Baseline Evaluation of XAFS Bending Magnet Beamlines

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

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- Rh coated first mirror is in front of the monochromator. Serves both harmonic rejection and vertical collimation. Angles changed to optimize for energy range, as noted below.
- Very handy laser level in the hutch for aligning samples is from http://www.overstock.com, ca. $20.
- Detectors: Oxford 30 cm gas ionization chambers operated at –1,500 VDC. Gases optimized for energy range (see notes). Chambers are filled and sealed for up to a week. Windows are 1 mil (25 micron) Kapton.
- Si(111) double flat crystals.
- Rh coated second mirror located after the monochromator used to bring the beam parallel to the experimental table in B hutch and for vertical focusing.
- Counting chain uses Keithley 428 current amplifiers, mounted on posts on the optical table so that the input is an 8 inch cable from the detectors. VME crate located inside the hutch.
- Feedback uses I00 (in air chamber) intensity feedback to the peak maximum. Detuning is also possible by setting the feedback to lock to some constant value on the side of the rocking curve. This is set up in software using P. Jemian's epics interface screens.
- 7 GeV electron synchrotron operating in Non top-up mode, 0 + 1296x1, 1% coupling, 99.7 mA at 10:55 am.
- I0 entrance slits

Some delays at the start of the experiment due to necessity of monochromator changeover, broken "x-ray eye" camera.

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 > 30 eV, step sizes as noted in table
- Vertical collimation using white-beam slits AND pre-I0 slits.
- 2nd mirror used to focus beam so that most of the beam is going through the slits. Focus is made slightly taller than the vertical I0 entrance slits, which are set to 1 mm V x 8 mm H at this time.
- monochromator in channel cut mode.

For Cu/Zn measurements, I0 has 80% N2, 20% He; IT has 80% N2, 20% Ar. 2nd crystal theta tweaked between Cu and Zn energies.

file name	foil	start time	edge energy		step size (eV)
			nominal†	measured‡	
Cufoil.001	Cu		8980.48(2)	calibrate	0.4
Cufoil.002	Cu	11:33	8980.48(2)	8980.75	0.4
Znfoil.001	Zn	11:42	9660.76(3)	9660.44	0.4

Leave detector gases the same for Cr and V measurements.

Crfoil.001	Cr	11:49	5989.02(4)	5989.9	0.3
Vfoil.001	V	11:57	5463.76(5)	5464.94	0.3

Mirror angle to 2 milliradians, detectors I0 50% N2, 50% Ar; IT pure Ar. Refocus. This measurement taken after removing mirrors and then putting them back in. Jenia found some misalignment with the mirror, so the Mo edge may not fit in with the lower energy data.

file name	foil		edge energy		step size
			nominal†	measured‡	
Mofoil.001	Mo	16:29	20,000.36(2)	mis-entered E0	0.8 eV
Mofoil.002	Mo	16:29	20,000.36(2)	I0 saturated	0.8 eV
Mofoil.003,4,5,6	Mo	16:29	20,000.36(2)	stuff happened while JX took a break	0.8 eV
Mofoil.007	Mo	16:29	20,000.36(2)	20,009	0.8 eV

Mirror out for Ag and Cd edges. Will throw off energy calibration.

Agfoil.004	Ag	25,515.6(3)	out of range	0.8 eV
Agfoil.005	Ag	25,515.6(3)	25,546	0.8 eV

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

‡Using first peak in first derivative of XANES calculated at beamline with UNI-CAT 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.
- Scan details (used baseline parameters)

filename	slit size		scan start time
	V	H	
V2O5.001	1 mm	8 mm	12:05

FLUX: Experiment log

Monochromator set to 10 keV
Nitrogen flowing at STP
1,000 VDC across detector plates
Incident beam slits 1 mm V x 10 mm H
10 cm ADC gas ionization chamber

Output voltage: 2.5 10^7 gain
Offset voltage:
V-F conversion factor

It took some time to set this up because Matt didn't get us the detector in time to overlap with the low energy experiments. Then Jenia had to set up a nitrogen gas bottle, since they use the Oxford detectors, which are sealed and have a special filling station. Also, we needed to get an adapter from Sector 20 to connect to Matt's ion chamber, which has CPC quick-connect couplings.

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 sector 20.

filename	scan start time	sec/pt
Harmonic.003	12:17	0.5

Break for lunch.

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	scan start time
koa_scan1_20060419_131517.dat	10 keV	13:11
koa_scan1_20060419_132139.dat	beam off	13:16
koa_scan1_20060419_132755.dat	1/2 blocked vertically	13:24

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.
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	
Znsolution.001	1 sec/pt	1 mm V x 8 mm H	0.15	13:43
Znsolution.002	repeat	same		

Change mirror settings and detector gases for higher energy edges (Mo, Ag, Cd). Cd K = 26711 (orange book value). White beam slit used for collimation (energy resolution). 0.5 mm V x 20 mm H. In hutch slits still 1 mm x 8 mm. No mirrors for Ag and Cd edges. Cutoff is too low.

filename		slit size	edge step height	
Cdsolution.002	1 sec/pt	0.8 mm x 10 mm		15:20
Cdsolution.003	repeat	repeat		

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

N.B. Unable to do this measurement as slits can only scan over 2mm range.