Baseline Evaluation of XAFS Bending Magnet Beamlines

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

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- Rh coated torroidal focusing mirror after the monochromator. Secondary Rh coated flat mirror for harmonic rejection.
- Detectors: Custom 10 cm gas ionization chambers operated at 300 VDC with He, ca. 400 VDC with other gases (nitrogen).
- Beamline optimized for low energies (e.g., sulfur K), so detectors run windowless and effectively serve as part of the flight path. Changing samples requires re-equillibration time of ca. 1 minute.
- XAFS scanning is done using spec. Software automatically measures offsets for 10 seconds at the start of every scan.
- Monochromator Si(111) double flat crystals in fixed-exit geometry.
- SRS 570 current amplifiers > V/F converter from Nova > VME Scaler
- Vertical feedback uses PSD on torroidal mirror pitch. Horizontal feedback uses PSD on monochromator 2nd crystal chi.
- 7 GeV electron synchrotron operating in Non top-up mode, 0 + 1296x1, 1% coupling, 98.7 mA at 11:43 am.
- Beam size at sample position varies, but depends on torroidal focusing mirror settings.

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.
- XANES scan: -20 to 30 eV, step sizes as noted in table
- Switch to N2 in IT for Cu and Zn measurements.

file name	foil	edge energy		step size (eV)
		nominal†	measured‡	
copper_foil_XANES_Apr21_2006_1515.1	Cu	8980.48(2)	8984.22	0.4
zinc_foil_XANES_Apr21_2006_1528.1	Zn	9660.76(3)	9665.07	0.4
changed 2^{nd} mirror tilt so cutoff is ca. 12keV (tilt is measured relative to torroidal mirror tilt). Detectors at 1 kV, Detector gases changed to nitrogen.				
zinc_foil_XANES_Apr21_2006_1606.1	Zn	9660.76(3)	9665.36	0.4

He in IT for Cr and V measurements.

file name	foil	edge energy		step size (eV)
		nominal†	measured‡	
chromium_foil_xanes_apr21_2006_1356.1	Cr	5989.02(4)	5990.43	0.3
vanadium_foil_xanes_apr21_2006_1259.1	V (5μm)	5463.76(5)	<5465> calibrated	0.3
vanadium_foil_xanes_apr21_2001_1313.1	V	5463.76(5)	5464.72	0.3

N.B. Mo, Ag and Cd not accessible

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

‡Using first peak in first derivative of XANES calculated at beamline with CMC-CAT regional software (Xplot).

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.
- Scan details:
  - –100, -20, 5eV steps
  - –20, 30, 0.2 eV steps
  - 2.81, 8, 0.075 Å^{-1} steps
  - 0.5 s/pt (w/1 s/pt settling time)

filename	beam size (focused)		Notes
	V	H	
vanadate_EXAFS_Apr21_2006_1325.1	ca. 1 mm	ca. 1 mm	Aborted; sample chamber gas not at equilibrium

vanadate_EXAFS_Apr21_2006_1331.1	"	"	

FLUX: Experiment log

Monochromator set to 10 keV
Nitrogen flowing at STP
1,000 VDC across detector plates
Incident beam focused by torroidal mirror to ca. 1 mm V x 1 mm H
10 cm ADC gas ionization chamber (GSECARS)

Unsure of v/f conversion factor. Can probably figure that out from these number:
ring current = 98.8 mA, scaler output is 472560 counts/sec
centering beam on IT using a table scan (slow). Discover that the Zn foil is still in the beam. Oops.

Output voltage: 20 nA/V
Offset voltage: 1 pA
V-F conversion factor
Scaler counts 11770 – 280
For comparison, in I0 10 cm, 1kV, 10 keV, Nitrogen,
Scaler counts 149056 – 1
gaoin settings are 20 nA/V, 1pA offset.

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. Set delay time to 0 seconds.

filename	condition	notes
timescan.dat #S 1	10 keV	
timescan.dat #S 2	beam off	
timescan.dat #S 3	1/2 blocked vertically	using slit, from bottom

DETECTOR LINEARITY: Experiment Log

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			
timescan.dat S# 5	ca. 1 mm	ca. 1 mm	2 nA/V	2 nA/V	check uniformity of the ionization chambers from side to side using a 0.1 mm wide slit

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. Scan parameters are the same as for the Vanadate, but with larger steps in the XANES region.

- Nominal edge position for Mo is 20,000 eV. Run a XANES scan with E0 = 6,667.
- 25 μm thick Mo foil from sector 20.
- Scan details:
    - –100, -20, 5 eV steps
    - –20, 30, 2 eV steps
    - 2.81, 8, 0.075 $Å^{-1}$ steps
    - 0.5 s/pt (w/1 s/pt settling time)

filename	notes
moly_EXAFS_Apr21_2006_1426.1	signal too low in IT; need to go to more absorbing detector gas
moly_EXAFS_Apr21_2006_1457.1	with nitrogen in IT.

Changed over to nitrogen in IT. Starting at 14:53, with connecting a regulator to the gas bottle.

DATA QUALITY: Experiment log

Transmission EXAFS of solutions with 0.1 edge step in ca. 2 absorption lengths of water.

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

zinc nitrate

N.B. New solutions made. Need to update Matt's dilution notes for 12, 33 and 9 BM.

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.

filename		edge step height	notes
zn_solution_EXAFS_Apr21_2006_1716.1	1 sec/pt		XANES scan to check signal
zn_solution_EXAFS_Apr21_2006_1718.1	1 sec/pt		EXAFS
zn_solution_EXAFS_Apr21_2006_	1 sec/pt		repeat

BEAMLINE OPERATIONS

Practical limits on energy range for EXAFS (highest and lowest measured spectra)

Have done Ti. Limiting factor is a lot of Be in the beamline. Highest is Ba. Can go up to 38-40 keV. Mirrors out at the high energies, and take advantage of the large size of the crystals.

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)