Energy Dispersive Spectroscopy (EDS) Calibration in TEAM™

When environmental conditions (e.g. temperature, humidity) are controlled within narrow limits, EDS detectors are very robust and calibration hardly shifts over very long time periods.

Calibration of an EDAX EDS detector is done to adjust the energy scale for the EDS spectrum, making sure PeakID does a perfect job identifying elements correctly. This is the only calibration that a customer might have to do. No calibration is needed for EDS quantification, as this is done standardless by default.

Calibration is needed when either PeakID is incorrect (for instance, if FeKα is misidentified as CoKα) or when the fitted spectrum (Halographic Peak Deconvolution (HPD) trace) does not exactly match the peaks. In some specific cases, there might be a little shift, which is caused by elements overlapping in EDS (Figures 1 and 2).
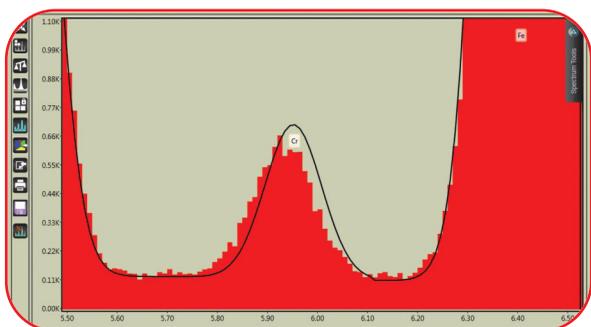

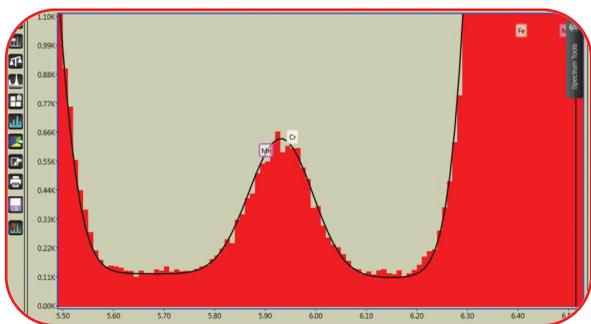*Figure 1. A steel sample with only Cr identified (HPD misfit).*

Figure 2. Same steel sample with CrKβ and MnKα (overlapping) identified (perfect HPD fit).

Calibration is done using an Al+Cu sample. This is because copper tape is usually present in an electron microscopy lab and the stub material is Al. The big advantage of using Al and Cu is that the AlKα and CuKα peaks are far apart and will give a very accurate calibration.

Procedure

• Put the Al+Cu sample in the microscope and pump down.
• Set the high voltage to 25 or 30 kV.
• Use > 500x magnification and spot size and/or aperture to get count rates around 10,000-20,000 cps.

• Make AlKα and CuKα peaks roughly the same height.
• Go to Advanced Properties and select Spectrum Calibration. Check that Peak 1 (AlKα) is set to 1487 eV and Peak 2 (CuKα) to 8041 eV.
• Set the number of counts to 10,000-20,000.
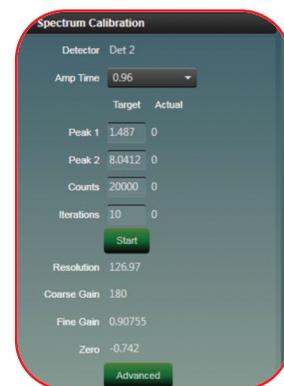• Choose the amp time and press Start (Figure 3).

Figure 3. Calibration panel in TEAM™.

Calibration should typically be finished using 2-3 iterations. Do this for all seven amp times. If it is not calibrated after 10 iterations, another 10 can be run.

Resolution at MnKα (peak width at half of peak height, called the Full Width Half Max (FWHM) value) is calculated from AlKα and CuKα peak width values. When checked by measuring MnKα, one often sees resolution is even a bit better than this calculated value.

Calibrations done in TEAM™ are saved in a logging file under c:/ProgramData/EDAX called calibrat.csv. It is good to check calibration every few weeks.

When a peak is seen at the very beginning of an EDS spectrum (often misidentified as BKα), it means the BLM (noise level) has not been adjusted properly. To solve this, go to the calibration section and click on the advanced button. Increase the BLM using small steps (increments of 1-2) until this peak is gone.

Figure 4 shows the BLM set incorrectly (at 18, in cyan) and the BLM being set correctly (at 23, in red). Note that the BLM is different for every amp time.
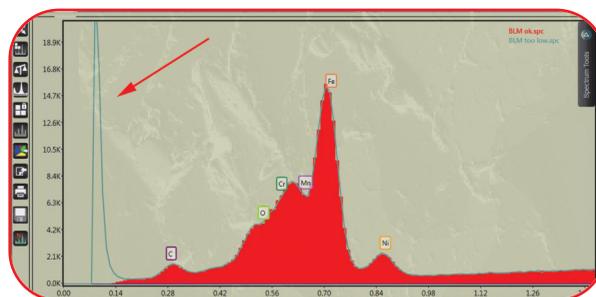

Figure 4. BLM set incorrectly (cyan) and set correctly (red).

EDAX
Smart Insight