

## Standardization and Metrology for Efficiency and Reliability in Microbeam Analysis – No pain, no gain

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Standardization and metrology are two terms which are used rather rarely at major conferences as well as in scientific publications in the field of microbeam analysis. For laboratories operating under an accreditation scheme the operator of the microscope/microprobe must have available internal, national or international written standards which should be applicable to any quantitative analysis. Hence, requirements, specifications, guidelines or characteristics of methods, instruments or samples are provided with the final goal that these can be used consistently. In this way it is ensured that microbeam analyses results are reliable and meet quality-management requirements [1].

Standardization organizations operating at national level such as ANSI (USA), BSI (GB), DIN (Germany), SAC (China) or JISC (Japan) synchronize their activities to the international organization for standardization, ISO. In the field of microbeam analysis the ISO technical committee TC 202 is in charge with the harmonization of technical specifications of products and services making industry more efficient and breaking down barriers to international trade. Since the TC 202 establishment in 1991 eighteen ISO standards including updates have been published. The current list and the projects in development is updated on the ISO webpage [2], see Fig. 1. A main and continuous task of TC 202 and its subcommittees is to identify and evaluate feasible projects/proposals needed to be developed into new international standards. A short overview of the ongoing projects will be given.

As regards the metrological aspects, The International Bureau of Weights and Measures (BIPM) through the Consultative Committee for the Amount of Substance (CCQM) is concerned with the metrological aspect in Chemistry and Biology. The development of the technical and organizational infrastructure of the International System of Units (SI) as the basis for the world-wide traceability of measurement results occurred here [3]. In particular, the Surface Analysis Working Group (SAWG) assists in identifying and establishing inter-laboratory work to test the consistency as well as to improve the traceability of spatially resolved chemical surface analysis at the micro and nanoscale. Examples of recent and ongoing projects related to microbeam analysis will be presented. The relevance of the evaluation of the complete chain of measurement uncertainties towards ensuring the traceability and comparability of the results will be emphasized through examples [4]. Thereby the compliance of the results of the microbeam analysis with the ISO document “Guide to the expression of uncertainty in measurement (GUM)” [5] will be explained.

Another international platform in the frame of which pre-standardization work can be organized is VAMAS (Versailles Project on Advanced Materials and Standards) [6]. International collaborative projects involving mainly national metrological institutes aim at providing the technical basis for

harmonized measurements, testing, specifications, and standards. One key point of VAMAS activities is constituted by inter-laboratory comparisons. In the field of microbeam analysis the technical working area (TWA) 37 Quantitative Microstructural Analysis as well as TWA 34 Nanoparticle Populations deal with corresponding projects.

The contribution is meant not only to give a short overview of the existing standardisation and metrology activities in the field of microbeam analysis, but to bring possibilities and needs closer to the users. Especially younger personnel should be aware of these rather disliked parts of the microbeam analysis. At the same time motivation of conference participants to generate potential new projects is a hidden scope of the present contribution.

#### References:

- [1] ISO/IEC 17025 (2005) General requirements for the competence of testing and calibration laboratories. ISO, Geneva.
- [2] <http://www.iso.org> (Standard developments / Technical Committees / ISO/TC 202 Microbeam analysis).
- [3] <http://www.bipm.org/en/committees/cc/ccqm/>
- [4] V-D Hodoroaba et al., Microsc. Microanal. 20 (Suppl 3), 2014, p. 730.
- [5] ISO/IEC Guide 98-3:2008 Uncertainty of measurement Part 3: Guide to the expression of uncertainty in measurement (GUM:1995), Geneva: Internat. Org. Stds, 101 pp.
- [6] <http://www.vamas.org/>

#### ISO/TC 202 - Microbeam analysis

Items to be displayed:

Published standards  Standards under development  Projects deleted (last 12 months)

#### Standards and projects under the direct responsibility of ISO/TC 202 Se

Standard and/or project
<input checked="" type="checkbox"/> ISO 15632:2012 Microbeam analysis -- Selected instrumental performance parameters for the specification and checking of energy-dispersive X-ray spectrometers for use in electron probe microanalysis
<input checked="" type="checkbox"/> ISO 14595:2014 Microbeam analysis -- Electron probe microanalysis -- Guidelines for the specification of certified reference materials (CRMs)
<input checked="" type="checkbox"/> ISO 14594:2014 Microbeam analysis -- Electron probe microanalysis -- Guidelines for the determination of experimental parameters for wavelength dispersive spectroscopy
<input checked="" type="checkbox"/> ISO 29301:2010 Microbeam analysis -- Analytical transmission electron microscopy -- Methods for calibrating

#### ISO/TC 202 - Microbeam analysis

Items to be displayed:

Published standards  Standards under development  Projects deleted (last 12 months)

#### Standards and projects under the direct responsibility of ISO/TC 202

Standard and/or project
<input checked="" type="checkbox"/> ISO/CD 16700 Microbeam analysis -- Scanning electron microscopy -- Guidelines for calibrating image magnification
<input checked="" type="checkbox"/> ISO/AWI 19463 Microbeam Analysis -- Quality assurance procedures and reference materials for the calibration and diagnostic assessment of electron probe microanalysis instruments utilizing wavelength dispersive spectrometers
<input checked="" type="checkbox"/> ISO/DTR 19640 Guide to practical chemical state analysis in EPMA
<input checked="" type="checkbox"/> ISO/AWI 20171 Tag image file format for Scanning electron microscopy(TIFF/SEM)
<input checked="" type="checkbox"/> ISO/WD 20263 Microbeam Analysis -- Analytical transmission electron microscopy -- Methods for

**Figure 1.** Screenshots with standards published by ISO/TC 202 (left) and with standards under development (right) [2].