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Technical Note 2015-03

Guidelines for Calibration of ED-XRF Spectrometers for Sub-Microgram Measurements Using Micromatter Reference Materials

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Micromatter has manufactured XRF calibration standards and reference samples for several decades. Our products contain highest quality materials, such as ultra-pure metals or stable inorganic compounds, which are deposited onto polyester or track-etched polycarbonate by evaporation methods.

Micromatter standards are NIST Traceable Reference Materials™. For details about traceability, please refer to Micromatter Technical Note 2015-01. General considerations for energy and intensity calibration of XRF spectrometers are outlined in Micromatter Technical Note 2015-02.

Recommendations for Quantitative Trace Analysis in the Sub-Microgram Range

XRF Spectrometry is the method of choice for multi-elemental analysis of traces of materials. In the field of air quality monitoring, very low particulate concentrations need to be determined with high accuracy.

Micromatter standards are pure elements or compounds, deposited onto polymer films by ultra-high vacuum techniques, such as resistance heating or electron beam evaporation. The contents of the metal or compound is given in units of area weight ($\mu g/cm^2$), as commonly used in the thin film industry. Users can calculate the thickness of the deposited layer using the theoretical density of the respective bulk material.

Reliable quantitative results can be obtained by calibrating a spectrometer with reference materials of different area weight. Following the general rule that 'Interpolation is better than Extrapolation', standards that cover the expected analytical range should be employed.

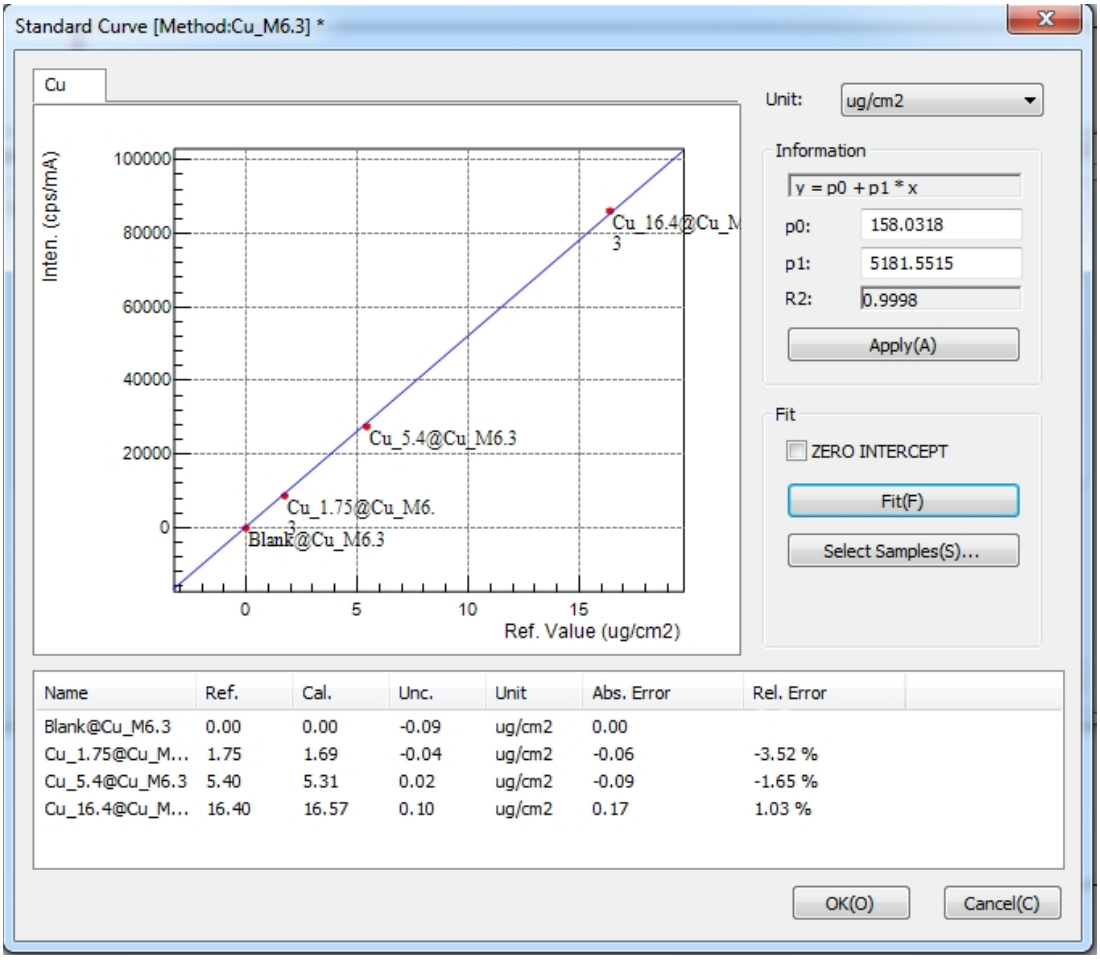

Fig. 1: Typical calibration diagram for light mono-element standards

For sub-microgram applications, we recommend recording a calibration curve such as the one shown above using standards of 15 µg/cm^2, ~5 µg/cm^2, ~1 µg/cm^2 as well as a blank membrane (0 µg/cm^2). This method has been validated to give reliable results for sub-microgram levels.

Micromatter generally advises against attempts to calibrate with ultra-light reference materials (0.1 µg/cm^2 or less) as they cannot be characterized by a primary method such as weighing with the same precision as regular standards. An additional calibration point in this weight range may in fact increase the discrepancies between instrument readings and actual sample loadings.