Optimal Source Characteristics for Micro-Beams for Macromolecular Crystallography

Gerd Rosenbaum

Structural Biology Center, ANL

and Dept. of Biochemistry, UGA

Workshop on Biology and Life Sciences

14 July 2006

Upgrade of the Technical Facilities of the SBC

Aims:

- More than Doubling Capacity of SBC Sector
- Adding Microbeam Capability
  (no PX beamline with microfocus at the APS at present)
- Upgrading Existing Beamlines to 3rd Generation Technology
- Improving Stability and Reliability of Beam on Sample (R&D)
- Preparing X-ray Optics for 200 mA Beam Current (R&D)
- Fully Integrated Auto-Mounting and Auto-Centering of Samples (R&D)

Doubling Capacity of SBC Sector

- Adding two undulator beamlines
- Three independent undulators in the straight section
- Undulator sequence: short - long - short
- X-ray beam directions:
  0.5 mrad outboard; center line; 0.5 mrad inboard
- Short undulators (new design): 1.0 m long, 3.1 cm period
- Long undulator (new magnetic structure) : 2.1 m long, 3.1 cm period
- All three beamlines can be operated independently of each other:
  independent wavelength change, independent access to endstation
- Flux into central cone ~ $L^{1.05 \ldots 1.2}$ (L = length of undulator)

Three Undulators in Straight Section

- One beam pipe for all three undulators, 4.8 m long, new design (with integral particle BPMs before, between, and after undulators)
- Between undulators:
  Deflector magnets (+ corrector magnet ?): each set about 0.35 m long
- Length budget: 1.0 m + 0.35 m + 2.1 m + 0.35 m + 1.0 m = 4.8 m
- Need new front end with triple beam mask (design, manufacture by APS)
- Need to adapt new hard X-ray BPMs for triple beam (R&D by GR & APS)

Source Sizes, X-ray Beam Divergences and Flux into Central Cone vs. Undulators Length

APS in lower emittance mode (http://www.aps.anl.gov/asd/oag/beamParameters.html):

natural emittance 2.5 nm-rad, coupling 0.01, β_x = 19.5 m, β_y = 2.87 m, α_x = 0, α_y = 0,
η_x = 0.172 m, η_y = 0, $\eta_{x'}$ = 0, $\eta_{y'}$ = 0, $\delta E/E$ = 0.000965
➔
σ_x = 275.3 µm, σ_y = 8.5 µm, $\sigma_{x'}$ = 11.3 µrad, $\sigma_{y'}$ = 3.0 µrad

Diffraction limited radiation source size and angle for undulators of length L:

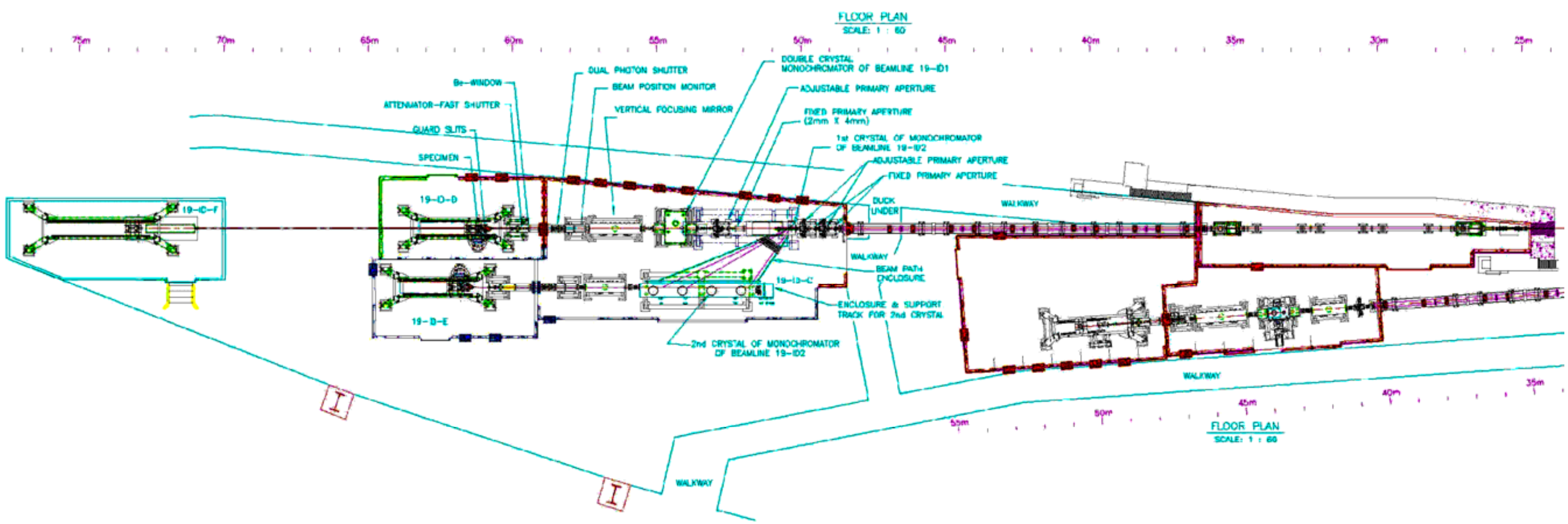
at E_{phot} =	6 keV	12 keV	18 keV	6 keV	12 keV	18 keV
L (m)		σ_r (µm)			$\sigma_{r'}$ (µrad)	
2.4	2.5	1.7	1.4	6.7	4.7	3.9
2.1	2.3	1.6	1.3	7.1	5.0	4.1
1.0	1.6	1.1	0.9	10.5	7.5	6.1

Radiation source size and angles and flux into central cone at 12 keV for undulator of length L and magnetic period P:

L (m)	P (cm)	Σx (µm)	Σy (µm)	$\Sigma x'$ (µrad)	$\Sigma y'$ (µrad)	Flux 1st (10^{14} ph/s/0.1%BW)	Flux 3rd
2.4	3.3	275.3	8.7	12.2	5.6	2.30	3.67
2.1	3.1	275.3	8.6	12.4	5.8	2.88	3.56
1.0	3.1	275.3	8.6	13.6	8.1	1.17	1.44

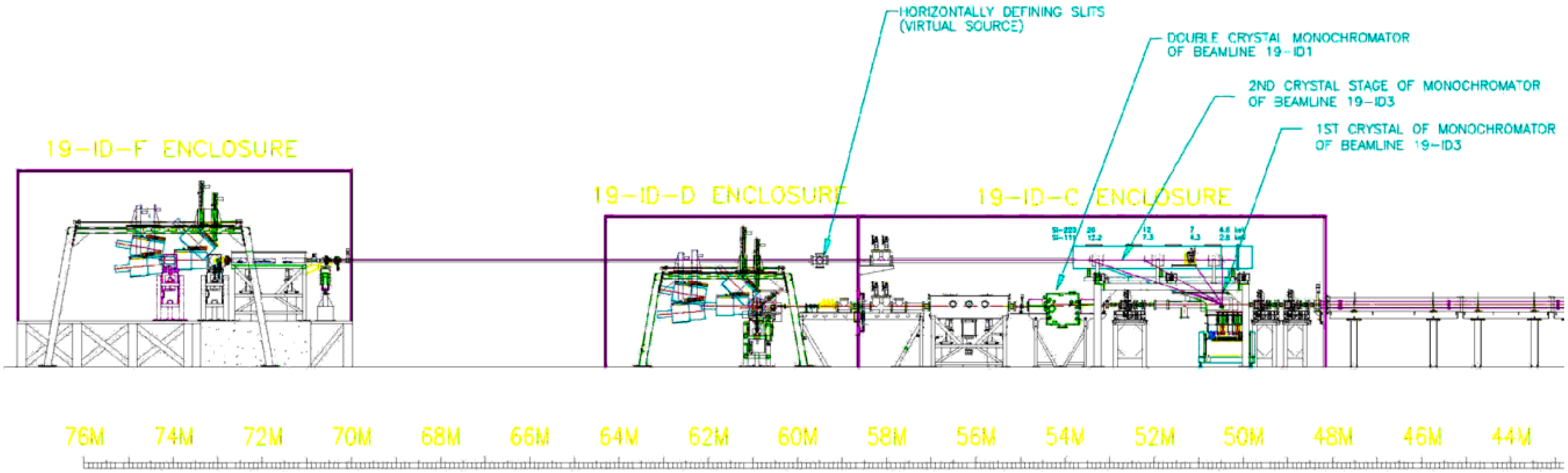
Three Undulator Beamlines Upgrade

Overview Plan View



Three Undulator Beamlines Upgrade

Overview Elevation



Vertically Offset Beamline
(Outboard Beam)

- Vertically deflecting monochromator
- Dual cut 1st crystal:Si-111 and 220
- Monochromatic beam 1 m vertically offset
- 2nd crystal stage on long track with crystal changer (111 and 220)
- Photon energy range: 3 - 20 keV (0.62 - 4 Å)
  (Bragg-angles 9.2°-45°, Si-111: 2.8 -12.2 keV, Si-220: 4.6 -20 keV)
- Minimum focal size: 1 µm x 3 µm FWHM (hor x ver)
- Beam transport through center beam line endstation above detector positioner
- New endstation enclosure and beam transport

Need for Microbeam

- Very small crystals: reduce scatter from non-crystalline material
- Selective exposure of small crystal volumes:
  - very asymmetric shape of crystal (e.g. needle): reduce scatter from non-crystalline material
  - very small, well ordered domains
- Reducing radiation damage in exposed volume:
  - photo-electrons travel several µm (~6µm for 18 keV initial energy)
  - large fraction of damaging energy is not deposited in the illuminated volume for micrometer size beams
  - energy deposit per distance traveled is not uniform, very high at end of travel
- Photo-electrons ejected predominantly in direction of electric field vector

From: Colin Nave & Mark A. Hill, JSR (2005), 12, 299-303

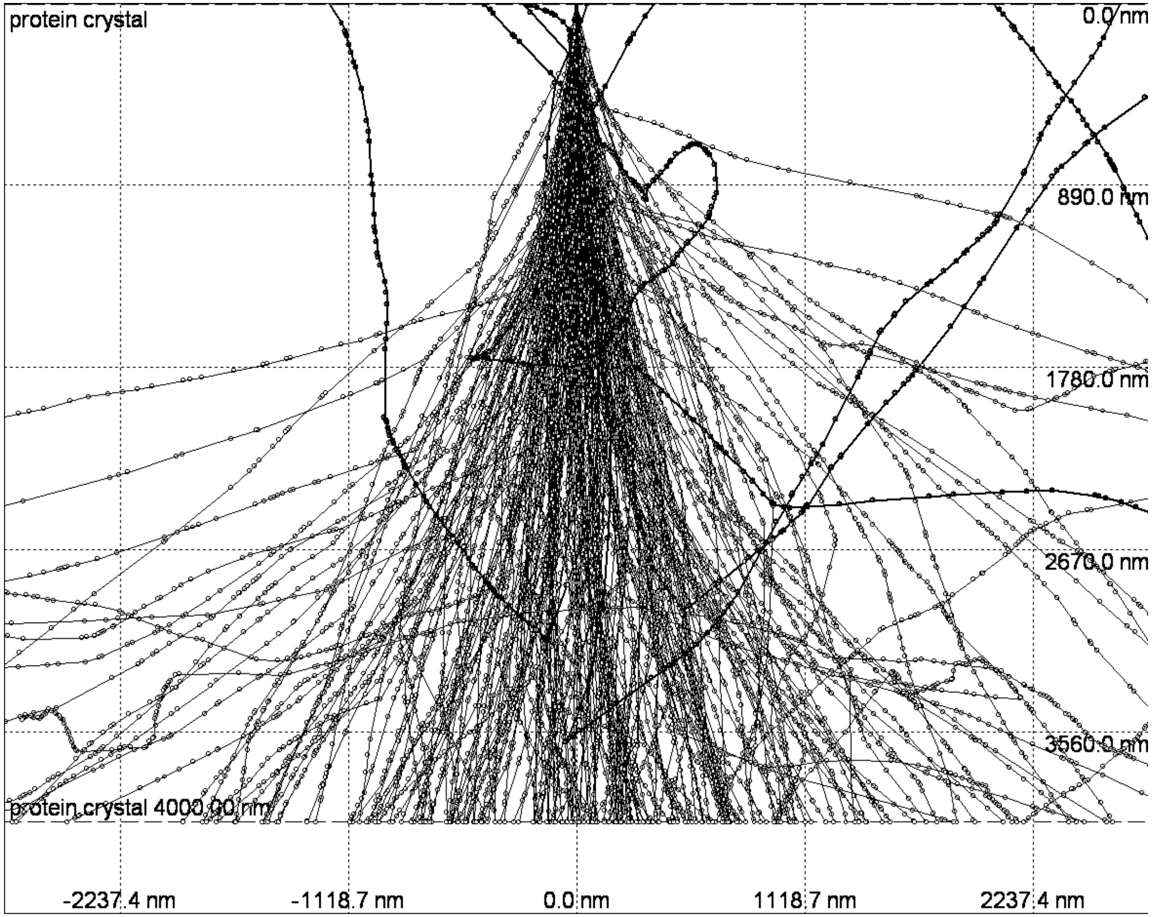


Figure 3. A simulation of tracks, created by electrons of 30 keV energy inside a protein crystal, calculated using the Monte-Carlo program Casino.

From: Colin Nave & Mark A. Hill, JSR (2005), 12, 299-303

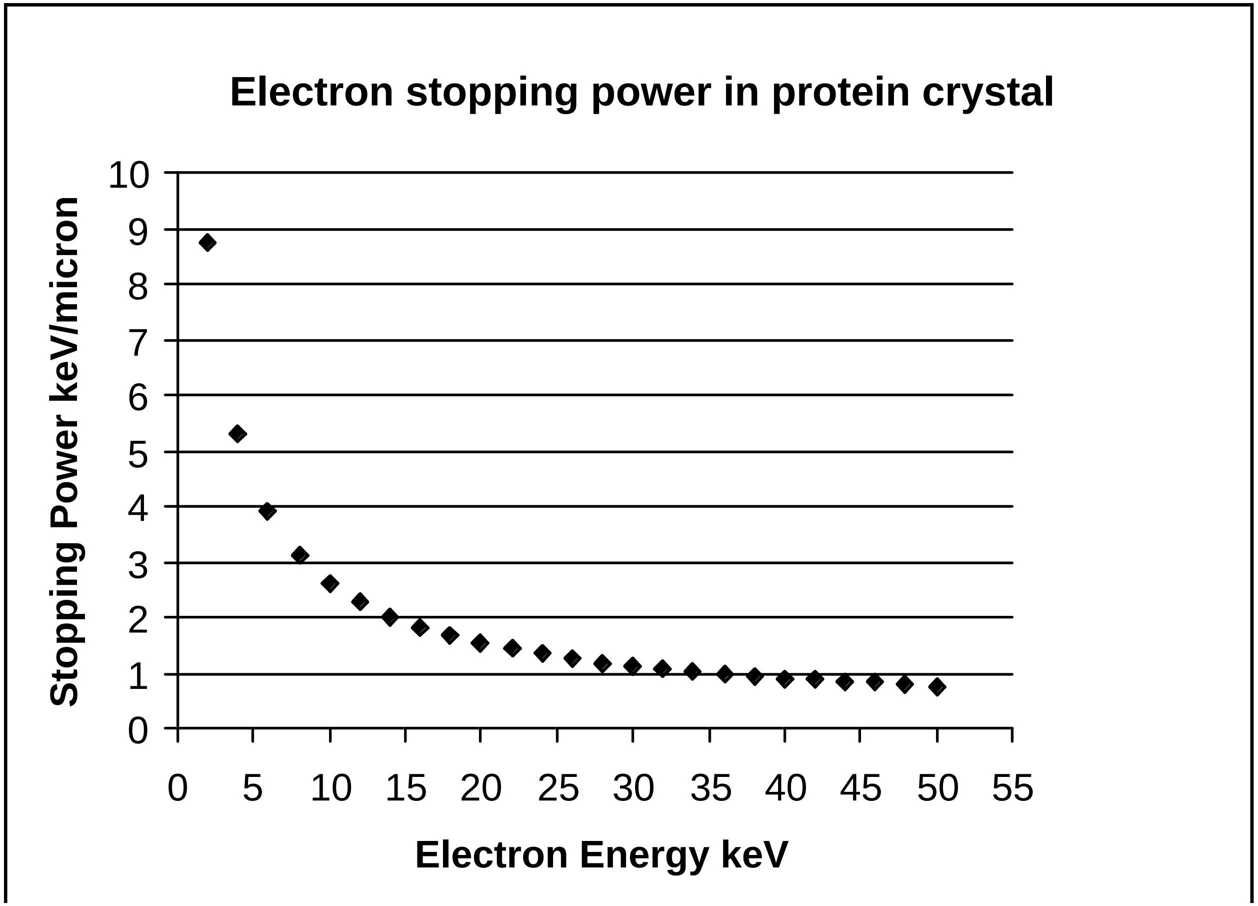
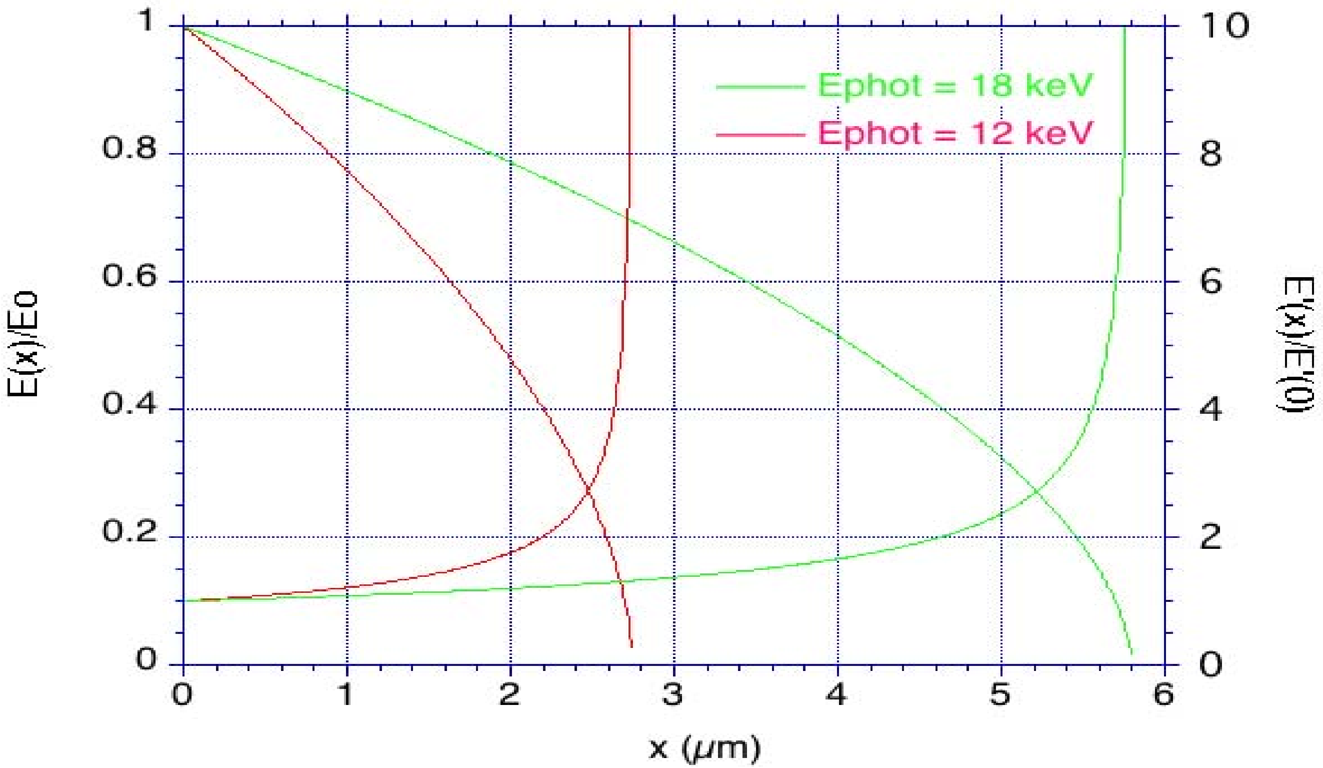


Figure 1. The electron stopping power as a function of electron energy inside a protein crystal of density 1.17g/cm³.

Energy Loss of Photo-Electrons and Deposited Dose vs. Distance Traveled

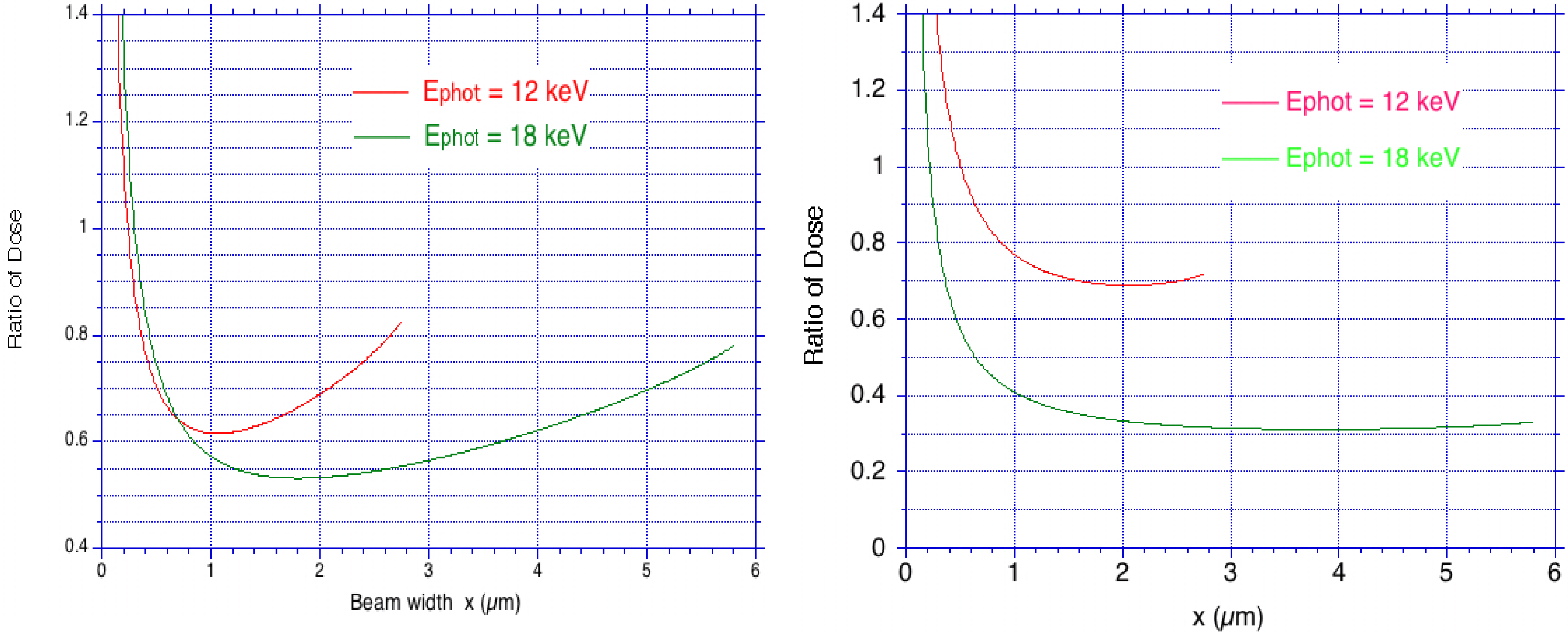


Ratio of energy E(x) of photo-electron at distance x from starting point to initial energy Eo (left scale).

Ratio of energy loss dE/dx of photo-electron at distance x from starting point to energy loss at x = 0 (right scale)

Average binding energy = 600 eV

Reduction of Dose to Exposed Crystal Volume vs. Beam Width



Ratio of Dose to Exposed Crystal Volume with Beam of Width x vs. Uniform Illumination

Left panel: Exposure of width x and no exposure of damaged volumes either side vs. uniform illumination. Exposure adjusted for equal integrated intensities. Average binding energy = 600 eV

Right panel: Exposure of width x vs. uniform illumination of microcrystal of 5 μm size. Exposure adjusted for equal integrated intensities. Average binding energy = 600 eV

Need for Microbeam

- Need beam of 1 - 2 µm horizontal width at 18 keV photon energy
- Vertical beam size can be larger:
  - is not the predominant direction of photo-electrons
  - due to rotation around horizontal axis, exposed volume is larger than beam size
- Source size is large (650 µm FWHM) in direction of polarization (horizontal, only 20 µm FWHM in vertical direction)
- Large horizontal demagnification required:
  - two step demagnification:
    (1) normal demagnifying beamline optics
    (2) focusing on horizontal defining slits = virtual source
    (3) re-imaging virtual source on sample

Vertically Offset Beamline
(Outboard Beam)

- Optical design:

  Horizontally (vertically after 45° mirror): two stage demagnification
  - imaging source on slits with focusing 2nd crystal, 8.35:1 demag
  - reducing effective horizontal source size with slits, ~4x
  - re-imaging virtual source with horizontally focusing mirror, 8.25:1 demag, 500 mm long bimorph, 1.46 m from sample

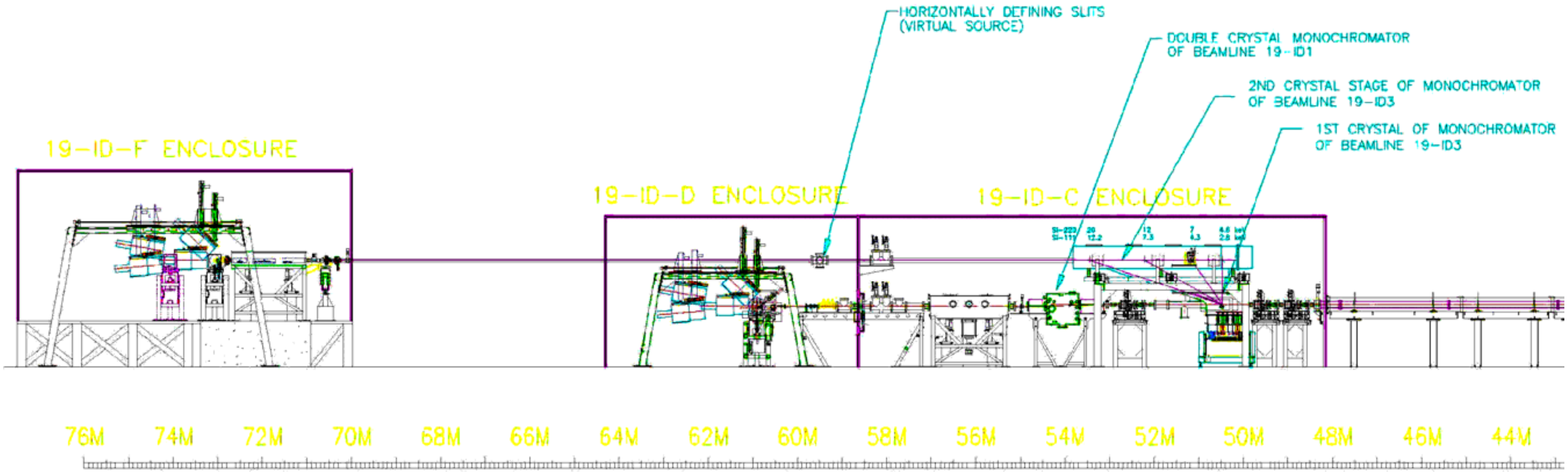
  Flat mirror at 45° with horizontal plane flips horizontal and vertical virtual source, 500 mm long, 0.93 m from sample

  Vertically (horizontally after 45° mirror): one stage demagnification
  - horizontally focusing mirror, 300 mm long bimorph, ~ 500 mm from sample

- Endstation instrumentation:

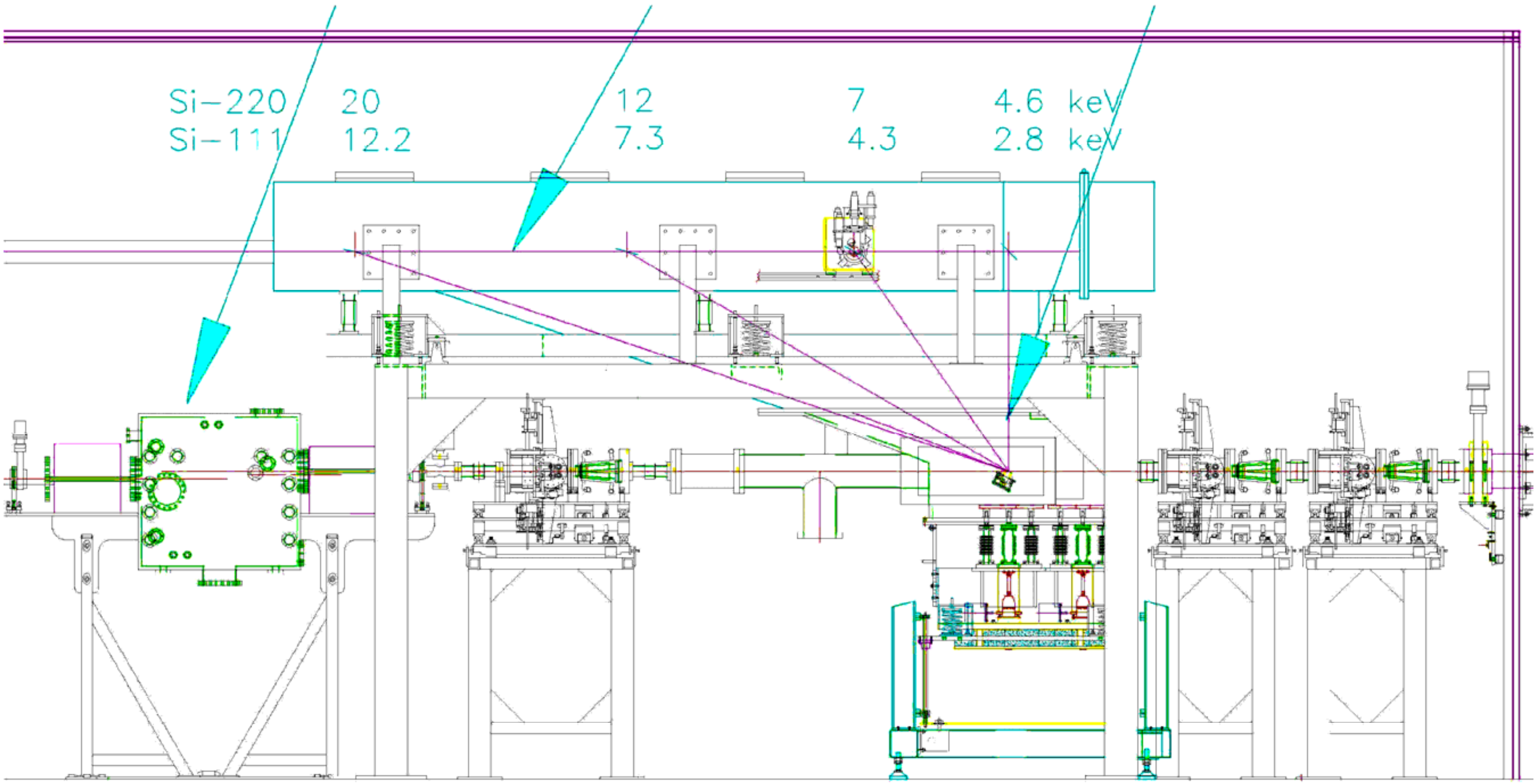
  Single axis goniostat, sub-micrometer runout
  On-axis, high magnification sample alignment microscope
  Standard detector positioner

Three Undulator Beamlines Upgrade
Overview Elevation



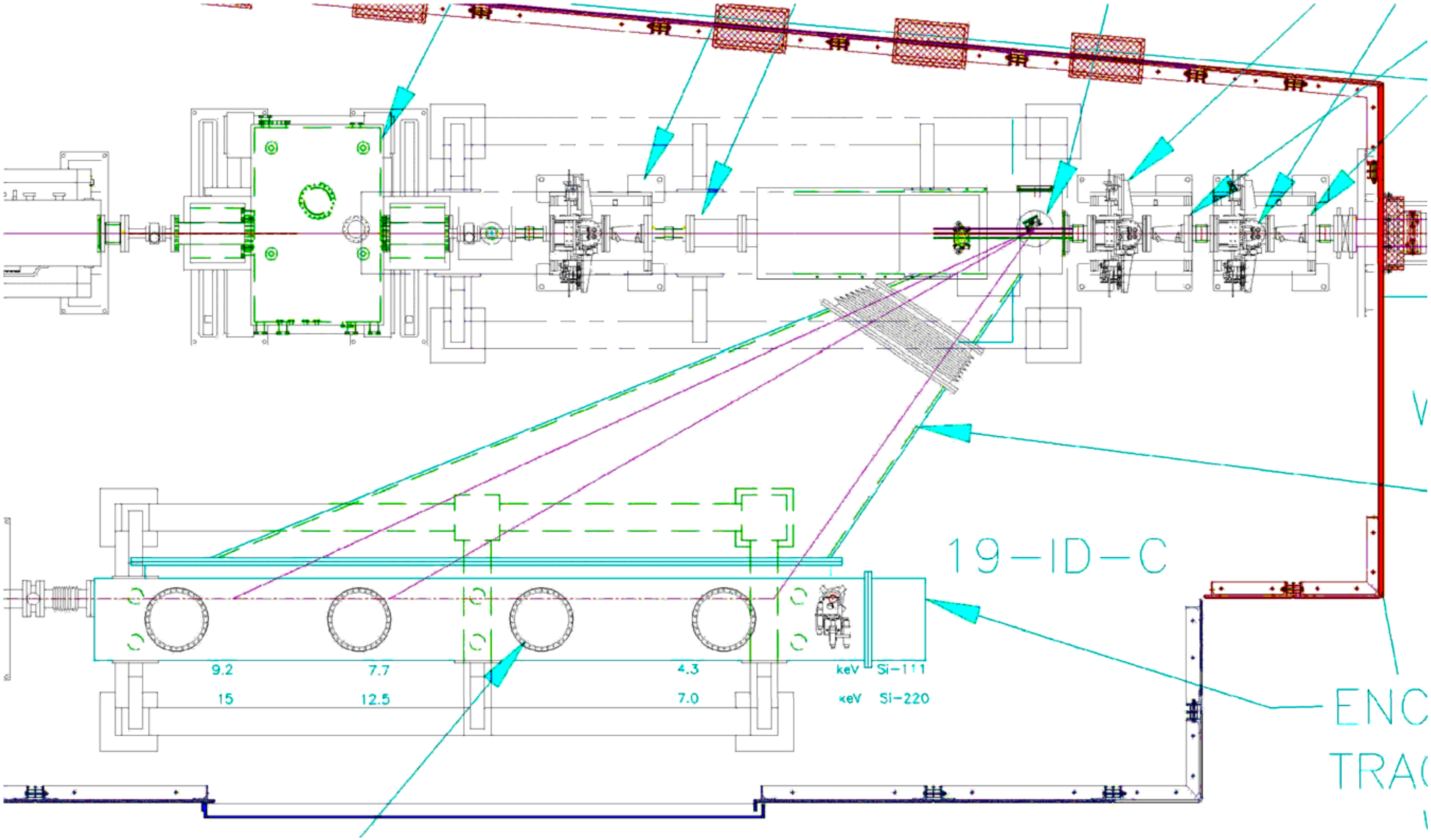
Three Undulator Beamlines Upgrade

Vertically Deflecting Monochromator



Three Undulator Beamlines Upgrade

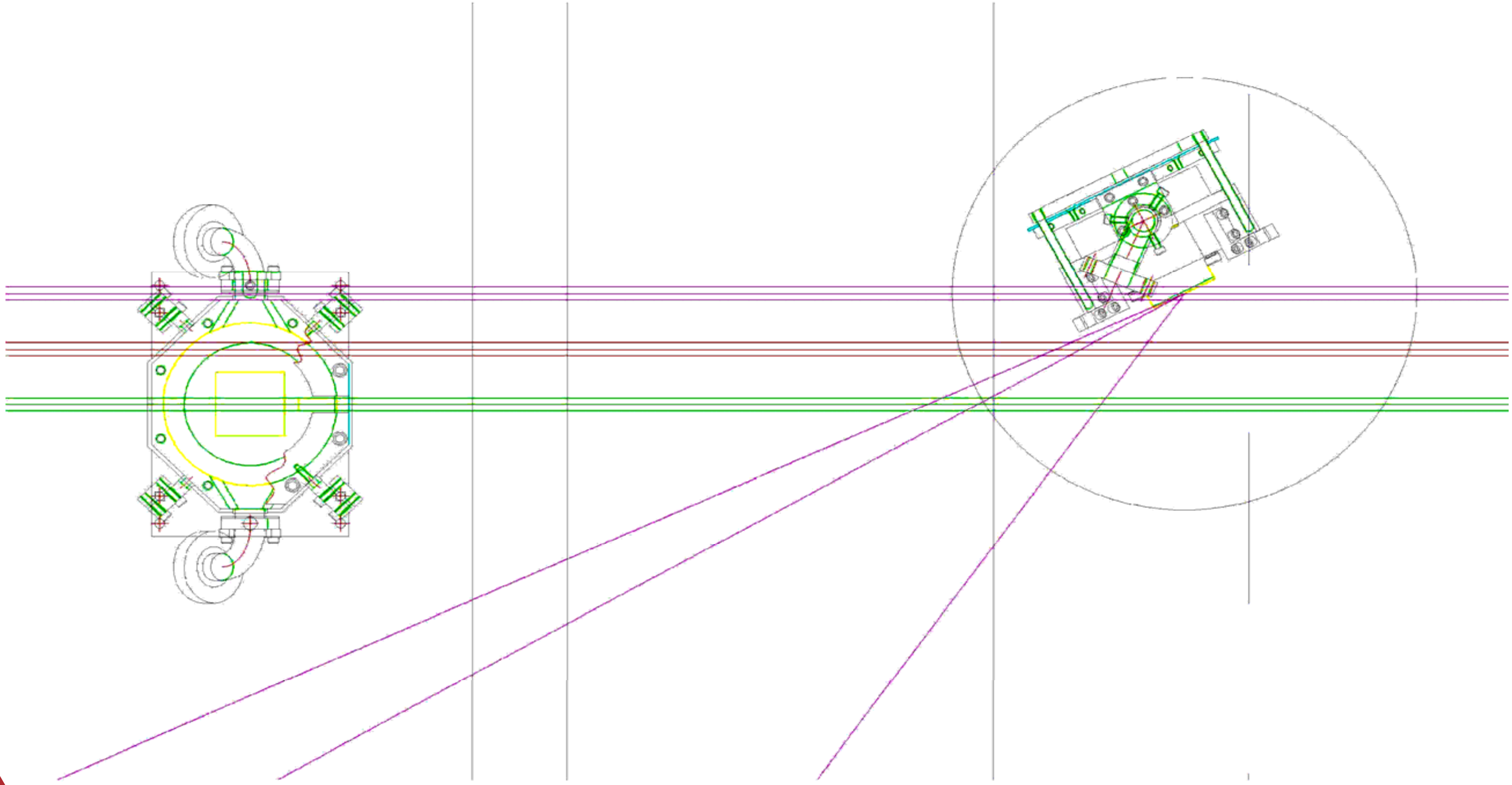
Horizontally Deflecting Monochromator



Three Undulator Beamlines Upgrade

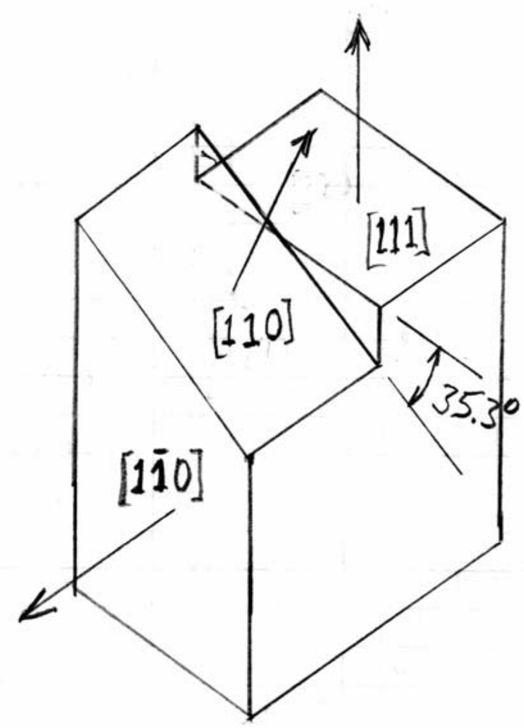
Three Undulator Beams and First Monochromator Crystals

Vertically Deflecting | *Horizontally Deflecting*



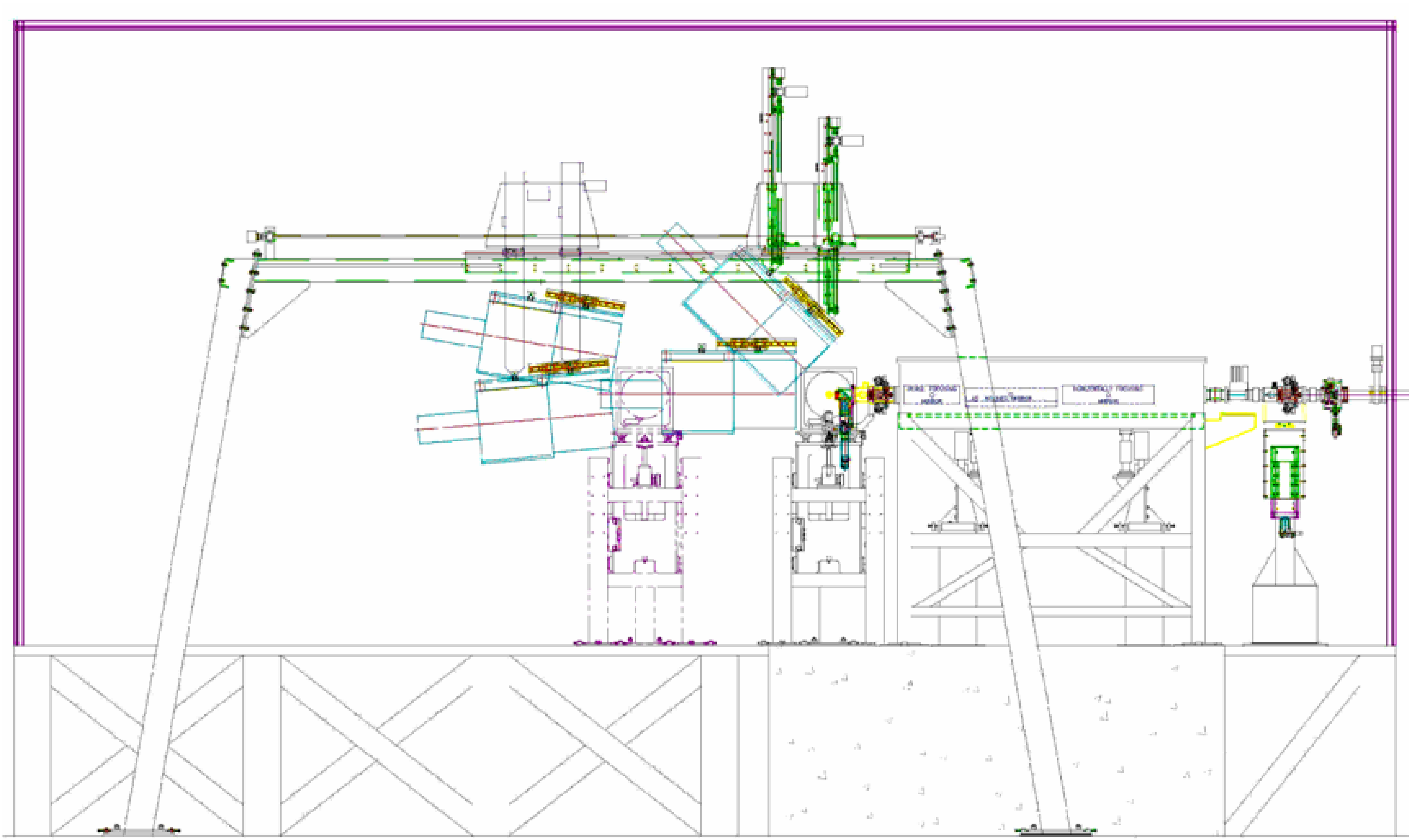
Dual Cut First Monochromator Crystal (Liquid Nitrogen Cooled for ID)

- Change of energy range by lateral translation of crystal
- No warm-up of cryo-cooled crystal and change of LN2 connections
- Extends energy coverage for limited range of Bragg-angles
- Requires robotic changer of focusing second crystal

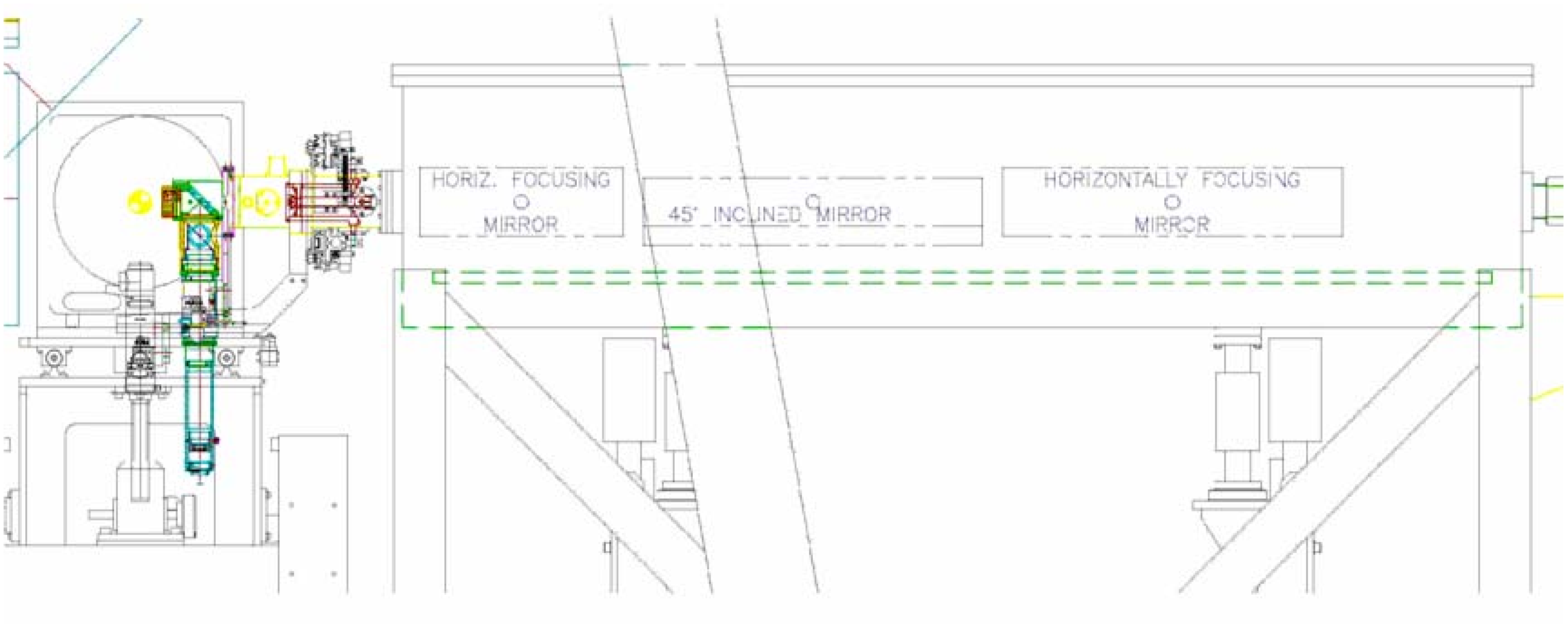


Vertically Offset Beamline

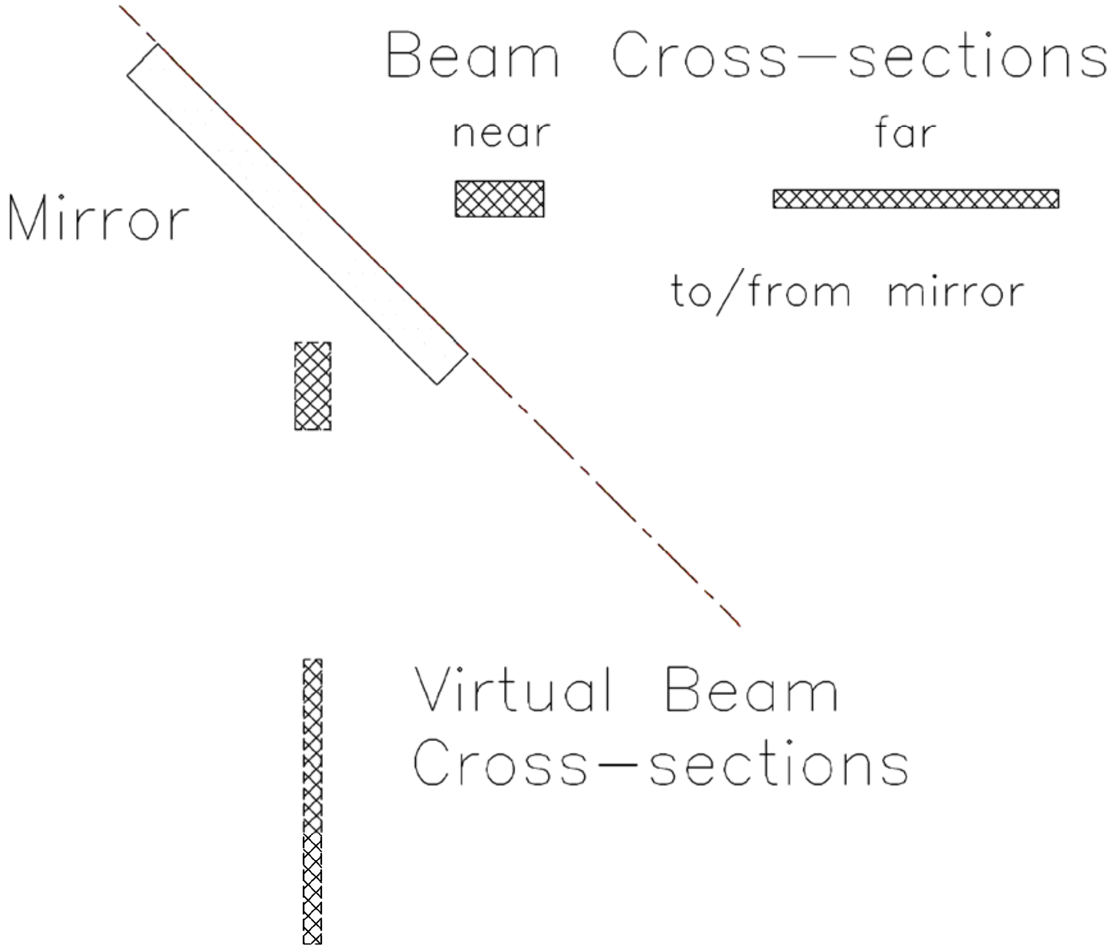
Endstation



Vertically Offset Beamline
Focusing Mirrors and Sample Stage

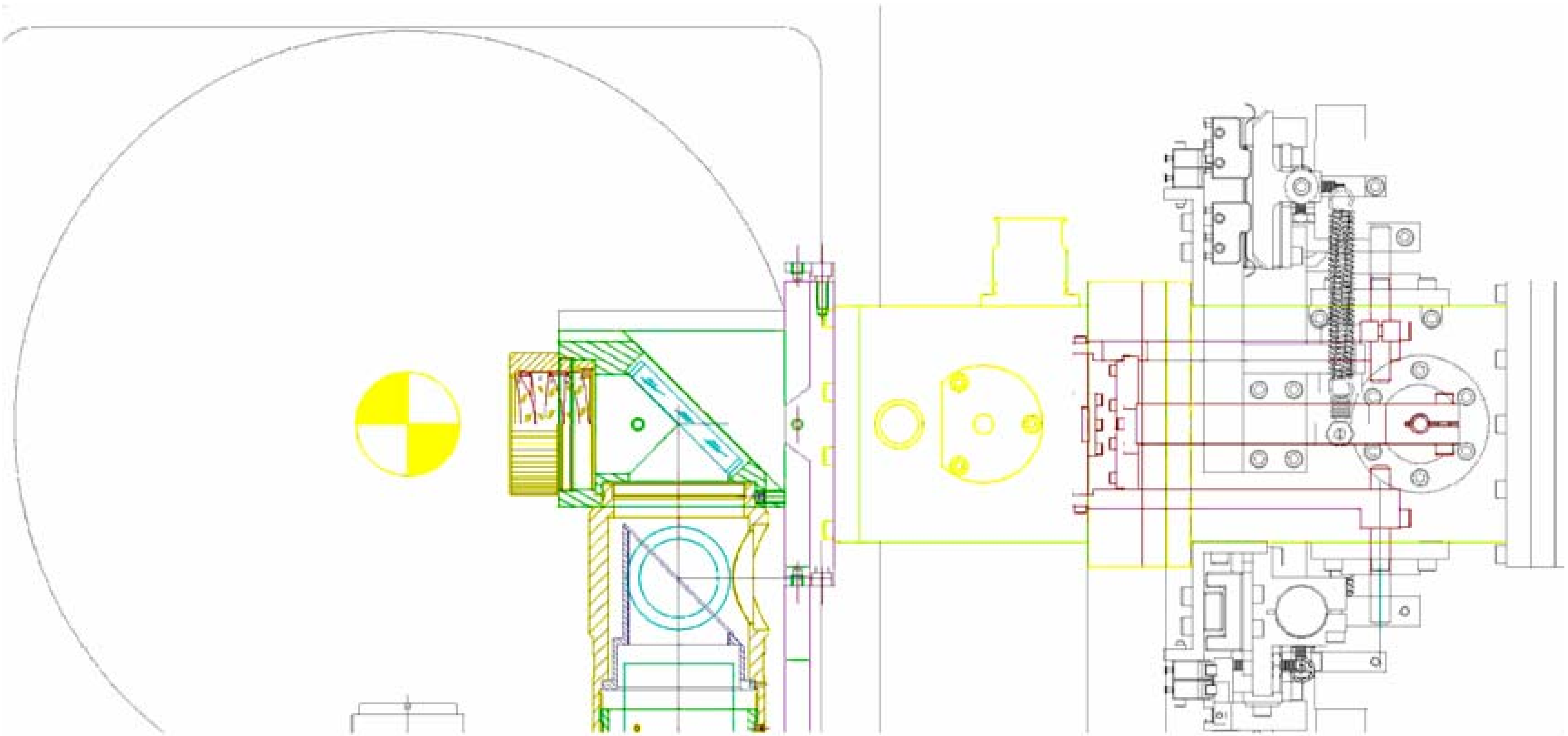


Rotation of Beam Cross-Section by 45° Mirror



Vertically Offset Beamline

Sample Area



Size and Flux Estimates of Microbeam

• Imaging horizontal source on slits	
- source to 2nd crystal at 18 keV (Si-220)	53.30 m
- 2nd crystal to slits	6.38 m
- demagnification	8.35:1
- width of horizontal focus	78 µm FWHM
• Imaging virtual source (slits) on sample	
- slits to focusing mirror	12.04 m
- focusing mirror to sample	1.46 m
- demagnification	8.25:1
• Size of image of slits at sample	
- aberration free image of 20 µm wide slits	2.4 µm
- bimorph allows to shape mirror to required elliptical figure	
- aberrations determined by residual surface figure error	
- Gaussian figure error of 0.3 µrad rms creates beam size of	2.1 µm FWHM
- convoluted size of image of 20 µm slits	3.2 µm FWHM
of 10 µm slits	2.4 µm FWHM

Size and Flux Estimates of Microbeam

- Imaging vertical source on sample
  - source to focusing mirror 72.68 m
  - focusing mirror to sample 0.50 m
  - demagnification 145:1

- Size of image of vertical source at sample
  - aberration free image 0.14 μm FWHM
  - bimorph allows to shape mirror to required elliptical figure
  - aberrations determined by residual surface figure error
  - Gaussian figure error of 0.3 μrad rms creates beam size of 0.7 μm FWHM
  - convoluted size of image 0.7 μm FWHM

Size and Flux Estimates of Microbeam

Flux at sample

- Loss at slits

- fraction of 78 µm wide beam passing 20 µm slits	0.238
10 µm slits	0.121

- Angular acceptance of mirror imaging slits

- mirror length 500 mm, effective	400 mm
- grazing angle	4 mrad
- distance to slits	12.04 m
- angular acceptance from slits	133 µrad
- angular acceptance from source at 8.35:1 demagnification	15.9 µrad

- Angular acceptance of 2nd focusing mirror

- mirror length 300 mm, effective	200 mm
- grazing angle	4 mrad
- distance to source	72.68 m
- angular acceptance from source	11.0 µrad

Size and Flux Estimates of Microbeam

• Flux from undulator	$2.37*10^{13}$ ph/s/0.1%BW
- 1m long (eff. 0.93 m), 3.1 cm magnetic period - at E_{phot} = 18 keV - into 15.9 µrad horizontal and 11.0 µrad vertical angle	
• Passing through 20 µm slits	0.238
• Si-220 double crystal bandwidth	$\sim 4.6*10^{-5}$
• Losses in windows, other	0.8
• Estimate of flux on sample	$2.1*10^{11}$ ph/s
• Estimate of flux density at sample	$9*10^{16}$ ph/s/mm^2
• Convergence angles of flux on sample	
- horizontal	1.1 mrad
- vertical	1.6 mrad
• Exposure time to reach dose limit $D_{1/2} = 4.3*10^7$ Gy	2 seconds

Microfocus Beamline After APS Upgrade

- Optical design:
  - basically the same
  - need maximum beamline length for high demagnification
  -but can be simplified:

  Horizontally: one stage demagnification
  horizontally focusing mirror, 300 mm long bimorph, ~ 500 mm from sample

  Vertically: one stage demagnification
  vertically focusing mirror, 400 mm long bimorph, ~ 900 mm from sample

- Endstation instrumentation: same

Size and Flux Estimates of Microbeam After APS Upgrade

- APS Upgrade parameters (Case: small size):
  σ_x= 35 μm, $\sigma_{x'}$= 22 μrad, σ_y= 7.3 μm, $\sigma_{y'}$= 1.1 μrad
  2.1 m long undulator, E_{phot}= 18 keV: σ_r= 1.3 μm, $\sigma_{r'}$= 4.1 μrad
  Σ_x= 35 μm, $\Sigma_{x'}$= 22.4 μrad, Σ_y= 7.4 μm, $\Sigma_{y'}$= 4.2 μrad
- Imaging horizontal source on sample
  - source to focusing mirror 72.68 m
  - focusing mirror to sample 0.50 m
  - demagnification 145:1
- Size of image of horizontal source at sample
  - aberration free image 0.57 μm FWHM
  - bimorph allows to shape mirror to required elliptical figure
  - aberrations determined by residual surface figure error
  - Gaussian figure error of 0.3 μrad rms creates beam size of 0.7 μm FWHM
  - convoluted size of image 0.9 μm FWHM

Size and Flux Estimates of Microbeam After APS Upgrade

- APS Upgrade parameters (Case: small size):
  σ_x= 35 µm, $\sigma_{x'}$= 22 µrad, σ_y= 7.3 µm, $\sigma_{y'}$= 1.1 µrad
  2.1 m long undulator, E_{phot}= 18 keV: σ_r= 1.3 µm, $\sigma_{r'}$= 4.1 µrad
  Σ_x= 35 µm, $\Sigma_{x'}$= 22.4 µrad, Σ_y= 7.4 µm, $\Sigma_{y'}$= 4.2 µrad
- Imaging vertical source on sample

- source to focusing mirror	72.28 m
- focusing mirror to sample	0.80 m
- demagnification	90:1

- Size of image of vertical source at sample

- aberration free image	0.19 µm FWHM
- bimorph allows to shape mirror to required elliptical figure	
- aberrations determined by residual surface figure error	
- Gaussian figure error of 0.3 µrad rms creates beam size of	1.1 µm FWHM
- convoluted size of image	1.1 µm FWHM

Size and Flux Estimates of Microbeam After APS Upgrade

Flux at sample

- Angular acceptance of horizontally focusing mirror

- mirror length 300 mm, effective	200 mm
- grazing angle	4 mrad
- distance to source	72.68 m
- angular acceptance from source	11.0 μrad
- angular divergence from source (FWHM)	52.6 μrad

Angular acceptance of vertically focusing mirror

- mirror length 400 mm, effective	300 mm
- grazing angle	4 mrad
- distance to source	72.28 m
- angular acceptance from source	16.6 μrad
- angular divergance from source (FWHM)	9.9 μrad

Size and Flux Estimates of Microbeam After APS Upgrade

• Flux from undulator - 200 mA beam current - 2.1m long (eff. 2.05 m), 3.1 cm magnetic period - at E_{phot} = 18 keV - into 11.0 µrad horizontal and 16.6 µrad vertical angle	$8.1*10^{13}$ ph/s/0.1%BW
• Si-220 double crystal bandwidth	$\sim 4.6*10^{-5}$
• Losses in windows, other	0.8
• Estimate of flux on sample	$3.0*10^{12}$ ph/s
• Estimate of flux density at sample	$3*10^{18}$ ph/s/mm^2
• Convergence angles of flux on sample - horizontal - vertical	 1.6 mrad 0.9 mrad
• Exposure time to reach dose limit $D_{1/2} = 4.3*10^7$ Gy	0.06 seconds

Gain with APS Upgrade for Microcrystal Macromolecular Crystallography

- Optical design: easier, less components
- Focal size: horizontally ~1/2
- Flux: 14-times ($3*10^{12}$ ph/s)
- Flux density: 30-times ($3*10^{18}$ ph/s/mm^2)
- Sample life: 0.06 seconds instead of 2 seconds
- Increase in samples measured / hour: none
- Gain for microcrystal macromol. xtallography: little
- Gain worth 2 years shutdown: **NO**

Vibration Reduction and Beam Stabilization

- Vibration isolation base
  - heavy platform on springs
  - supports optical elements
  - reduces higher frequencies more than 40x
  - low natural frequencies (<5 Hz)

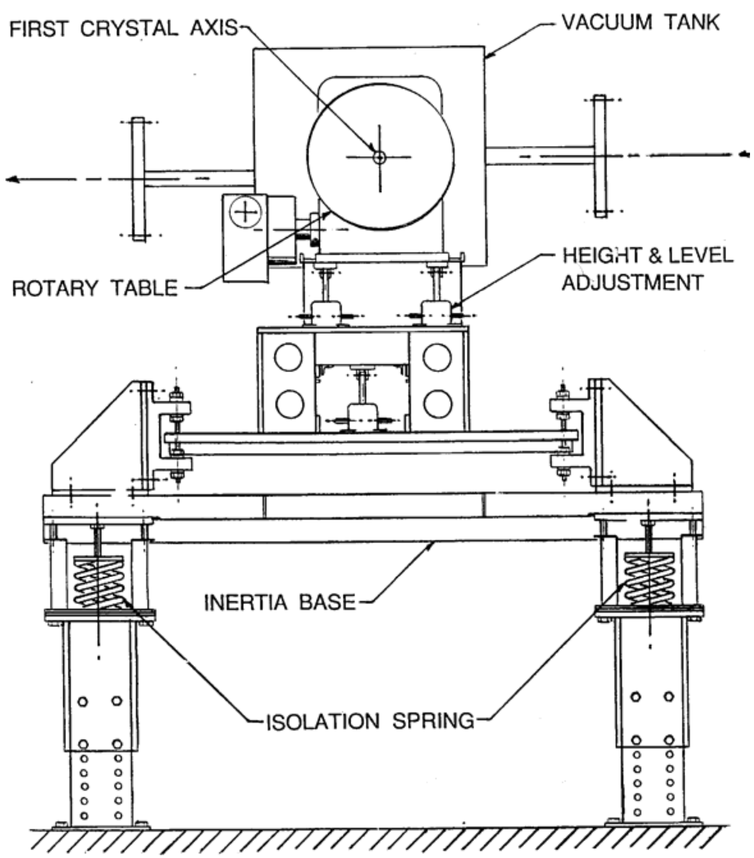
- Stabilization of vibration isolated platform
  - position sensors (platform vs. support frame)
    (x-ray BPM design with laser, 0.5 µm sensitivity)
  - angle sensors (laser beam reflected from mirror on platform)
  - feedback to linear motors after low pass filter

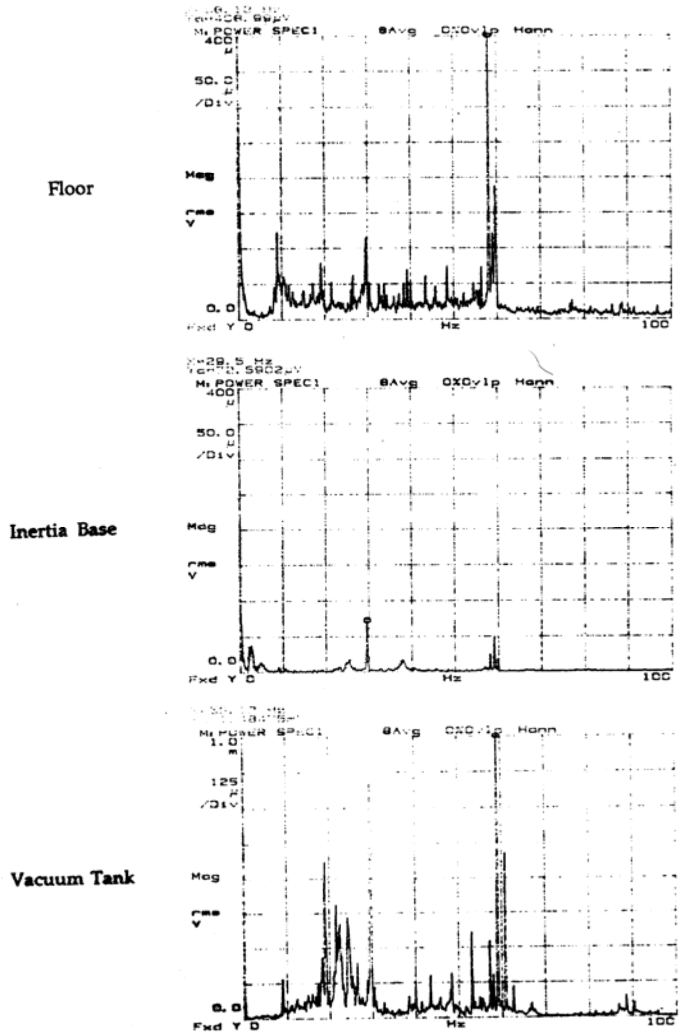
Vibration Reduction and Beam Stabilization

Stabilization of x-ray beam:

- X-ray tracking
  - white beam position monitor (hard x-ray BPM)
  - x-ray BPM before horizontally defining slits
  - x-ray BPM before mirrors
  - x-ray BPM before sample
- Optical tracking
  - laser beam parallel to x-ray beam path
  - mirrors on first and second crystal rotation axis
  - laser beam BPM before horizontally defining slits

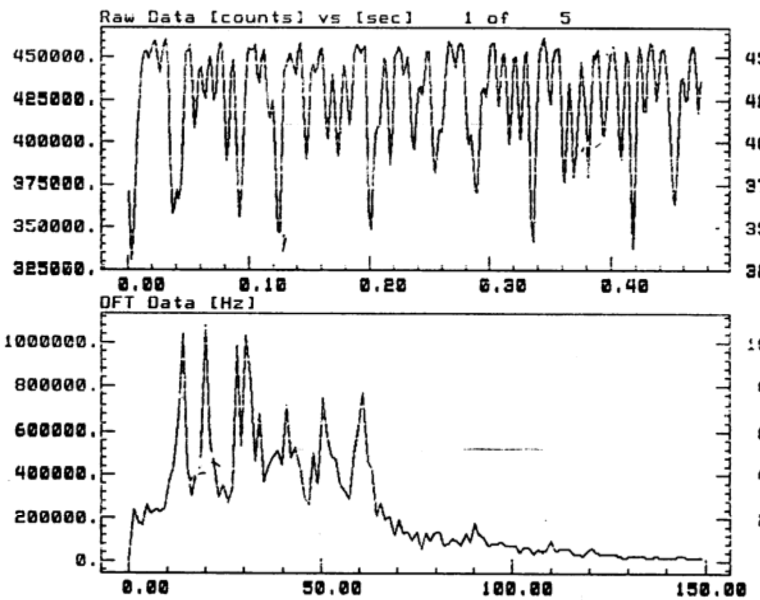
Vibration Isolation Base





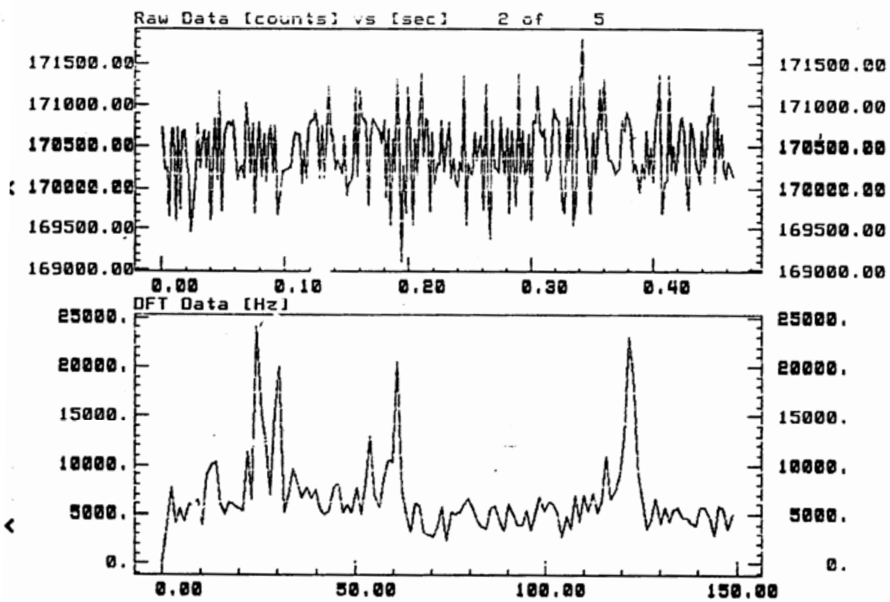
Vibration Isolation Base

Noise Spectrum without Vibration Isolation
Si-220 Crystal, Fully Tuned



Top: real time, 1 scan
Bottom: Fourier transform, average of 5 scans

Noise Spectrum with Vibration Isolation
Si-220 Crystal, Fully Tuned

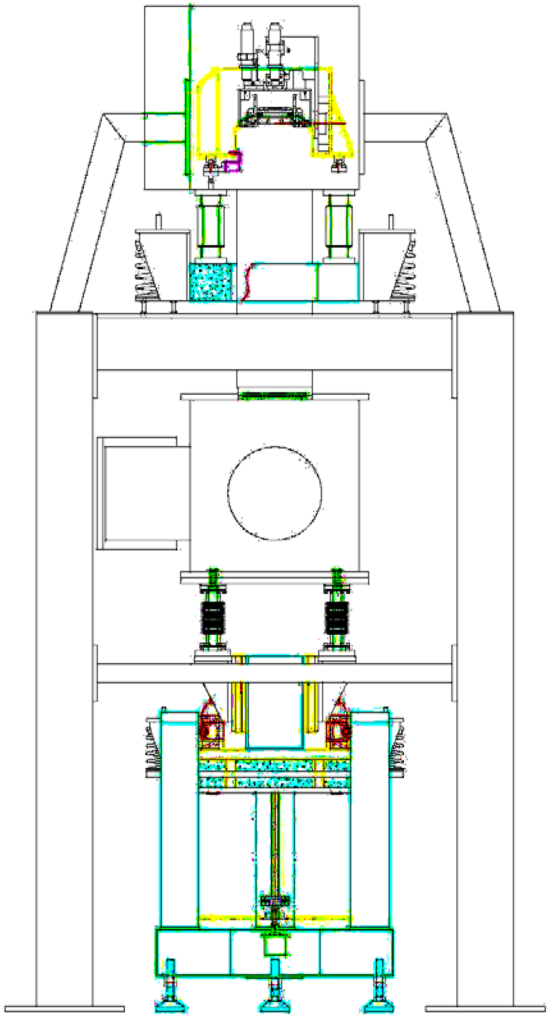


Top: real time, 1 scan
Bottom: Fourier transform, average of 5 scans

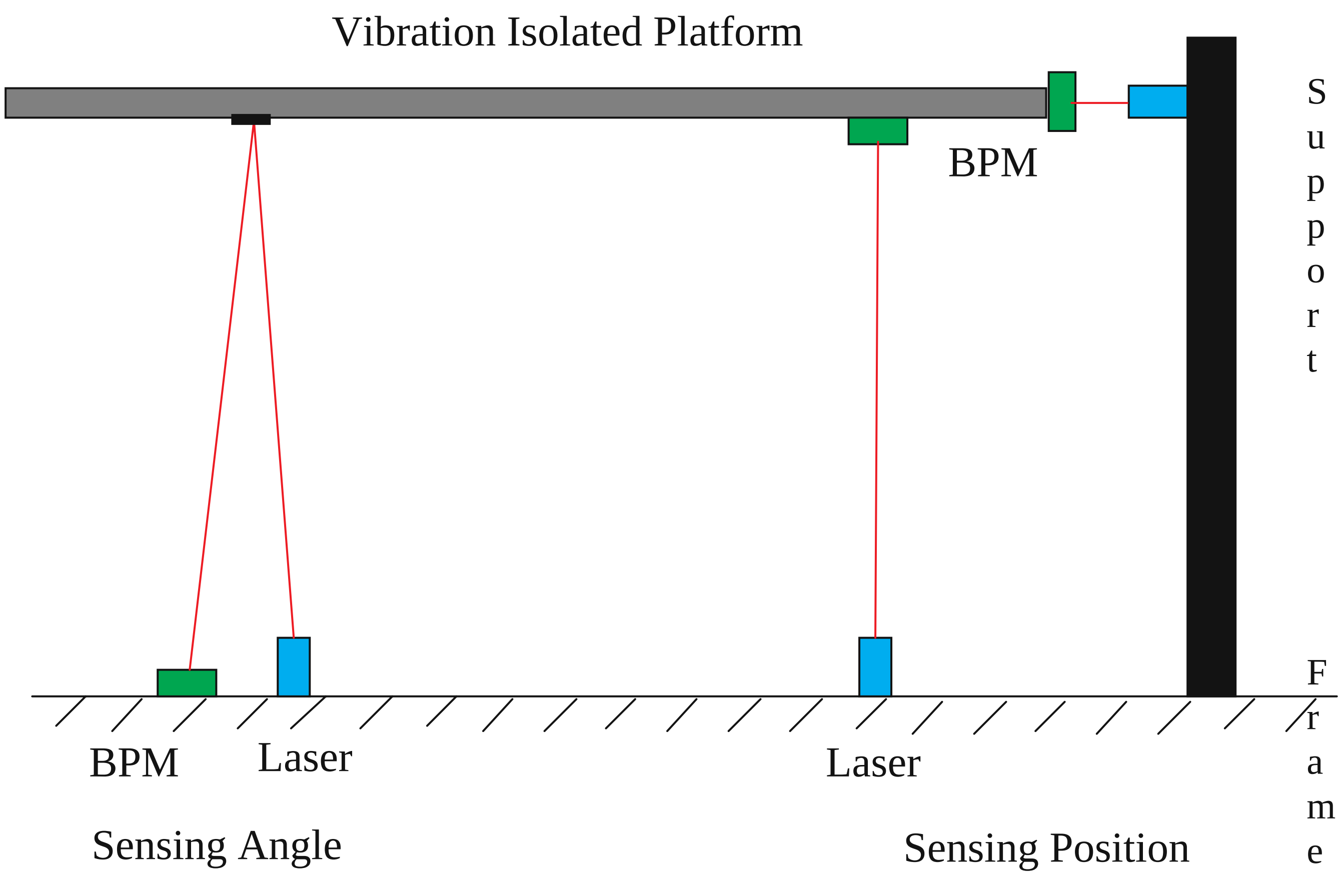
The vibration isolation system reduces the amplitudes of the vibration spectrum by a factor 40.

Three Undulator Beamlines Upgrade

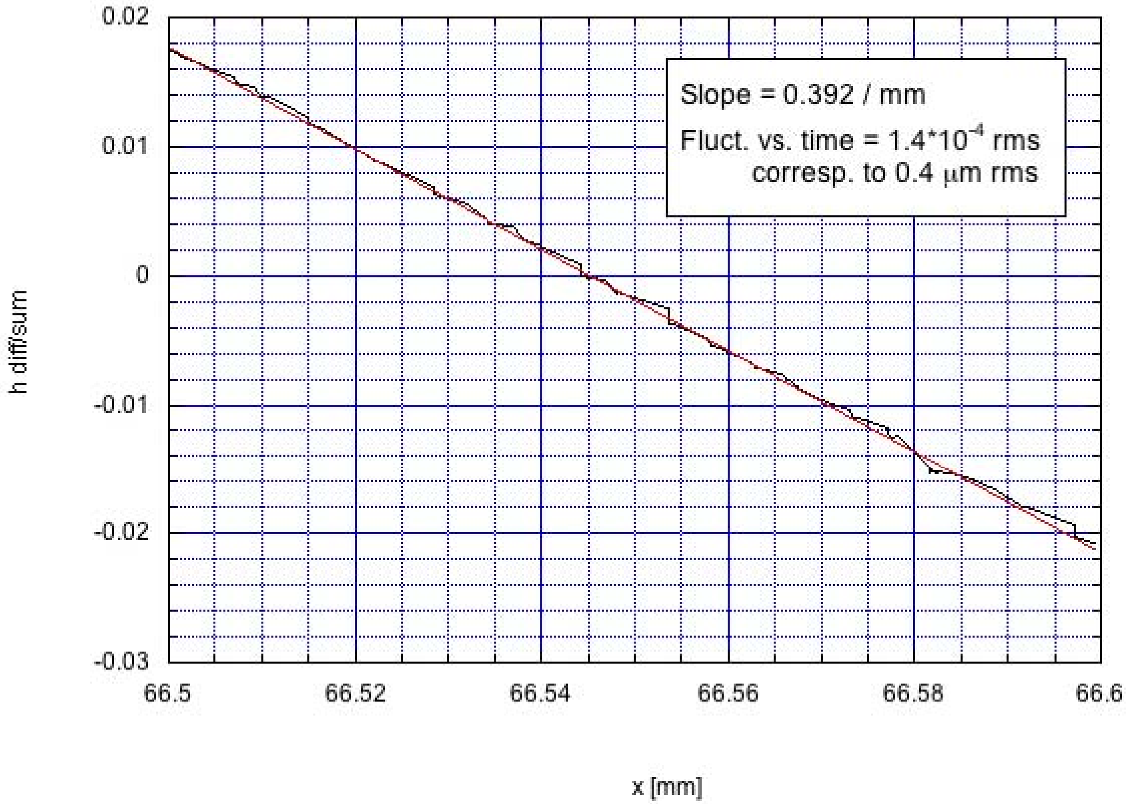
Vertically Offset Beamline, View in Beam Direction



Sensing Angle and Position of the Vibration Isolated Platform



Quad PIN Diode BPM Position Sensing



Laser BPM Signal For 0.1 mm Horizontal Scan

Construction Time Line

FY 2006	upgrade BM beamline start upgrade of center ID beamline
FY 2007	finish upgrade of center ID beamline start design & construction of triple ID beamlines
FY 2008	start modification of hutch 19-ID-C and 19-ID-D start construction of additional hutches
FY 2009	rebuilt center beamline 19-ID1 recommission 19-ID1 finish construction of triple ID beamlines
Summer 2010	commission new ID beamlines
Fall 2010	start user operation of new ID beamlines

Front End and Undulator Construction Timeline

1. design modification of 2.1 m undulator for 3.1 cm period	1 month
2. fabrication of 2.1 m undulator with 3.1 cm period	6 months
3. design of 1 m undulator with 3.1 cm period	6 months
4. fabrication of two 1 m undulators with 3.1 cm period	12 months
5. design of triple undulators vacuum chamber	3 months
6. fabrication of triple undulator vacuum chamber	6 months
7. fabrication of two sets of deflector and steering magnets	6 months
8. fabrication of additional RF BPMs and electronics	3 months
9. design modification dual to triple undulator front end	3 months
10. fabrication of triple undulator front end	6 months
11. installation of 2.1 m undulator and new vacuum chamber	during 1 month shut down
12. installation of new front end	during 1 month shut down
13. installation of two 1 m undulators	during 1 month shut down