



# Introductory notes for the Acta IMEKO thematic issue on the 2023 SBM Metrology Conference – part 2

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Dear Readers,

This special issue of Acta IMEKO continues the dissemination of selected contributions presented at the 2023 SBM Metrology Conference, highlighting recent advances in measurement science and its diverse applications. The 15 papers collected here showcase innovative research in areas ranging from chemical and environmental metrology to industrial processes and health-related measurements. Together, they reflect the growing relevance of metrology in supporting technological development, regulatory frameworks, and societal needs. By sharing these peer-reviewed studies, this issue reinforces the role of measurement as a foundation for scientific progress, quality assurance, and international collaboration across multiple disciplines.

The study presented in [1] evaluates Tip-Enhanced Raman Spectroscopy (TERS) as a metrological tool for determining the number of graphene layers at the nanoscale, with emphasis on transitions between single-layer graphene (SLG) and bilayer graphene (BLG). Although widely used, conventional micro-Raman spectroscopy is limited by diffraction, leaving regions of uncertainty in distinguishing adjacent SLG and BLG domains. By combining optical and scanning probe techniques, TERS overcomes these limitations, achieving a spatial resolution of ~60 nm, compared to ~500 nm for micro-Raman. Through hyperspectral line profiling and mapping, we demonstrate that TERS enables clear clusterization of data into SLG and BLG categories, using the intensity ratio ( $I_{2D}/I_G$ ) and spectral positions of the G and 2D bands as criteria. These results highlight TERS's enhanced reliability and precision in scenarios where micro-Raman yields inconclusive measurements. Moreover, systematic comparison reveals that TERS substantially reduces the indetermination region and provides robust hyperspectral images at the nanoscale. The findings reinforce the potential of TERS as a complementary technique

to current ISO/TS 21356-1:2021 standards, supporting its future incorporation for graphene characterisation. This work thus positions TERS as a powerful tool for advancing metrological approaches to 2D material analysis.

Paper [2] presents the development of a low-cost, mobile network-based system for environmental radiation monitoring in Brazil, addressing the absence of a nationwide surveillance infrastructure. The proposed device integrates a Geiger counter, ecological sensors, GPS, and a microcontroller with mobile connectivity, enabling autonomous data collection and transmission without user intervention. Mounted on vehicles from public and private fleets, these mobile stations can cover vast geographic areas, providing real-time geolocated radiation measurements. A preliminary study collected more than 180,000 readings, demonstrating the feasibility of continuous large-scale monitoring. Calibration against standard sources confirmed the system's accuracy. At the same time, post-processing techniques ensured reliable data visualisation despite challenges with network connectivity, and adaptive solutions, such as multiple APN lists, improved performance. The results highlight the potential of this approach as a citizen science-inspired framework for radiation protection, combining low-cost technology, existing mobile infrastructure, and participatory data collection to enhance public safety and preparedness.

Authors of paper [3] discuss the legal control of moisture meters for cereals, grains, and oilseeds in Brazil, highlighting their critical role in fair trade and agricultural quality assurance. As commodities such as soybeans, corn, rice, beans, and coffee represent a significant share of Brazilian exports, accurate moisture measurement is essential for determining grain value and preventing deterioration during storage. Following the 2013 national regulation based on OIML R 59, Brazil has been consolidating a legal metrology infrastructure, including verification laboratories, manufacturers, and repairers. Data from 2017 to 2024 show steady growth in type approvals, initial verifications, and after-repair verifications, reflecting increasing

compliance despite the provisional period extending until 2028. The air-oven method remains the reference standard, though it poses logistical and operational challenges for widespread verification. Ongoing studies explore alternatives to improve efficiency, including moisture meters as working standards. The results underscore the importance of robust metrological control to ensure transparency, fairness, and economic stability in agricultural trade.

The reliability of commercial caesium standards is crucial for maintaining atomic time scales, as instabilities can compromise navigation, telecommunications, and scientific applications, as proposed in [4]. This study investigates statistical monitoring methods to detect anomalies in caesium standards used in the Brazilian atomic time scale. Data from minute-by-minute records were analysed using multivariate and univariate control charts, specifically Hotelling's  $T^2$  and Exponentially Weighted Moving Average (EWMA). Principal Component Analysis (PCA) was applied to reduce dimensionality, while preserving variance information. Results demonstrated that Hotelling's  $T^2$  detected the first anomaly on January 1, 2019, well before significant instability appeared in the Allan deviation, an established frequency stability metric, 18 days later. EWMA charts further identified which parameters deviated first, with the E-multiplier emerging as the earliest indicator. The combined approach provided an effective early-warning system, enabling corrective actions before severe degradation of clock performance. These findings highlight the potential of statistical process control for ensuring robust and predictive monitoring of atomic timekeeping systems.

Paper [5] examines the suitability of a surface roughness measurement process through a comparative study using Measurement System Analysis (MSA) and the recently developed  $C_{dl}^*$  capability coefficient. Ten carbon steel samples were prepared by planning, and two trained metrologists measured roughness ( $R_a$ ) under controlled conditions. Based on repeatability and reproducibility (R&R), the MSA study revealed a high %R&R ( $\approx 86\%$ ), classifying the process as unsuitable. Parallel evaluation with the  $C_{dl}^*$  coefficient, which integrates systematic and random errors, while incorporating calibration uncertainty, confirmed inadequacy with values below the accepted threshold of 1.33 across all datasets. Both approaches consistently pointed to deficiencies in the measurement process, likely linked to variability introduced during machining, rather than instrument instability. Control chart analysis further indicated that the manufacturing process was not under statistical control. These findings validate the  $C_{dl}^*$  coefficient as a robust and complementary tool to MSA for assessing the reliability of measurement processes in industrial metrology.

The following paper presents a preliminary methodology for quantifying biodiesel content in diesel blends using ultraviolet-visible (UV-Vis) spectroscopy, aiming at applications in optical sensors for online fuel monitoring [6]. Absorbance measurements in the 400–500 nm range revealed a linear correlation between the optical signal and biodiesel concentration (0–20 %), with the strongest performance around 450 nm. Calibration curves were constructed from blends prepared with diesel and biodiesel components from different suppliers, and their quality was validated using multivariate Partial Least Squares Regression and cross-validation. The study highlights the influence of both biodiesel feedstock and diesel type (S10 or S500), the latter containing a compulsory dye that shifts spectral response. Despite these variations, consistent correlations were achieved, with relative prediction errors around

3 % for commercial samples. The results demonstrate the potential of UV-Vis spectroscopy as a low-cost, practical method for biodiesel quantification, supporting its integration into sensor-based systems for quality control and regulatory compliance in biofuel distribution.

Paper [7] investigates the transferability of water calibration to energy-transition relevant fluids in Coriolis mass flowmeters (CMFs). Traditionally, calibrations rely on water under reference conditions, but expanding applications to gases and viscous fluids demands the validation of calibration factors beyond water. A calibration round was conducted across multiple facilities, testing CMFs, calibrated initially with water and subsequently operated with natural gas, hydrogen, CO<sub>2</sub>, and high-viscosity oils. Results showed deviations within maximum permissible errors specified by OIML R 137 and R 117, confirming the robustness of the water-derived calibration factor. High zero-point stability, repeatability, linearity, and low Reynolds number compensation enabled consistent performance under challenging conditions, such as low-density gases and viscous flows. These findings provide strong experimental support for adopting transferability as a reliable approach in custody transfer and energy-transition applications, reducing costs and infrastructure demands, while ensuring metrological reliability across diverse process conditions.

In paper [8], the authors reported the production and certification of MRC 8757.0001, a potassium calibration solution developed at the Brazilian National Metrology Institute (Inmetro) to strengthen Brazil's chemical metrology infrastructure. The solution was gravimetrically prepared from a high-purity KCl salt, certified for purity at Inmetro, and characterised by the formulation method in compliance with ISO 17034, ISO 33405, and internal standards. Homogeneity, transport stability (50 °C), and long-term storage stability (20–25 °C) were evaluated using High-Performance ICP-OES (HP-ICP-OES), which also served as a confirmation technique. The certified potassium mass fraction was  $(1002 \pm 11)$  mg/kg, with  $k = 2.02$  at a 95 % confidence level. Compared to conventional approaches, the integration of primary materials, buoyancy correction, and HP-ICP-OES led to a 47 % reduction in measurement uncertainty. Recognition of this CRM within the CIPM MRA ensures its international acceptance, providing Brazilian laboratories with a reliable traceability source for elemental analysis. The work establishes a foundation for producing additional single- and multi-element calibration solutions critical for regulatory and industrial needs.

Paper [9] presents the electrical characterisation and modelling of a commercial polyvinylidene fluoride (PVDF) thin-film sensor for application in Piezoelectric Energy Harvesting (PEH). PVDF is a flexible polymer with strong piezoelectric properties, making it a promising material for converting low-frequency mechanical vibrations into electrical energy. Impedance analysis confirmed its capacitive behaviour, with measured capacitance around 500 pF, consistent with manufacturer data. Based on these results, an equivalent electrical circuit was derived and simulated in LTspice®, allowing an accurate representation of the sensor's frequency-dependent response. The model revealed a resonant frequency near 7 Hz and behaviour analogous to a low-pass filter, highlighting limitations due to high reactance at low frequencies. Integration of the model with a power management chip (LTC3588-1) demonstrated successful rectification and storage of harvested energy, achieving a regulated 1.8 V output. This study provides a methodological foundation for optimising PVDF-based PEH

devices, supporting future development of sustainable micro-power sources for low-energy electronics.

Accurate measurement of transient pressure in small-calibre ammunition tests is essential for weapon safety, ammunition certification, and performance evaluation [10]. Piezoelectric transducers are widely used for this purpose, but their reliability depends on proper calibration procedures. This study evaluates two indirect calibration methods currently employed in ballistics laboratories: Indirect Quasi-static Calibration (IQsC) using the HPI B630 system, and Indirect Dynamic Calibration (IDC) using the TMS K9905D system. Three HPI GP6 transducers were calibrated with both approaches, and the results were compared in terms of sensitivity, linearity error, and uncertainty. While both methods produced acceptable sensitivity deviations, IQsC showed lower uncertainties and linearity errors, whereas IDC revealed operator-dependent variability and higher errors, often exceeding normative limits. The findings highlight the impact of automation, drift control, and hydraulic system integrity on calibration quality. The study emphasises the need for refined procedures, traceability, and verification practices to minimise undue transducer rejection and ensure metrological reliability in ammunition testing.

Paper [11] reports a metrological assessment of the speed of sound in oil-based materials aimed at developing durable Tissue-Mimicking Materials (TMMs) for ultrasonic diagnostic equipment. Traditional agar-based TMMs, while widely used, are limited by poor long-term stability and transportability. To address these drawbacks, mineral oil, lubricating oil, and ethylene glycol were characterised using a pulse-echo ultrasonic method under controlled thermal conditions. The speed of sound was derived from time-of-flight measurements and evaluated following the Guide to the Expression of Uncertainty in Measurement (GUM). Results showed ultrasonic velocities of  $(1727.0 \pm 4.6) \text{ m s}^{-1}$  for ethylene glycol,  $(1397.0 \pm 5.7) \text{ m s}^{-1}$  for mineral oil, and  $(1472.0 \pm 4.7) \text{ m s}^{-1}$  for lubricating oil. Mineral and lubricating oils demonstrated acoustic properties closest to soft tissue and exhibited low measurement uncertainties, making them promising candidates for stable, transportable commercial phantoms. These findings support oil-based formulations as reliable alternatives to conventional agar gels for calibration and performance evaluation of ultrasound systems.

The next paper describes the development and certification of a methamphetamine hydrochloride (MA·HCl) reference material to support forensic laboratories in achieving reliable, traceable measurements [12]. Methamphetamine is a potent central nervous system stimulant with significant public health and legal implications, requiring accurate analytical standards for law enforcement and judicial processes. A batch of high-purity MA·HCl was prepared and evaluated for homogeneity, transport stability, and storage stability, following ISO 17034 and ISO/IEC 17025 requirements. High-Performance Liquid Chromatography with Photodiode Array Detection (HPLC-PDA) was used to assess homogeneity and short-term stability, while long-term stability and final characterisation were determined by quantitative  $^1\text{H}$  Nuclear Magnetic Resonance (qNMR), a primary ratio method. The material showed no significant degradation under transport (50 °C, 21 days) or storage (20–25 °C, six months). The certified mass fraction was established at  $(999 \pm 12) \text{ mg/g}$  ( $k = 2$ ). This CRM provides a robust, SI-traceable tool to enhance the quality and comparability of forensic drug analyses.

The following paper compares the qualitative performance of real-time Reverse Transcription quantitative PCR (RT-qPCR)

and droplet digital Reverse Transcription PCR (RT-ddPCR) for detecting Bovine Viral Diarrhoea Virus (BVDV) RNA, to support the development of molecular reference materials [13]. BVDV, a model for RNA virus diagnostics, was analysed using serial dilutions of both a commercial RNA standard and virus grown in cell culture. Using identical primers and probe sets, RT-qPCR achieved 100 % sensitivity but showed lower specificity (83 % for the standard and 67 % for cultured RNA) when benchmarked against RT-ddPCR, considered the primary reference method. Discrepancies were most evident at low RNA concentrations, where RT-qPCR yielded possible false positives. These findings highlight the critical need for rigorous validation of molecular *in vitro* diagnostic assays, especially when establishing reference materials for metrological traceability. The results provide guidance for laboratories developing reliable molecular standards and evaluating the limits of qualitative PCR detection.

Paper [14] evaluates the dynamic modulus of elasticity of concrete using two non-destructive acoustic techniques—ultrasonic pulse velocity and natural vibration (impulse excitation)—and compares their results with static modulus values prescribed by ABNT NBR 6118:2023. A total of 48 cylindrical specimens, representing four strength classes, were tested at 14 and 28 days of curing. Dynamic modulus estimates obtained by ultrasound and Sonelastic® vibration analysis consistently exceeded static modulus values, reflecting the reduced influence of microcracks and energy dissipation mechanisms in dynamic testing. Both acoustic methods showed low measurement uncertainties (2–3 %) and captured the expected modulus growth with curing age. However, normalised error analysis indicated statistically significant differences among the methods, confirming that their results cannot be used interchangeably, despite internal consistency. The findings demonstrate the reliability of ultrasonic and vibration-based evaluations, while underscoring the importance of metrological validation when applying non-destructive testing to structural concrete characterisation.

Finally, paper [15] presents a conceptual model for monitoring and evaluating Brazil's regulation on pesticide and wood preservative labelling and packaging leaflets (RDC 296/2019). The model integrates fuzzy logic with multicriteria decision-making methods (Analytic Hierarchy Process and TOPSIS) to address key gaps in defining and ranking indicators for regulatory assessment. Using a logic model framework, the study maps inputs, activities, outputs, and outcomes to identify and prioritise 69 indicators aligned with the regulation's expected results, such as improved toxicological information, more explicit safety instructions, and alignment with the Globally Harmonised System of Classification and Labelling of Chemicals (GHS). Expert judgments guided the weighting and ranking of indicators to ensure relevance, reliability, and traceability. The results provide regulatory agencies, particularly the Brazilian National Agency for Sanitary Surveillance (Anvisa), with a systematic tool to strengthen compliance monitoring, enhance hazard communication, and support international harmonisation. The framework offers scalability for broader applications in chemical safety and other regulatory contexts.

We hope you will enjoy reading this special issue.

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Daniel Ramos Louzada  
Rodrigo Costa-Felix  
Section Editors

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