Baseline Evaluation of XAFS Bending Magnet Beamlines

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

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- Monochromator: Si(111) fixed exit, DC servomotors
- Harmonic Rejection: two Rh coated flat mirrors that can also be bent for focusing; detuning when mirrors are not appropriate.
- Detectors: Oxford ionization chambers, 30 cm. Takes about 10 minutes to change gases.
- Counting Chain: amp= SRS570; v/f = Nova N101VTF 4-channel NIM module 10V/MHz; digitization = pc card CYDDA06.
- Controls and Software spec for the Huber slits. Tcl/Tk on top of scipe (John Quintana's linux drivers). Data stored in ASCII format.
- Feedback Intensity feedback, piezo on 2nd crystal.
- Synchrotron operating mode during test 0+24x1 1% coupling, top-up mode. 102.3 mA at 10 am. Second day of 2006-2 user operations.

ENERGY CALIBRATION: Experiment log

XANES scans of metal foil reference standards collected over a large energy range without recalibrating the monochromator. (1 sec/point).

- Metal foils from EXAFS Materials (Joe Wong's company). Set provided by M. Newville.
- XANES scan details
  - -20 to 30 eV
  - step sizes 0.3, 0.4 and 0.8 eV for low, mid and high energies, as noted in table
  - 1 s/pt or less. Not critical.
- Detector Settings: I0 He:N2, 500 Torr, 80:20; IT He:N2, 1400 Torr, 8:92, both at 1 kV
- I0 is 50nA/V w/5nA offset, 20/0.1 nA offset current +/- 1000; IT is 50 nA/V, 5nA offset, 24/0.12 nA offset current. Have the ability to "dial in" the offset current on a fine scale (?).
- No mirrors, detuned to 70% of peak, vertical feedback on
- Beam size is 2 mm V x 10 mm H

file name	foil	edge energy		step size (eV)	Notes
		nominal†	measured‡		
CuFoil_004.dat	Cu	8980.48(2)	8978.9 <calibrated>	varies	check calibration, long scan
CuFoil_001.dat*	Cu	8980.48(2)	same	0.4	fin. 11:25
ZnFoil_001.dat	Zn	9660.76(3)	9659.2	0.4	√
VFoil_001.dat	V (5μm)	5463.76(5)	5463	0.3	√
CrFoil_001	Cr	5989.02(4)	5988	0.3	detune may have been >70%

*new directory APS_TEST, standard beamline_eval XANES scan.

file name	foil	edge energy		step size (eV)	Notes
		nominal†	measured‡		
Mofoil_001	Mo	20,000.36(2)	@	1.5[a]	fin. 16:24
AgFoil_002	Ag	25,515.6(3)	@	1.5	edge is broad, extend range
AgFoil_003	Ag	25,515.6(3)	@	1.5	-50 eV to 30 eV, ΔE=1.5 eV

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

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

[a] N.B. this is a larger step size than we used for ß 9, 12, 20 and 33.

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)
- Detector Settings: I0 He:N2, 500 Torr, 80:20; IT He:N2, 1400 Torr, 8:92, both at 1 kV
- I0 is 10nA/V w/1nA offset, 20/0.02 nA offset current +/- 1000; IT is 1 nA/V, 100pA offset, 18/1.8 pA current offset current. Have the ability to "dial in" the offset current on a fine scale (?). I think this means that it is done by hand, but I don't quite get what is being "done."
- No mirrors, detune to 70% of peak, vertical feedback on
- Beam size is 2 mm V x 13 mm H

filename	beam size		Notes
	V	H	
V2O5_001.dat	2 mm	13 mm	Needed to change gain in IT, abort.
V2O5_002.dat	"	"	fin. ca. 15:00 (jox went for coffee, etc.)

FLUX: Experiment log

Monochromator set to 10 keV
Nitrogen flowing at STP
1,000 VDC across detector plates
Incident Beam @
10 cm ADC gas ionization chamber (GSECARS)
ring current = @, scaler output is @ counts/sec

Output voltage: @
Offset voltage: @
V-F conversion factor @
Scaler counts @
For comparison, in I0 10 cm, 1kV, 10 keV, Nitrogen,
Scaler counts @
gain settings @, @ offset.

Skip this experiment. Too hard to set up, not especially informative, and anyway we don't have the detector.

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 Vertical for 3 minutes. Set delay time to 0 seconds.

- Detector Settings: I0 He:N2, 500 Torr, 80:20; IT He:N2, 1400 Torr, 8:92, both at 1 kV
- No mirrors, detune to 70% of peak, vertical feedback on
- Beam size is 2 mm V x 13 mm H

filename	condition	notes
aps_test_counters S# 4 (spec file)	10 keV	I0 50nA/V, 5nA, 20/0.1nA offset current IT 50nA/V, 5nA, 24/0.12nA offset current IR 100nA/V, 10nA, 40/0.4 nA offset current
aps_test_counters S# 5	short scan	to get offset (dark current)
aps_test_counters S# 6	beam off	finished at 11:59 a.m.
aps_test_counters S# 7	1/2 blocked vertically	Closed slit to 1/2 intensity

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			
					Executive decision to skip this test on 5BM. Not all beamlines were capable, and it didn't show much on 20BM.

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)
- Detector Settings: I0 He:N2, 500 Torr, 80:20; IT He:N2, 1400 Torr, 8:92, both at 1 kV
- I0 is 10nA/V, 1nA offset, 20/0.02nA offset current
- IT is 100pA/V, 10pA offset, 18/0.18pA current offset current
- No mirrors, detune to 70% of peak, vertical feedback on
- Beam size is 2 mm V x 13 mm H

filename	notes	step height
MoFoil_001	25µm Mo foil from Matt's foils box, start 15:28, large step at "edge"	0.3
MoFoil_002	Increase detuning to 65%	0.25
MoFoil_003	Increase detuning to 56%	

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 and cadmium nitrate.

Dilution notes for cells filled 05/31/2006 (MGN) @

- Scan details (from 20 BM gif image file):
  - –200, -20, 5 eV steps
  - –20, 30, 0.4 eV steps @ Zn, 0.8 eV steps @ Cd
  - 2.81, 16, 0.05 $Å^{-1}$ steps
  - 1.0 s/pt

Amplifier Gain Settings

I0=50 nA/V; 5nA; 20/0.1nA offset current

I1=20 nA/V; 2nA; 18/0.036 nA offset current

IR=20 nA/V; 2nA; 95/0.19 nA offset current

Zinc Nitrate Solution filename		edge step height	notes
Zn_solution1_001.dat	1 sec/pt	about 0.1	EXAFS
Zn_solution1_002.dat	1 sec/pt	√	repeat

- Scan details (from 20 BM gif image file):
  - –200, -50, 5 eV steps
  - –50, 30 eV in 1.5 eV steps @ Cd
  - 2.81, 16, 0.05 $Å^{-1}$ steps, kweighted counting by factor of 1.5
  - 1.0 s/pt
- Detector Settings: I0 He:N2, 500 Torr, 80:20; IT He:N2, 1400 Torr, 8:92, both at 1 kV
- I0 is 10nA/V, 1nA offset, 20/0.02nA offset current
- IT is 100pA/V, 10pA offset, 18/0.18pA current offset current
- No mirrors, detune to 70% of peak, vertical feedback on
- Beam size is 2 mm V x 13 mm H

Cadmium Nitrate Solution filename		edge step height	notes
Cd_solution_001.dat	1 sec/pt	@	EXAFS, start at 16:37
Cd_solution_002.dat	1 sec/pt	@	repeat

BEAMLINE OPERATIONS

Practical limits on energy range for EXAFS (highest and lowest measured spectra) Highest is Pr, Lowest is Ti.

Ease of changing energy easy as eating pie.

Availability of detectors 13 element Ge, Lytle, complete set of bent Laue, 3 Cyberstars

Availability of special sample environments (high/low temp., vacuum, pressure, etc.) Displex (can get to 10 K and 800 K, gas handling and characterization for catalysis is available)

Ease of integrating APS Pool Detectors and Equipment typically don't need for XAFS, but could if 13 element broke down. Sometimes borrow image plate or CCD.

Data collection software Tcl/Tk GUI on top of scipe

On-line data processing and analysis Athena on main computer, most users process data on their laptops.

Known sources of systematic errors (random electronic noise, known monochromator glitches, etc.) None

Tools: Milwaukie 2-speed screwdriver.

Travelogue:

- The experimental hutch at 5 BM is the nicest we've seen.
- 75% of time is for XAFS
- Overhead ca. 1/2 second, includes settling time for feedback. Haven't tried going faster.
- Quick XAFS is enabled. Fastest so far is about 1 minute for 1 keV range.
- N.B. end of XANES range needs to be specified in k, not energy.
- Motorized, automated sample chamber
- Macro capability in the software.
- "The first mirror works really well. It collimates the beam."
- Setup for harmonic rejection by detuning to 70% of peak: scan the piezo, look at the rocking curve, determine the position for the lock by eye/cursor.