First Results from a Dispersive EXAFS beamline at Indus-2 SR facility

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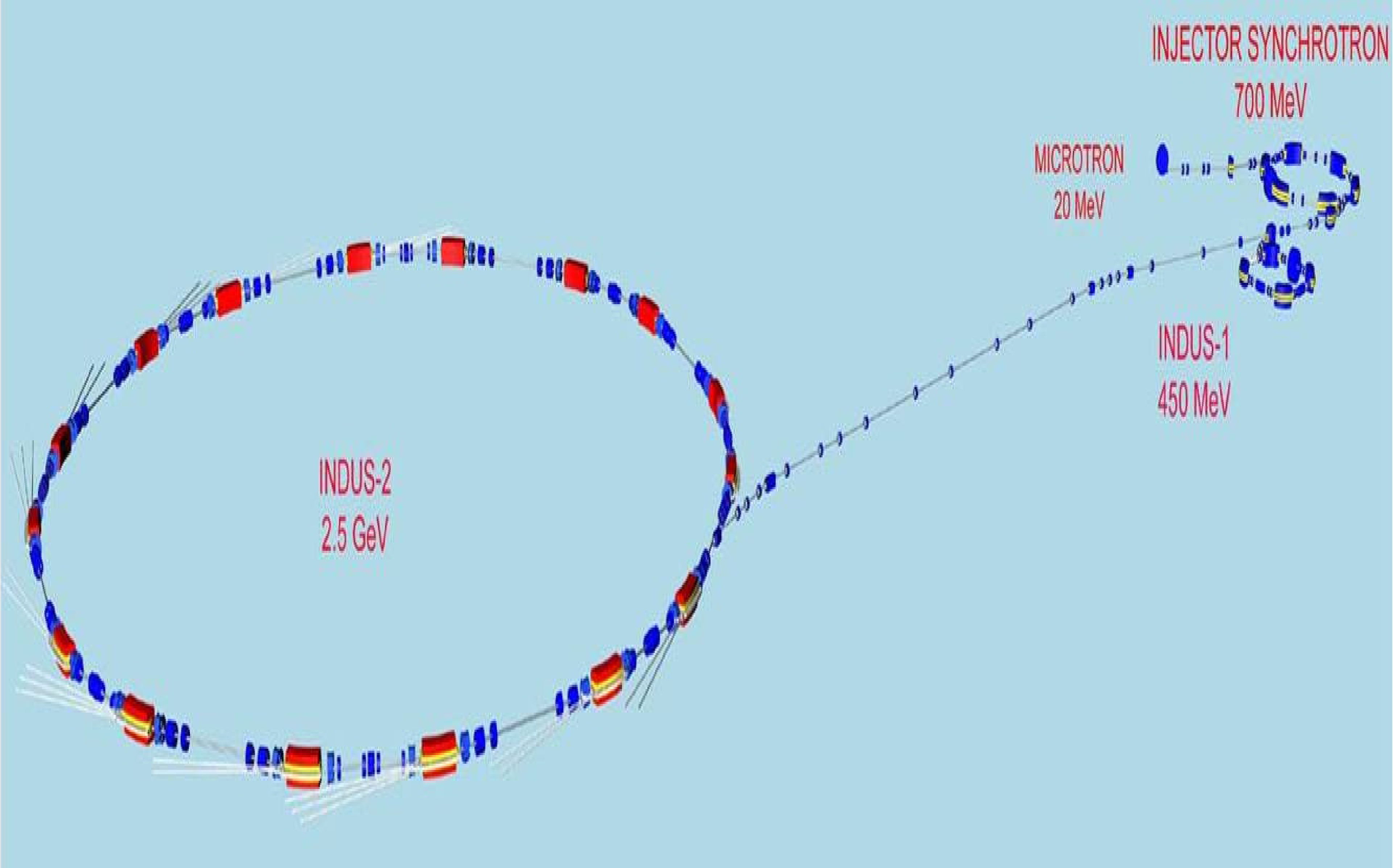
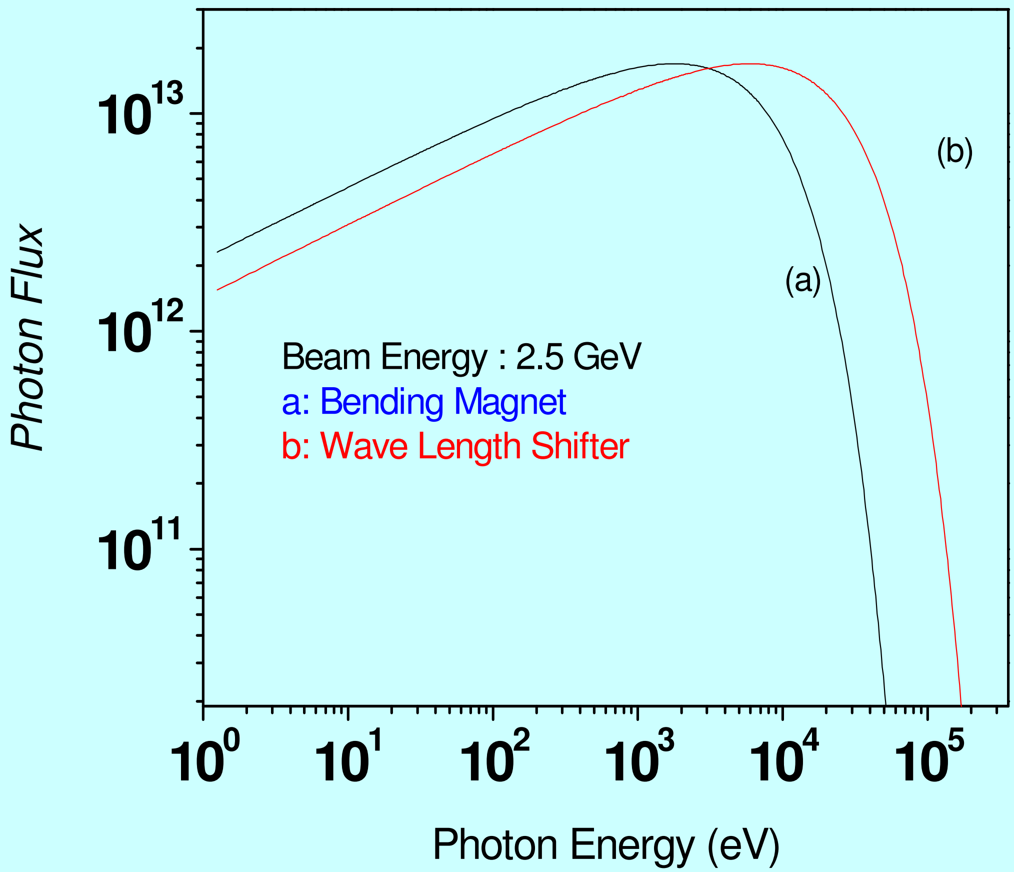
Outline

- **Introduction to Indus SR**
- **Beamline description**
  - **Basic working principle**
  - **Optical layout**
  - **Design procedure**
  - **Mechanical Layout**
  - **Experimental station**
- **Commissioning results**

Schematic view of Indus complex



Indus-2 synchrotron spectrum



Indus-2 Parameter

Energy : 2.5 GeV

Current : 300mA

Field : 1.5 T (BM)

Circumference:172.5m

Lifetime: 15 Hrs

λ_c =1.98 Å (6.23 KeV)

Beam size

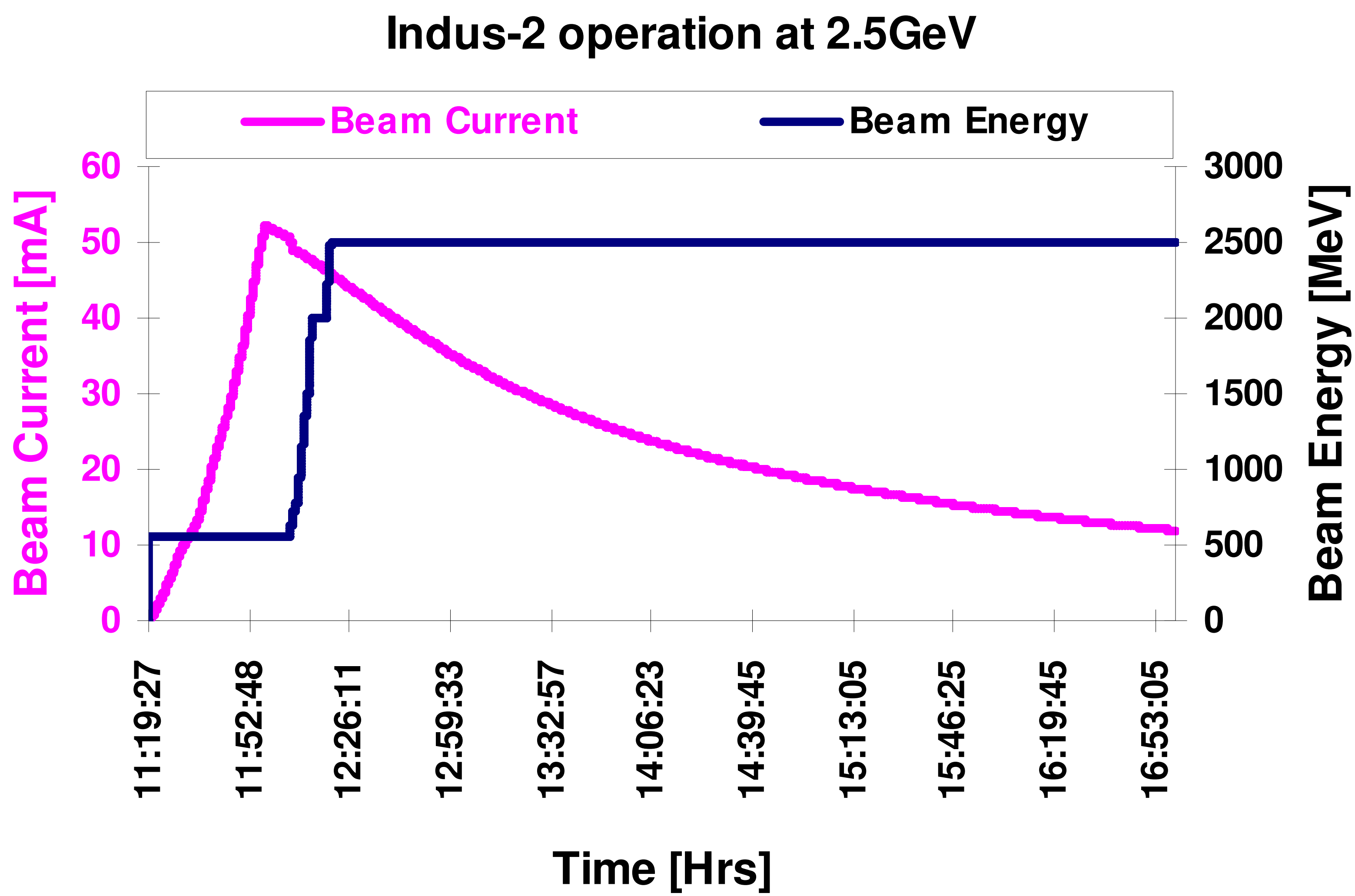
σx : 0.234mm

σy : 0.237 mm

RF frequency

:505.812 MHz

Status of Indus-2



Beamline Layout view of Indus-2

37 BLs

09:EXAFS-scanning

14:PES

16:XRFμ

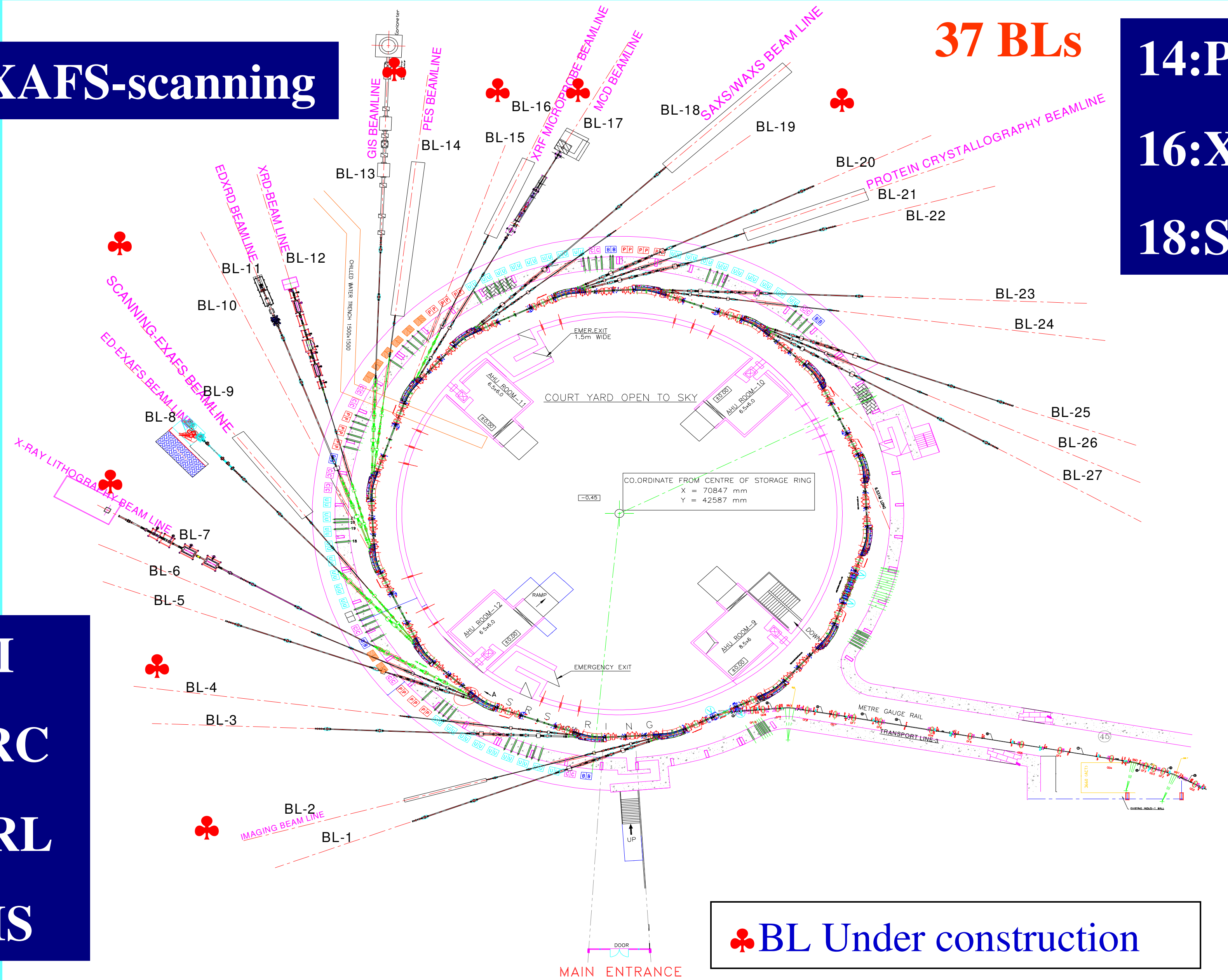
18:SAXS

02:MI

04:PRC

07:XRL

13:GIS

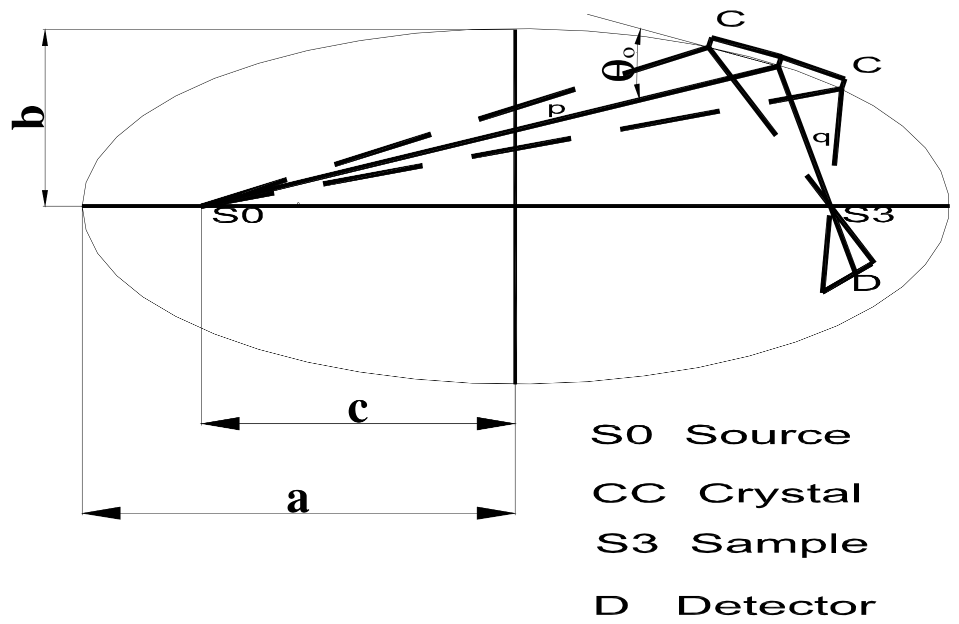


♣BL Under construction

Characteristics of ED XAFS BL at Indus-2

- Source: Bending magnet
- Energy range: 5-20keV
- Resolution: 10^{-4}
- Band pass: 300ev to 2000eV
- Flux: 10^{12} photons/sec/1000eV
- Polychromator: Si(111)
- Detector: CCD(2k x 2k ; pixel: 13.5μ)

Working Principle of EXAFS Beamline

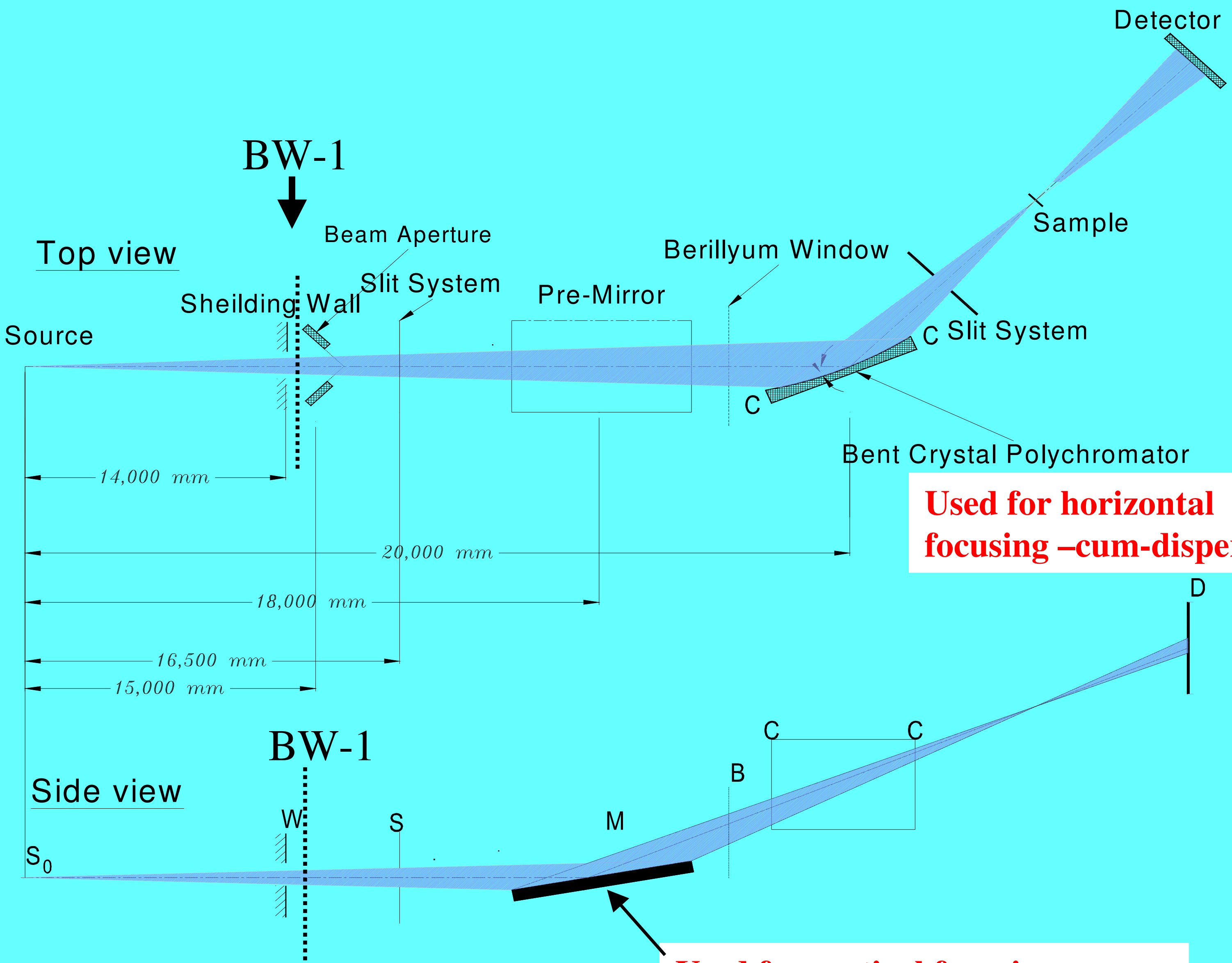


Energ y (eV)	θ_o (deg)	a (mm)	b (mm)	c (mm)
5,000	23.28	10285	1335.5	10198
10,000	11.40	10320	708.1	10296
20,000	5.67	10702	524	10689

Principle of Action:

- Synchrotron Radiation is diffracted and focused on sample by a perfect crystal, bent elliptically such that the sample and the source are at focii of the ellipse. Transmitted intensity is detected by a position sensitive CCD detector.

Optical Layout of EXAFS Beamline at INDUS-2



Used for horizontal focusing –cum-dispersion

Used for vertical focusing –cum-higher harmonic rejection

Design Procedure

- Fixed parameters
- Variable geometrical parameters
- Ray tracing : check the performance
- Fixing the specs of optical components
- Simulation of heat load on each optical components
- Mechanical specs of different subsystem
    - » Mirror mount, crystal bender et
- Simulation of radiation dose
- Fixing specs of Hutch

Ref.: N.C. Das, S.N. Jha,& D. Bhattacharyya, Proc. 8th International Conference on SRI, AIP conference proceedings, 705, 301(2003)

Fixed Parameters

Crystal type	**Si (111)**
2d Value	**6.2709 Å**
Source to Crystal Distance (p)	**20,000 mm**
Detector length (L)	**25 mm**
Horizontal Beam Divergence (U_m)	**1.5 mrad**
Vertical Beam Divergence	**0.2 mrad**

E_o → θ_o

$$Sin\theta_o = \frac{\lambda}{2d} = \frac{12.398}{2dE_o(keV)}$$

p, U_m, θ_o → l

$$U_m = (\frac{l}{p})Sin\theta_o$$

$l, \theta_o, \Delta E, p$ → R

$$\Delta E = E_o l Cot\theta_o(\frac{1}{R} - \frac{Sin\theta_o}{p})$$

R, p, θ_o → q

$$\frac{1}{Sin\theta_o} = \frac{R}{2}(\frac{1}{p} + \frac{1}{q})$$

l, q, θ_o → $U_{m'}$

$$U'_m = (\frac{l}{q})Sin\theta_o$$

$L, U_{m'}$

$$r = \frac{L}{U'_m}$$

P.M. Lee etal, Review Scientific Instrum, 65,1(1994)

Derived Parameters

Photon Energy E_o (eV)	5000	10,000	20,000
Band Pass ΔE (eV)	297	1123	2000
Bragg Angle (θ_o)	23.28°	11.40°	5.67°
Crystal length l (mm)	75.9	151.8	303.5
Crystal Radius R (mm)	2803	6287	26,550
Crystal to Sample distance q (mm)	570	641	1404
Sample to Detector Distance r (mm)	475	534	1170

Ray tracing result

	5 keV	10 keV	20 keV
Focal spot	20	40	50
Spatial resolution at detector	0.3eV/pixel	1.1eV/pixel	2.1eV/pixel

Optical components

- Polychromator

- Pre-mirror:

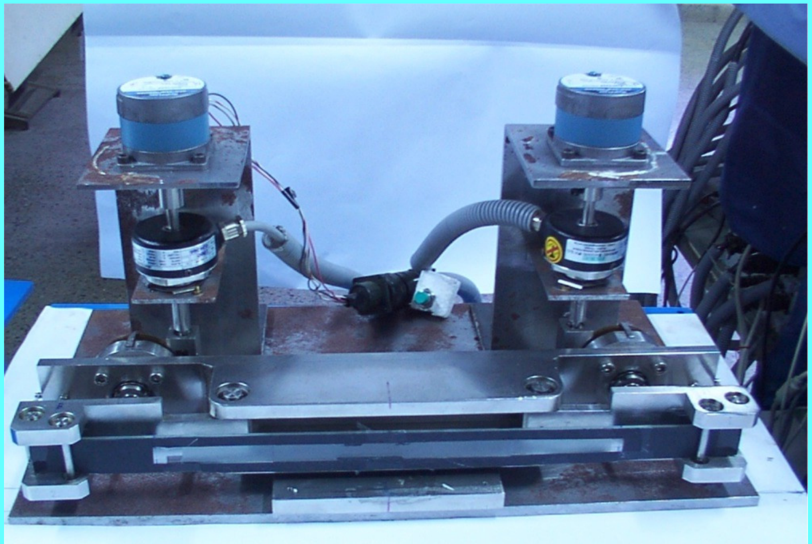
Polychromator

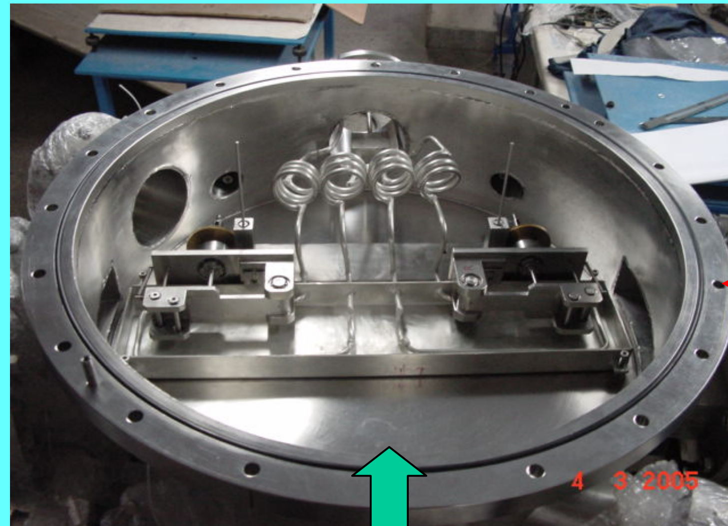
Crystal Bender

Indigenously built

Generates elliptical curvature on a profiled crystal of ½ m length

Mean Radius of Curvature: 2 m to 20 m (within +/-0.5%)





Stepper motor driven shafts
Stroke: 0-12 mm (required 3-4 mm)
Resolution: 0.5 μm

Actual bender inside a vacuum chamber with Ga/In based cooling arrangement

460 mm long profiled Si (111) Crystal ;Darwin width: 0.6 arc sec

Harmonic Rejection Mirror :

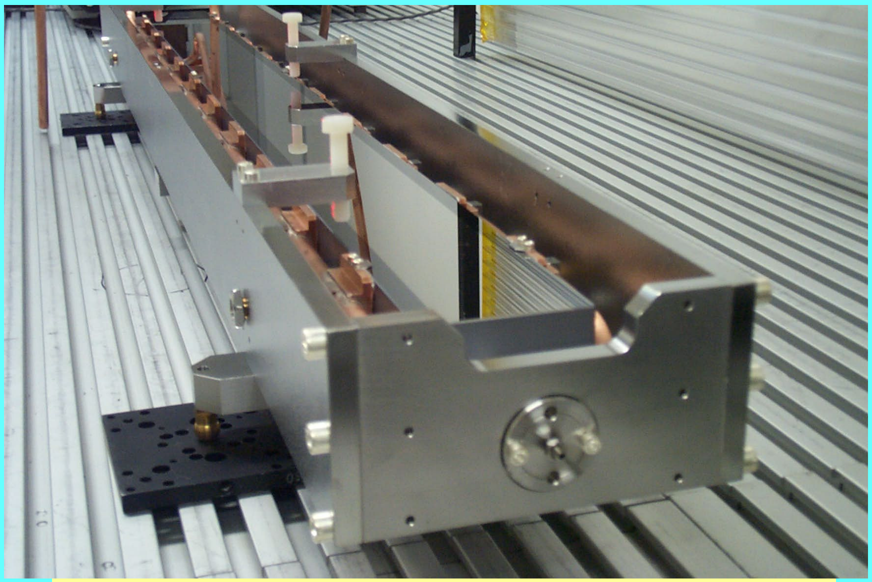
Substrate: Si

Coating: Rh

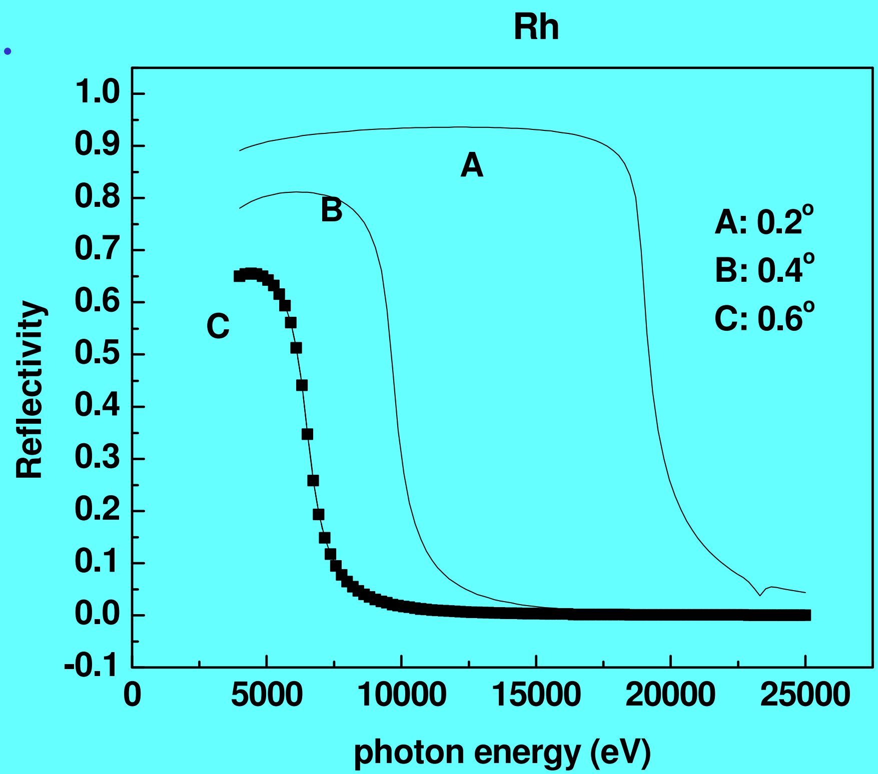
Radius: 1319 m

Size : 1 m × 30 mm

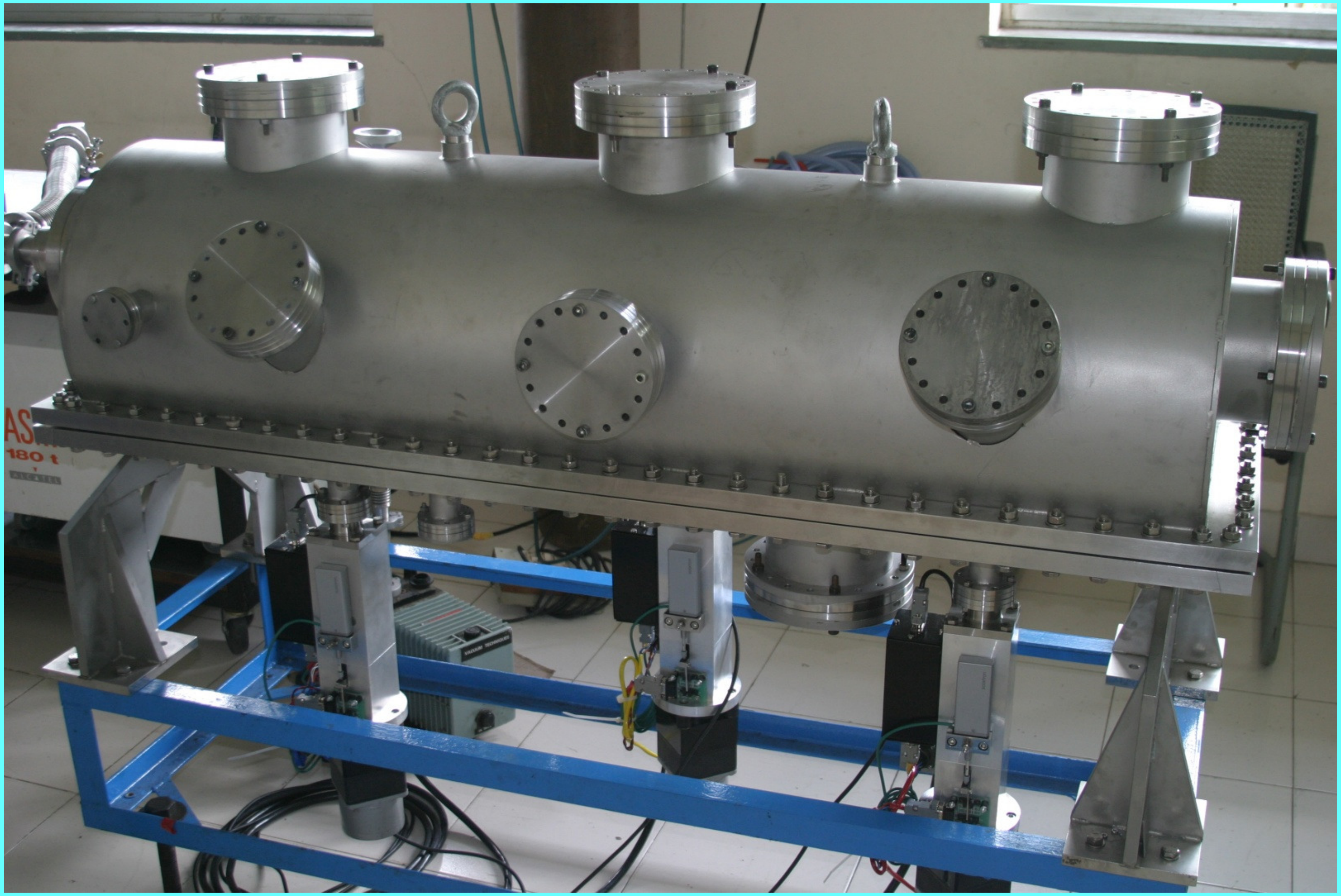
Surface Roughness: 3 Å r.m.s.



Cylindrical Mirror
Side cooled, gravity compensated



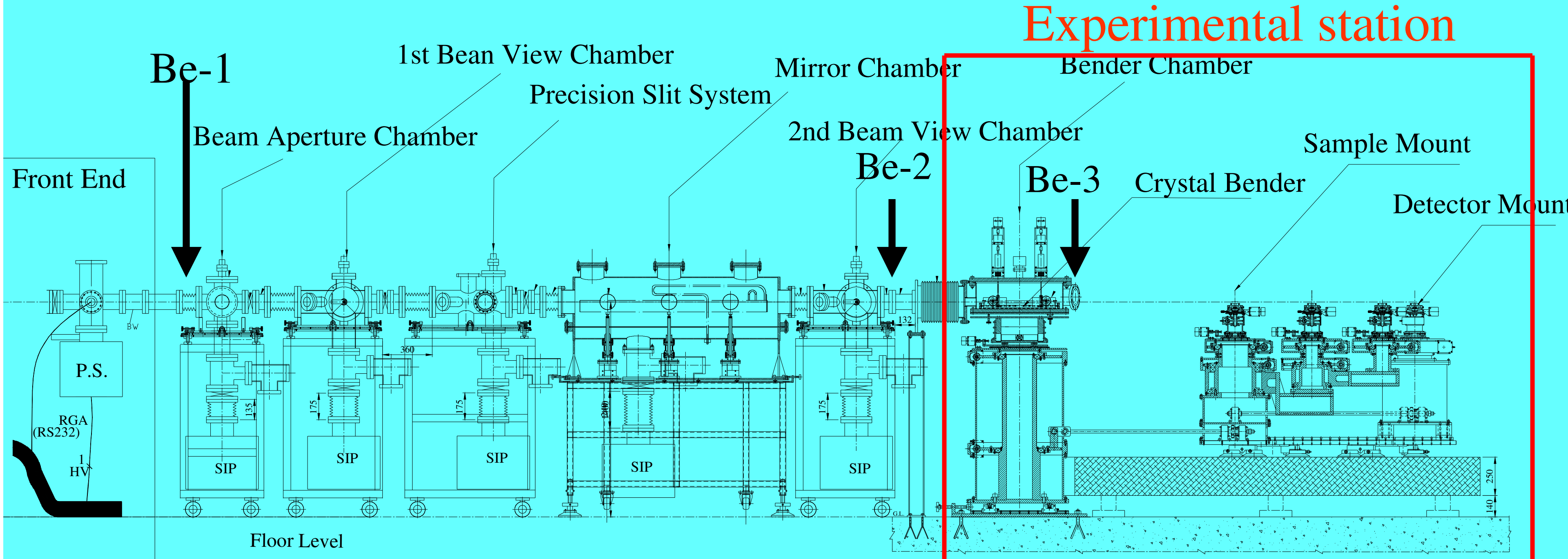
Mirror



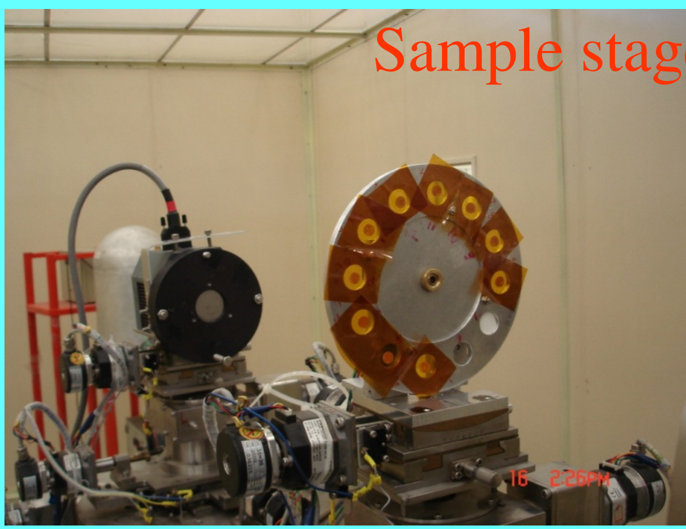
Movement resolution of mount
Shafts inside UHV : 1μm

Indigenously built Mirror chamber with mirror mount

Mechanical layout of EXAFS BL at Indus-2



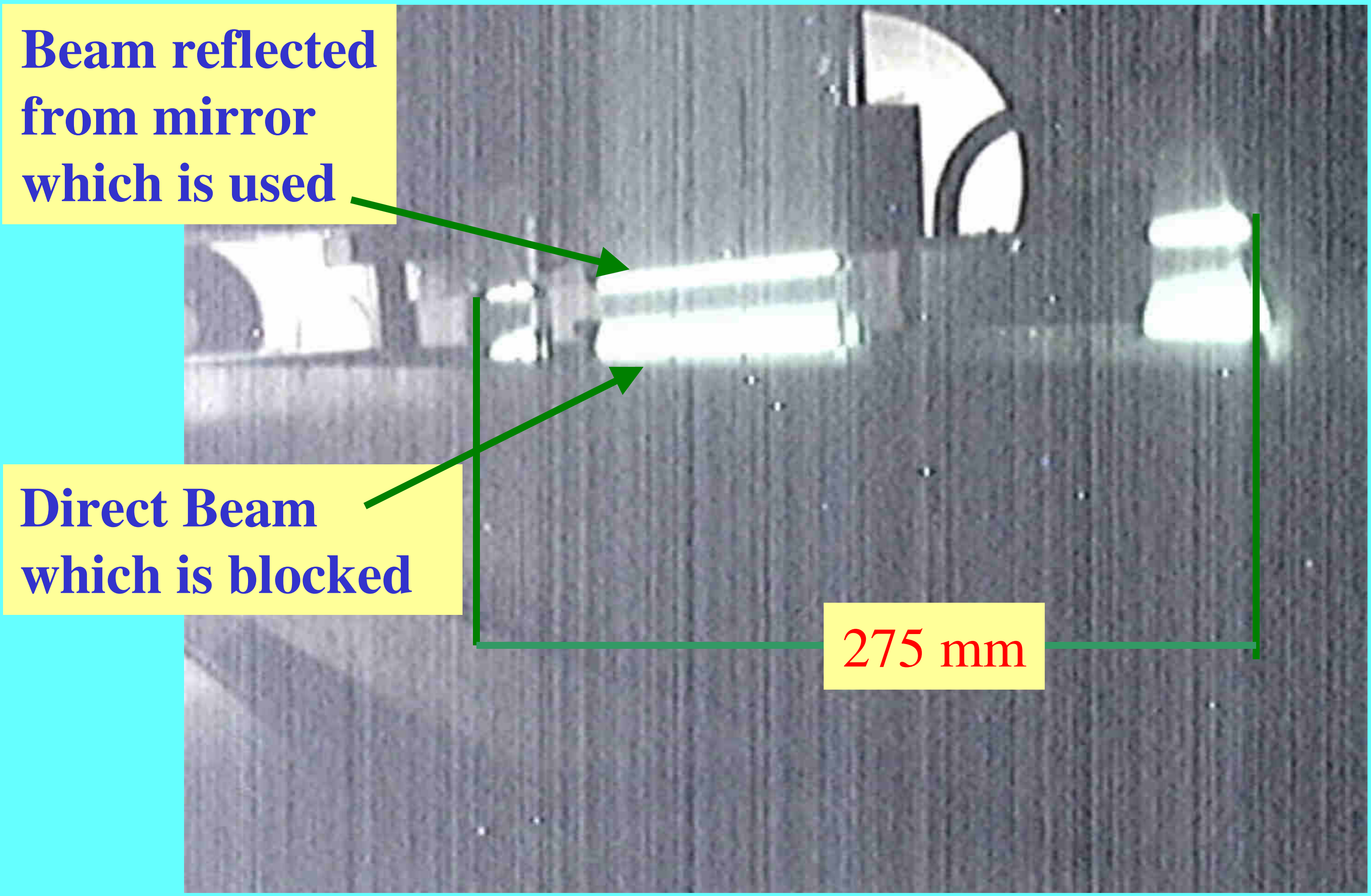
Shielding wall



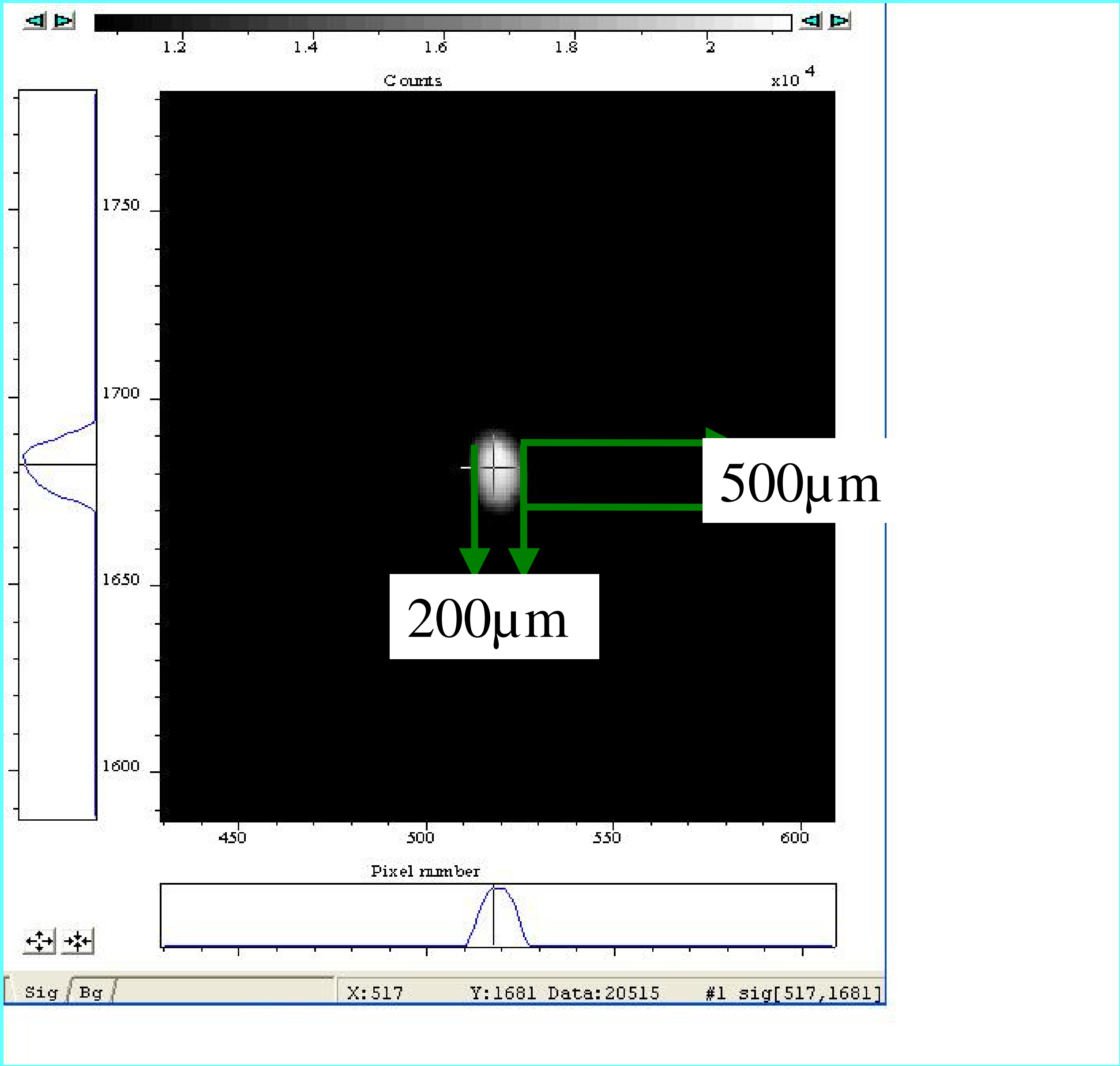
Commissioning stages

- Alignment of Front-end with SR
- Integrating the front end with beamline and achieving vacuum in subsystems
- Optical Alignment of with SR using BV systems and CCD detector
- Checking the SR foot prints at different locations of the beamline
- Checking the performance of the bender
- Recording absorption of standard samples to check the spatial resolution and band pass energy

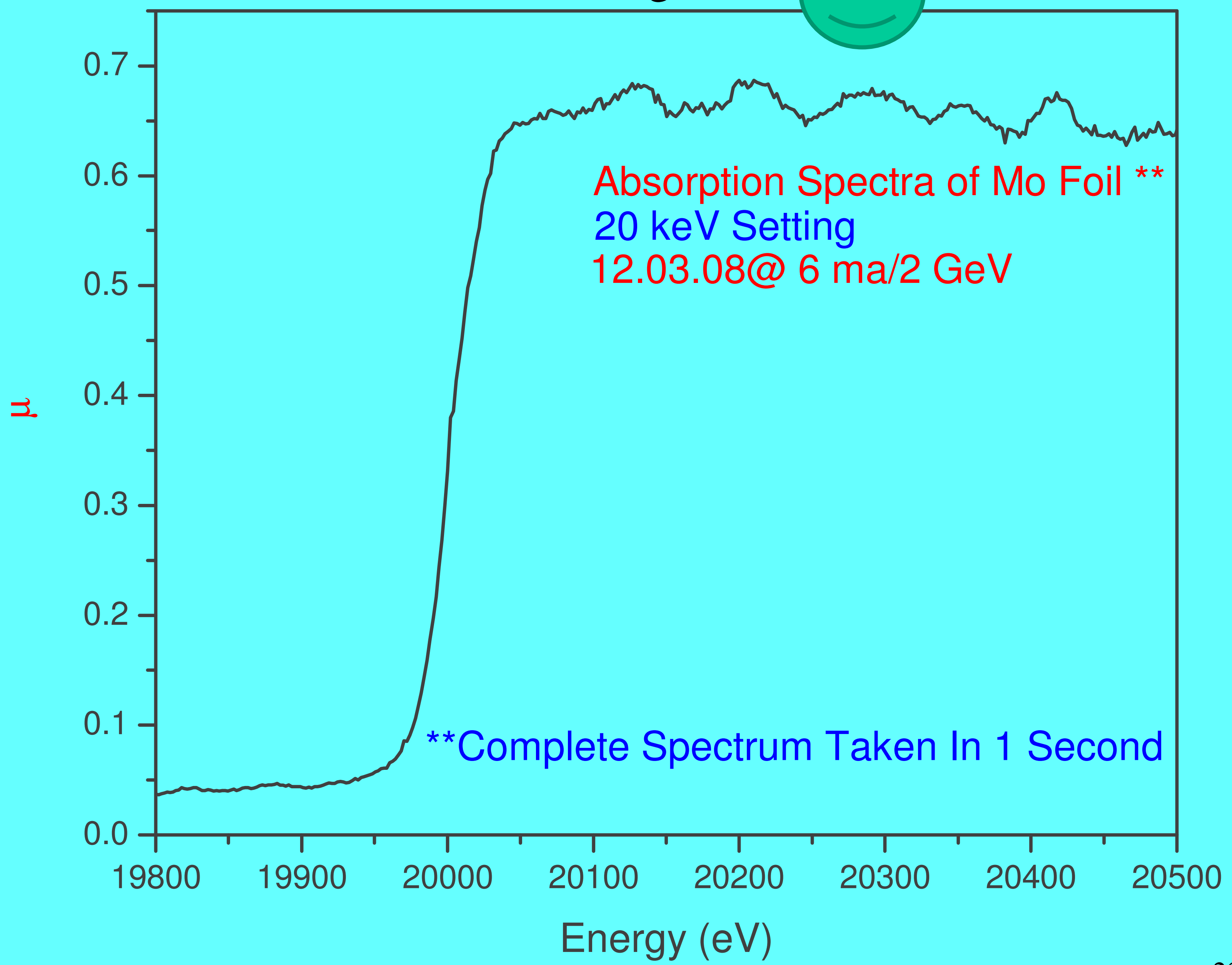
Illumination on crystal for 20 keV setting



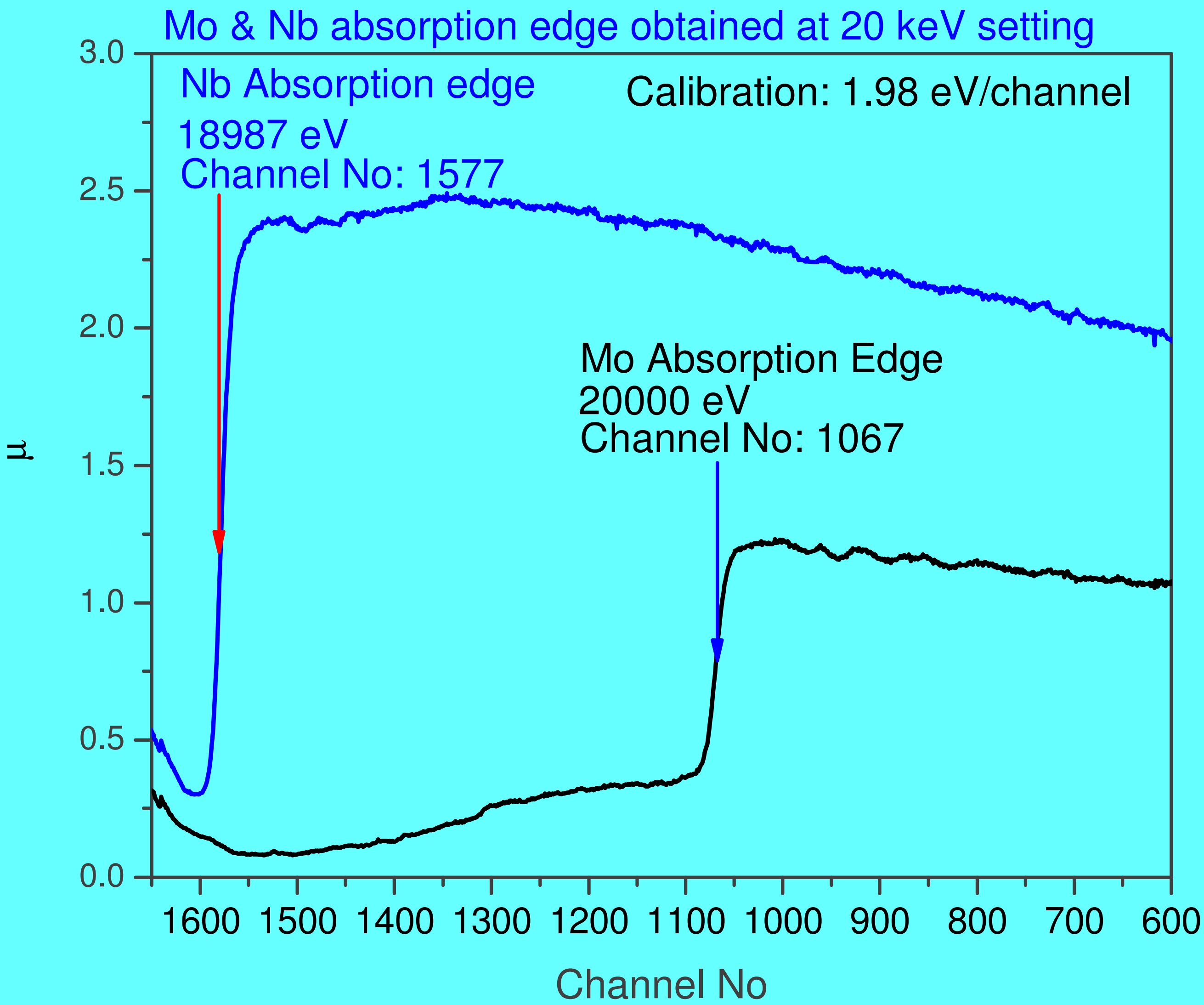
Beam spot at sample position



First EXAFS Signal



Mo-Nb

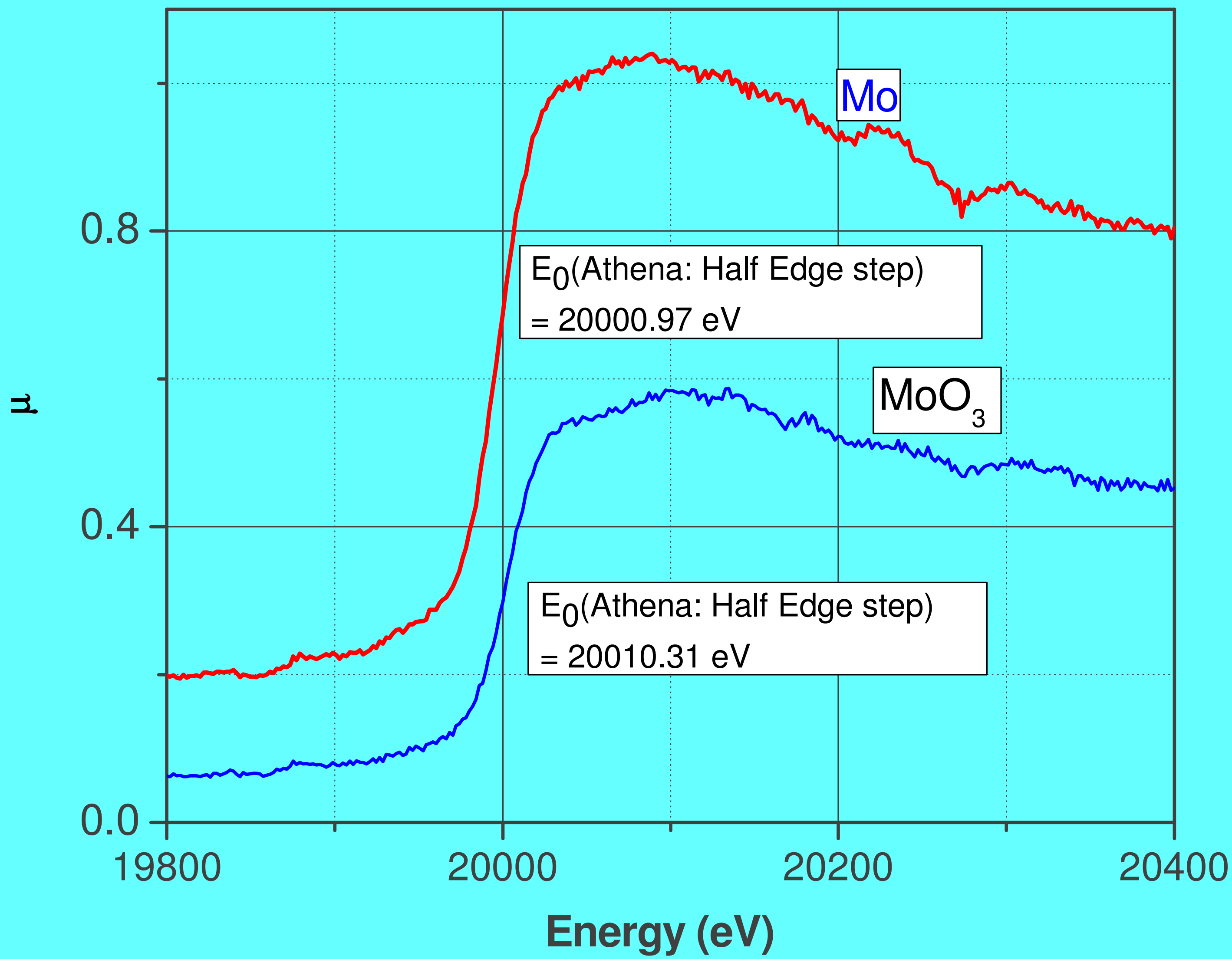


Mo & Nb absorption edge obtained at 20 keV setting
Nb Absorption edge
18987 eV
Channel No: 1577
Calibration: 1.98 eV/channel
Mo Absorption Edge
20000 eV
Channel No: 1067
μ
3.0
2.5
2.0
1.5
1.0
0.5
0.0
1600 1500 1400 1300 1200 1100 1000 900 800 700 600
Channel No

Mo MoO_3

Reported Difference in E_0: ~9 eV

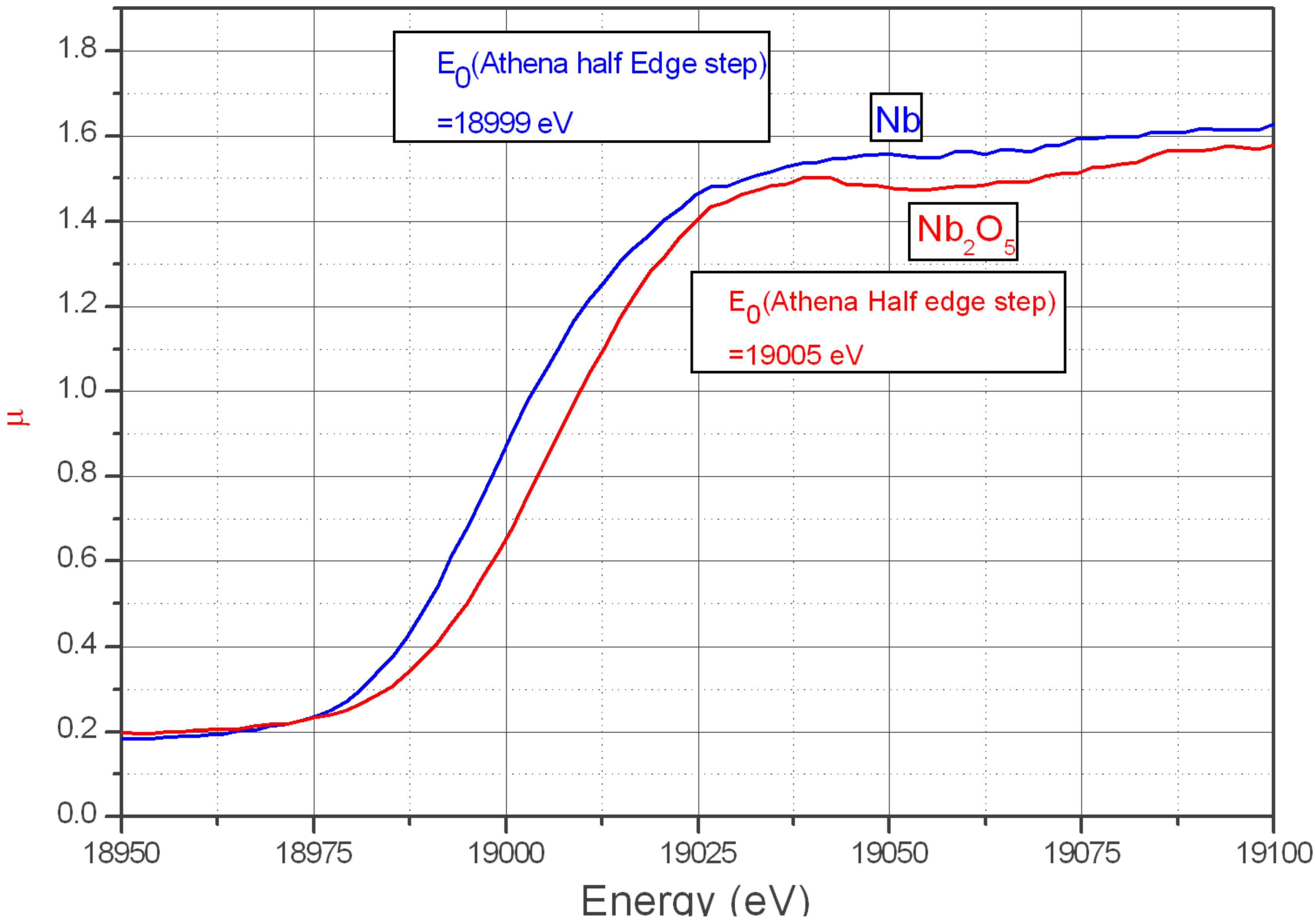
D. Lutzenkrichen-Hecht and R. Frahm, J. Synch. Rad., 6 (1999) 591.



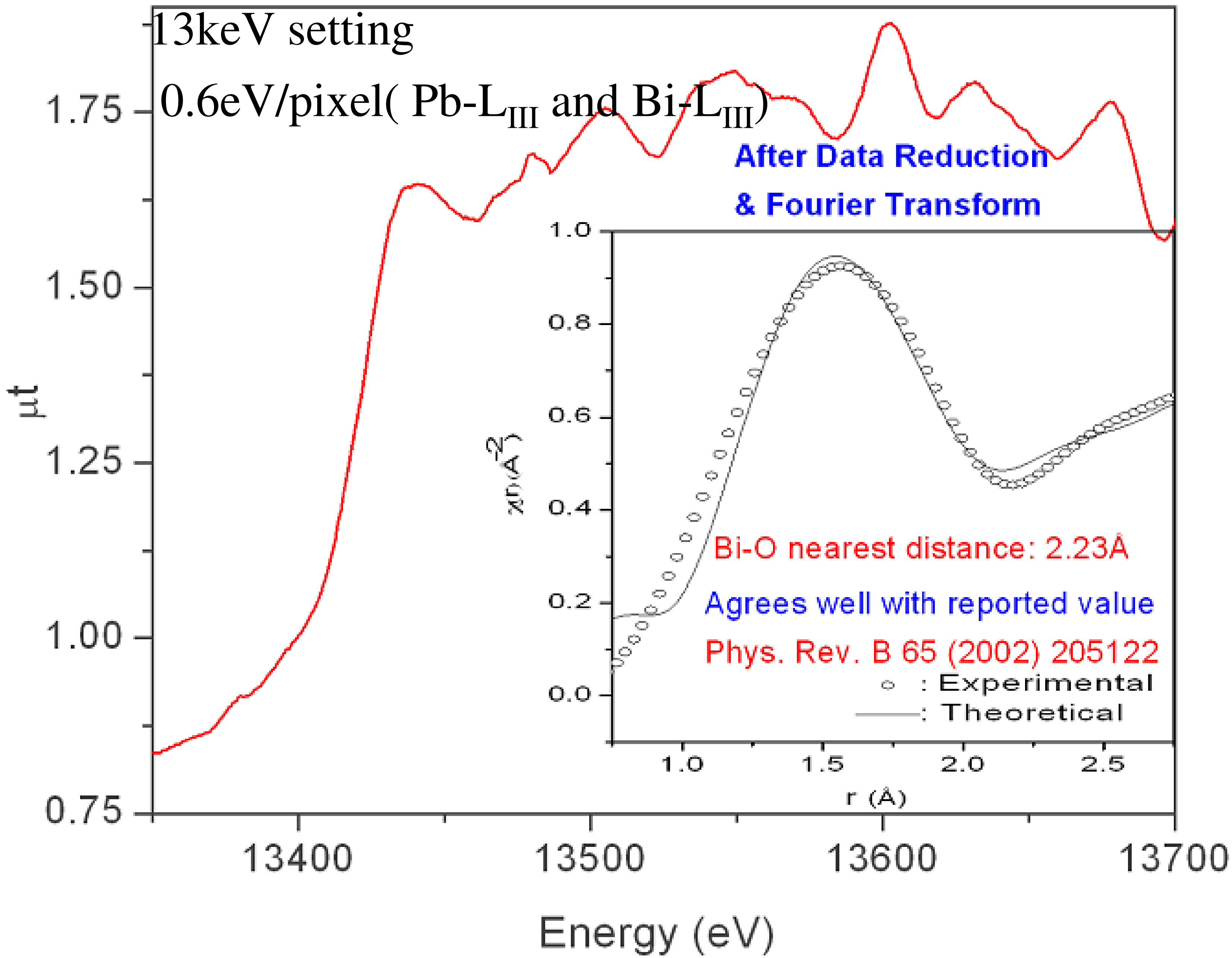
Chemical shift

Reported Value: 5 eV

Sacerdoti et al. , J. Sol. St. Chem. 177 (2004) 1781



EXAFS Spectra of Bi_2O_3 taken at L_{III} edge



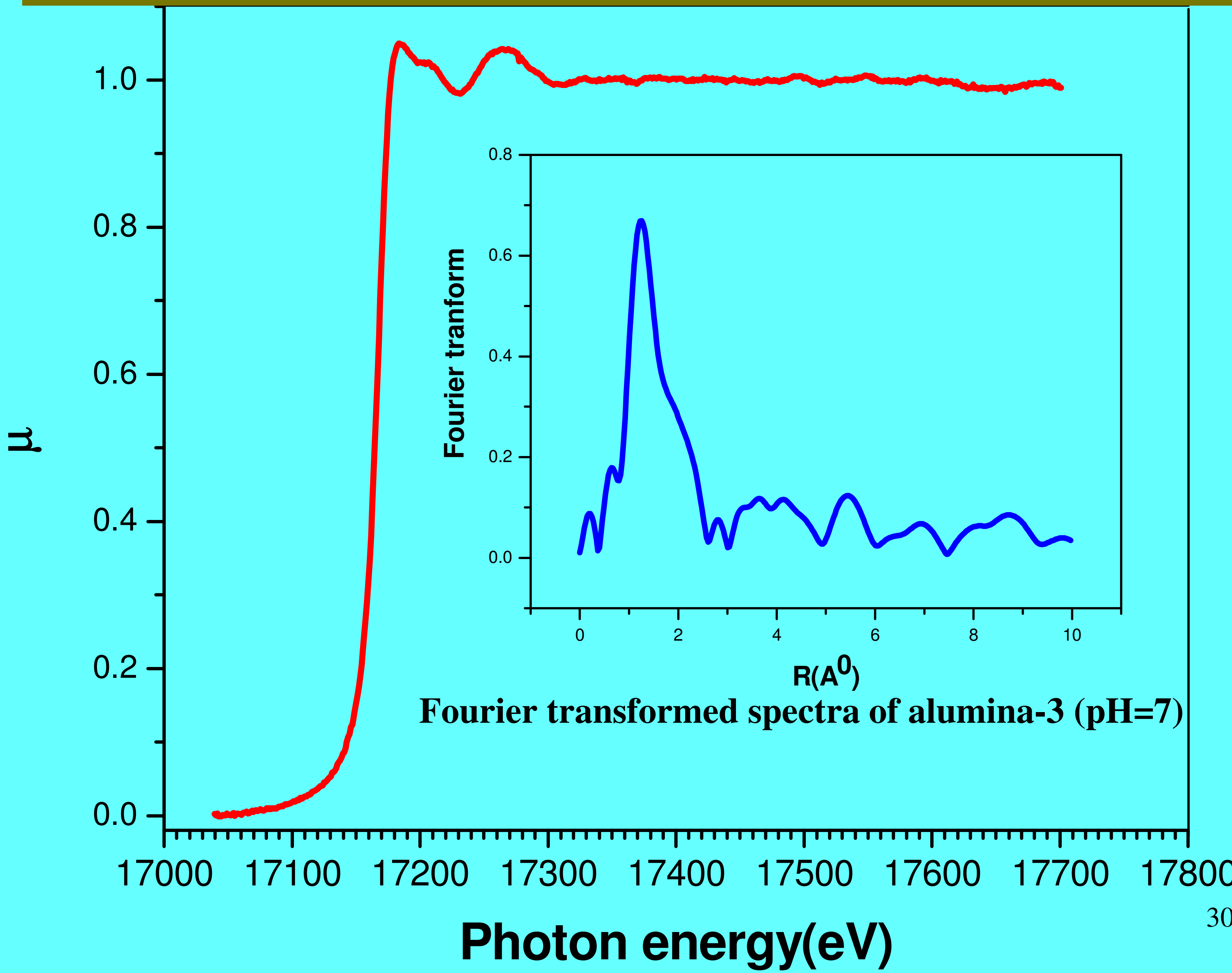
EXAFS MEASUREMNENTS

- U(VI) sorbed on alumina and kolinite
- Crystalline $Pb_5Ge_3O_{11}$

X-ray Absorption Spectroscopy of U(VI) sorbed onto Alumina

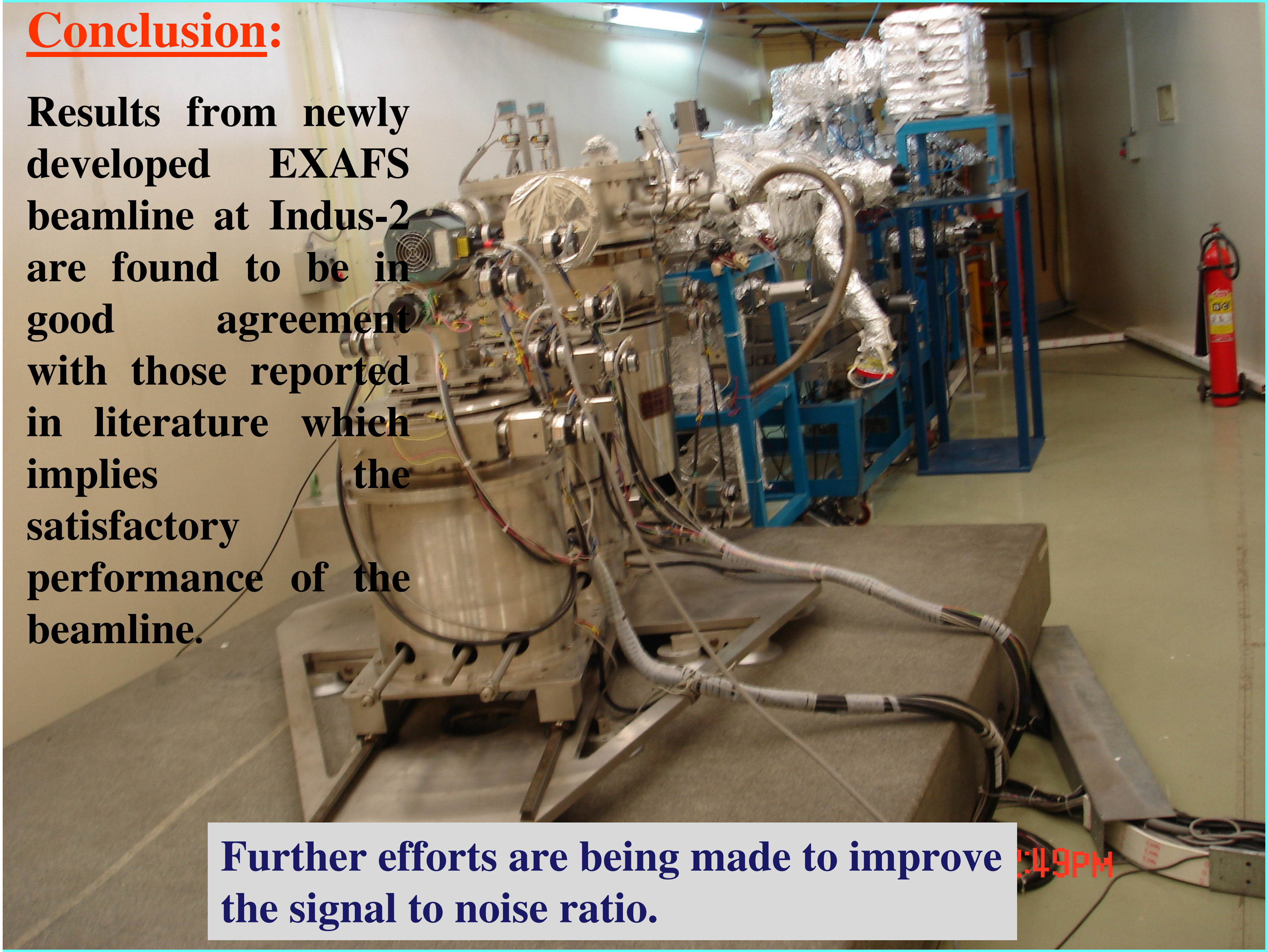
Sorption of U(VI) by alumina and kaolinite at varying pH has been studied by X-ray absorption Spectroscopy. The absorption intensity was found to increase with increasing pH of the suspension.

X-ray absorption spectrum of U(VI) sorbed onto Alumina



Conclusion:

Results from newly developed EXAFS beamline at Indus-2 are found to be in good agreement with those reported in literature which implies the satisfactory performance of the beamline.



Further efforts are being made to improve the signal to noise ratio.

Acknowledgements

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