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Technical Note 2015-01A

Quantification and NIST Traceability of Micromatter Thin Film Standards for XRF Analysis

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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™.

Production Process and Characterization of Micromatter Thin Films

Micromatter standards are exclusively manufactured from 99.9% or higher purity metals or compounds by ultra-high vacuum deposition methods. Materials with low melting/boiling point are volatilized by resistance heating using tantalum or tungsten crucibles. High melting materials, such as the platinum metals, are generally evaporated by electron beam.

Evaporation processes entail the transfer of heat to the backing foils (substrates), which need to consist of thermally stable polymers. Mylar® polyester films or Nuclepore® polycarbonate membranes show excellent heat tolerance and consistency and are hence preferable to e.g. Teflon®.

Micromatter standards are characterized by precision gravimetry. Each individual backing disc is weighed before and after the deposition of the metal or compound with an accuracy of one tenth of a microgram (0.1 µg). The area density in $\mu g/cm^2$ is then calculated by dividing the mass of the deposit by its area. Finally, the standard is mounted according to the customer's specification.

Precision weighing at Micromatter is performed on Mettler analytical balances (Mettler M3 and Mettler ME30; Figure 1), which are calibrated routinely with an ANSI/ASTM/OIML/NIST compliant set of weights as per Micromatter's Quality Assurance Program.

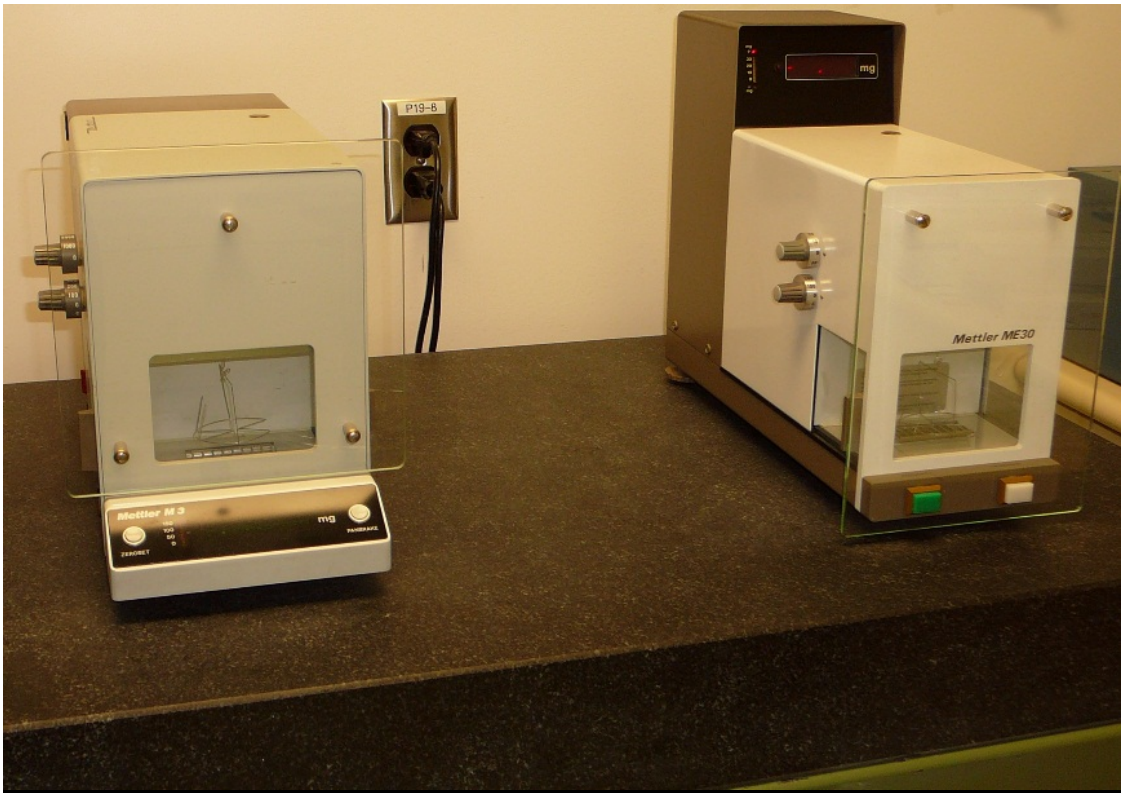

Fig. 1: Micromatter Precision Balances

NIST Traceability of Micromatter Standards

What is traceability, and how is it achieved?

The definition of traceability[1] accepted by the global metrology community is contained in the International Vocabulary of Metrology as the *"… property of a measurement result whereby the result can be related to a reference through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty. …."* [2]

It is worth emphasizing that traceability is the property of a measurement, not of an instrument, a calibration report, a laboratory or an institution. Merely having an instrument calibrated, even by NIST, is not sufficient to ensure traceability.

Micromatter strictly follows a measurement system that fulfills the elements of traceability as described in Ref. 1, Section I.A.5.

Which definitions does NIST use to describe various classes of reference materials, and which one applies to Micromatter XRF Standards?

Reference Material (RM) - Material, sufficiently homogeneous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in a measurement process

1. RM is a generic term.

2. Properties can be quantitative or qualitative, e.g. identity of substances or species.

3. Uses may include the calibration of a measurement system, assessment of a measurement procedure, assigning values to other materials, and quality control.

4. A single RM cannot be used for both calibration and validation of results in the same measurement procedure.

5. International Vocabulary of Metrology – Basic and General Concepts and Associated Terms has an analogous definition[3], but restricts the term "measurement" to apply to quantitative values and not to qualitative properties. However, Note 3 of ISO/IEC Guide 99:2007, 5.13, specifically includes the concept of qualitative attributes, called "nominal properties".[4]

Certified Reference Material (CRM) - Reference material characterized by a metrologically valid procedure for one or more specified properties, accompanied by a certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability.

1. The concept of value includes qualitative attributes such as identity or sequence. Uncertainties for such attributes may be expressed as probabilities.

2. Metrologically valid procedures for the production and certification of reference materials are given in, among others, ISO Guides 34 and 35.

3. ISO Guide 31 gives guidance on the contents of certificates.

4. VIM has an analogous definition.[5]

Reference Material Certificate - Document accompanying a certified reference material stating one or more property values and their uncertainties, and confirming that the necessary procedures have been carried out to ensure their validity and traceability.

NIST Standard Reference Material® (SRM) - A CRM issued by NIST that also meets additional NIST-specific certification criteria and is issued with a certificate or certificate of analysis that reports the results of its characterizations and provides information regarding the appropriate use(s) of the material (NIST SP 260-136). Note: An SRM is prepared and used for three main purposes: (1) to help develop accurate methods of analysis; (2) to calibrate measurement systems used to facilitate exchange of goods, institute quality control, determine performance characteristics, or measure a property at the state-of-the-art limit; and (3) to ensure the long-term adequacy and integrity of measurement quality assurance

programs. The terms "Standard Reference Material" and the diamond-shaped logo which contains the term "SRM," are registered with the United States Patent and Trademark Office.

NIST Reference Material - Material issued by NIST with a report of investigation instead of a certificate to: (1) further scientific or technical research; (2) determine the efficacy of a prototype reference material; (3) provide a homogeneous and stable material so that investigators in different laboratories can be ensured that they are investigating the same material; and (4) ensure availability when a material produced and certified by an organization other than NIST is defined to be in the public interest or when an alternate means of national distribution does not exist. A NIST RM meets the ISO definition for a RM and may meet the ISO definition for a CRM (depending on the organization that produced it).

NIST Traceable Reference Material™(NTRM™) - A commercially-produced reference material with a well-defined traceability linkage to existing NIST standards for chemical measurements. This traceability linkage is established via criteria and protocols defined by NIST to meet the needs of the metrological community to be served[6]. Reference materials producers adhering to these requirements are allowed use of the NTRM trademark. A NIST NTRM may be recognized by a regulatory authority as being equivalent to a CRM .

Micromatter XRF standards belong to the category of 'NIST Traceable Reference Materials'. Their traceability is validated through Micromatter's Quality Assurance Program, which is based on a primary measurement method (precision weighing). Micromatter has traditionally used the term 'Micromatter XRF Standard' for its products. Although our standards fulfill NIST requirements, we do not use the NTRM trademark.

How does e.g. NIST's Standard Reference Material for air particulates on filter matter (SRM® 2783) compare to Micromatter XRF Standards?

NIST SRMs® are manufactured to closely resemble naturally occurring substances, such as plant parts or soil, or typical samples found in environmental measurements (pollution control). SRM®2783 is a certified multi-element sample on a porous polycarbonate air sampling membrane that simulates PM2.5 air particulate matter (particles with an aerodynamic equivalent diameter of 2.5 μm). It is primarily intended for use in the evaluation and calibration of methods of analysis for common and toxic elements contained in various fractions of airborne particulate matter collected on filter media.[7]

Micromatter's product philosophy differs from NIST's in that we do not attempt to produce reference materials that simulate the sample to be analyzed. Our XRF standards contain single elements or compounds and are intended be used for the calibration of spectrometers independent of matrix effects and sample composition.

Several elements in NIST's SRM®2783 are verified by at least two analytical methods, one of them being energy-dispersive X-ray fluorescence analysis (EDXRF). In turn, EDXRF spectrometers are commonly calibrated using Micromatter XRF Standards[8].

In conclusion, in the analytical practice Micromatter XRF Standards and NIST SRMs® can be used as complementary calibration and validation materials. See Ref. 9 for a practical example of a Standard Operating Procedure involving both Micromatter Standards and NIST SRMs®.

References

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2. JCGM 200:2008, International vocabulary of metrology – Basic and general concepts and associated terms. VIM Third Edition (2008).
3. VIM - ISO/IEC Guide 99, 5.13 (2007).
4. ISO Guide 30:1992/Amd 1 (2008).
5. ISO/IEC Guide 99, 5.14 (2007).
6. NIST SP 260-136 - Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements (2000).
7. NIST SRM®2783 Certificate of Analysis (2011).
8. 40 CFR Part 50, Appendix Q to Part 50 - Reference Method for the Determination of Lead in Particulate Matter as PM10 Collected From Ambient Air. Section 6.2: Analysis.
9. RTI International – Standard Operating Procedure for the X-Ray Fluorescence Analysis of Particulate Matter Deposits on Teflon Filters. Revision 5 (2009).