

HXMA BL Operation Notes: Monochromator Energy Calibration Stability - Crystal Si(220)

Ning Chen, Weifeng Chen

Dec 21, 2009

1. Intention

It was reported recently by a HXMA user group that the energy calibration of HXMA monochromator drifted during this experiment between the two storage ring injections. The reported issue was investigated at HXMA, and the monochromator was tested to examine its operation status regarding energy calibration stability. During the testing, effort was made to reproduce the reported problem through repeating scan monochromator energy (in XANES mode) at a selected fixed energy range by using a well-known XANES spectra feature. Here the Cu K edge first inflection point (E0) was used as energy calibration testing feature.

2. Experiment procedure

The testing condition was summarized as table 1, and the experimental Cu K edge XANES data are included in Figures 1 and 2.

Table 1. Summary of experimental detail

BL components	Operation status	
HXMA Wiggler	1.9 T	
Storage operation mode	250 mA	
Graphite filter(s)	Out of beam path	
Primary slits	$1^{(v)} \times 8^{(H)} \text{ mm}^2$	
mirror	collimating	Pt stripe (pre-mono)
	Toroidal	Pt stripe (post-mono)
mono	Crystal	Si(220)
	Energy	Cu K edge
Detector	Straight ion chamber filled with 100% N ₂	
Beam pipe setup	Between WB pipe end and JJ slits, He filled	
JJ slits	$1^{(v)} \times 2^{(H)} \text{ mm}^2$	
Experiment time scope	Injection to injection	
Detune	Rate	50 % before each XANES scan
	Energy position	at XANES scan upper energy end
	Detector	Photo diode at WB pipe end
XANES scan config	Pre-edge	-200 to -30, $\Delta E=10 \text{ eV}$
	XANES	-30 to 80, $\Delta E=0.25 \text{ eV}$
	Post -edge	80 eV to 9 k (\AA^{-1})
Scan mode	Single scan	
Sample	standard Cu metal foil, located between I0 and I1	
Experiment date	Dec 17, 2009	
Operator	Weifeng Chen, Ning Chen	

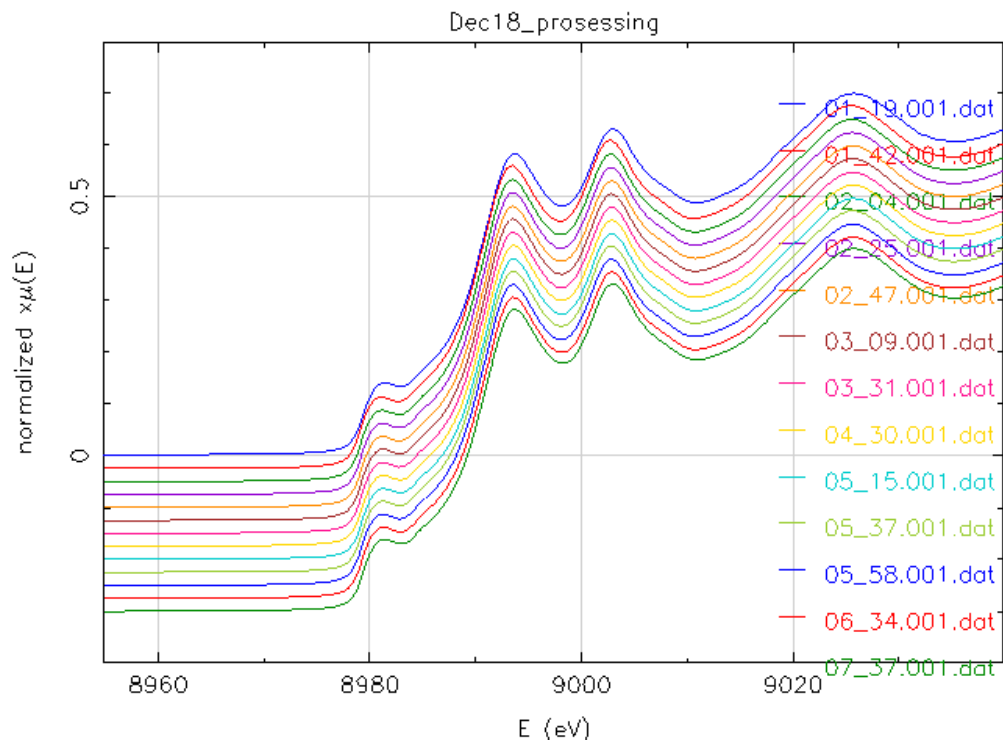


Figure 1. XANES data, Cu K edge. Single scan for each data trace.

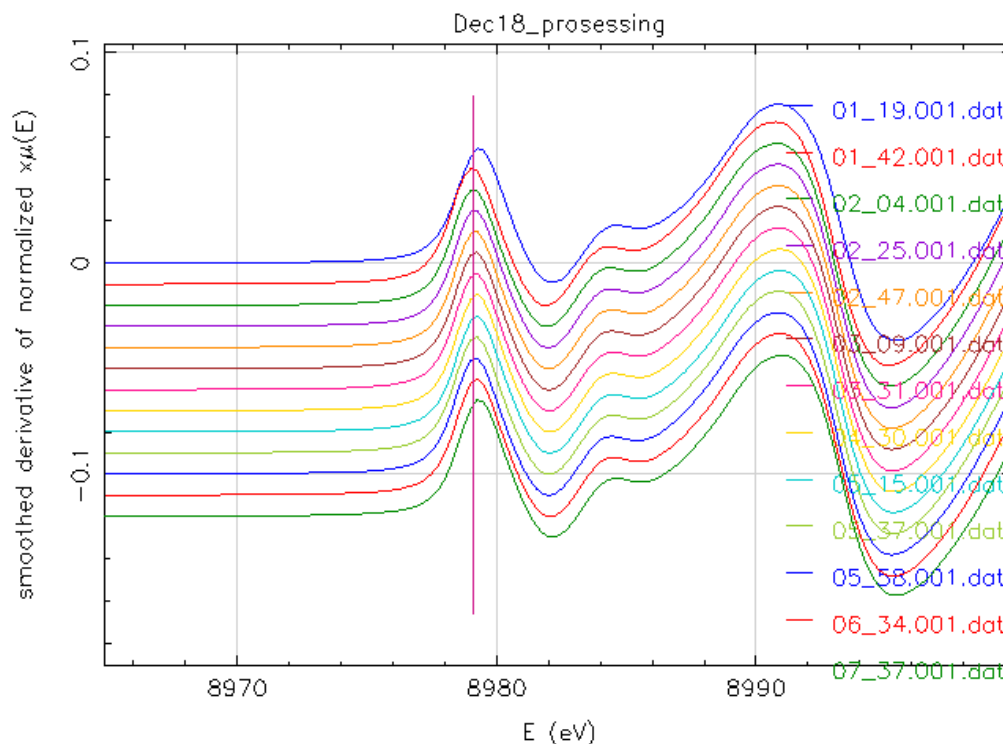


Figure 2. The 1st derivative of XANES data (Fig. 1). The vertical bar indicating the stabilized (thermal equilibrated) mono energy position. Notice that the energy drifting right after the injection (blue trace on the top of the data stack, 01_19_001), and the last two data points (03_34_001 and 07_37_001) before the injection at the bottom of data.

3. Summary of the experimental data

3.1 E0 drifting

Table 2 summarizes the experiment data, i.e., time, storage ring current (I_{ring}), piezo setup, and measured E0. Figure 3 shows the E0 drifting trend versus the I_{ring} decay, namely, the E0 stability corresponds to power loading changing at monochromator crystal. In general the behaviour of E0 drifting follows an unsymmetrical “U” shape trace, composed by high current (I_{ring}) wing, plateau region, and low current (I_{ring}) wing, respectively.

- High current (I_{ring}) wing: right after the injection within ~30 minutes. Within this wing
 - ring current decayed by ~7 mA;
 - Cu K edge E0 drift up to 0.85 eV;
 - Slope of E0 drift trend 1.2×10^{-1} (eV/mA);
- The plateau region is maintained when the ring current was between 244-184 eV, where the detected E0 is essentially constant;
 - Linear fit resulting in a slope of 4.6×10^{-4} (eV/mA) at an average value of 8979.16 (± 0.03) eV with fit residual sum of squares of 0.01 eV²;
 - Notice that the energy resolution at near edge region was set at 0.25 eV, which was high enough for most XANES feature detecting requirement scenario. The detected average E0 (8979.16 eV) is within the detecting resolution;
- Low current (I_{ring}) wing: it was observed when the ring current was lower than 180 mA, where E0 started to drift with a slope of 1.5×10^{-2} (eV/mA), which is 2 magnitudes larger than that of plateau region, but 10 times smaller than that of high current wing.

Table 2. Experimental data

Scan No	Time (h:min)	I_{ring} (mA)	Piezo ^a	E0 (eV) ^b
1	01:09	251.4	803	8979.97
2	01:42	244.3	293	8979.12
3	02:04	238.1	440	8979.12
4	02:25	231.9	563	8979.18
5	02:47	226.1	588	8979.18
6	03:09	220.5	617	8979.12
7	03:31	215.2	659	8979.18
8 ^c	03:53	210	691	8979.99
9	04:30	201.7	722	8979.18
10 ^c	04:52	197	835	8979.96
11	05:15	192.4	752	8979.18
12	05:37	188.2	636	8979.18
13	05:58	184.1	570	8979.12
14	06:34	177.7	733	8979.26
15	07:37	171	1198	8979.36

a. Piezo setup for the 50% detune (high side);
 b. The 1st inflection point of XANES at energy position around the vertical bar shown in Figure 2.
 c. Data No. 8 and 10 collected at 03:53 am and 04:52 am, respectively, are not included the following discussion, and the results shown in Figs 3 and 4, see Fig. 5 for reason.

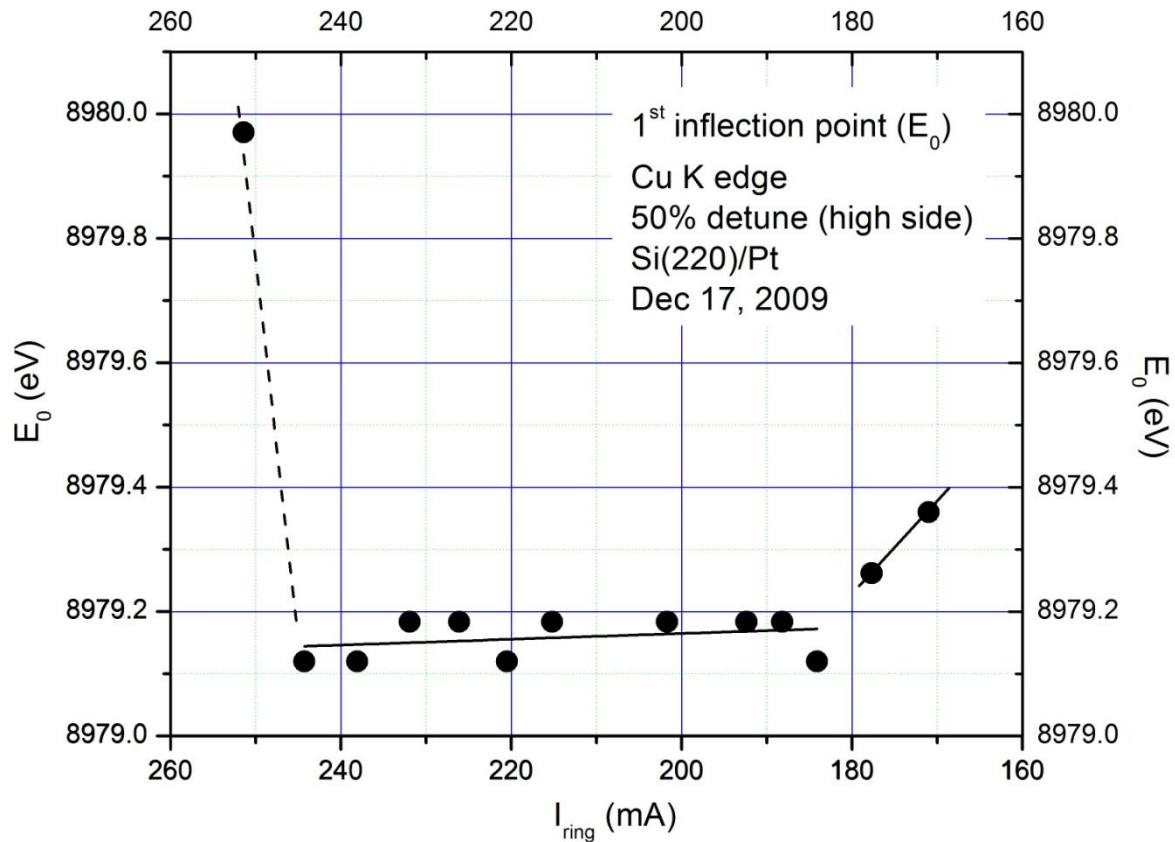


Figure 3. The three regions for the monochromator energy stability versus ring current (I_{ring}), i.e., high current (I_{ring}) wing (dash line), plateau region, and Low current wing. Notice that since there is not enough data for high current wing, the trend of the ring is predicted, therefore presented in dash line.

3.2 Piezo drifting

The second temperature effect is piezo drifting when targeting at a same identical detune rate on the same detune direction (e.g., positive in this investigation), for data see Table 2 and Figure 4. Similar to that of E_0 drifting the piezo drifting trend follows three sectioned traces, namely, high current (I_{ring}) wing, smooth curved region, and low current (I_{ring}) wing. The optimized operation region is again 30 minutes after the injection and when the storage ring current is between 244 – 184 mA.

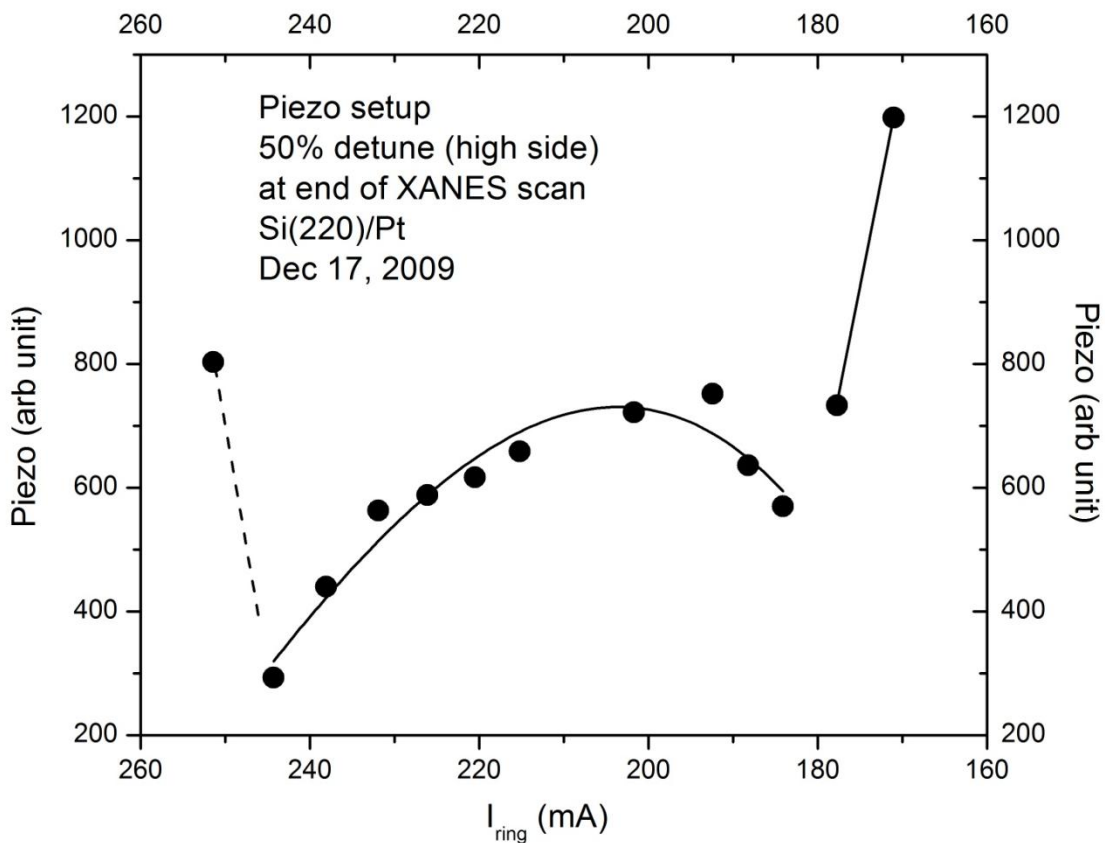


Figure 4. Piezo setup for the 50% detune (high side) at the end of XANES scans (Cu K edge, Figure 1). Notice that similar to the testing for the E0 drifting since there is not enough data for high current wing, the trend of the ring is predicted, therefore presented in dash line.

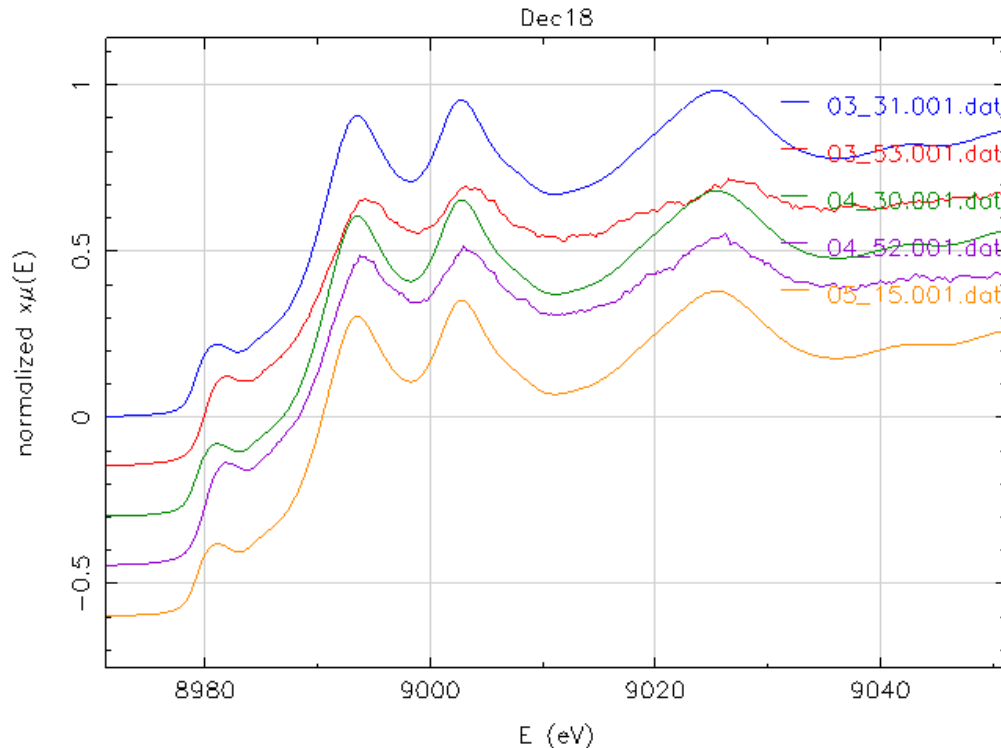


Figure 5. Comparison of data set# 8 (red trace, label: 03-35.001) and #10 (purple trace, label: 04-52.001) with neighbouring data sets, namely the data set #7 (blue trace, label: 03-31.001) collected right before the set#8, data set #9 (green trace, label: 04-30.001) collected between the sets #8 and #10, and the data set#11 (yellow trace, label: 05-15-001) collected right after the set#10. The quality for the date sets #8 and 10 is apparently worse than the three neighbouring sets, and not good enough in quality to provide accurate E0, therefore not included in the data analysis.

4. Discussion

Based on the above observation, following discussion can be made regarding E0 drifting:

- Slope of E0 drift is not only storage ring current related, but also ring current decay rate dependent.
 - After injection at least 30 min is required to let the mono crystal reaches their thermal “equilibrium”;
 - The equilibrium between the monochromator LN₂ cooling and the thermal loading on the mono crystal is dynamic in nature, and will be disrupted when the ring current is lower than a certain level, because the total cooling power from the LN₂ is constant. The later was determined by the pumping rate and pressure set at LN₂ cryocooler. Normally the setup at cryocooler is not changed during operation.
- The time required for the equilibrium t will be longer if the injection last longer (or shutter closed longer);
- The equilibrium is also determined or at influenced by the following factors
 - the wiggler operation condition;
 - the ring operation condition;
 - BL primary slits alignment, and their opening;

- With the graphite filter(s) in the beam path upstream of the monochromator, and the number and their combination;
- With the collimating mirror in the beam path, which mirror stripe is in used, and the pitch angle of the incident beam to the mirror as well;
- Which monochromator crystal used, and what working energy range used during data collection;
- The vacuum condition of the BL section from monochromator upstream to the BL primary slits/graphite section might also have impact the equilibrium process;
- The ring life time, namely the ring current decay rate, has direct impact to the equilibrium process.

5. Procedure

If the XANES/XAFS experiment is performed beyond the monochromator E0 stabilized segment and extended into low current wing of the E0 performance region, following procedure will provided in-step energy calibration which has been available at BL:

- Transmission mode: also set up standard sample between I1 and I2;
- Fluorescent mode:
 - also set up standard sample between I1 and I2 if there is enough flux seen by the standard;
 - when the single element Vortex is used in the experiment, and CLS data acquisition software IDA is applied for data collection, a standard sample should be set up on top of the photo diode for detune purpose. Therefore the signal form the diode associated the signal from I0 can provide in-step energy calibration;
 - when 32 element Ge detector is used in the experiment, and SSRL data acquisition software is applied for data collection, then the standard sample should be set up again on top of photo diode for detune purpose, and a second photo diode also set up at WB pipe end will provide I0 for the in-step energy calibration.

6. Conclusion

HXMA mono energy calibration (Si220 investigated for this test run) is stabilized half hour after the storage ring injection, and the crystal keeps stable for a ring current range between ~ 244 -184 eV. It is expected that there might be error bars at the upper and lower end of this estimated stabilized range because of the data availability (time is required for the XANES data scan).

Based on the observation include herein, shortening the injection time, improving in the storage ring electron beam lift time, and introducing the injection top-up mode (or injection without close BL frontend photon shutter) will improve monochromator performance at HXMA.