Compact XAFS Spectrometer

- # Background

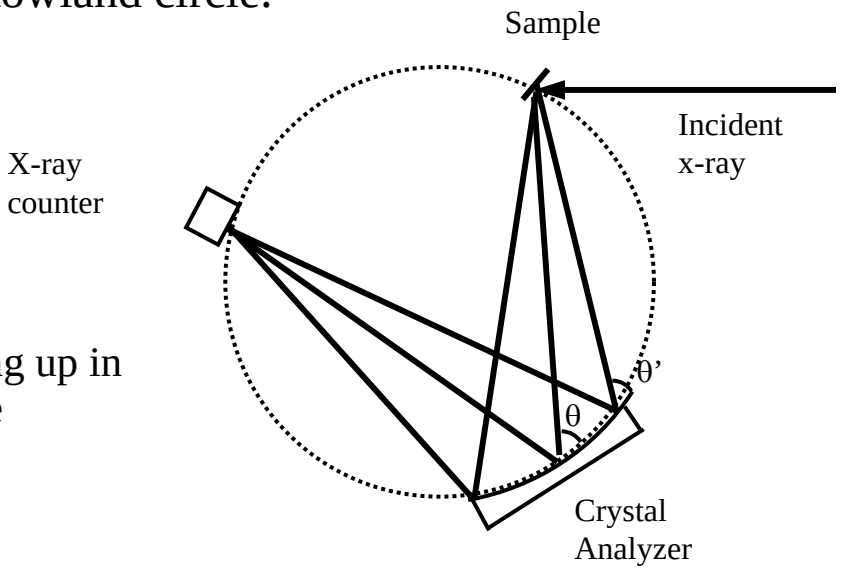
Current XAFS instruments include ion chamber, scintillation detector, germanium detector, and silicon draft detector. But they all have low energy resolution or none. For some measurements, such as in case of complicated combined samples and requiring extra low background measurements, high energy resolution better than 60eV is demanded. The crystal analyzer based spectrometer is the only choice for those requirements today.

Detector	Energy resolution
Ion chamber	None
Scintillation	keV range
Germanium	300 eV
Si Draft	130 eV

1. Principle

The spectrometer is based on spherically bent single crystal analyzer which can diffract x-ray energy on Bragg angles. Each Bragg angle refers to each energy with some bandwidth (also called energy resolution of the analyzer) . When we tune the Bragg angle to the energy of target fluorescence line, the detector, or x-ray counter can only receive the target fluorescence signal. If we fix the Bragg angle of the spectrometer and scan the incident x-ray beam energy, we can get XAFS spectrum in fluorescent model.

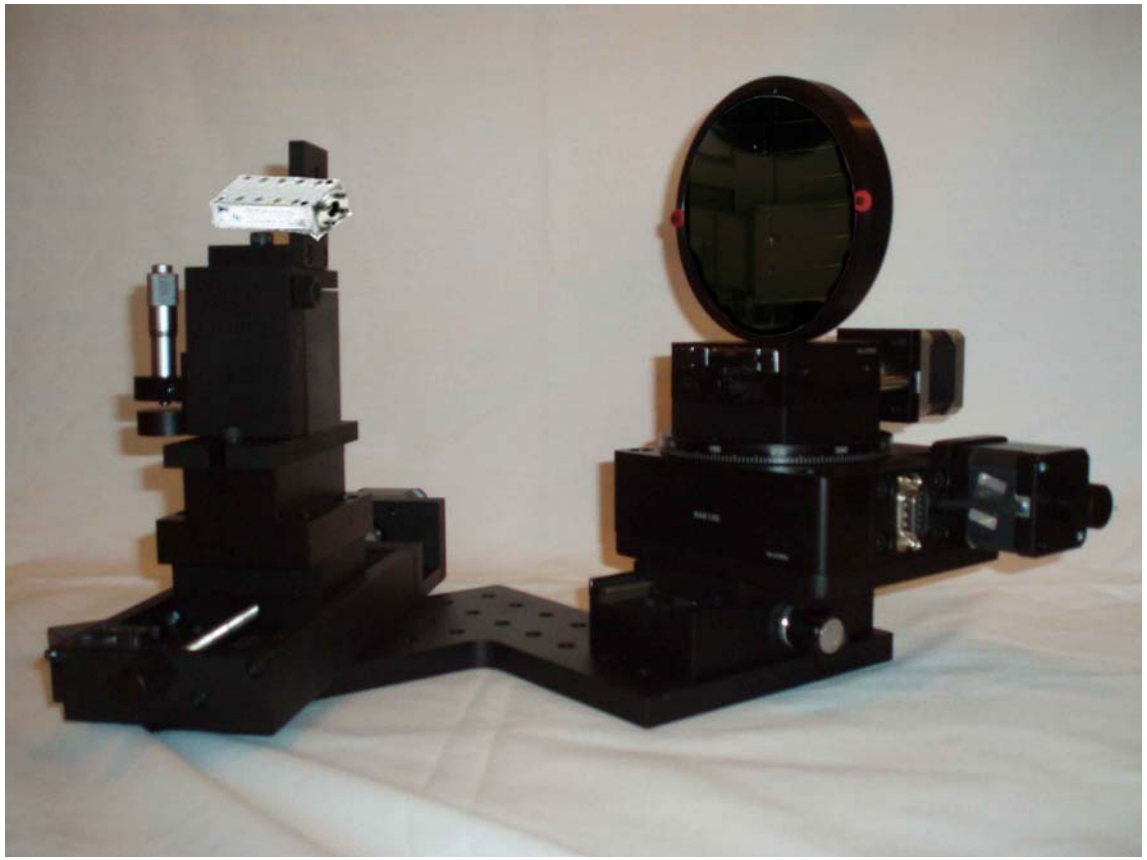
Normally, the sample, crystal and detector are set on Rowland circle to get the best energy resolution, where crystal bent radius is equal to the diameter of this Rowland circle.



Spectrometer setting up in Rowland circle

2.mini-spectrometer

The spectrometer consists of a crystal analyzer, a detector and attached motion stages/rotators, which can adjust their positions to fit in Rowland circle and expected Bragg angles. The sample holder is not included since it mostly sits on beamline. The crystal is 100mm diameter size, and bent to 182mm radius. Its entry solid angle is 2% of 4π. The spectrometer physical size is about 300mm x 300mm x 300mm.



3.spectrometer properties

- The most important property of the spectrometer is its energy resolution, or its crystal analyzer energy resolution. It is varied with Bragg angles due to bending distortion and geometry distortion.
- There is relation between x-ray energy resolution and Bragg angle.
  - $\Delta E=E\Delta\Theta/\tan\Theta$

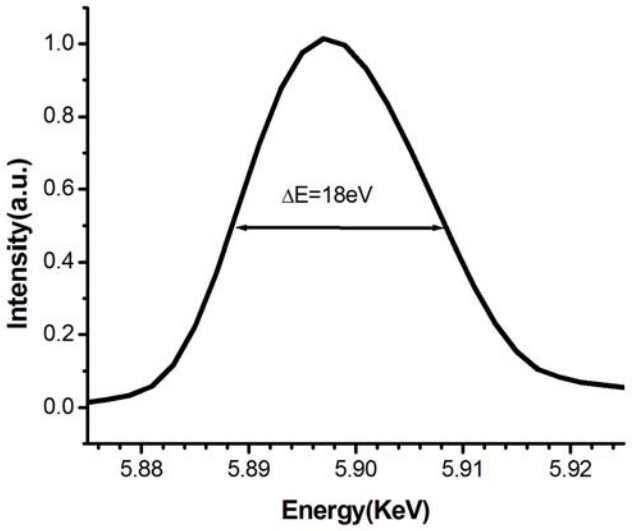
  The larger Bragg angle, the better energy resolution.

  For different target element there is different fluorescence energy, one need to choose suitable orientation crystal to match its Bragg law to maximum the Bragg angle in order to get better energy resolution.

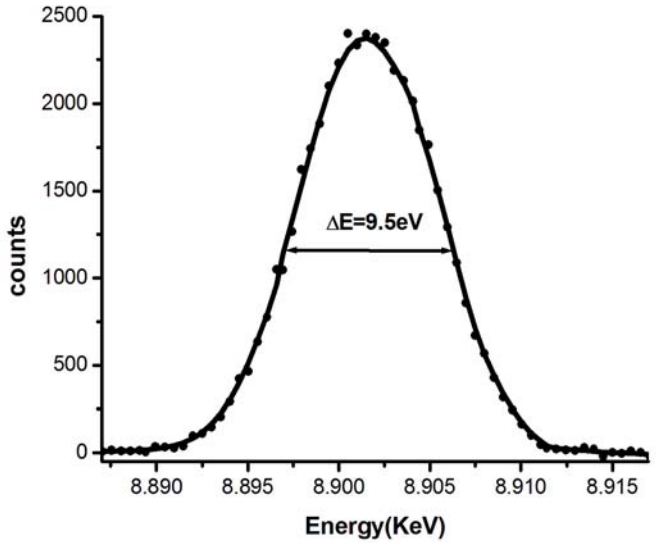
  We normally run elastic scan on synchrotron beamline to measure spectrometer energy resolution.
- For example, if we want to measure Mn K-edge XAFS, we need monitor Mn K-alpha line with this spectrometer. Its energy is around 5900eV, the suitable Si crystal orientation is (422) which gives the largest Bragg angle 71.4 deg. (note, we can only bend Si currently). This bent crystal analyzer can give energy resolution about 18eV at this energy, which can cover both Mn K-alpha1 and K-alpha2.

4.Energy resolution of the mini-spectrometer

- Si(422) elastic line at E=5900eV



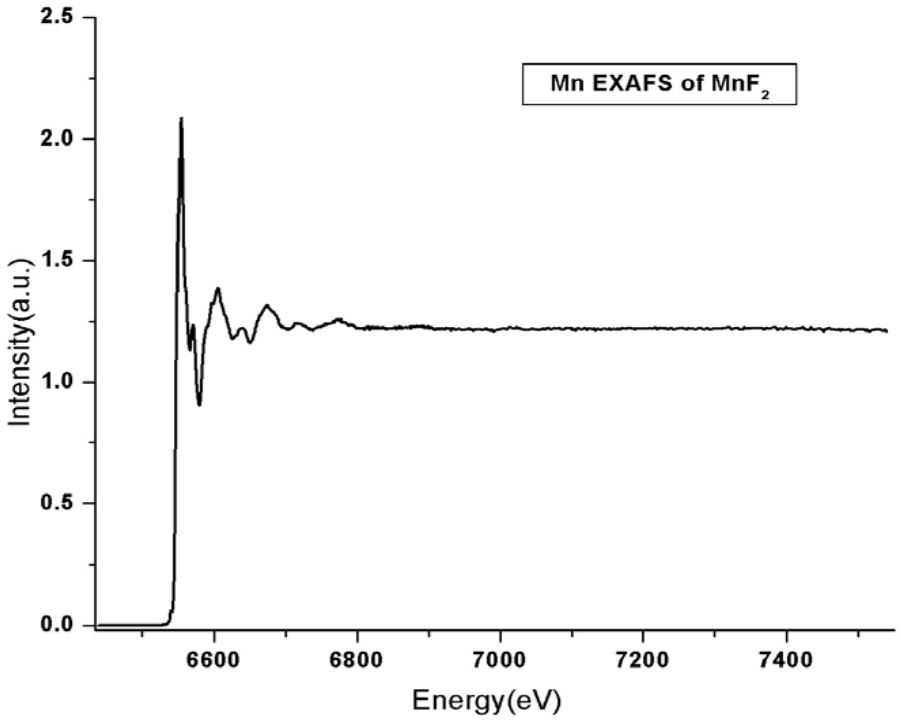
- Si(553) elastic line at E=8904eV



X-ray energy falls in these range can be diffracted to detector. So energy resolution ΔE, can also be regarded as energy through bandwidth, or energy window for this spectrometer. Typical energy resolution for most of the ten third row transit element K-edges is around 4eV to 40eV.

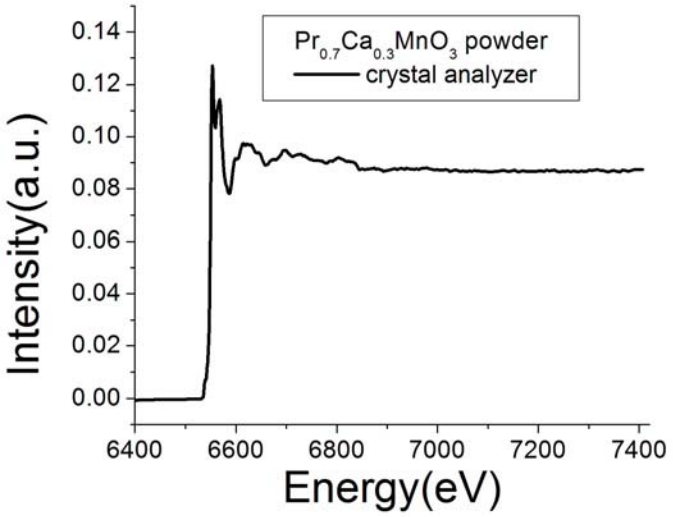
5.XAFS measurement

- Mn K-edge of MnF_2 by Si(422), signal count rate at 200k/sec above edge with x-ray beam intensity at 10^{10} photons/sec

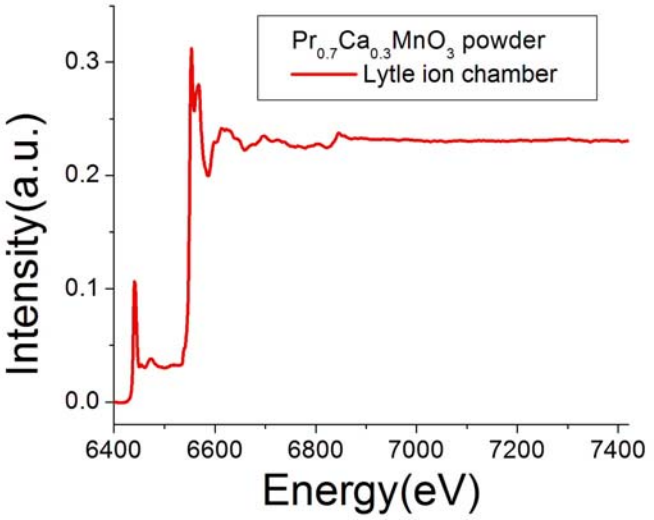


Mn-K edge of powder $Pr_{0.7}Ca_{0.3}MnO_3$

- Measured with mini-spectrometer with Si(422)



- Measured with Lytle ion chamber



Note there is low energy side peak at right panel from Pr L edge

- # Conclusions

- Mini-spectrometer can offer 4eV to 40eV energy resolution or energy window for most third row transit elements. Its good energy resolution can maximally reduce/eliminate background from environment.
- If analyzers can separate K-beta main peak and its satellite peak(normally 16eV lower than main peak), one can measure spin selected XAS(SSXAS) since these two peaks come from different spin states.(ref: V60, 4665, Physical Review B, 1999).
- The spectrometer has big entry solid angle due to its small bent radius and big size in order to get most fluorescence signal from samples. It is better suitable to the third generation synchrotron facility due to its much higher intensity beam.
- The spectrometer's compact size can let it to be mounted easily and combined to other instruments in using, such as Hube diffractometer.