XPS – BE Calibration and Atom % Accuracy Checks

All We Need is Copper (Cu)

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This poster explains that BE Calibration and Atom % Accuracy can be quickly and reliably checked by using just one piece of freshly cleaned, sputtered, (99.9%) Copper (Cu) for both tasks.

Existing methods are solid, but are slow and time consuming, and therefore seldom used. The end result of this consensus is that published BEs and atom % values are unnecessarily inaccurate.

As evidence that the problem continues, please review the Statistical Analyses of the BEs shown in Periodic Table format. The results were derived from the on-line version of NIST Database of BEs v3.5. The NIST database and other collections of BEs all suffer from the use of maker recommended calibration values (i.e. Cu 2p3 ranges from 932.2 eV to 932.8 eV), and the assumption that the hydrocarbon C 1s BE should equal (~) 284.8 ±0.2 eV. The evidence of this still ongoing problem is revealed in a Histogram.

To escape the BE error problem, and to publish, have and use reliable XPS BEs to assign chemical states reliably, we must start to check and calibrate our Energy Scales, so a fast and reliable method is needed.

The Atom % (TF) Check:
After collecting, either one routine, wide-scan survey spectrum (0-1000 eV), or two smaller, wide-scan spectra (i.e. 50-350 eV and 900-1000 eV), we can check Atom % Accuracy by generating atom % values from two or more of the major XPS signals produced from freshly cleaned and strongly sputtered (99.9%) Copper (Cu).

If the Transmission Function (TF) or Instrument Response Function (IRF) is setup correctly, and we are using Scofield’s theoretically calculated, relative sensitivity factors (RSFs), then each Copper peak must have the same atom % value within 10% of that value. If any of the Atom % values differ by ≥12%, then there is, most likely, a problem.

The BE Calibration Check:
Check BE Calibration by using the routine conditions used to separate and identify different chemical states. If more than one pass energy and step size (i.e. step) is routinely used, then consider checking those settings at least one time.

Check the Energy Scale by measuring the Cu 2p3 peak (932.67 eV) and either the Cu 3s peak (224.52 eV) or Cu 3p3 peak (75.14 eV) from pure (99.9%) Copper metal that was freshly cleaned (i.e. sputtered) and strongly sputtered.

If any BEs are wrong by more than ~0.15 eV (practical conditions), we can use the measured BEs to adjust the work function (energy offset), energy scale factor (DAC) or pass energy of that XPS instrument.

Each time one instrument voltage is adjusted, the Cu 2p3 and Cu 3p3 (or Cu 3s) must be re-measured.

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Summary of BE Uncertainties
Ag 3d5/2 as Listed in NIST SRD v3.5 On-Line Database of BEs vs. v3.5 On-Line (free)

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