Baseline Evaluation of XAFS Bending Magnet Beamlines

Experiments performed under "*standard optimized operating conditions*," as recorded.

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- Rhodium coated torroidal mirror at 2.7 mrad for spectroscopy set up at front end of hutch.
- Cutoff at Rh edge, i.e., not defined by mirror angle.
- Normal 12-BM XAFS is in fluorescence mode using a multi-element detector, since user samples are too dilute for transmission measurement.
- Normally runs with 2nd crystal translation on because the 2nd crystal is short (ca. 4" long).
- Energy range: upper limit (on first harmonic) limited to the first edge of Cd. Lowest edge normally run is Ti.†
- Matched 15 cm ion chambers for I0 and IT. Start out running dry N_2 for first measurements.
- SR 570 run normally without offset, though it can be added.
- No filters.
- Intensity feedback (A/C lock). Set point can be set on the side of the rocking curve for detuning. Normally run at 0.8, so 20% detuned. Using SIS-2900 MOSTAB NIM module from Struck (http://www.struck.de).
- Gas ionization chambers operating at 1,000 VDC.
- 7 GeV electron synchrotron operating in top-up mode, 102.1 mA.
- I0 entrance slits 3 mm vertical x 3 mm horizontal. Beam normally slightly defocused for spectroscopy experiments (set by torroidal mirror angle), beam size is ca. 2.1 mm Vertical x 1.4 mm Horizontal. Focus can be made as small as 0.8 mm Horizontal x 1.2 mm Vertical at the diffractometer (farther downstream than the spectroscopy table). Many spectroscopy samples run at 12-BM are actinides. Users have noticed that fully focused beam causes some beam-induced chemstry of the samples. Therefore they often request the beam slightly defocused.

† Web site needs to be updated.

Some problem at the start of the experiment, unable to find the Zn edge in a Zn foil. Monochromator was apparently recalibrated at high energies by previous users, making Zn K off by about 100 eV.

N.B. Beamline discussion about energy ranges posted on the APS web site. High energies quoted are usually for scattering (fixed energy), but not applicable to spectroscopy (scanning energy). Perhaps this should be taken into account on the web pages for spectroscopy beamlines.

ENERGY CALIBRATION: Experiment log

XANES scans of metal foil reference standards collected over a large energy range without recalibrating the monochromator.

- Metal foils from EXAFS Materials (Joe Wong's company). Set provided by M. Newville.
- All XANES collected at 0.2 sec/point, unless otherwise noted.
- Scan details for V scan, -20 to 30 eV around edge. Step size as noted in table. Other edges, used the same range as for the ENERGY RESOLUTION measurement, but this took too long. Suggest using shorter time/point, as well.

file name	foil	notes	edge energy		step size
			nominal†	measured‡	
Cu_K-1.txt	Cu	0.5 s/pt. feedback off	8980.48(2)	8980.5	0.4 eV
Zn_K-1.txt	Zn	0.5 s/pt. feedback off	9660.76(3)	9659.2	0.4
Cr_K-2.txt	Cr	0.5 s/pt. feedback on 0.8 of peak	5989.02(4)	5987.9	0.3
V_K-1.txt	V	0.5 s/pt feedback on 0.8 of peak	5463.76(5)	5462.9	0.3

file name	foil	edge energy		step size
		nominal†	measured‡	
	Mo	20,000.36(2)		0.8 eV
	Ag	25,515.6(3)		0.8 eV

○

file name	foil	edge energy		step size
		nominal†	measured‡	
	Ag	25,515.6(3)		0.8 eV

†Rev. Sci. Instrum., **67** (1996) 686.

‡Using first peak in first derivative of XANES calculated at beamline with BESSRC regional software.

ENERGY RESOLUTION: Experiment log

Measure the full width at half maximum of the V_2O_5 pre-edge feature.

- The sample is powder-on-tape prepared by Matt Newville.
- Harmonic Rejection by detuning to 80% of fully tuned intensity.
- Scan details: pre-edge –100 to –20 eV, Post-edge 30 eV to 8k (240 eV) for accurate normalization.

filename	slit size		FWHM of feature	step size
	V	H		
V2O5_K-1.txt	1.1 mm	6 mm		0.2 eV

Count time at 12 BM, 0.5 sec/pt.

HARMONIC CONTENT: Experiment log

Scan the energy around 6.66 keV through a Mo foil to look for emergent Mo XANES from the third harmonic.

- Nominal edge position for Mo is 20,000 eV. Run a XANES scan with E0 = 6,667.
- 25 µm thick Mo foil from the PNC set of foil standards.

filename	IT	I0	v/f counts	sec/pt
Mo_Harmonic-1				0.5

BASE NOISE LEVEL: Experiment Log

Record at 10 keV for 3 minutes. Record with beam off for 3 minutes. Record data with knife edge 1/2 way through beam, Horizontal and Vertical, for 3 minutes.

filename	condition	p-p on µ	topoff event
	10 keV	2 x 10^-4	0.5%
	beam off		
	1/2 blocked Vert.		

DATA QUALITY: Experiment log

Transmission EXAFS of solutions with 0.1 edge step in ca. 2 absorption lengths of water. Cover a range of energies (Cl, Zn, Cd).

Solutions and transmission cells prepared by Matt Newville using dilution calculations by Bruce Ravel.

zinc nitrate

445 mg $Zn(NO_3)2•6H_2O$ (Alfa #22403)

was dissolved into ≈40 ml H_2O (Fischer W2-4 DIVF water) and stirred for 5 minutes.

The solution was then brought to 50 ml.

cadmium nitrate

626 mg $Cd(NO_3)2•6H_2O$ (Alfa #21853)

was dissolved in ≈25 ml H_2O (Fischer W2-4 DIVF water) and stirred for 5 minutes.

The solution was then brought to 30 ml.

filename		slit size	edge step height	
Zn_Nitrate-1	1 sec/pt			
Zn_Nitrate-2	repeat	repeat		

I0	IT
50 nA/V	50 nA/V

filename		slit size	edge step height	
	1 sec/pt	0.8 mm x 10 mm	0.14	
	repeat	repeat		

-200, -20, 30, 16k

DETECTOR LINEARITY: Experiment Log

Move a knife edge or aperture across the incident beam and monitor the I0/IT ratio.

Apparently there are several different types of linearity tests one could perform. We debate the merits and applicability, and decide to perform a slit scan: scan a narrow slit across the beam horizontally, to see how uniform the detector is from side to side.

filename	beam size		IO	IT	comments
	H	V			
	1 mm	1 mm	2 nA/V	2 nA/V	check uniformity of the ionization chambers from side to side
	1 mm	10 mm	20 nA/V	20 nA/V	Also used IRef at 10 nA/V. Dummy scan, with manually inserted layers of 12.5 μm Mo foil

BEAMLINE OPERATIONS

Practical limits on energy range for EXAFS (highest and lowest measured spectra)
Ti K (4966) to Cd K (26711).
Ease of changing energy
<comment>
Availability of detectors
<comment>
Availability of special sample environments (high/low temp., vacuum, pressure, etc.)
<comment>
Ease of integrating APS Pool Detectors and Equipment
<comment>
Data collection software
<comment>
On-line data processing and analysis
<comment>
Sources of systematic errors (random electronic noise, known monochromator glitches, etc.)
<comment>

SOFTWARE CAPABILITIES (need to make a table for this)

maximum number of regions for energy scans
k-space scanning
k-weighted integration time
automatic offset correction
macro capabilities
plotting capabilities
on-line analysis capabilities
example of data file header (what information is automatically recorded)

9.1 % absorption at Cd, 15 cm Ar. estimated flux is 8x10^9 above the edge.