A Quick Guide to XAS

This quick-guide is intended as a general overview of procedures for successfully performing X- ray Absorption Spectroscopy experiments.
In general, a good XAS experiment will be guided by the **HALO** principle:

- **H**armonics
  - reduce energy harmonics by properly tuning monochromator and/or using harmonic rejection mirror

- **A**lignment
  - align mono, slits, detectors, and sample

- **L**inearity
  - operate ion chambers, Lytle detector, and Germanium detector in a linear response regime.

- **O**ffsets
  - take offsets after any change to detector electronics and before collecting data

XAS Support Staff

XAS support cell phone

- Mon – Fri: 7 am – 10 pm
- Sat – Sun: 8 am – 5 pm
- Matthew Latimer 510-259-8102
- Erik Nelson 415-218-5505

beam-line optimization, detectors, cryostats, data collection

- Matthew Latimer X4944
- Erik Nelson X5103

equipment: rails, cryostats, liquid helium

- Jeff Maske x 3833

Ionization Chambers

high voltage

- check that detector is **not** space charge limited
    - check at beginning-of-scan energy, fully tuned
        - if space charge limited, may be tricky to tell if fully tuned
    - increasing high voltage should **not** increase I0
    - check when changing energy regime, especially when changing gas

gas

- select gas based on energy
    - energy < 5 keV ⇒ helium
    - 5 keV < energy < 15 keV ⇒ nitrogen
    - energy > 15 keV ⇒ argon
- set flow to a few bubbles per second (do **not** leave bubbler bubbling)

amplifiers

- if using SR570 amps
    - run "XASUTILS" program and select "ION CHAMBER CONTROLS"
    - under Controls / More Controls, check filter settings
        - filter type = low pass 6 dB
        - low frequency = 30 Hz
    - set gain such that beam-on gives ≈ 1 (< 5)
        - check value at beginning-of-scan energy
        - check gain after changing offset
    - set offset such that beam-off gives ≈ 0.004 (> 0)
        - check offset after changing gain
- if using Keithley preamps
    - set Rise Time on amplifiers to 30 (Lytle) or 10 (Germanium), 10 is a good all-purpose setting
    - set Gain such that beam-on gives 1 to 5 (< 10)
        - check value at beginning-of-scan energy
        - check gain after changing offset
    - set Suppression on inverse setting of Gain
    - adjust Zeroing Knob such that beam-off gives ≈ 0.004 (> 0)
        - check offset after changing gain

Lytle Detector

gas

- select gas based on fluorescence energy
  - fluorescence energy < 3 keV ⇒ nitrogen
  - 3 keV < fluorescence energy < 7 keV ⇒ argon
  - fluorescence energy > 7 keV ⇒ krypton (ideal), argon (< 12 keV)
- gas change procedure: **use care** – windows are fragile
  1) start with gas tubing disconnected from detector and gas flow set to 0
  2) slowly turn up gas flow to a barely detectable level
  3) connect gas tubing to detector: source to bottom port, return to top port

Soller slits and filter

- can use filter to reduce scatter signal
  - in general, for measuring Kα radiation of element with atomic number Z, use filter with atomic number Z-1
  - Kβ of element Z-1 will overlap Kα of element Z
    - is the sample's fluorescence signal greater than the filter's?
- Soller slits reduce fluorescence from filter

preamplifier

- set gain such that beam on gives 1 to 5 (< 10), but avoid gain of 1000
  - check value above fluorescence-edge-energy
  - check gain after changing offset
- set offset such that beam off gives ≈ 0.004 (> 0)
  - adjust using potentiometer R9
  - check offset after changing gain
- Rise Time is 45 ms, so set Keithleys to < 30 ms or SR570s to > 10 Hz

alignment

1) set MONO above fluorescence-edge-energy
2) scan Lytle position and maximize Lytle signal
   - on 2-3, 4-1, 4-3
     a) visually align horizontally
     b) scan VERT
   - on 7-3 and 9-3
     a) scan VERT
     b) scan HOR
     c) scan VERT again

Germanium Detector

moving detector

- **Be Careful! -** be inside the hutch and prepared to abort the move if necessary

count rates

- operate in linear response regime if not dead-time correcting
    - maximum Incoming Count Rate (ICR) versus shaping time
        - 0.125 μsec ⇒ 110,000 counts/sec
        - 0.250 μsec ⇒ 70,000 counts/sec
        - 0.500 μsec ⇒ 40,000 counts/sec
    - maximum Single Channel Analyzer (SCA) count rate versus shaping time
        - 0.125 μsec ⇒ 30,000 counts/sec
- 30 element detector anomalous behavior
    - ICRs > 200,000 counts/sec may cause gain-shifting
    - if observed, please contact XAS staff

windowing

1) run "GE13" or "GE30" program, depending on detector
2) insert calibration sample into beam
3) check counts
4) set SCA window for one detector element
    a) activate button for selected SCA window
    b) select Anticoincidence Gating Mode
    c) set SCA width and centroid position in energy
    d) adjust SCA window position: optimal width and centroid depends on effect of scatter on pre-edge
5) set SCA windows for remaining detector elements
    a) after adjusting first window, select APPLY TO ALL
    b) adjust window for each detector element, but do **not** select APPLY TO ALL afterwards

Soller slits and filter

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    - in general, for measuring Kα radiation of element with atomic number Z, use filter with atomic number Z-1
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        - is the sample's fluorescence signal greater than the filter's?
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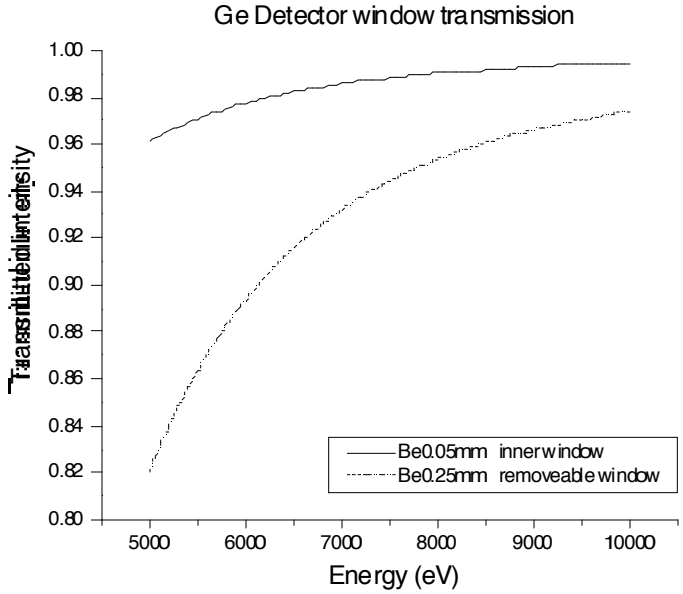
- align using Lytle detector

Germanium Detector (continued)

Beryllium window

- removable outer window protects the thin inner window
  - outer, removable Be window is 0.25 mm
  - inner Be window:
    - Systems 1, 2, 3: 0.05 mm (0.002")
    - Systems 4, 5: 0.025 mm (0.001")
- use the plot below to decide when to remove the window (for example, at Mn ~11% of the fluorescence signal is absorbed by the outer window)
- please contact XAS staff to remove window



Cryostats

Please refer to "User Guide: SSRL Biotechnology Continuous Flow Liquid Helium Cryostats" manual for background and details.

cool-down

1) start with the sample-port under vacuum
2) close sample-port valve on cryostat (otherwise, clog may form)
3) set Heater mode on temperature controller to MANual
4) set Heater power on temperature controller to 0
5) increase Gas Flow Pump's Exhaust to ≈ 2 liter/hour
6) as desired temperature is approached, stabilize the temperature
   - adjust the Gas Flow Pump's Exhaust
   - may need to set Heater mode on temperature controller to AUTO and set SET temperature

sample change

1) open helium gas toggle valve
2) open sample-port valve on cryostat
3) check for gas flow from relief valve on cryostat
4) change samples
5) cycle between vacuum and helium gas five times, ending with helium
6) check for gas flow from relief valve on cryostat
7) close sample-port valve on cryostat
8) close helium gas toggle valve

warm-up

1) cap Germanium detector and retract
2) open vacuum toggle valve
3) open sample-port valve on cryostat (otherwise, explosion may result)
4) decrease Gas Flow Pump's Exhaust to ≈ 0.2 liter/hour
5) set Heater mode on temperature controller to MANual
6) for quick warm-up, set Heater power on temperature controller to 15
7) as desired temperature is approached, stabilize the temperature
   - set Heater power on temperature controller to 0
   - adjust the Gas Flow Pump's Exhaust
   - may need to set Heater mode on temperature controller to AUTO and set SET temperature

Cryostats (continued)

stuck sample rod

1) do **not** force the sample rod
2) cap Germanium detector and retract
3) warm-up to 45 to 65 K (see warm-up procedure above)
4) **slow** warm-up above 85 K (reduce Heater power in temperature controller)
5) **gently** remove sample rod

alignment

1) scan CRYOVERT
   - protein cell ⇒ minimize I1
   - powder cell ⇒ maximize I1
2) adjust CRYOHOR
   - fluorescence screen with camera is easier
   - film or "burn paper" is more precise

temperature range considerations

- photoreduction
- Debye-Waller effect
- sample's state and integrity

liquid helium usage

- keep a log
  1) record "gas flow pump exhaust" rate (liter / hour)
  2) calculate usage: (time) x (exhaust rate)

Data Collection

offsets

- take offsets after any change to detector electronics
  - do before collecting data
  - under Detectors : Current, select Offsets, collect for 10 seconds

crystal glitches

- glitch library at http://www-ssrl.slac.stanford.edu/smbin/dataextractnew.pl , Beamlines, Interactive Crystal Glitch Library
- determine glitch's effect on EXAFS
  - run it: check for spike in data multiplied by k^3
  - if necessary, shorten EXAFS region

signal-to-noise ("Am I done with this sample?")

- always at least two sweeps: do they agree?
- visually check on signal-to-noise of sweep
- more quantitative check:
  1) start with sum of sweeps
  2) do background subtraction
  3) multiply by k^3
  4) calculate Fourier transform
  5) compare successive averages and make judgment on improvement with number of sweeps
  6) estimate signal and noise amplitudes

Checks

daily

- liquid helium dewar level for cryostat
- liquid nitrogen level for Germanium detector dewar
- gas cylinders for the ionization chambers and Lytle detector
- He gas for cryostat sample space

after each beam fill

- mono slit vertical translation
- table vertical translation

after each mono-region change

- mono calibration
- table alignment
- gains and offsets

after each sample change

- if using Germanium detector - count rates

after each sweep

- monochromator detuning (the higher the energy, the more necessary)

always!

- quality of the data - look at it!
- cryostat temperature