Overview of Beamlines and Measurements

APS XAFS School

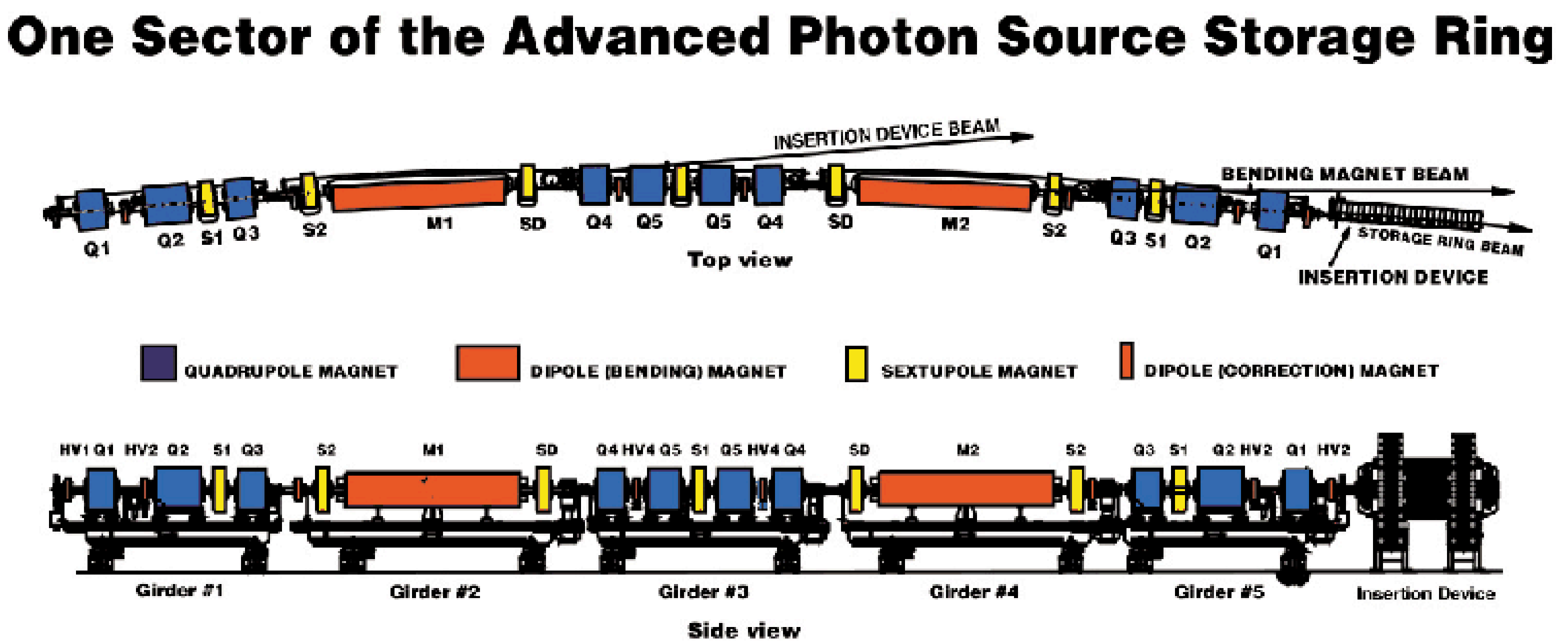
July, 2007

Outline

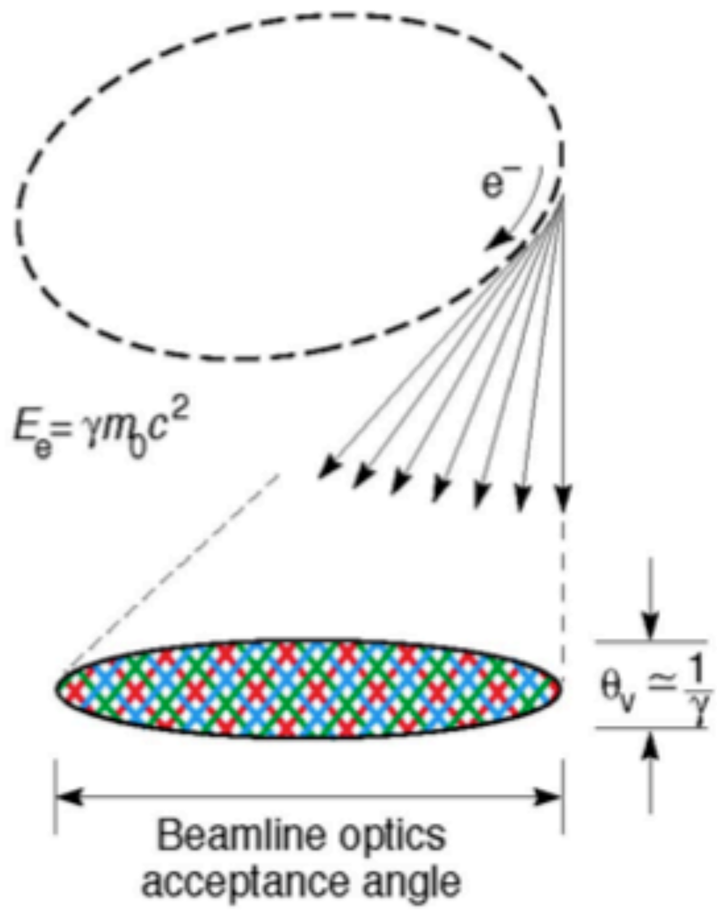
- APS synchrotron radiation and characteristics
- Example of an XAFS beamline and its optics
- Example of an experimental station and components inside of it
- Measurement Considerations

Overview (literally) of the Advanced Photon Source

Synchrotron Magnetic Lattice



Bending Magnet Radiation

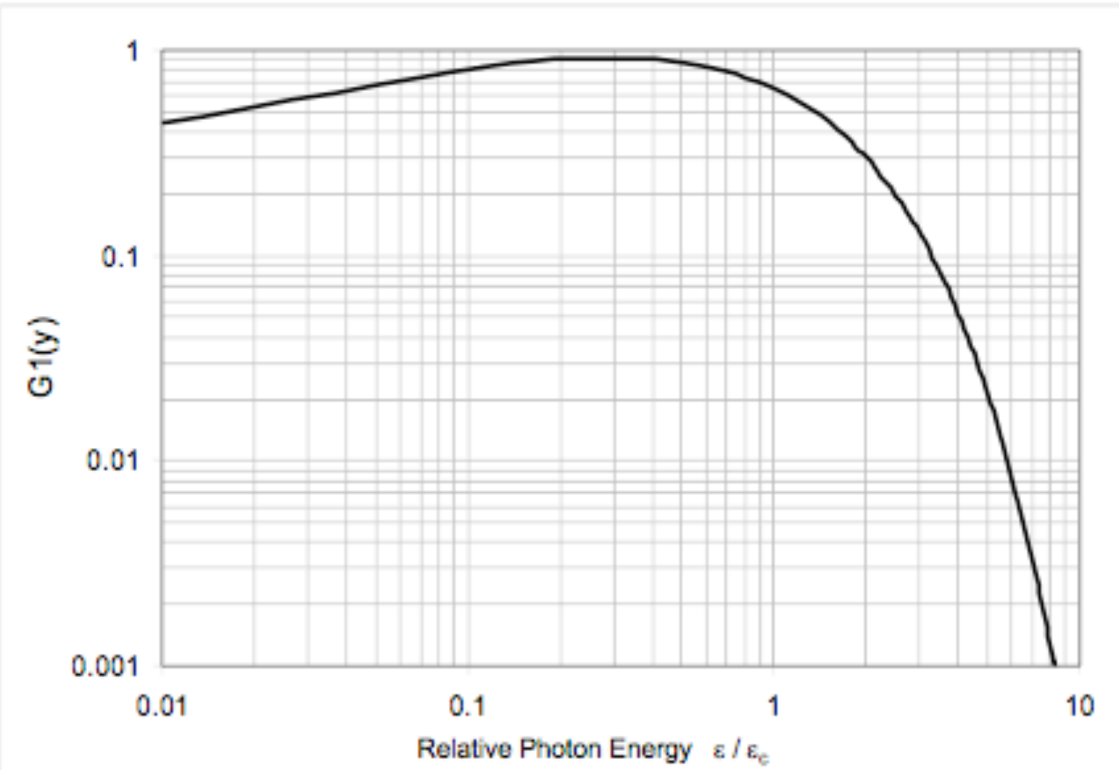


Emission limited to angle range $1/\gamma$.

γ= 1957E(GeV)

For APS: γ = 13699 or $1/\gamma$ = 73 μrad

Bending Magnet Spectrum



Emitted Radiation has Characteristic Photon Energy

$$\varepsilon_c = 0.665\, B_o\, E^2$$

ε_c – Critical Photon Energy [keV]

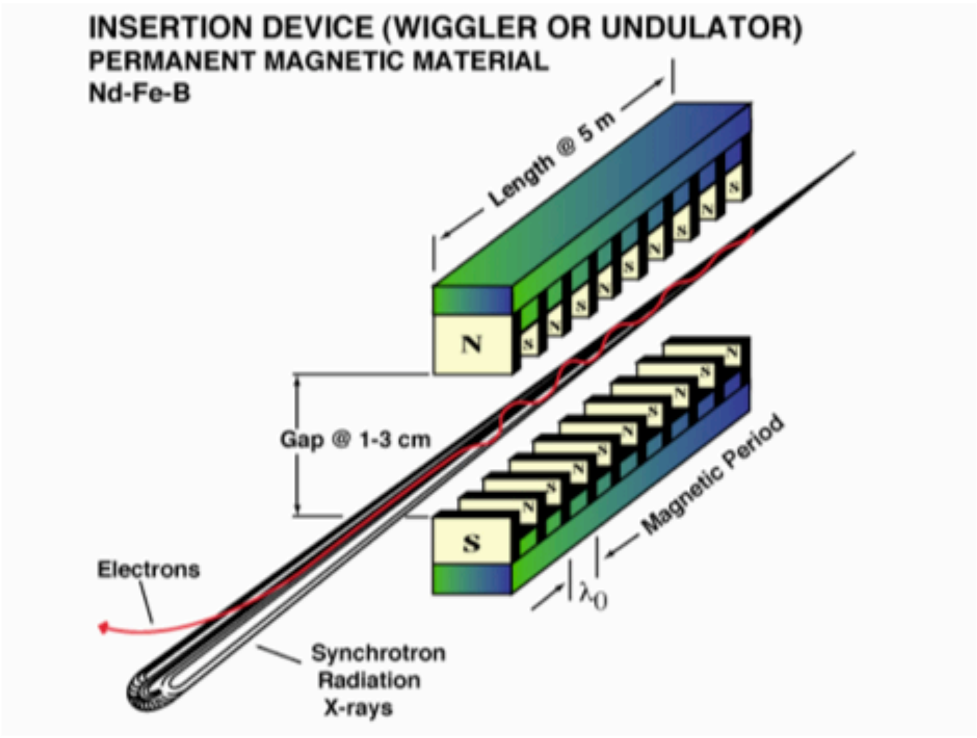
E – Electron Energy in [GeV]

B_o – Magnetic Field in [Tesla]

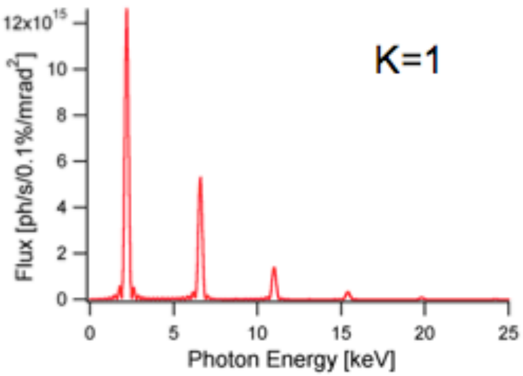
$$Flux\,/\,mrad\,/\,0.1\%BW = 2.457\times10^{13}\,E\,I\,G_1(y)$$

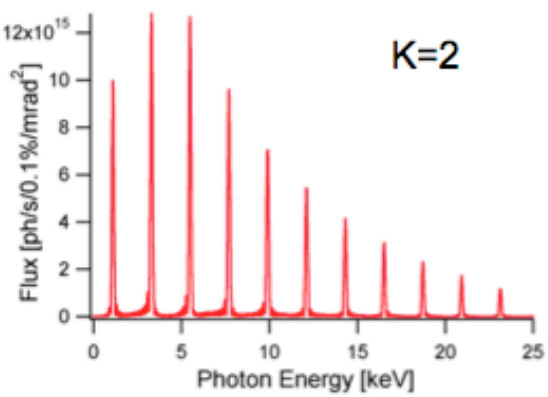
Insertion Device Radiation

Arrays of magnets bends the particle (electron) beam many times to increase x-ray flux



Insertion Device Spectrum





Undulator energy tuned by varying its K value – usually by tuning the magnetic gap which varies B

$K = 0.0934\ \lambda_u\ [mm]\ B_o\ [T]$

Some Parameters

APS Source Parameters

Undulator A
Period: 3.30 cm
Length: 2.4 m
K_{max}: 2.74 (effective; at minimum gap)
Minimum gap: 10.5 mm
Tuning range: 3.0-13.0 keV (1st harmonic)
3.0-45.0 keV (1st-5th harmonic)
On-axis peak brilliance:
4.6 x 10^{19} ph/s/mrad^2/mm^2/0.1%bw at 7 keV
Source size and divergence at 8.0 keV:
Σ_x: 273 µm Σ_y: 10 µm
$\Sigma_{x'}$: 12.6 µrad $\Sigma_{y'}$: 6.6 µrad

2.70-cm Undulator (sector 3)
Period: 2.70 cm
Length: 2.4 m
K_{max}: 2.18 (effective; at minimum gap)
Minimum gap: 8.5 mm
Tuning range: 5.1-16.0 keV (1st harmonic)
5.1-60.0 keV (1st-5th harmonic)
On-axis peak brilliance:
6.4 x 10^{19} ph/s/mrad^2/mm^2/0.1%bw at 8 keV
Source size and divergence at 8.0 keV:
Σ_x: 273 µm Σ_y: 10 µm
$\Sigma_{x'}$: 12.6 µrad $\Sigma_{y'}$: 6.6 µrad

5.50-cm Undulator (sector 2)
Period length: 5.50 cm
Length: 2.4 m
K_{max}: 6.57 (effective; at minimum gap)
Minimum gap: 10.5 mm
Tuning range: 0.4-7.0 keV (1st harmonic)
0.4-25.0 keV (1st-5th harmonic)
On-axis peak brilliance:
1.9 x 10^{19} ph/s/mrad^2/mm^2/0.1%bw at 4 keV
Source size and divergence at 4.0 keV:
Σ_x: 273 µm Σ_y: 10 µm
$\Sigma_{x'}$: 13.8 µrad $\Sigma_{y'}$: 8.7µrad

APS Bending Magnet
Critical energy: 19.51 keV
Energy range: 1-100 keV
On-axis peak brilliance:
5.6 x 10^{15} ph/s/mrad^2/mm^2/0.1%bw at 16.3 keV
On-axis peak angular flux:
9.6 x 10^{13} ph/s/mrad^2/0.1%bw at 16.3 keV
On-axis peak horizontal angular flux:
1.6 x 10^{13} ph/s/mradh/0.1%bw at 5.6 keV
Source size and divergence at the critical energy:
Σ_x: 91 µm Σ_y: 30 µm
$\Sigma_{x'}$: 6 mrad $\Sigma_{y'}$: 47 µrad

Circularly Polarized Undulator (sector 4)
Period: 12.8 cm
Length: 2.1 m
Circular mode:
K_{max}: 2.65 (effective; for both horizontal and vertical fields at maximum currents 1.2 kA horizontal and 0.34 kA vertical)
B_{max}: 0.26 T (peak fields)
Tuning range: 0.5-3.0 keV (1st harmonic)
On-axis peak circular brilliance:
3.4 x 10^{18} ph/s/mrad^2/mm^2/0.1%bw at 1.8 keV
Linear mode:
K_{max}: 2.80 (effective; for both horizontal and vertical fields at maximum currents 1.4 kA horizontal and 0.40 kA vertical)
B_{max}: 0.29 T (peak fields)
Tuning range: 0.8-3.0 keV (1st harmonic)
0.8-10.0 keV (1st-5th harmonic)
On-axis peak linear brilliance:
2.5 x 10^{18} ph/s/mrad^2/mm^2/0.1%bw at 2.1 keV
Switching frequency: 0-5 Hz
Switching rise time: 20 ms
Source size and divergence at 1.5 keV:
Σ_x: 273 µm Σ_y: 10µm
$\Sigma_{x'}$: 17.9 µrad $\Sigma_{y'}$: 14.4 µrad

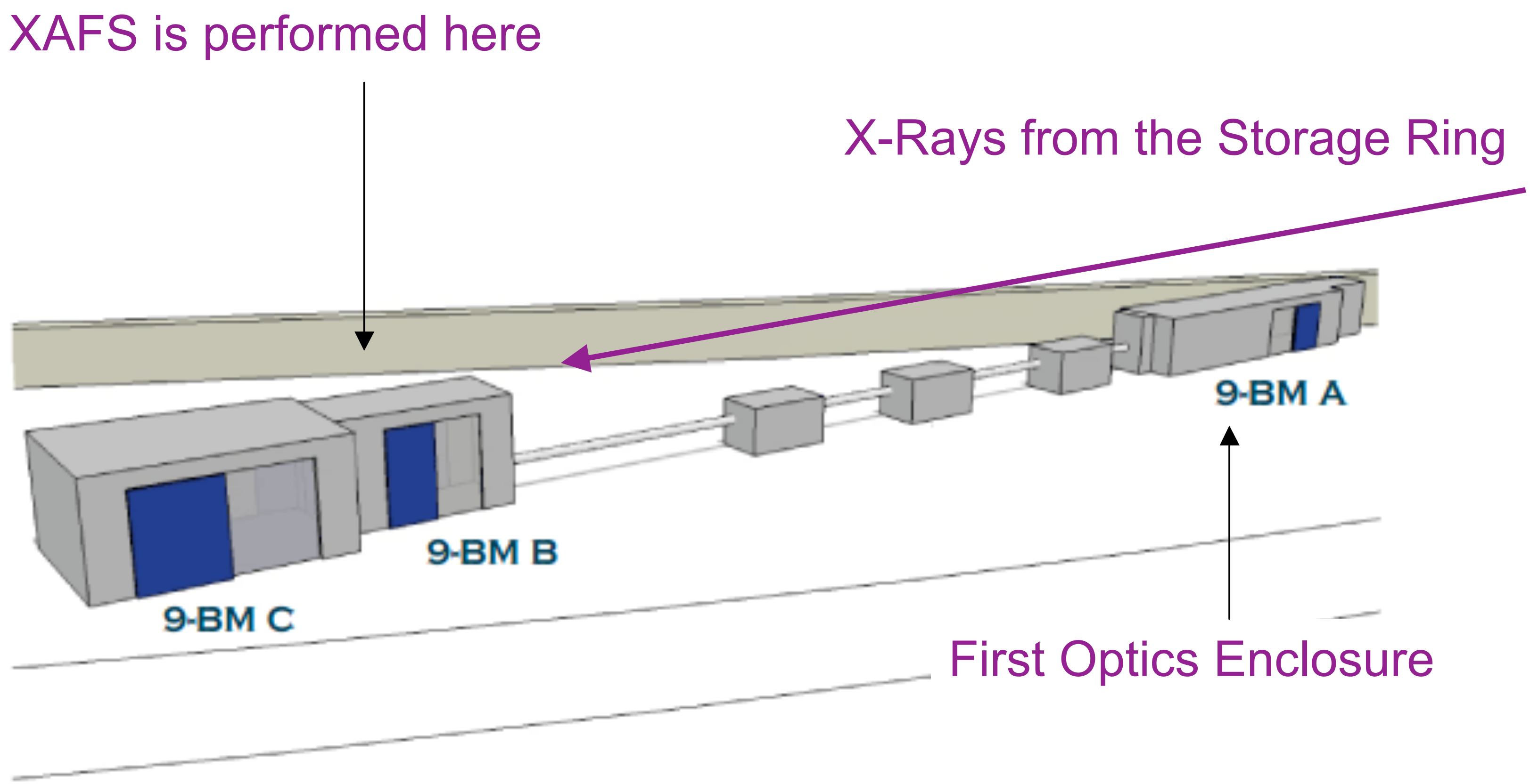
Elliptical Multipole Wiggler (sector 11)
Period length: 16.0 cm
Number of poles: 34 permanent magnets,
36 electromagnets
Length: 2.8 m
$K_{x\text{-}max}$: 1.3 (effective; at maximum current 1.15 kA)
$K_{y\text{-}max}$: 14.4 (peak; at minimum gap 24.0 mm)
Switching frequency: 0-10 Hz
Critical energy: 31.4 keV (at minimum gap)
Energy range: 5-200 keV
Source size and divergence at the critical energy:
Σ_x: 273 µm Σ_y: 10 µm
$\Sigma_{x'}$: 820 µrad (FWHM 1.9 mrad; non-Gaussian; linear mode)
$\Sigma_{y'}$: 47 µrad (linear mode)

Undulator A
Period: 3.30 cm
Length: 2.4 m
K_{max}: 2.74 (effective; at minimum gap)
Minimum gap: 10.5 mm
Tuning range: 3.0-13.0 keV (1st harmonic)
3.0-45.0 keV (1st-5th harmonic)
On-axis peak brilliance:
4.6 x 10^{19} ph/s/mrad^2/mm^2/0.1%bw at 7 keV
Source size and divergence at 8.0 keV:
Σ_x: 273 µm Σ_y: 10 µm
$\Sigma_{x'}$: 12.6 µrad $\Sigma_{y'}$: 6.6 µrad

APS Bending Magnet
Critical energy: 19.51 keV
Energy range: 1-100 keV
On-axis peak brilliance:
5.6 x 10^{15} ph/s/mrad^2/mm^2/0.1%bw at 16.3 keV
On-axis peak angular flux:
9.6 x 10^{13} ph/s/mrad^2/0.1%bw at 16.3 keV
On-axis peak horizontal angular flux:
1.6 x 10^{13} ph/s/mradh/0.1%bw at 5.6 keV
Source size and divergence at the critical energy:
Σ_x: 91 µm Σ_y: 30 µm
$\Sigma_{x'}$: 6 mrad $\Sigma_{y'}$: 47 µrad

APS Undulator A

9BM Layout



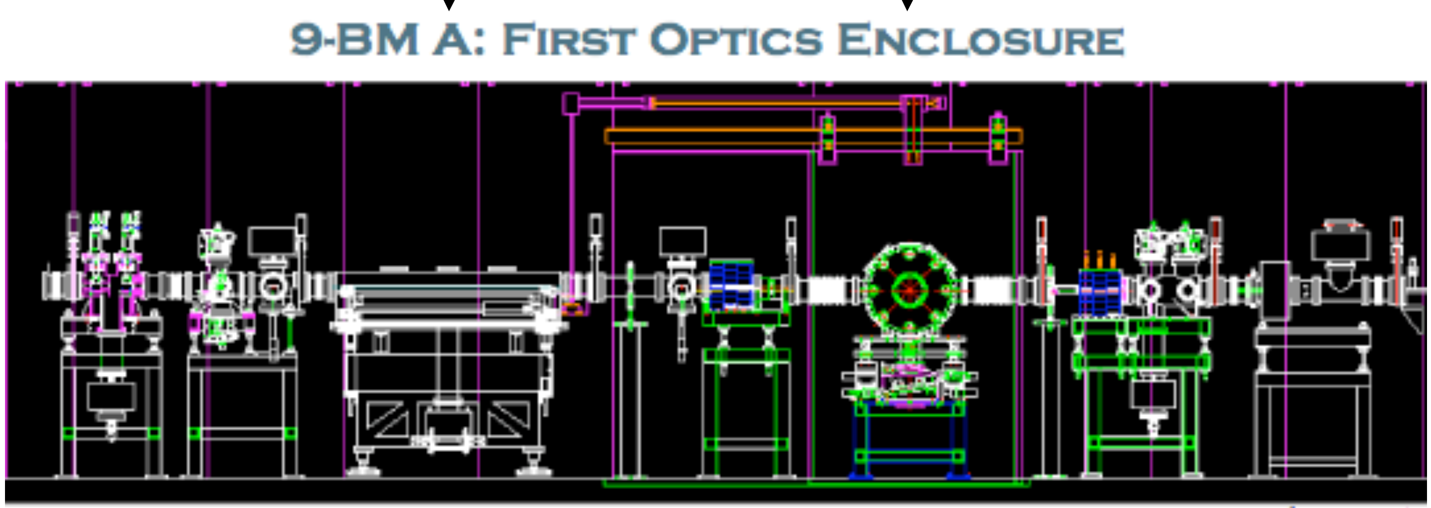
XAFS is performed here
X-Rays from the Storage Ring
9-BM A
9-BM B
9-BM C
First Optics Enclosure

9 BM First Optics Enclosure

9 BM's FOE contains the optical components used to focus and monochromatize the x-ray beam

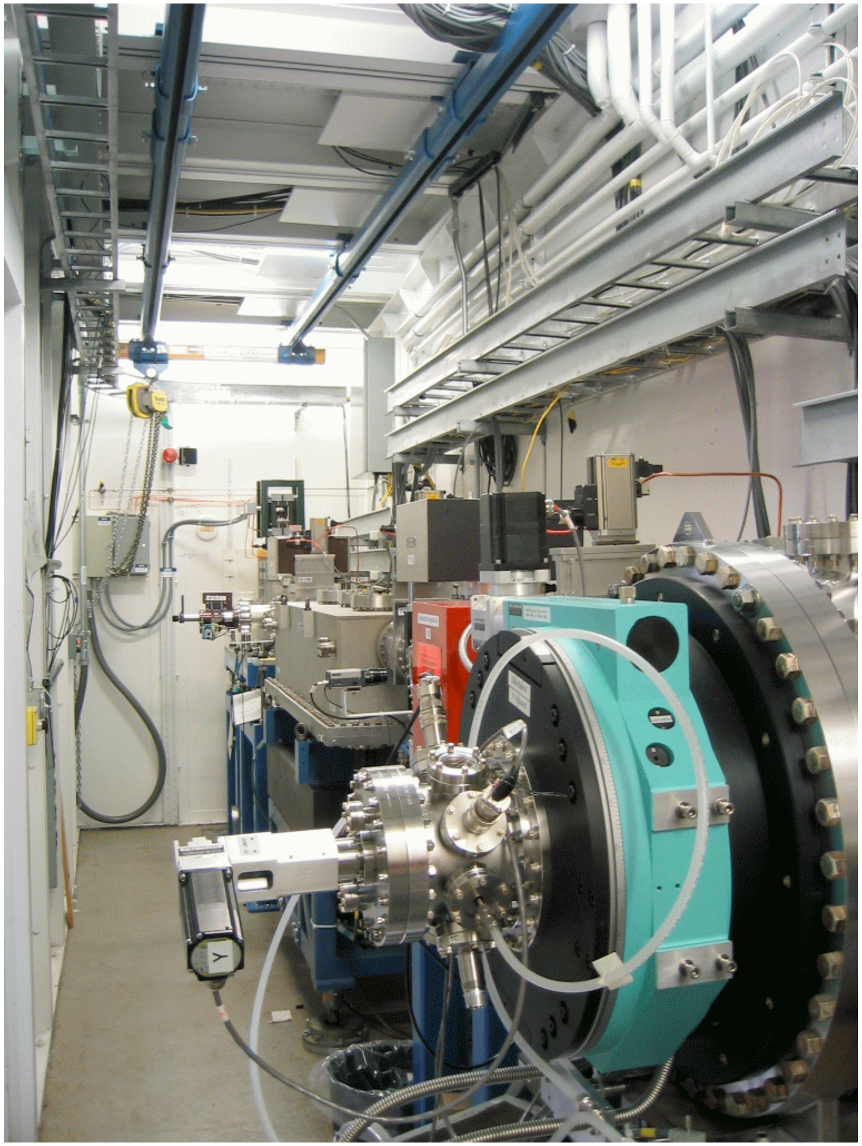
Toroidal Mirror

Monochromator



Beam

Some Photos of the 9 BM First Optics Enclosure



FOE Components

- White beam slits select “hot” spot from Storage Ring and play a role in defining the energy resolution at a given energy
- Monochromator controls energy selection (dual crystals-Si(111) for lower energies and Si(220) for higher energy resolution)
- Combined energy range is 2.1 to 22 keV
- Toroidal Mirror controls focusing

  Many degrees of freedom in movement (yaw, pitch, bending, etc.)

  – Typical beam size is .5mm by .5 mm in 9-BM-B

Monochromators

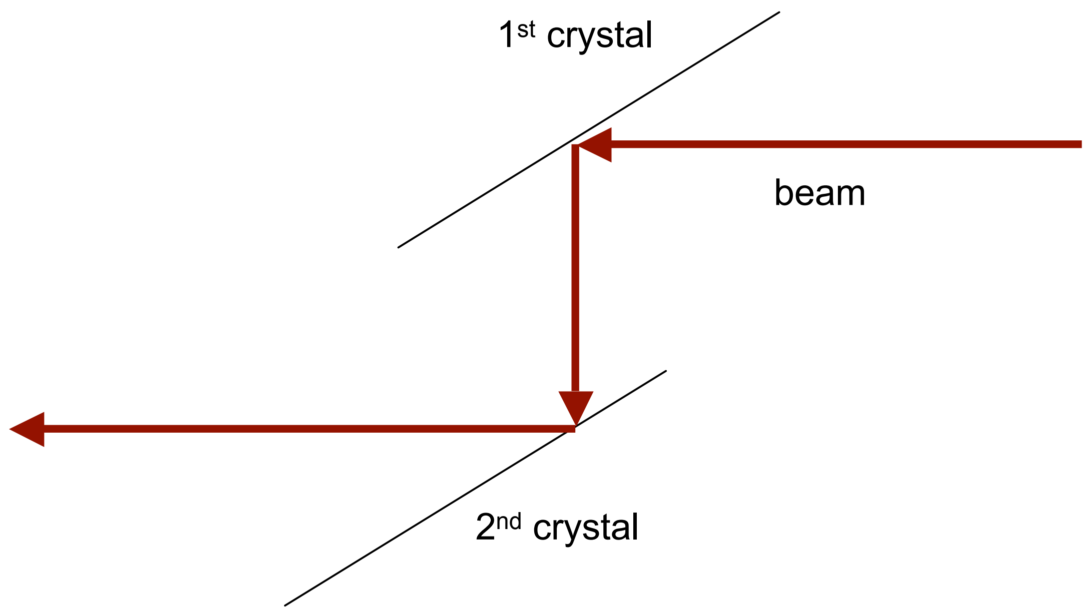
- A monochromator works by the use of Bragg's Law

$$n\lambda=2d\sin\theta$$

- Crystals cut along a specific set of planes, such as Si(111), will diffract specific wavelengths depending on their incident angle to the beam

More Monochromators

- Most monos at the APS are Double Crystal which will direct the exit beam in the direction of the incident beam

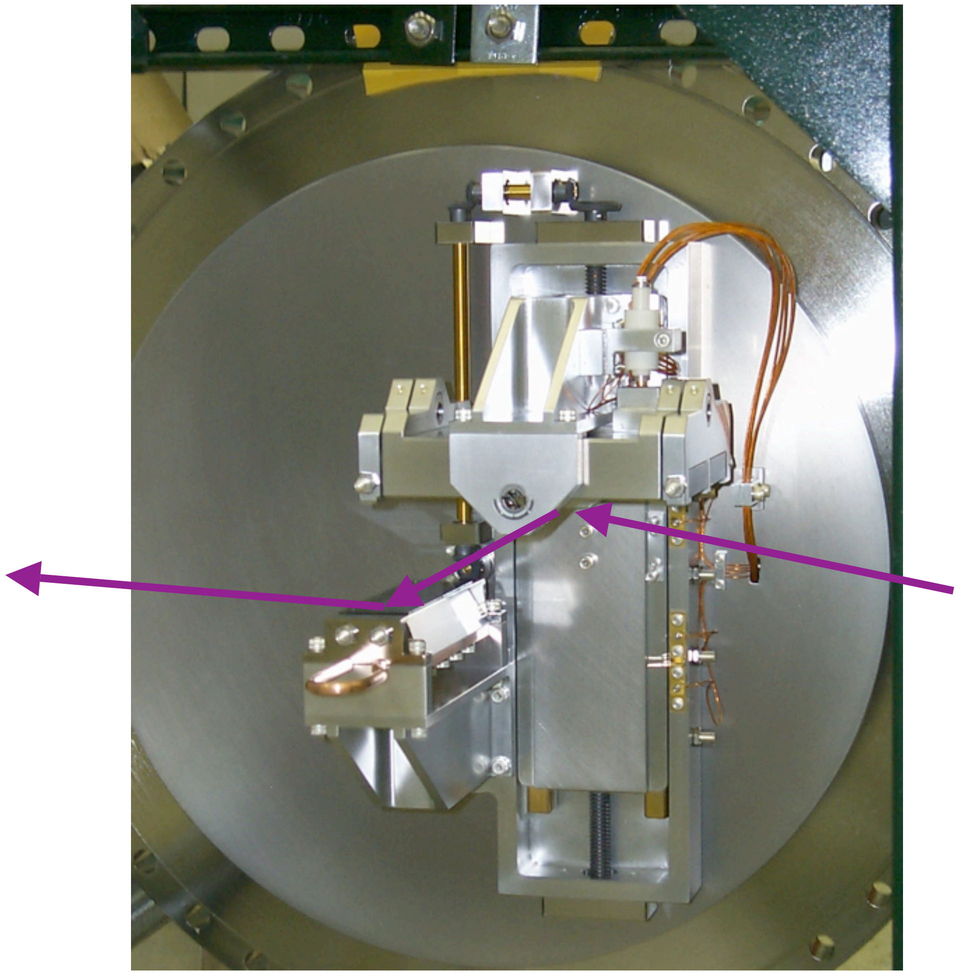


Some notes on monochromators in general

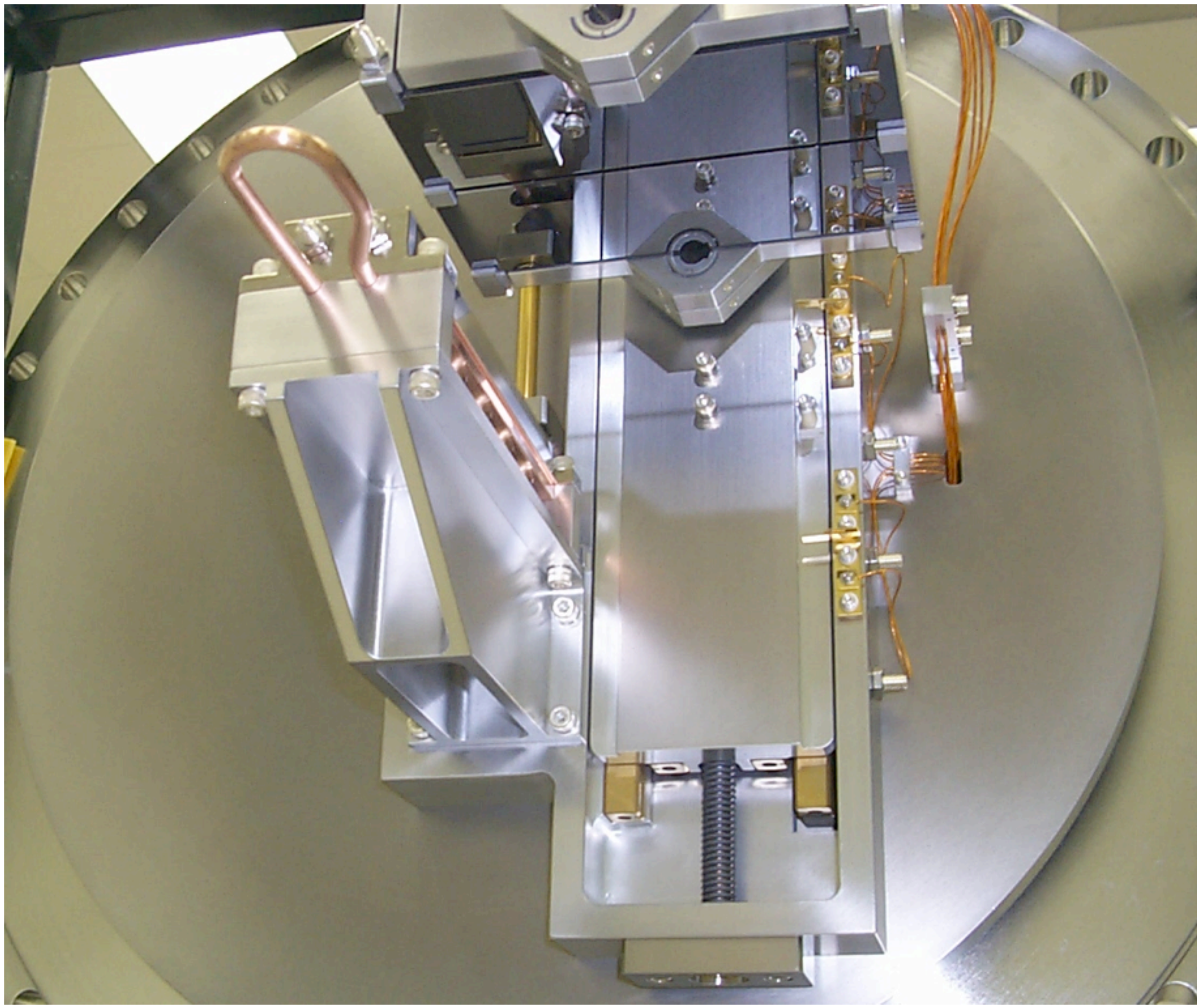
- A good monochromator is essential for an good XAFS experiment
- A few elements of a good monochromator
  - Good energy reproducibility/calibration
  - Thermal Stability (little beam drift due to heating effects)
  - Good use of energy resolution ($\Delta E/E \sim 1E-4$) intrinsic to mono crystals (mechanically, can the mono move in a step size much smaller than the resolution?)
  - Optimized energy range while keeping a good quality beam

9 BM Monochromator

- 2nd crystal translates up and down to keep a fixed exit height
- First set of crystals relatively long in order to keep the incident beam on them
- 2nd crystal motion controlled by Inchworm Motors (Burleigh)

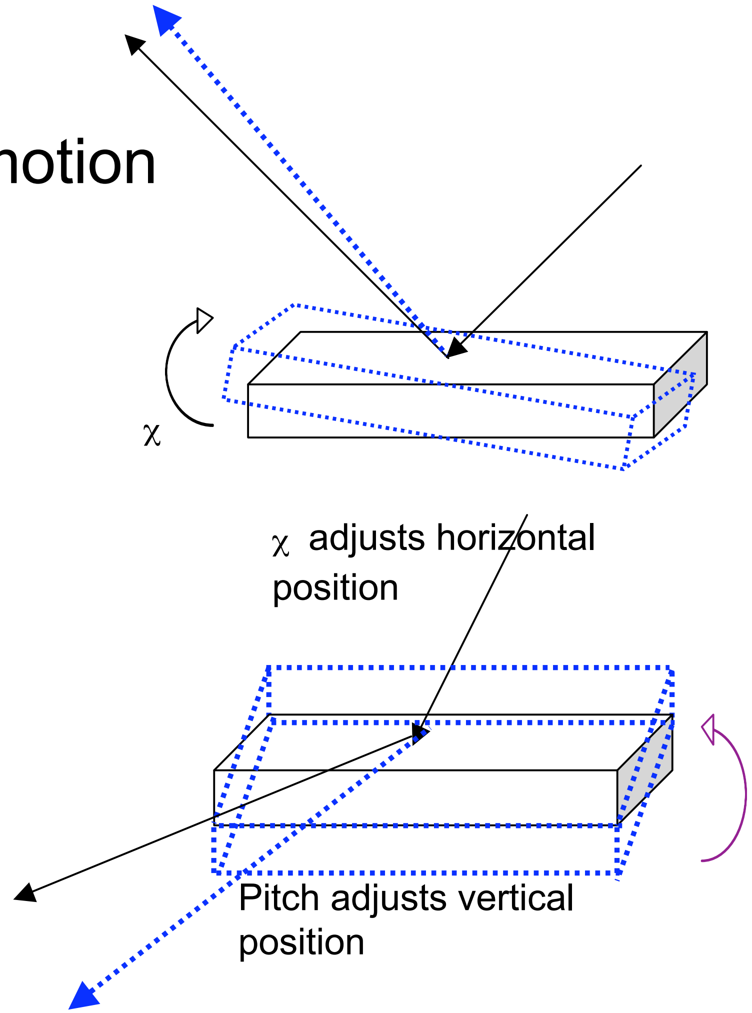


9 BM Monochromator

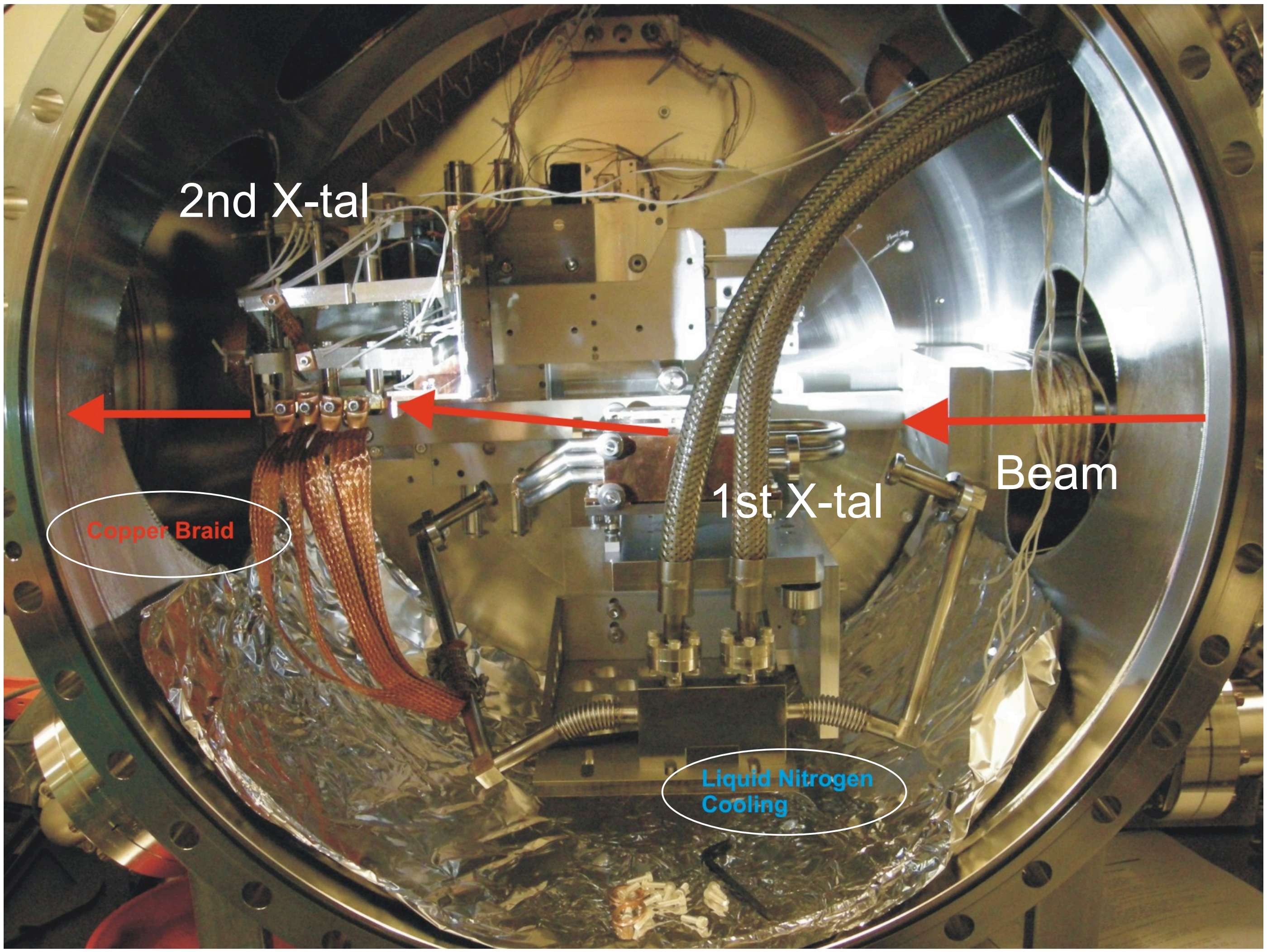


2nd crystal motion

- Typically, second crystal motion Consists of tilting either transverse to (adjusting χ) or along the beam (pitching)
- *Pitching is used to detune for harmonics rejection or to make small adjustments to intensity*
- *Adjusting χ affects horizontal beam motion*



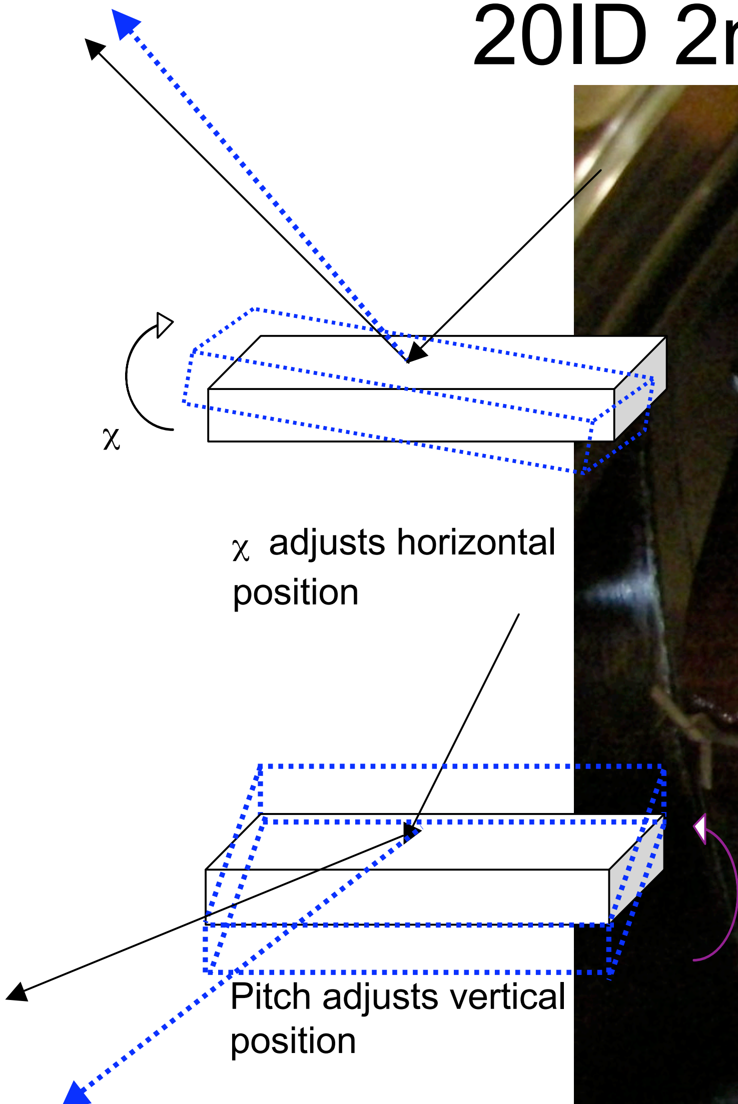
Sector 20 ID Monochromator

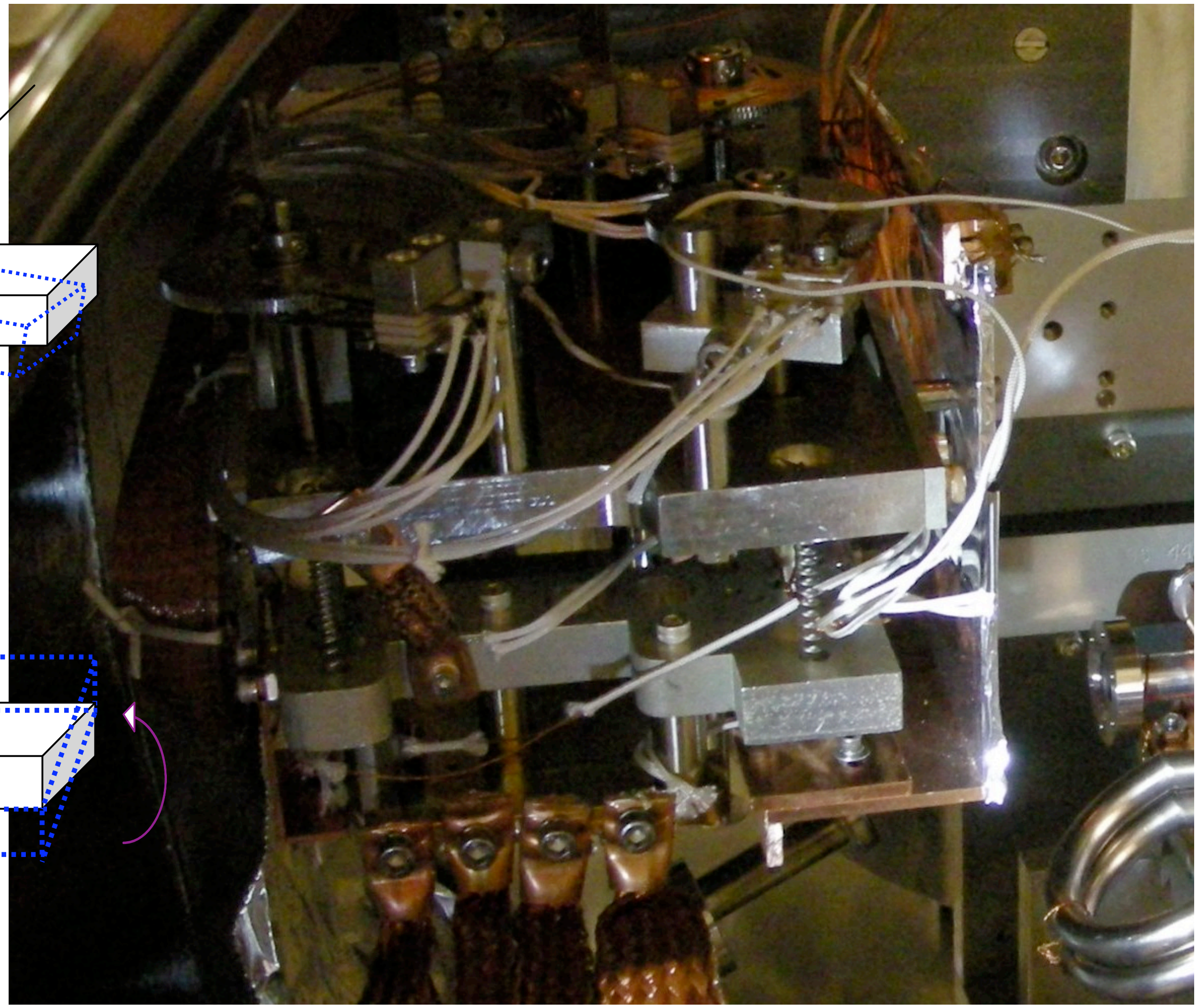


20ID Monochromator Details

- Designed by BESSRC
- Dual Crystal, Double Crystal Monochromator with Si(111) and Si(311) crystals
- Si(311) is for higher energies and higher energy resolution ($\Delta E/E<10e-4$)
- Lowest energy allowed by monochromator design: Si(111) 4.3KeV
- Combined Energy Range is 4.3 to 50 keV
- Smallest focused beam size for Si(111) is 1 micron by 1 micron

Showing the set of motors needed for 20ID 2nd X-tal motion…

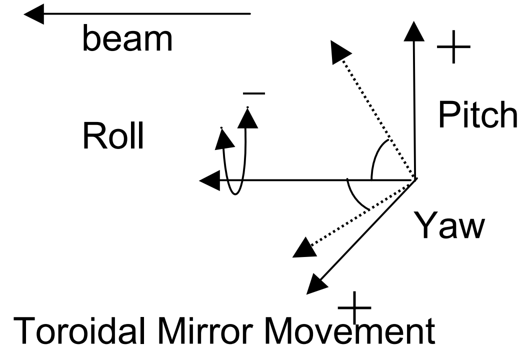


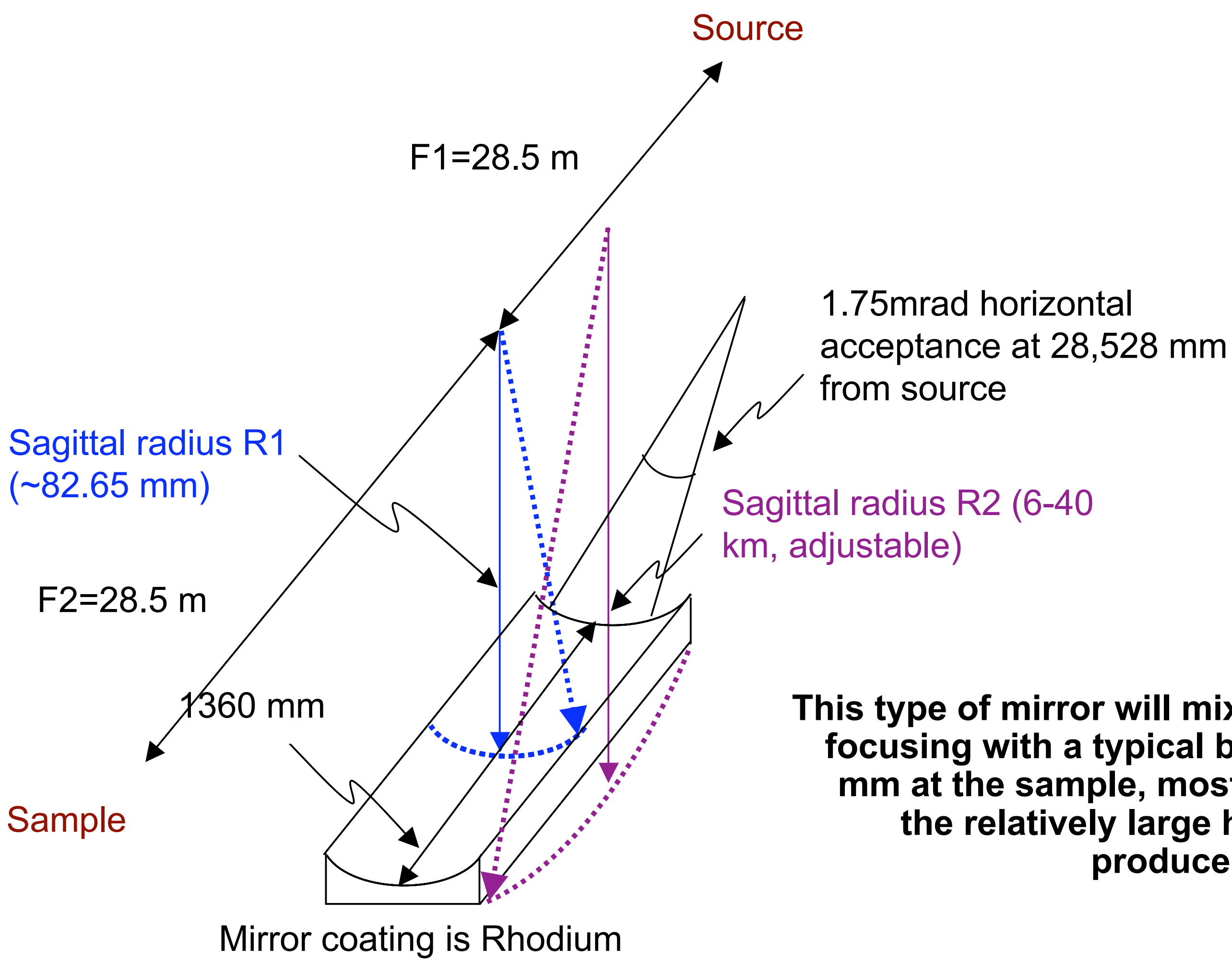


20ID Second Crystal Motion

- Two kinds of motors; piezo crystals for small motions and stepping motors for large
- 2nd X-tal has multiple degrees of motion, but most used are pitch (detuning/horizontal rejection) and yaw (χ adjustments)

Toroidal Mirror



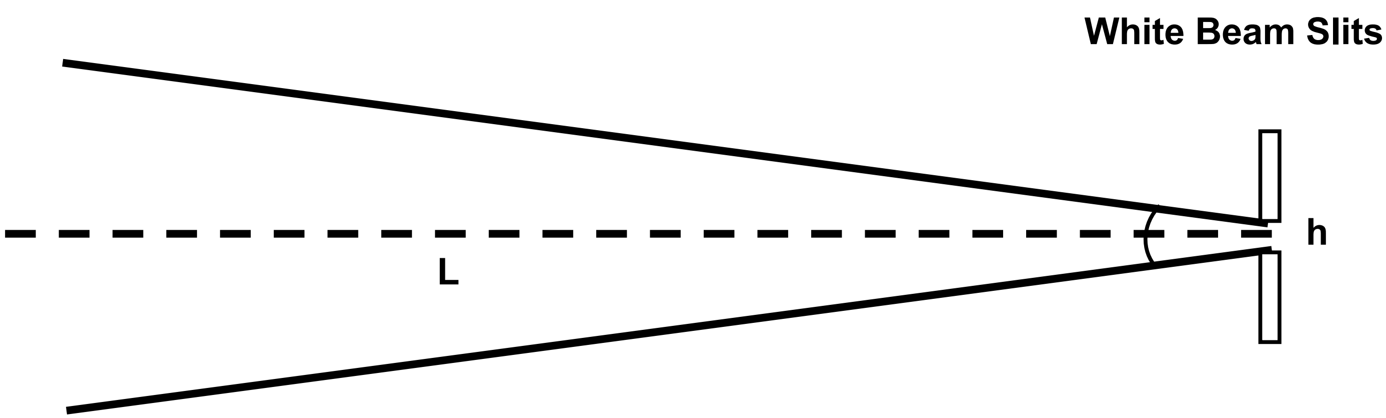


This type of mirror will mix horizontal and vertical focusing with a typical beam size of about .5 by .5 mm at the sample, most typically used to accept the relatively large horizontal divergence produced by BM's

Slits and Energy Resolution

$\Delta E/E = -\cot\theta\Delta\theta$

Resolution (ΔE) 0.5–4 eV Si(111)
0.25–2 eV Si(220)

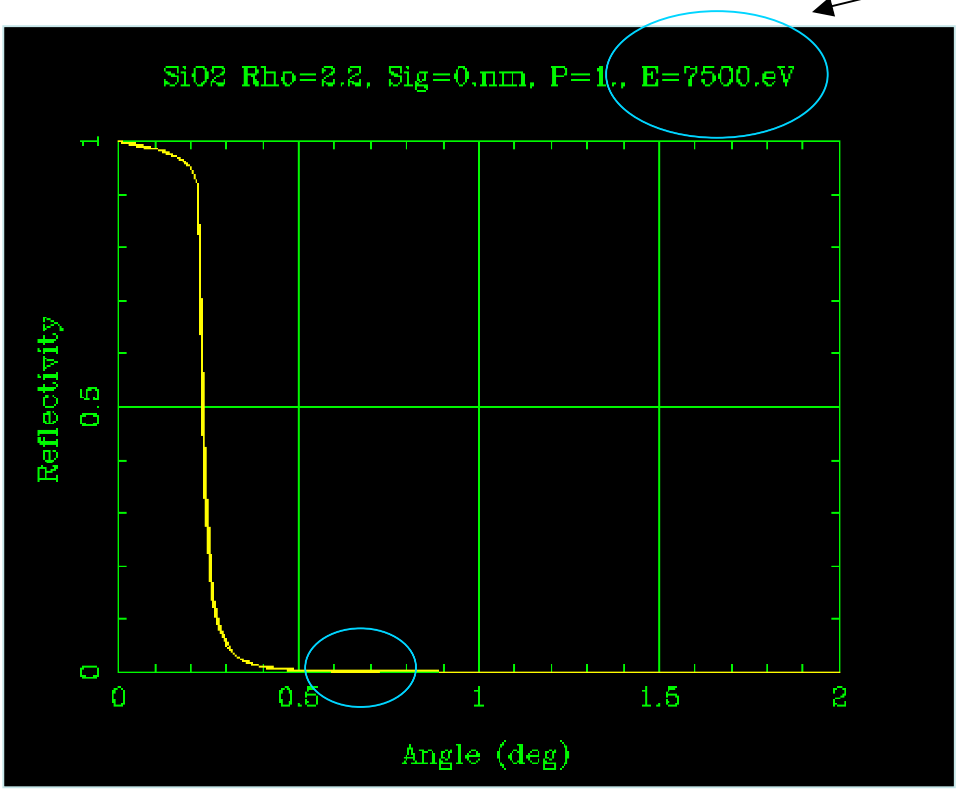


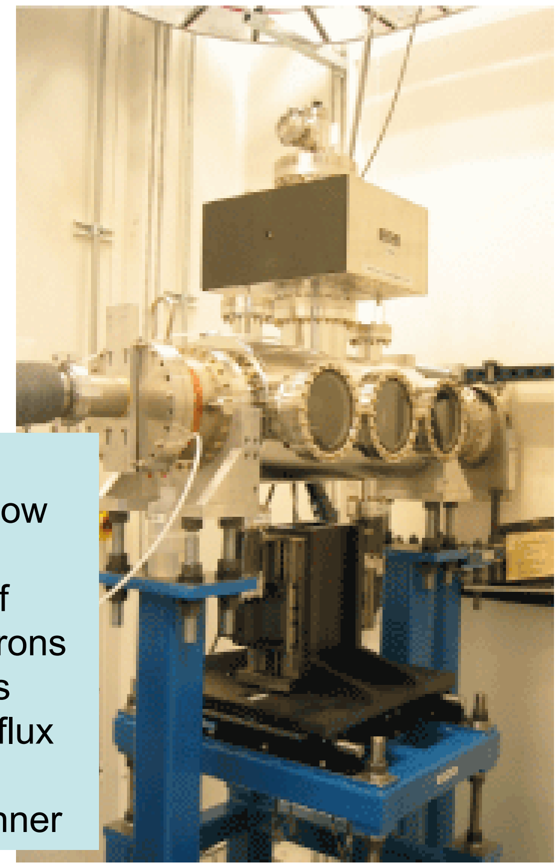
Approximating the vertical source size to be a point, then **h=.75mm** WBS slit height and **L= 25,158 mm** from the source leads to about **.3 eV** resolution at 2.5 keV

9 BM-B Optical Component

There is one other optical component, which is in 9-BM-B; a Rh-coated, flat harmonics rejection mirror (Smir)

3rd harmonic for E=2.5 keV

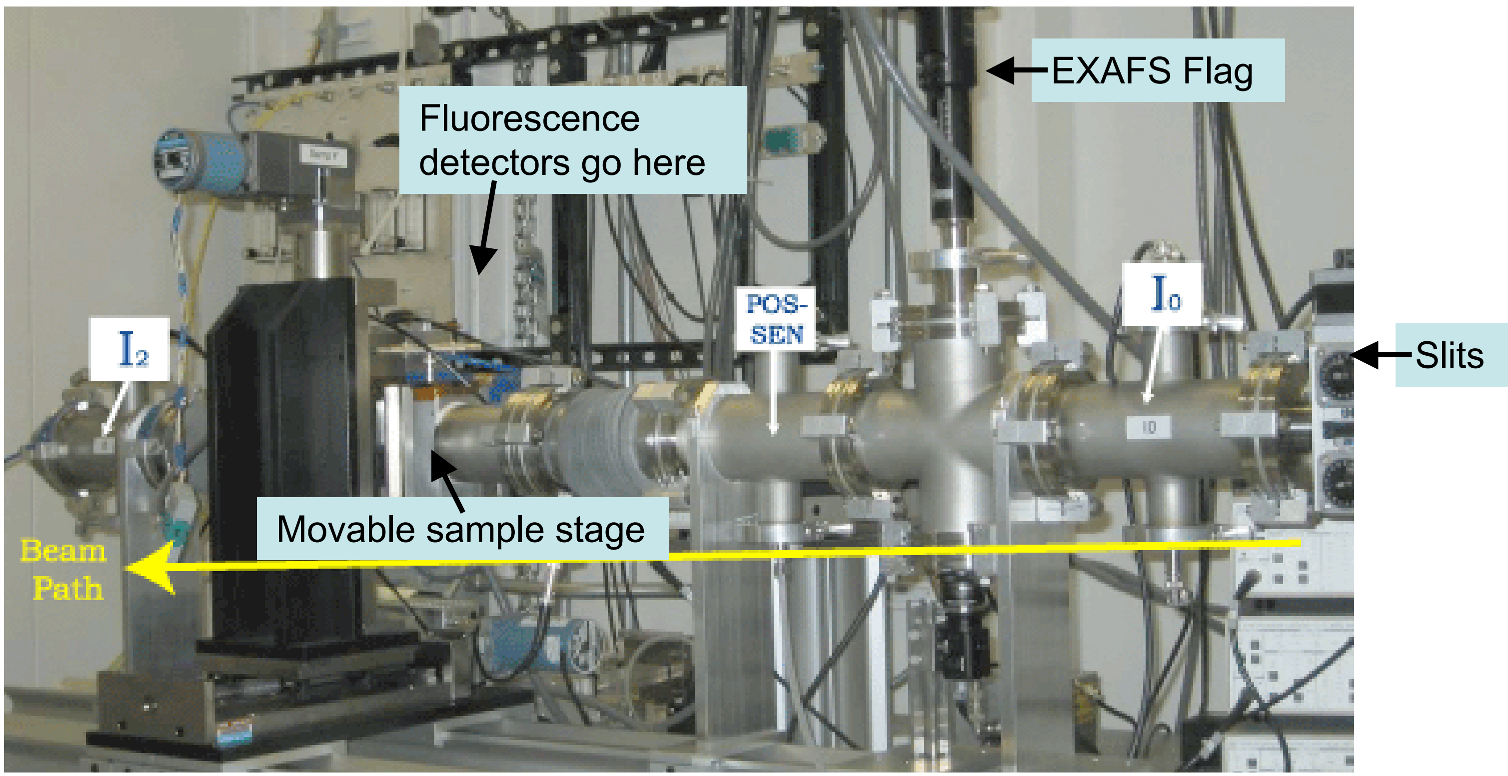




There is one beryllium window located just downstream of Smir (250 microns thick); this cuts down a bit on flux and we have plans for a thinner one…

A Picture of the Ion Chambers, etc…

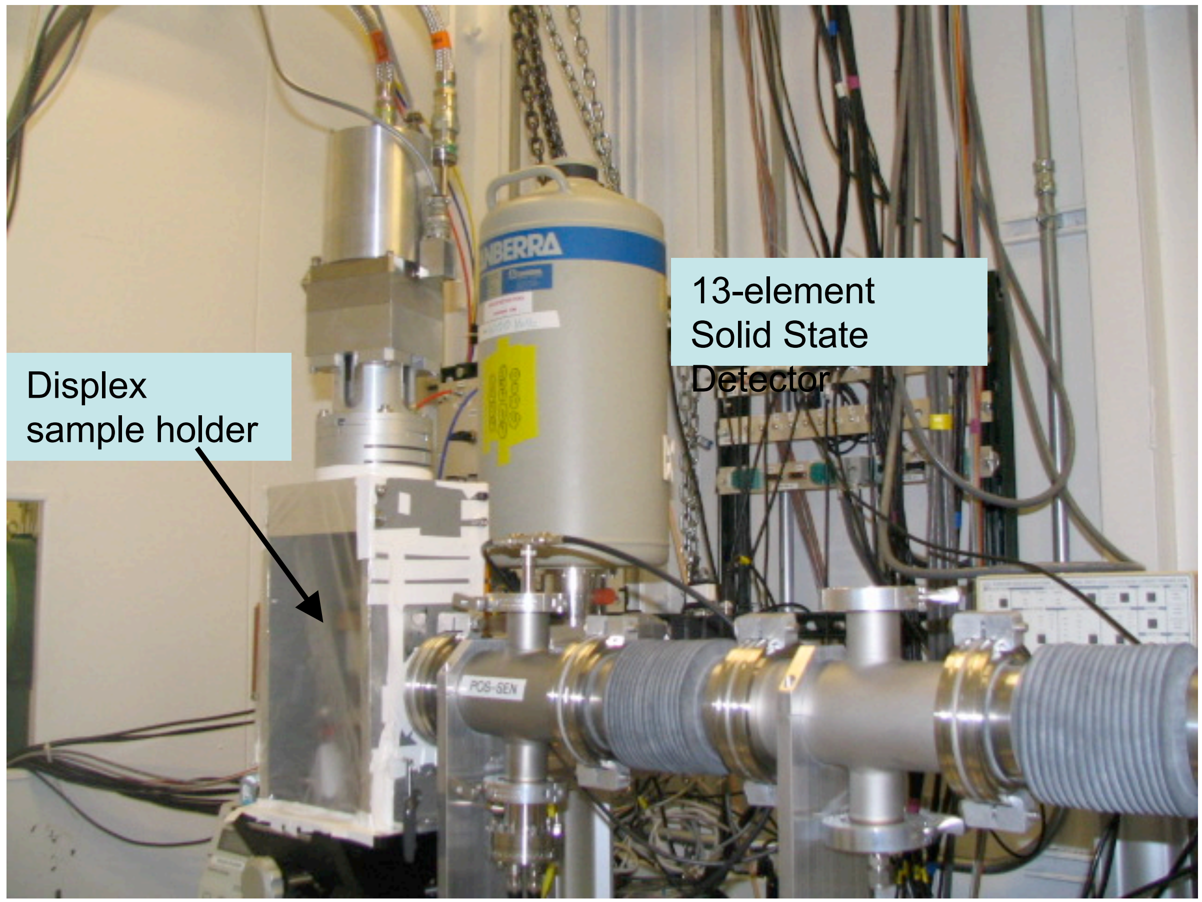
- Three beamline sections separated by polycarbonate (5 micron) windows- upstream (I0), sample chamber, and downstream (I2)
- Three sections can be purged independently depending on energy, etc.



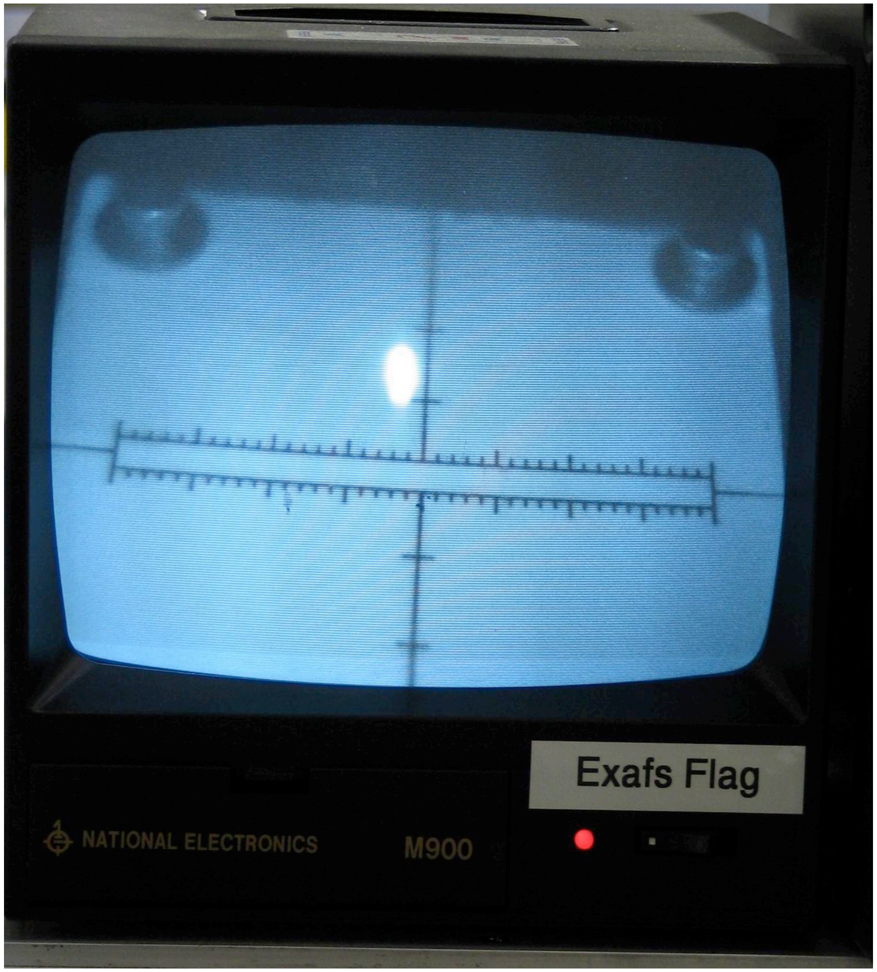
More 9 BM-B…

Sometimes sample cooling is needed. Here is one way to set that type of experiment up for measurements in fluorescence mode.

Two main types of fluorescence detectors are either Lytle Detectors and Energy Dispersive Detectors. Typical energy resolution is about 160-250 eV.



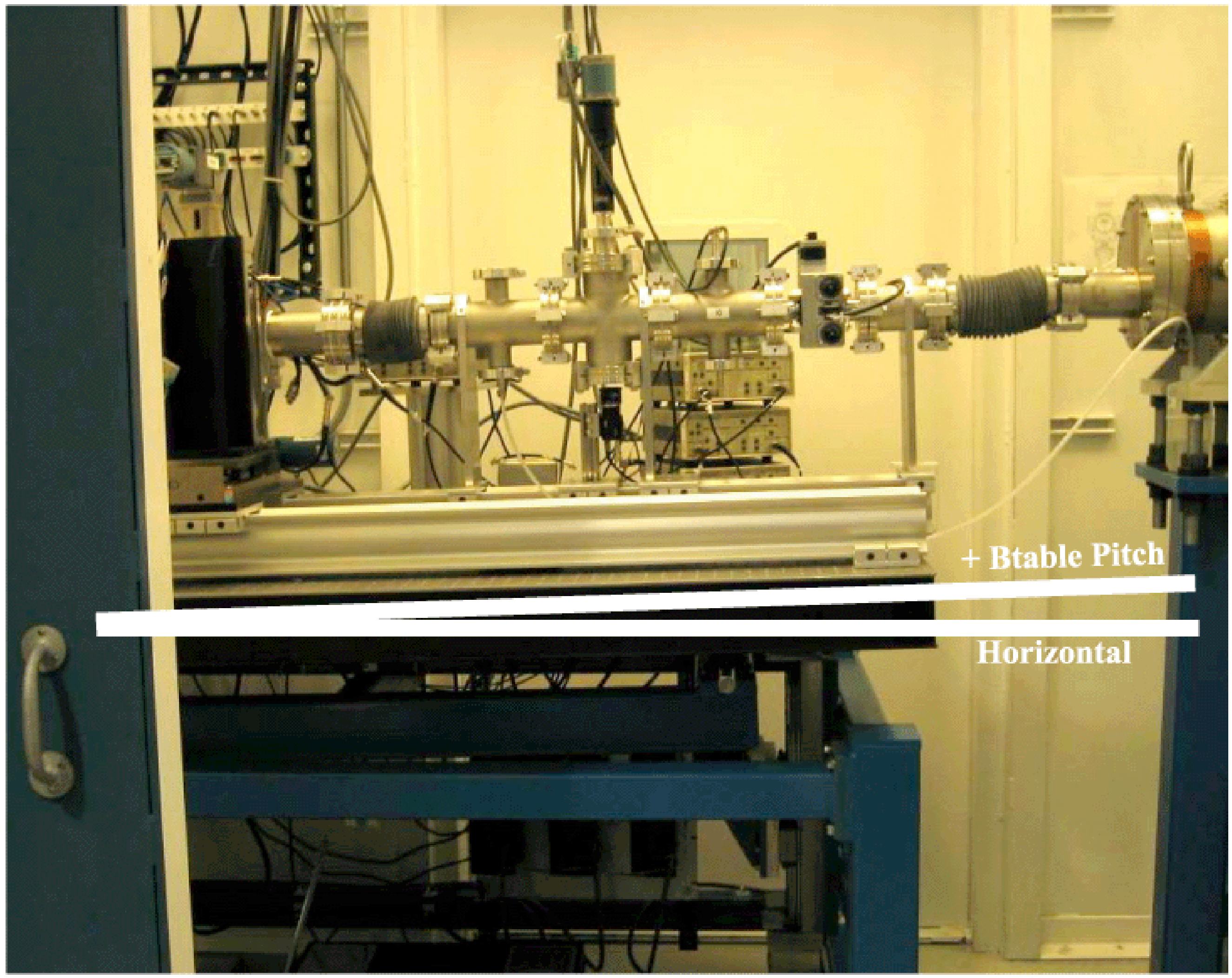
Picture of Beam on Flag in 9-BM-B…



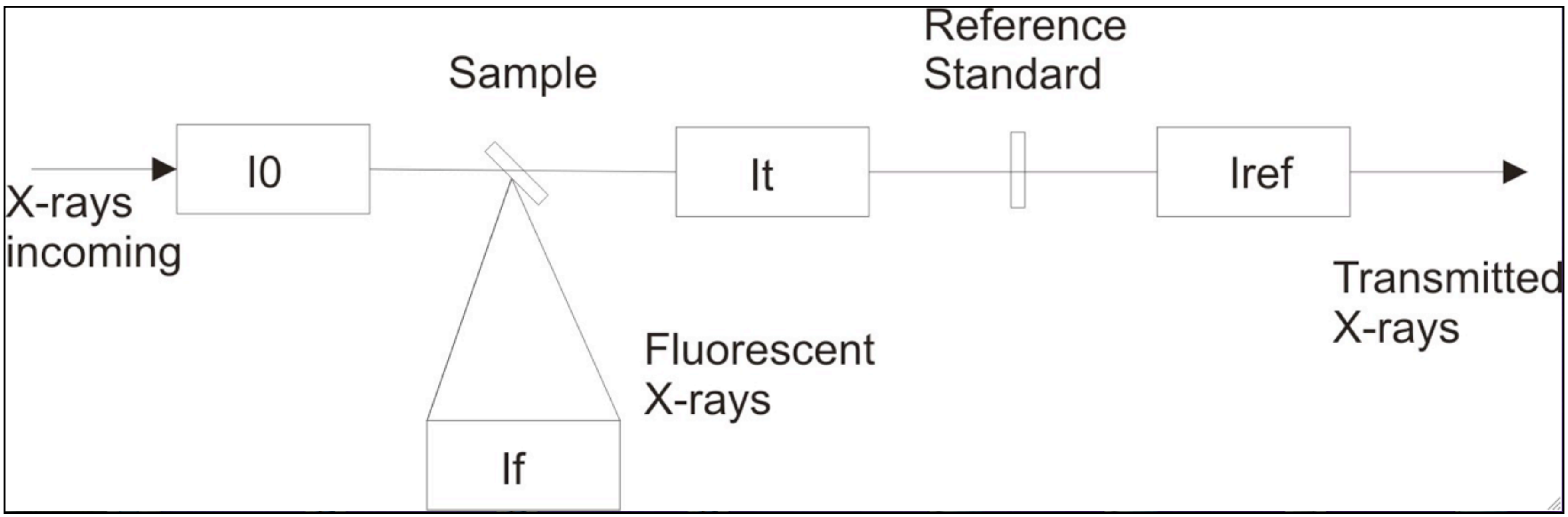
Last 9 BM-B Slide…

Sample Table with positive pitch for beam alignment

Whole table can move in various directions



Typical XAFS Experimental Setup



XAFS Measurements…

Typical points to consider:

- Concentration of element of interest
- Other elements in the sample that may interfere with XAFS spectrum of the desired element
- What energy? (air absorption and use of purging to limit it)
- In what form is the sample?
- What temperature is needed?
- Radiation damage/oxidation

Feedback Systems

- For example, needed for samples which have elements of interest that are not evenly distributed
- Typically, a feedback system tweaks the position of optical components in the horizontal and vertical direction. For example, at 9-BM the toroidal mirror pitch and χ on the 2nd mono crystal are tweaked
- System can be turned on and off according to experimental needs