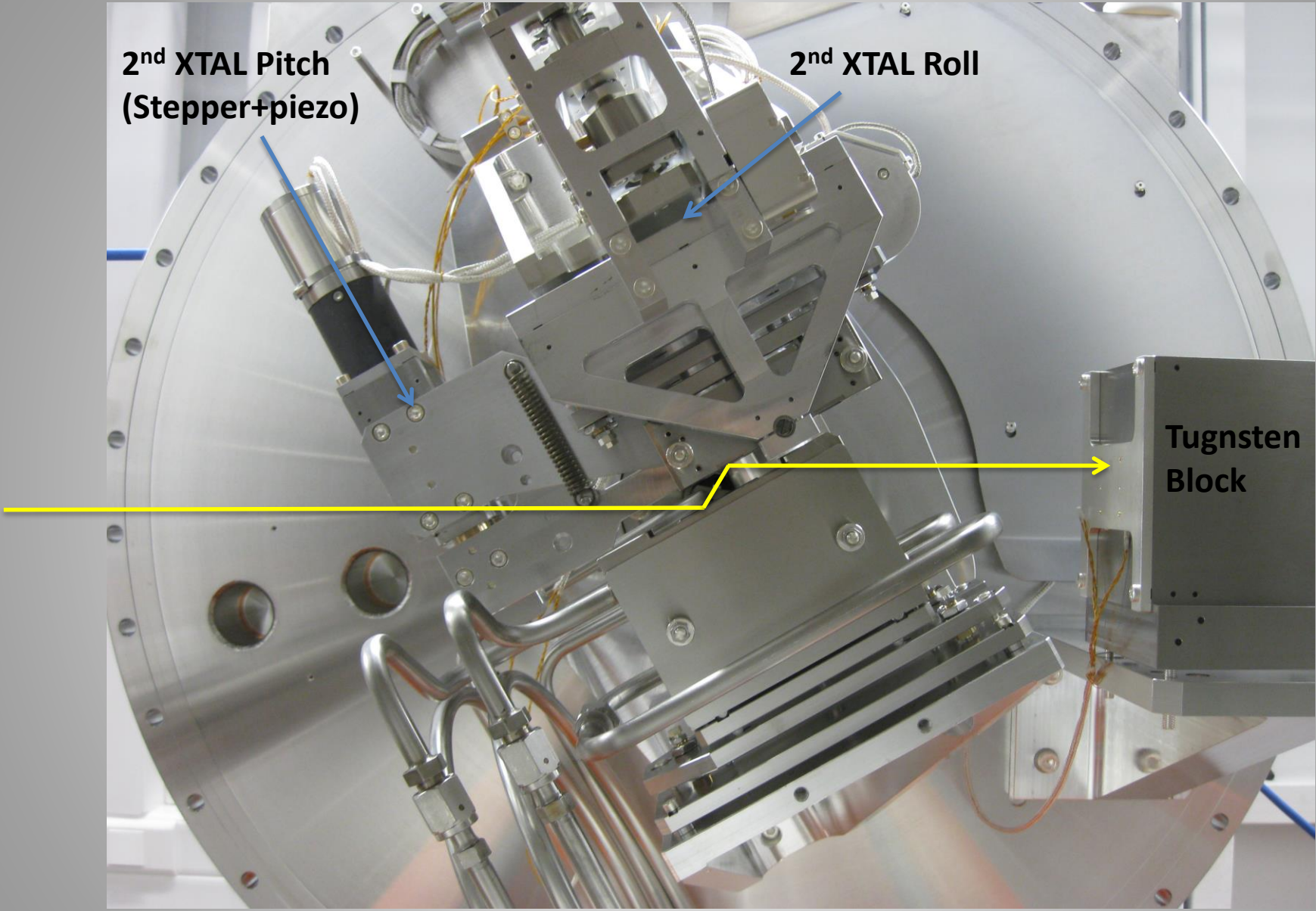
Measurement: Interferometer (Renishaw ML 10)

	Bragg Rotation	2 nd crystal pitch	2 nd crystal roll	2 nd crystal gap
	motor: Oriental PK 266 DB 200 full steps/rev Encoder: HEIDENHAIN RON 886 Interpolator: HEIDENHAIN IBV660B	motor: Phytron VSS42.200.2,5-E-VGPL42.2-HV Encoder: RENISHAW RGH25F 15 M 01 C Interpolator: RENISHAW REF0200 E 01 A	motor: Phytron VSS42.200.2,5-E-VGPL42.2-HV Encoder: RENISHAW RGH25F 15 M 01 C Interpolator: RENISHAW REF0200 E 01 A	motor: Phytron VSS42.200.2,5-E-VGPL42.2-HV Encoder: RENISHAW RGH25F 15 M 01 C Interpolator: RENISHAW REF0200 E 01 A
Resolution (specs/measured)	0.9μrad / 0.1 μrad ✓ Bragg Angle @ 35°, 58° Tz @ 0mm, Rx @ 0° Range : ±2 mrad 35 scans. 8 points per scan.	0.5μrad / <1.6 μrad ✗ Bragg Angle @ 10° Around Rx @ 0° 30 scans. 10 measures/step.	4μrad / <1 μrad ✓ Bragg Angle @ 10° Rz @ 0° Backlash 2.5 μrad 30 scans. 8 measures/step.	2μm / 2 μm ✓ Bragg Angle @ 10° Range: ±1mm 15 scans. 8 measures/step.
Repeatability (specs/measured)	1.8μrad / 3 μrad ✗ Bragg Angle @ 10° Tz @ 0mm, Rx @ 0° Range : ±2 mrad 16 scans. 8 points per scan.	0.9μrad / 3 μrad ✗ Bragg Angle @ 10° Tz @ 0mm, Rx @ 0° Range : ±2 mrad 16 scans. 8 points per scan.	8μrad / 2.1 μrad ✓ Bragg Angle @ 10° Range : ±2 mrad 15 scans. 8 points per scan.	1μm / 1 μm ✓ Bragg Angle @ 10° 30 scans. 8 measures/step.
Parasitic Motion (specs/measured)		Parasitic motion in moving Tz 1μrad/mm / <1 μrad/mm ✓ Bragg Angle @ 10° Tz range 9.6mm 7 scans. 47 steps per scan	Parasitic motion in moving Tz 1μrad/mm / <1 μrad/mm ✓ Bragg Angle @ 10° Tz range 9.6mm 7 scans. 47 steps per scan	

**2nd XTAL Pitch
(Stepper+piezo)**

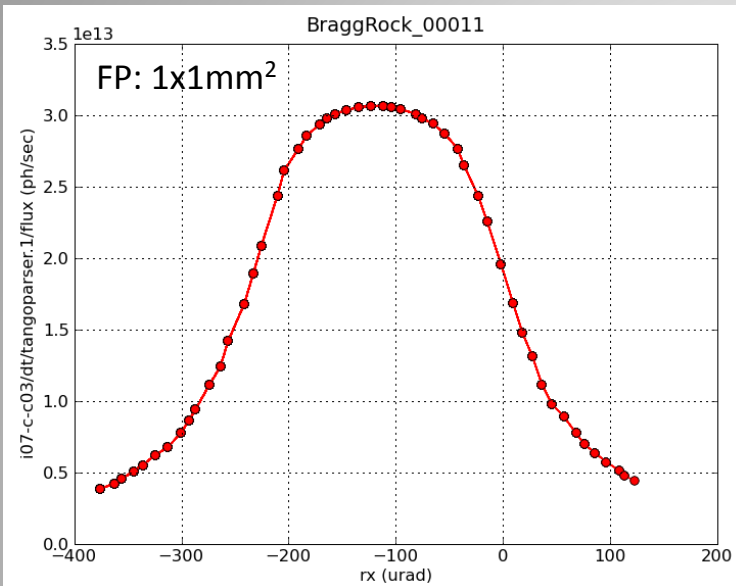
2nd XTAL Roll

**Tungsten
Block**



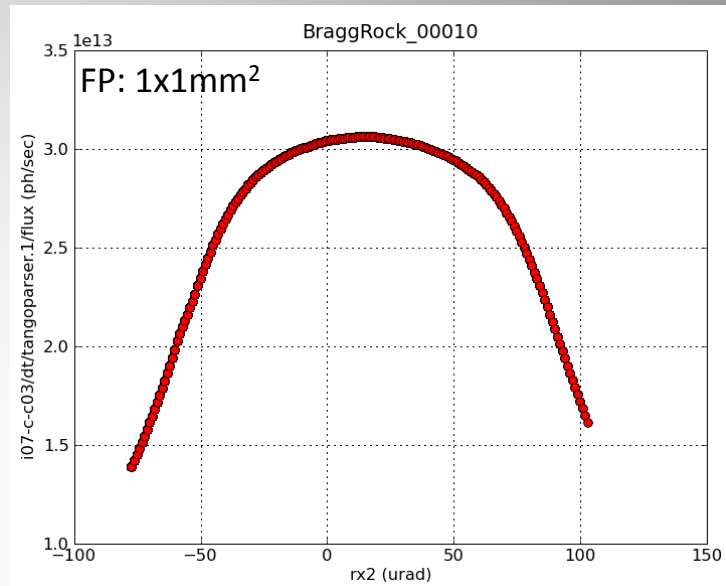
2nd XTAL Stepper and Piezo Capabilities

Stepper

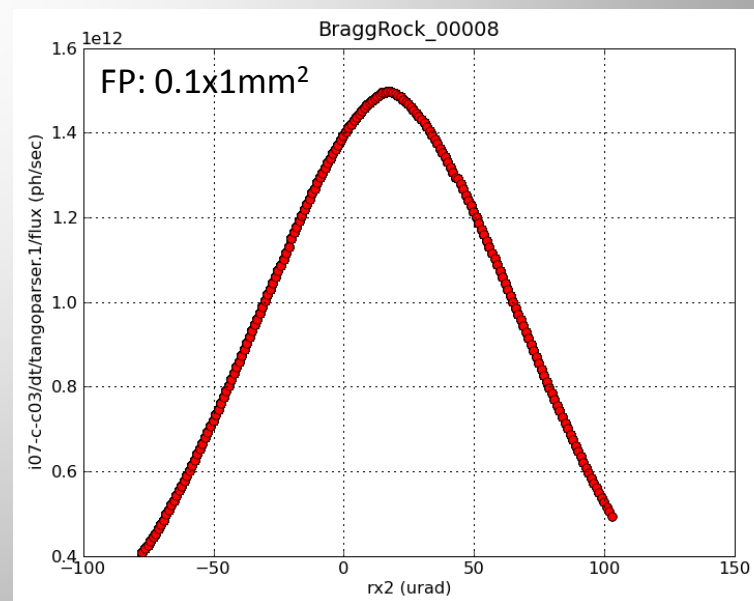
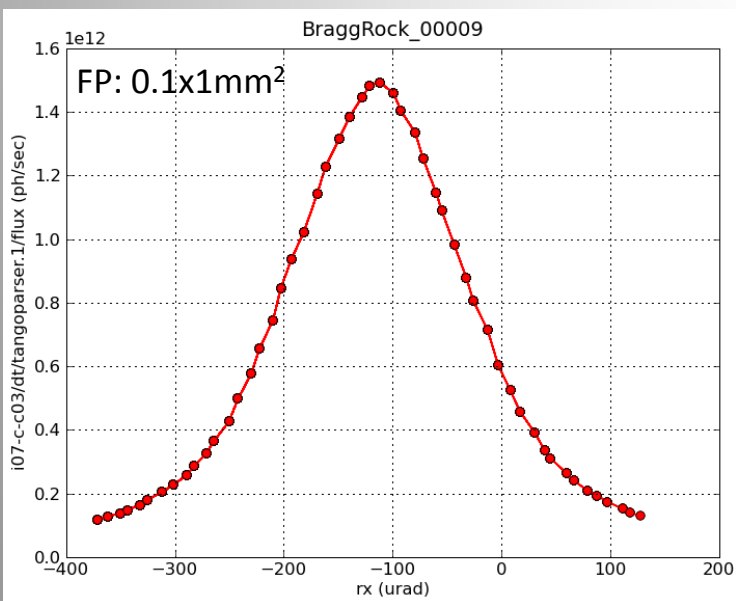


Date 05/08/2011
Energy 3 keV
(Si 111)

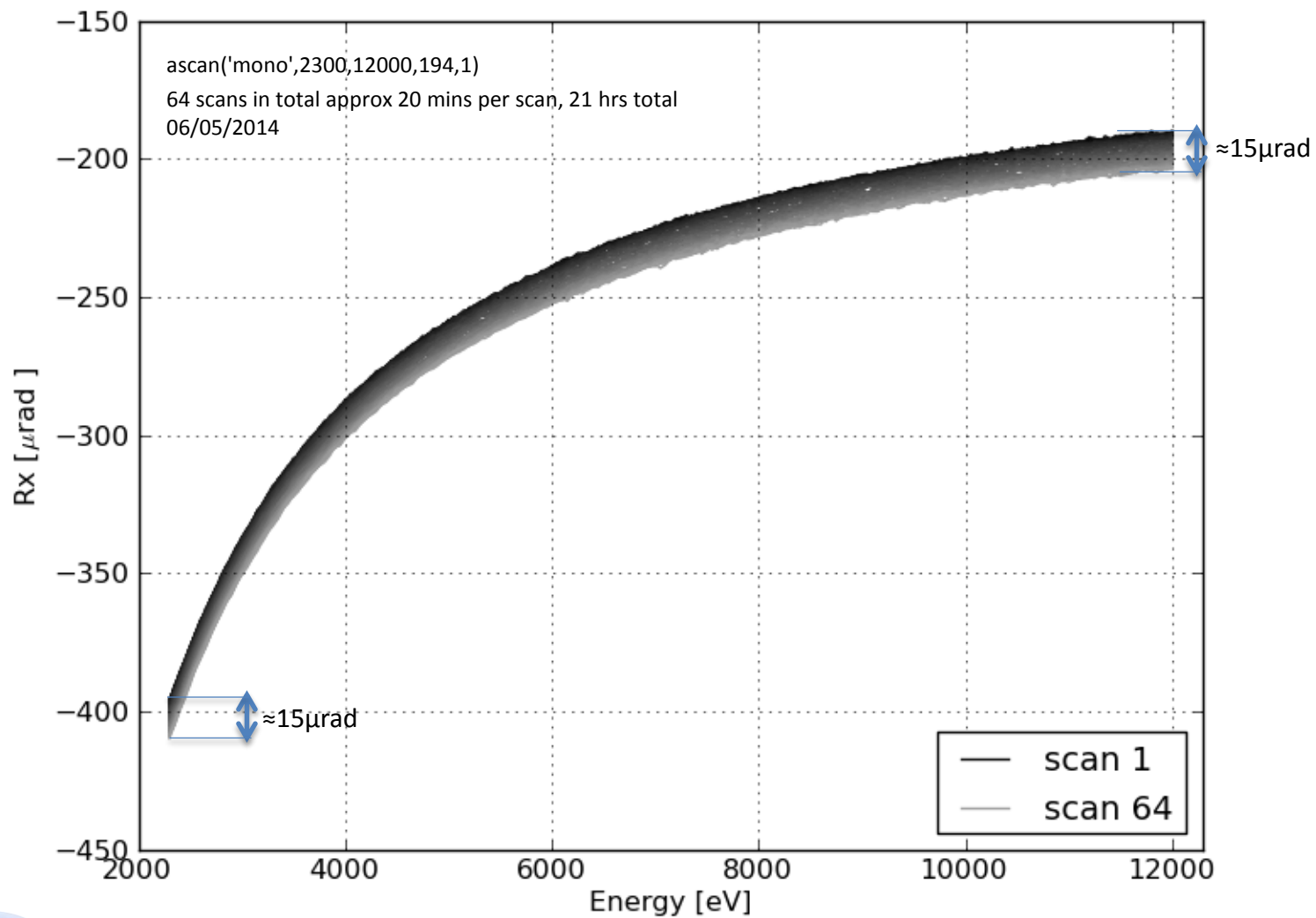
Piezo



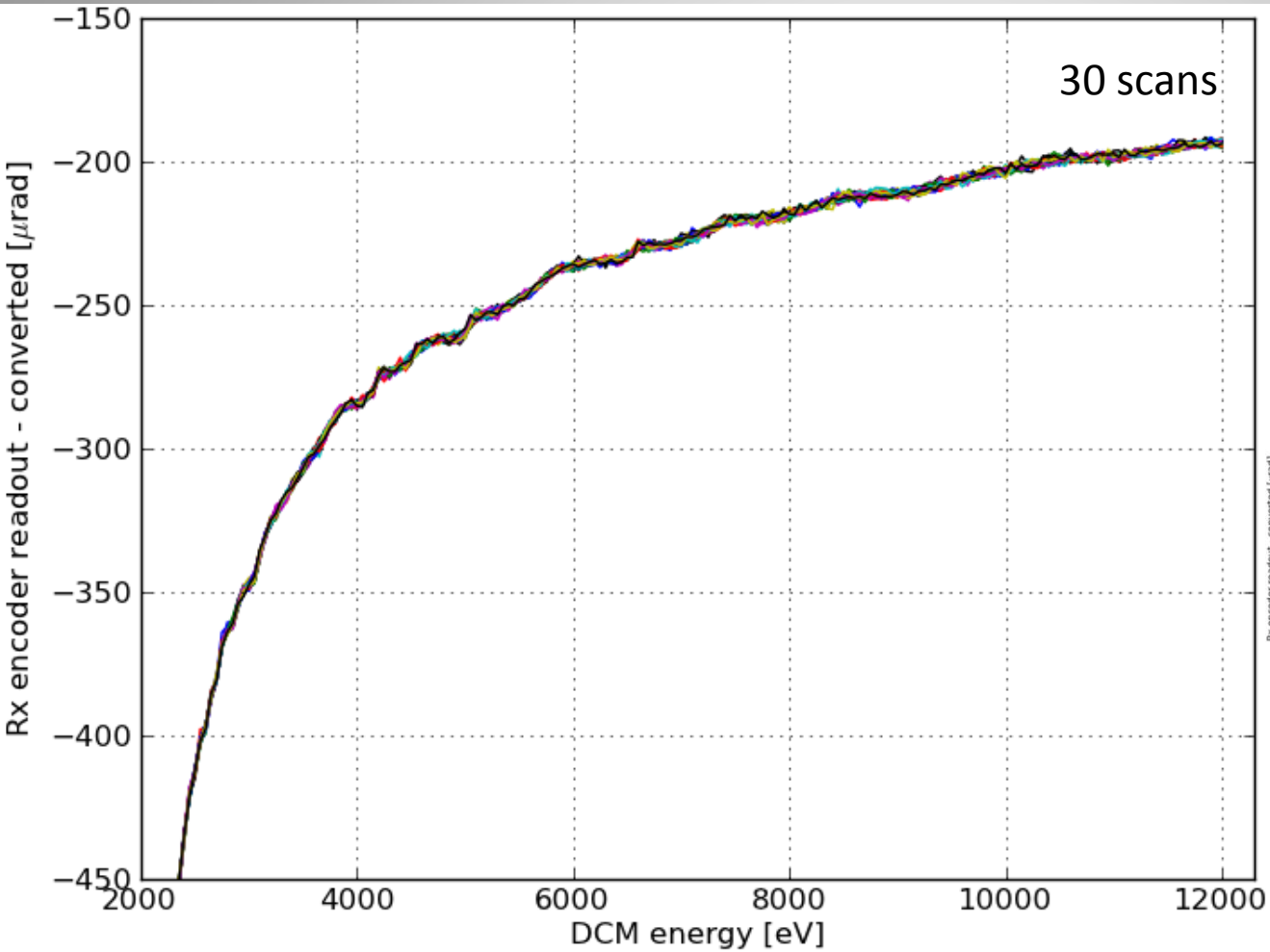
0.1x1mm²



2nd Xtal Pitch Encoder Readout as function of bragg angle (No Beam)



2nd Xtal Pitch Encoder Readout as function of bragg angle (No Beam): Rx Enabled



AtkPanel 4.8 : I07-C-C02/OP/MONO

File View Preferences Help

I07-C-C02/OP/MONO

All the motors are in STANDBY state...

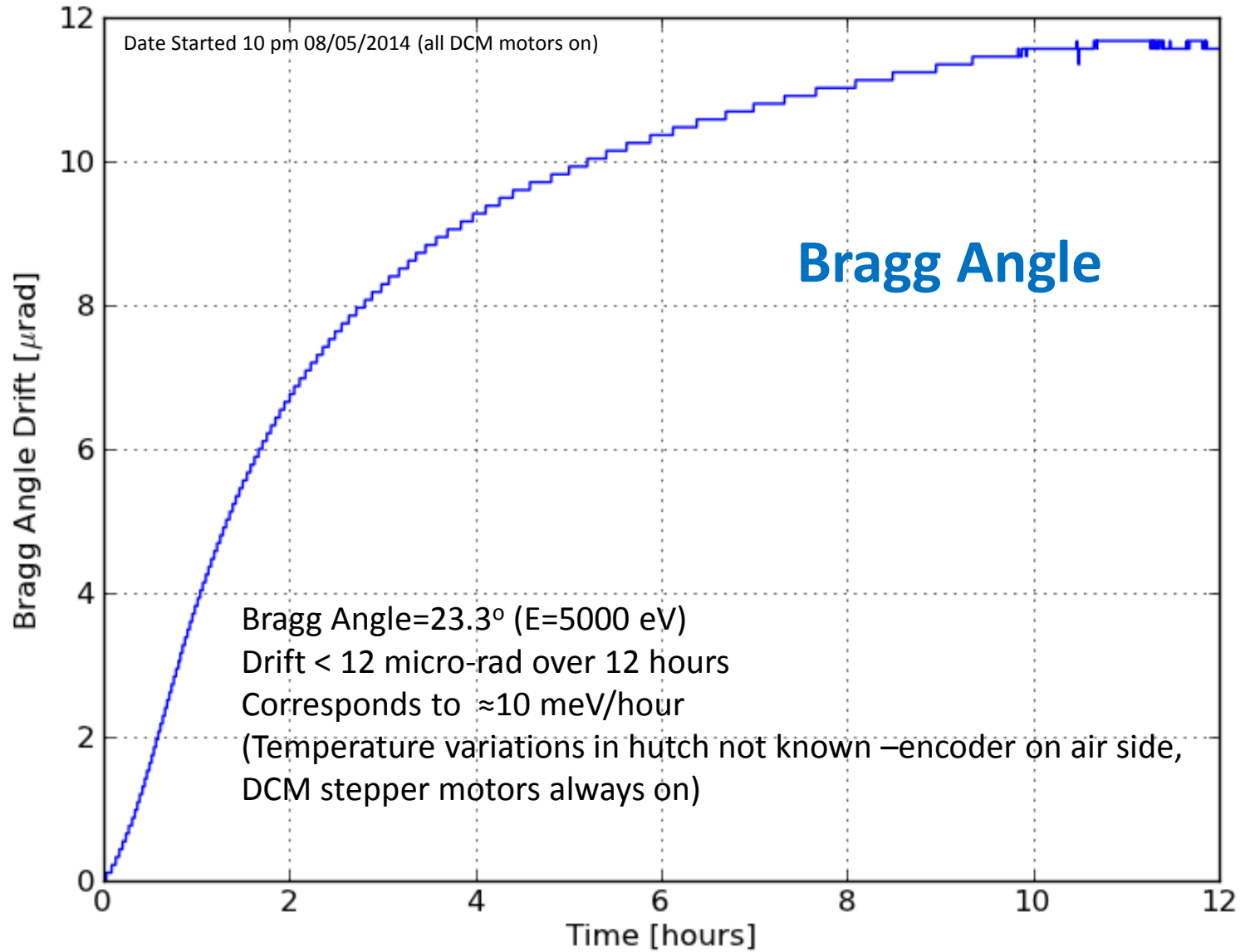
Theta motor --> STANDBY
Tz2 motor --> STANDBY
Rs2 motor --> STANDBY

energy	2299.99276 eV	02300.00000
Lambda	5.3906 Å	▲▲▲▲
Theta Bragg	59.29501 deg	10.00000
Current Functioning Mode	3: Custom	
Current Slot	Slot 1	
Crystal Name	Si(111)	
Crystal Temperature	-186.400 Celsius	
Crystal Inter Reticular Distance	3.134790 1/2	
Rs	-1 μrad	0000
Rx	-475.3 urad	-275.3
Tz	11.417 mm	10.220

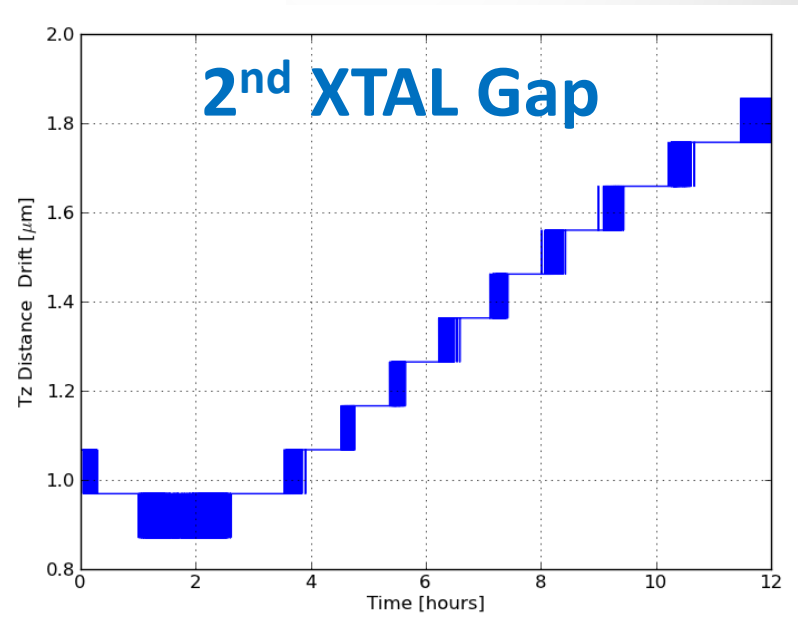
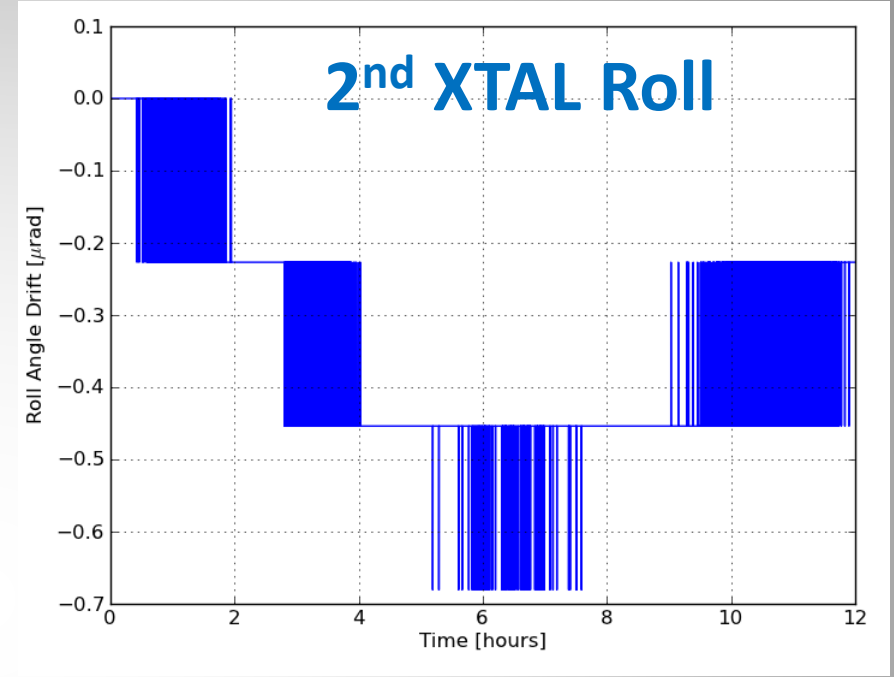
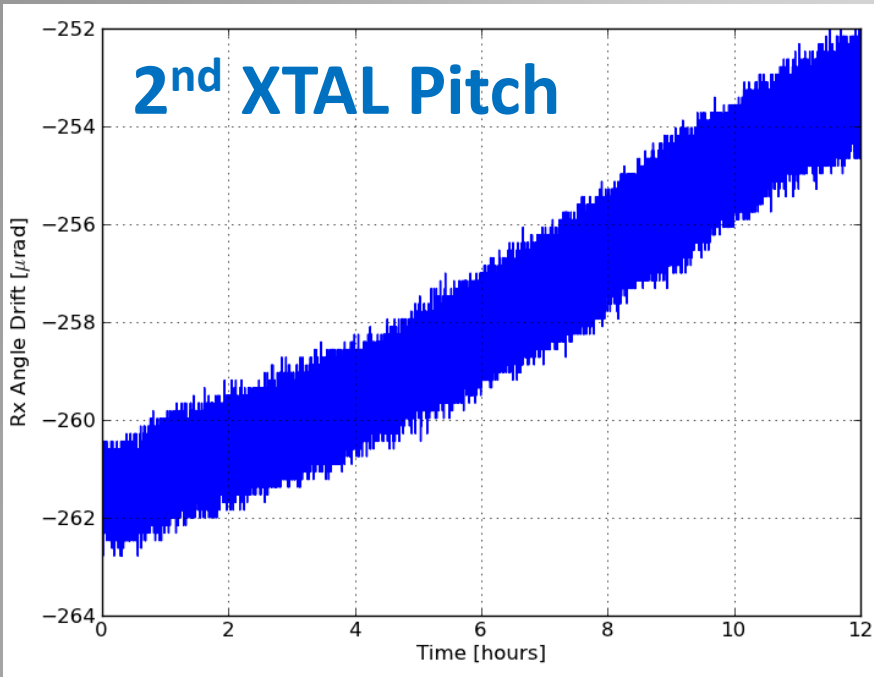
Enable Rs True
Enable Rx True
Enable Tz True

Scalar log

DCM Stability Tests (No Beam)



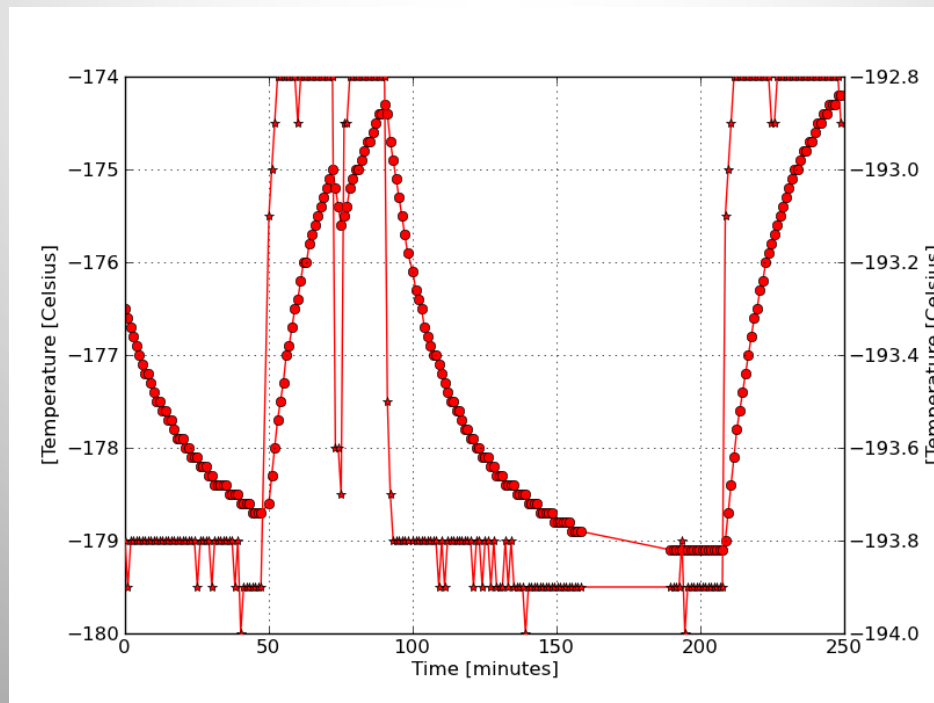
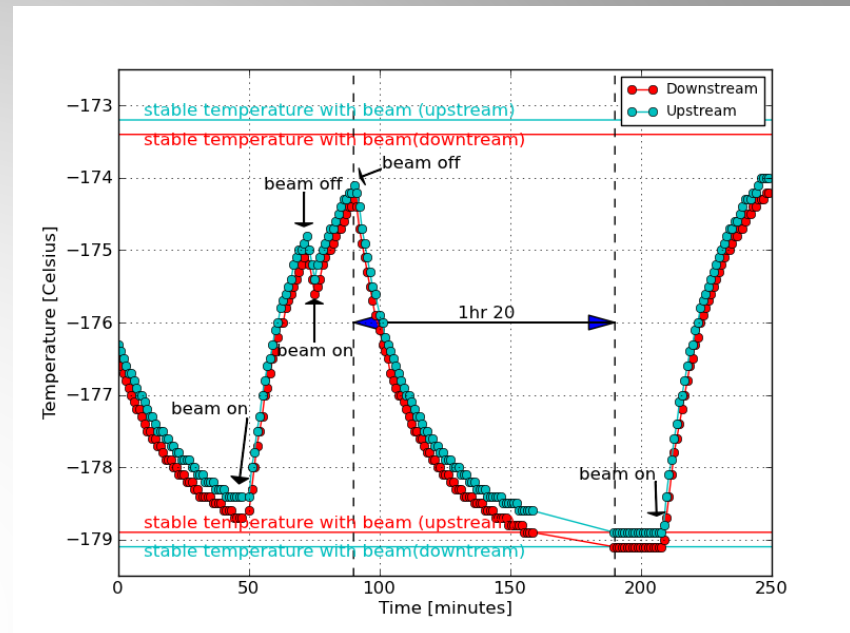
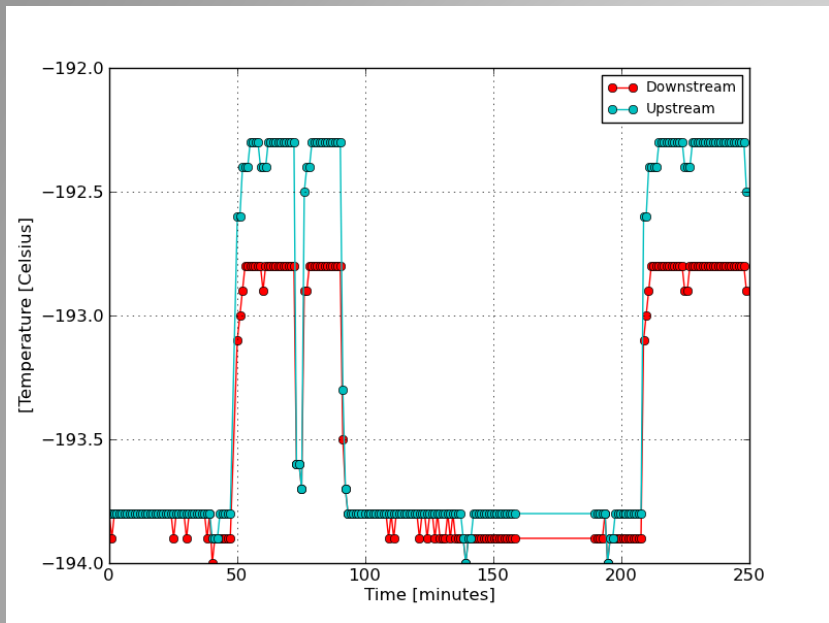
DCM Stability Tests (No Beam)



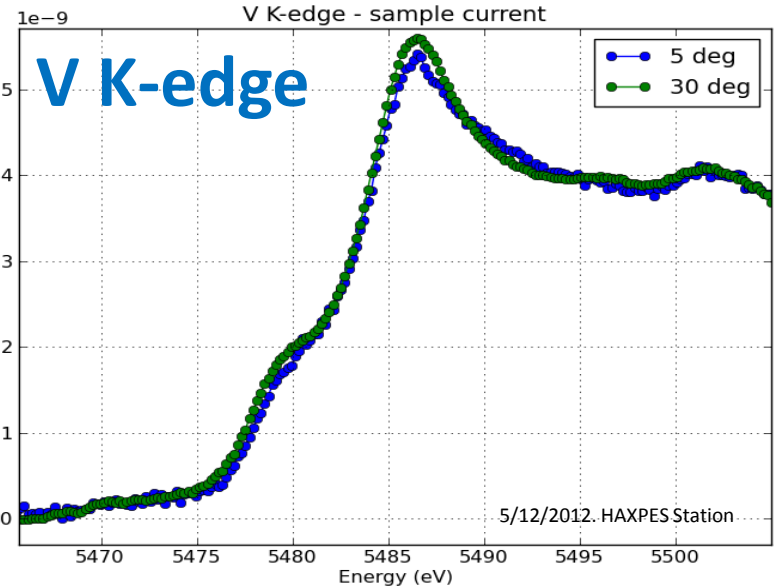
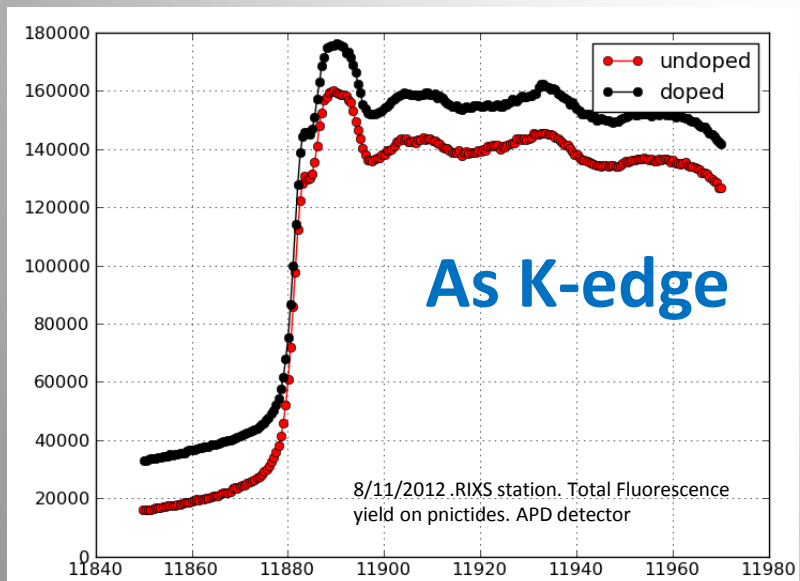
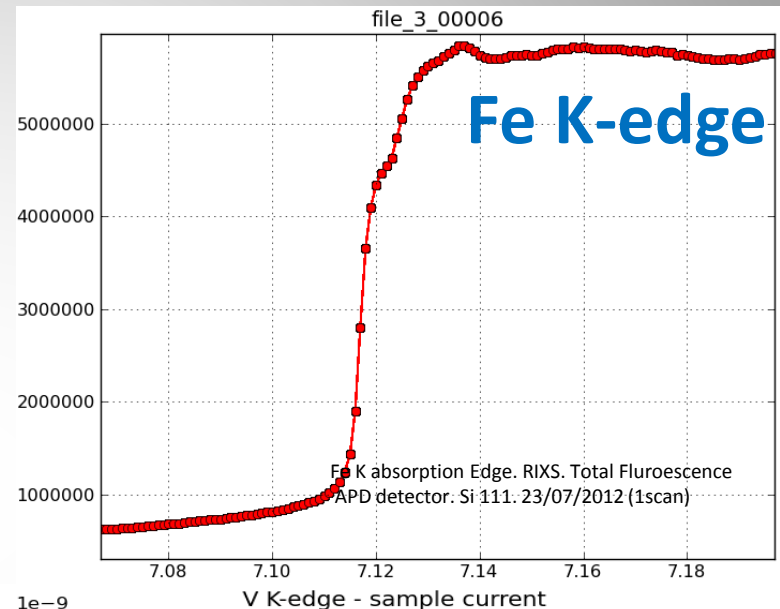
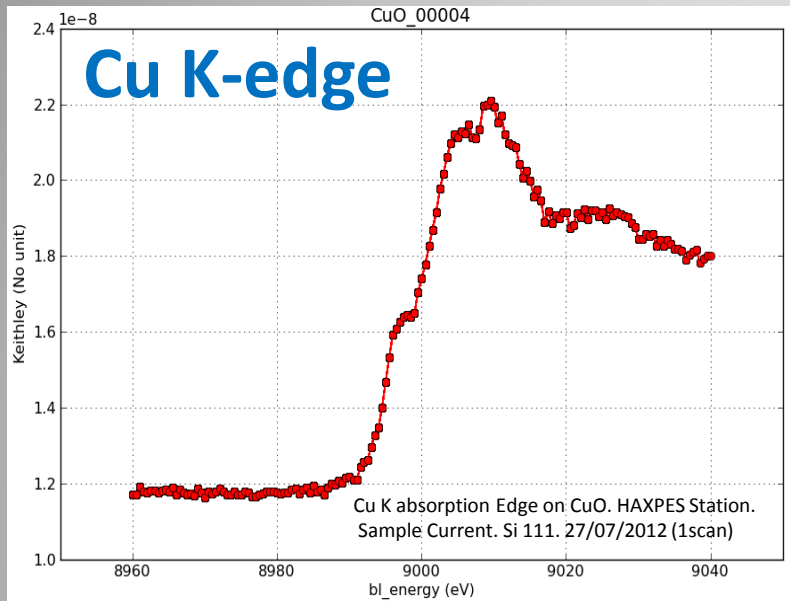
Date Started 10 pm 08/05/2014



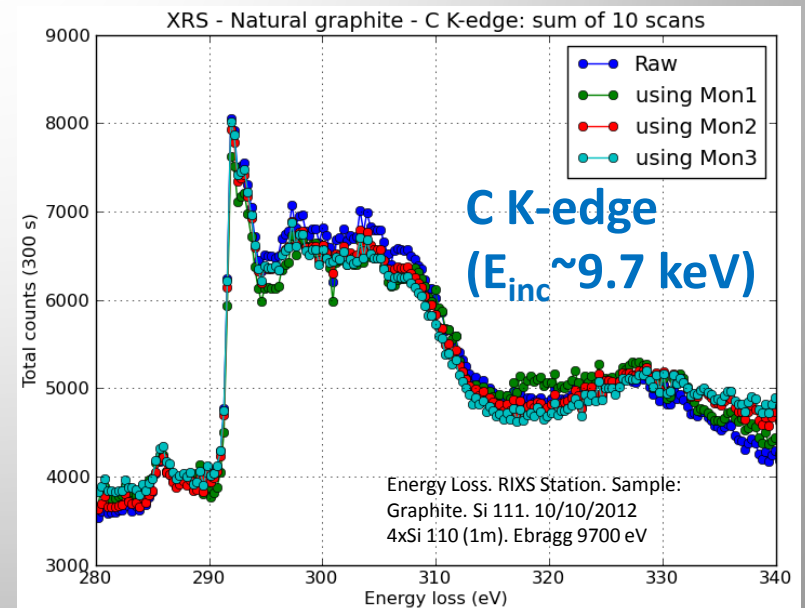
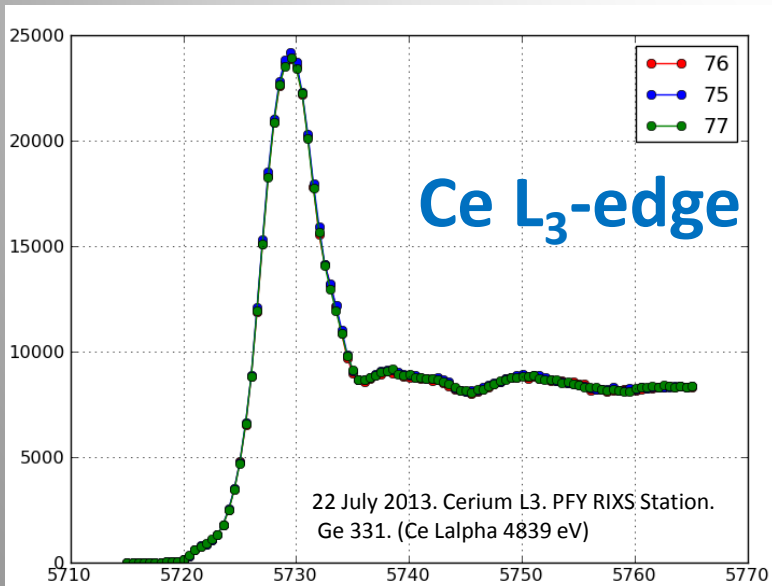
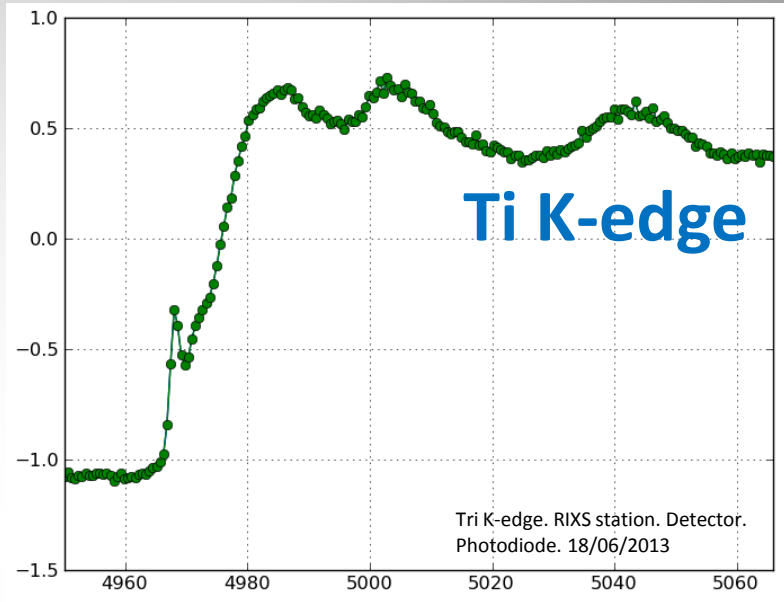
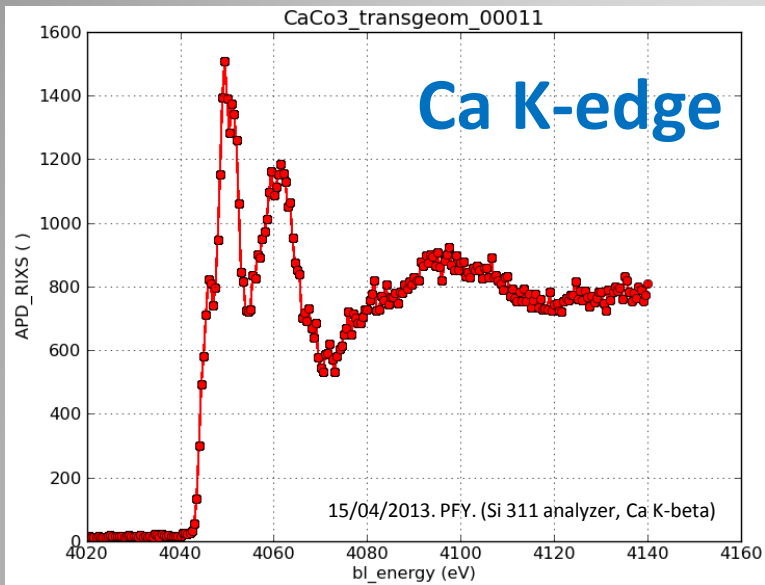
DCM warm up / cool down. Bragg angle = 11.7°



Example Energy Scans



Example Energy Scans



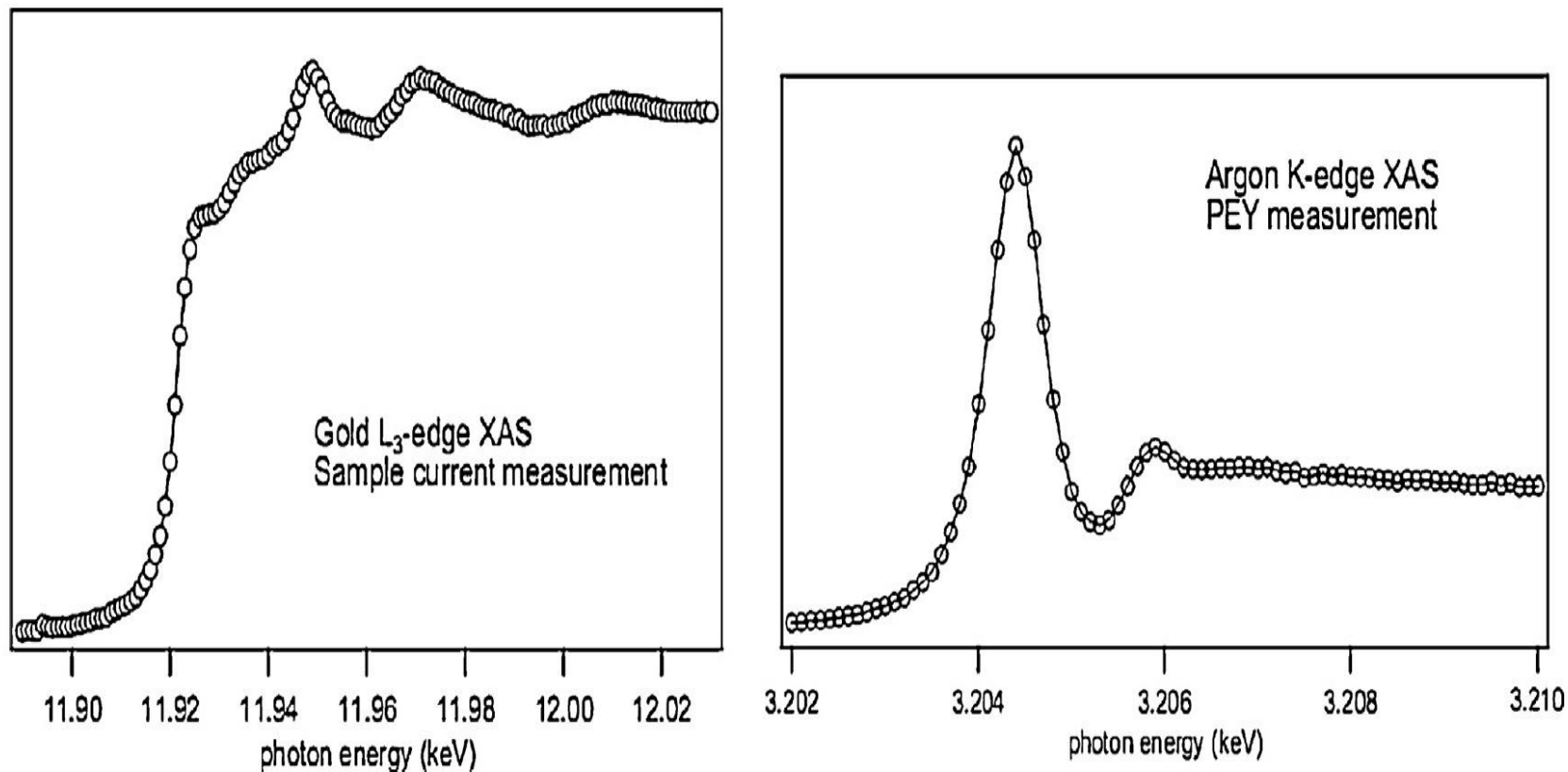


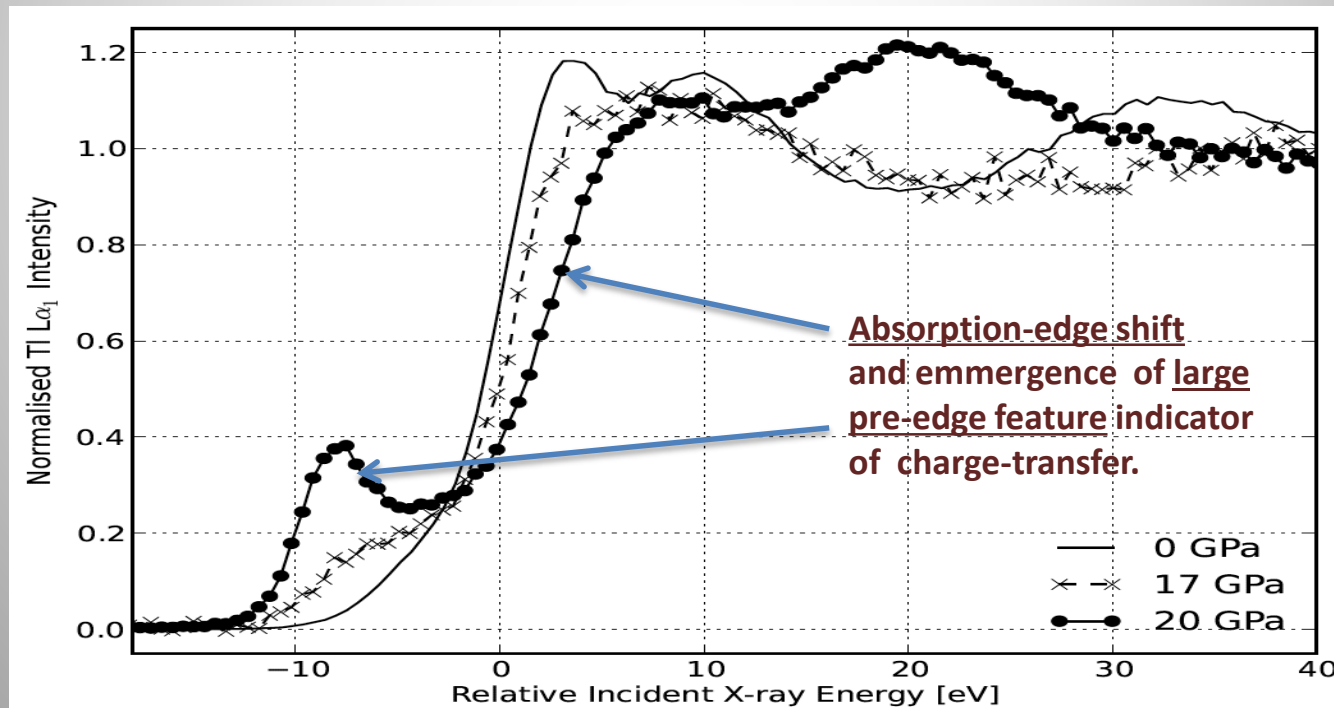
Fig. 5. Au L₃-edge XAS measured using the sample current and Ar K-edge XAS measured using the partial electron yield.

Experimental Study: High-Pressure XANES study of TlReO_4

"High-Pressure Induced Charge Transfer in Thallium-Rhenium Oxide at Room Temperature", J. M. Ablett et al., in preparation

- ❑ **Technique: High Energy Resolved Fluorescence Detected XANES (HERFD XANES) using DAC**
- ❑ **2 Analysers (Si 555). 1m radius. Bragg Angle 74.2° (10269 eV, $\text{Tl } L_{\alpha_1}: 3d_{5/2} \rightarrow 2p_{3/2}$)**
- ❑ **X-ray Energy Scans around the $\text{Tl } L_3$ -edge (12658 eV).**

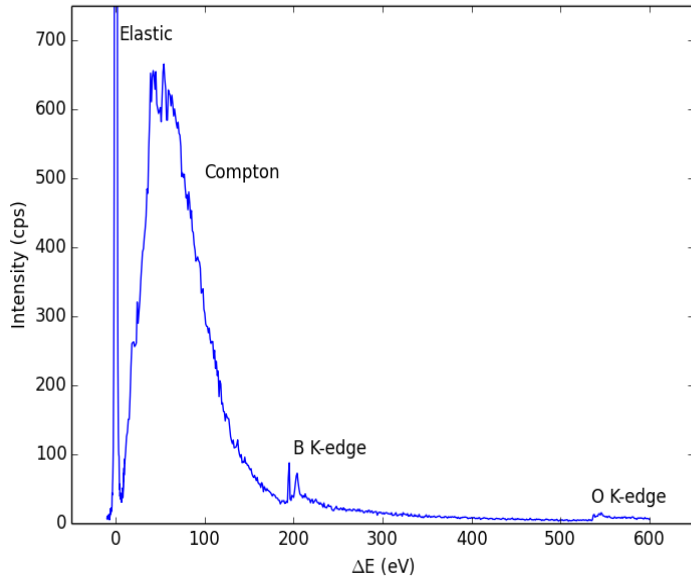
**Proposed high-pressure charge transfer: $\text{Tl}^{1+}(\text{Re}^{7+}\text{O}_4)^{-1} \rightarrow \text{Tl}^{3+}(\text{Re}^{5+}\text{O}_4)^{-3}$
(Jayaraman et al., PRB 36(16) 8547, 1987.)**



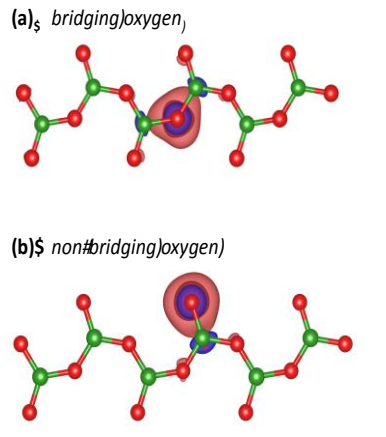
Experimental Study: Non-resonant IXS on Lithium Borate Systems

"Spectroscopic signature of non-bridging oxygen in the O K-edge of lithium borate crystals using inelastic X-ray scattering: an experimental and theoretical study", G. Lelong et al., submitted to Inorganic Chemistry.

- ❑ *Technique: Non-resonant IXS (Energy Loss)*
- ❑ *4 Analysers (Si 660). 1m radius. Bragg Angle 86° (9720 eV)*
- ❑ *X-ray Energy Scans around the Oxygen K-edge (10255 eV)*

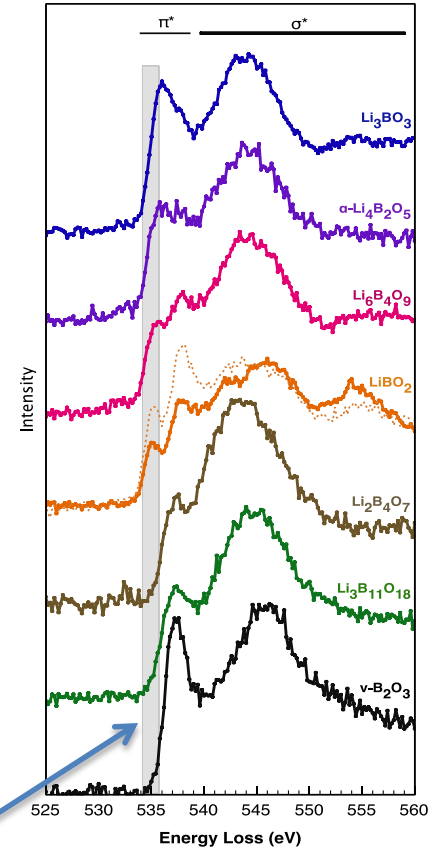


Wide-range energy-loss spectra on lithium borate crystal, clearly showing the B K-edge, OK-edge, the elastic peak, the Compton profile, as well as other features.



Calculated core-hole screening maps for a bridging oxygen (a) and a non-bridging oxygen (b) in the case of LiBO_2 .

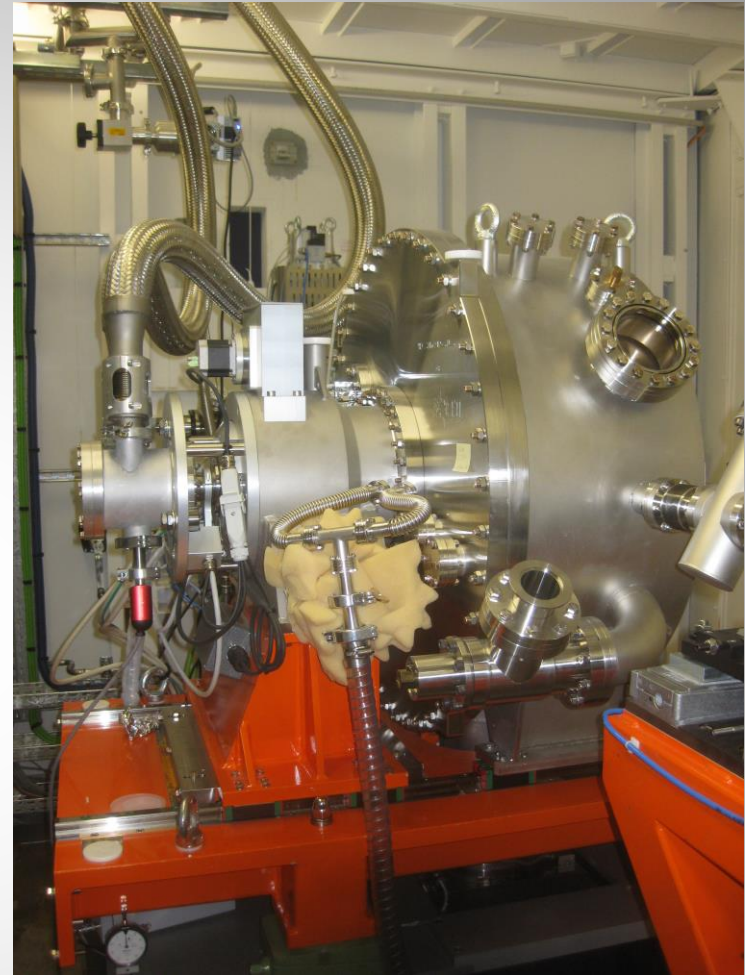
Grey-area : spectral signature of non-bridging oxygen atoms



O K-edge IXS spectra for the six crystalline compounds of the $\text{Li}_2\text{O-B}_2\text{O}_3$ system.

Conclusions

- CINEL DCM successfully designed and then installed on GALAXIES in February 2010.
- No major problems encountered with system to date.
- CINEL extremely cooperative throughout design phase (piezo mount redesigned), installation, and with small problems encountered afterwards (thermocouple breakage, position of limit switch on Tz).
- We also use Si 333 and Si 444 reflections for higher resolution measurements.
- Feedback to be implemented in future using 2nd xtal piezo.



“The GALAXIES Beamline at the SOLEIL Synchrotron: Inelastic X-ray Scattering and Photoelectron Spectroscopy in the Hard X-ray Range”, J.P. Rueff et al., submitted to Journal of Synchrotron Radiation.

“Hard X-ray Photoelectron Spectroscopy on the GALAXIES beamline at the SOLEIL synchrotron.”, D. Céolin et al., *Journal of Electron Spectroscopy and Related Phenomena*, 2013, 190 part B: 188–192