CITAC
Cooperation on International
Traceability in Analytical Chemistry

CITAC NEWS

2025

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The cover page picture is provided courtesy of author of CITAC best paper titled "Navigating the Complexity: Managing Multivariate Error and Uncertainties in Spectroscopic Data Modelling". Special thanks to Barbara Giussani.

FOREWORD BY THE CHAIR

CITAC ACTIVITY IN 2024

Zoltan Mester // National Research Council (NRC), Canada

Dear friends,

CITAC is an international organization with members from all continents aiming to improve the worldwide comparability of chemical measurements through collaboration. We are doing this by observing the principles of mutual respect, peace, basic human rights, dignity and worth of every person as the foundation of our work.

In Memoriam: Prof. Wolfhard Wegscheider

On March 3rd 2025, Prof Wolfhard Wegscheider, our past CITAC Chair serving between 1999 and 2002 has passed away. He was 75 years old.

Wolfhard served as the Rector and Professor of General and Analytical Chemistry at the University of Leoben, Austria, where he made significant contributions to the field of analytical chemistry. He received his education from Graz University of Technology, specializing in Technical Chemistry with an emphasis on Biochemistry and Food Chemistry. His academic journey included a diploma and doctoral thesis centered on trace and environmental analysis, reflecting his deep commitment to advancing scientific knowledge.

As a Fulbright Scholar, Wegscheider worked in Denver, Colorado, focusing on energy-dispersive X-ray fluorescence spectrometry, which was a testament to his dedication to research and innovation. His work in developing chemometric procedures for analytical chemical problems was highly regarded, and he co-authored a textbook on Analytical Chemistry, aligning with the DAC FECS-Curriculum.

Wolfhard was actively involved in several learned societies, including GDCh, GOECh, CITAC and EURACHEM, where he served as Chair and contributed significantly to the Working Groups on Education and Training, as well as Measurement Uncertainty and Traceability. His leadership extended to the Austrian Society of Analytical Chemistry, where he was a member of the Board of Directors after serving as President. Additionally, he was honored as a Fellow of the International Union of Pure and Applied Chemistry (IUPAC).

His expertise was sought by the Austrian Federal Ministry for Agriculture, Forestry, Environment and Water Management, where he served as a consultant. He also played a crucial role as a lead auditor in the Austrian Accreditation of Laboratories System and was an Editorial Advisory board member for journals such as Mikrochmica Acta, Accreditation and Quality Assurance, and Spectrochimica Acta.

Wolfhard Wegscheider was a family man, married to Susanne, a professional pharmacist, and was a proud father to his son, Stefan, and daughters, Beate and Eva. His legacy in the scientific community and his contributions to analytical chemistry and metrology in chemistry will be remembered fondly.

Source:
https://www.unileoben.ac.at/news/nachruf-altrektor-wolfhard-wegscheider/

Digital Metrology:

Digital transformation has remained the key theme of the year in metrology. In March 2024 signatories of the Joint Statement of Intent (https://www.bipm.org/en/liaison/digital-transformation) have organized a workshop on "Towards Digital Quality Infrastructure"

Machine-readable, findable, accessible and reusable information and data is essential for digitalization and also known as "FAIR principles". The aim of this workshop was to establish a common understanding on the FAIR principles and their role for the scientific and quality infrastructure. With several keynote presentations, Q&A elements and panel discussions, challenges and benefits were discussed.

https://www.bipm.org/en/committees/fo/forum-md/wg/forum-md-ws/2024-03-05

Later in the year the same group has organized another event in August 2025 exploring the concept and implementation of metrological traceability in the digital space.

https://www.bipm.org/en/committees/fo/forum-md/wg/forum-md-ws/2024-08-28

Even closer to home the Consultative Committee for Amount of Substance: Metrology in Chemistry and Biology (CCQM) has held a workshop in September 2025 on "Digital and FAIR Chemical and Biological Reference Data and Certificates: Challenges and Opportunities"

The workshop provided and excellent overview and some practical directions on ways forward on:

- Challenges with unique interoperable identifiers in the chemical and biological measurement space;
- Machine readable digital CRM certificates; and
- Best practice in developing databases for chem/bio data to meet FAIR principles.

The workshops has a dedicated website and all lectures are publicly accessible.

https://ccqmws2024.org/program/

Another significant, ongoing, digital transformation effort relevant to the chemical measurement science community is the IUPAC Gold Book project which aims to bring all currently analog, IUPAC recommendations and definitions into the digital era, following FAIR data principles of being findable, accessible, interoperable, reusable. Learn more about the IUPAC Gold Book and the current renewal process.

https://www.youtube.com/watch?v=uLkONO3MLOU

CITAC Award:

One key goal of CITAC is the building of chemical measurement science as distinct field of scientific enquiry. Celebrating outstanding contributions and contributors to our field is a key element of this discipline building effort.

The 2024 CITAC Best Paper in Metrology Award recipients are:

A. Henrion, C.-G. Arsene, M. Liebl and G. O'Connor, Label free quantifcation of host cell protein impurity in recombinant hemoglobin

materials, Anal. Bioanal. Chem. (2024) 416:387–396 (Published online 11/2023)
https://doi.org/10.1007/s00216-023-05024-8

E. Pagliano and J. Meija, Calibration model averaging in chemical analysis: a case study for the method of standard additions, Metrologia 60 (2023) 035003
https://iopscience.iop.org/article/10.1088/1681-7575/accd75

G.Holcombe, M. Walker, M. Singh, K. Gray, S. Cowen, S. Ellison, A. Rogers, A. Balasundaram, M. Burns, E. Mills, Clinically and industrially relevant incurred reference materials to improve analysis of food allergens, milk, egg, almond, hazelnut and walnut, Food Chem. 434 (2024) 137391
http://www.elsevier.com/locate/foodchem

Award Ceremony and Lectures of the CITAC Best Paper held on June 14, 2024.

https://www.youtube.com/watch?v=GRgLvtXhheY

Congratulations to the class of 2024 winners and I would like to thank all the CITAC members for supporting the nomination and selections process.

Our last in person CITAC meeting was held in April 2019 Paris right after the CCQM meetings. Although we have successfully pivoted to an online only operation I would very much hope that before the end of my chairmanship we can all shake hands again at an in-person annual meeting in 2026!

Keep well.

ADDRESS OF THE VICE-CHAIR

ETHICS AT THE TOP OF THE METROLOGICAL CHAIN

Ricardo Bettencourt da Silva // University of Lisbon, Portugal

No solid metrological or examinological[1] resources and practices survive ethical fragilities from analysts or those that condition analysts' work. In a time when we are suffering major ethical threats, we should also consider the ethical challenges that analytical chemists face.

I do not write this text from a position of "spectroscopic purity", as my 30 years as a professional chemist and 21 years as a technical assessor of accredited laboratories have brought both ethical challenges from others and introspective questions about the fairness of my own decisions. In the early days of my assessor work, it was not uncommon for laboratories to question my ethics after I identified serious shortcomings in their quality systems. Fortunately, such confrontations are less frequent today and generally easier to resolve. Undoubtedly, I have made mistakes across the 300+ external assessments I've conducted. Still, I can state with confidence that my only motivations were the protection of the accreditation system and my professional integrity.

After this introductory text, I feel I am in the position to reflect on how ethical fragilities can affect the application of state-of-art metrology and examinology to manage relevant societal interests. According to a UNESCO definition from this year, "Ethics refers to the principles of conduct governing an individual or a group, based on universal values such as respect for life, freedom, justice, equality, and solidarity."

In science, ethics should generally rule who should be listed as a research co-author, how research data is processed and interpreted, the impact of experiments on living organisms, etc. However, when we look at analytical chemistry's role in assessing a product's conformity with legislation or private specifications or the efficiency of an industrial process, more specific ethical issues can arise that should be considered.

Some ethical problems in Analytical Chemistry are driven by the fact that laboratories (the unquestionable experts in analysis) are frequently invited to set up the rules they have to apply themselves to ensure analytical results are fit for the intended purpose. This conflict of interest can lead to overly flexible and lax rules

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[1] Term related to examinology, the science of examinations and its application.

that result in delivering analytical information that is less fit or even unfit regarding the important interest being managed. Some examples of ethical fragilities in some options from analysts are:

- The option of not reporting the measurement uncertainty due to the additional work laboratories have to do and the impact of that parameter in the commercial competition between laboratories;
- The oversimplification of the evaluation of the measurement uncertainty due to the effort of performing metrologically sound calculations;
- The oversimplification of criteria for the identification of trace levels of compounds in complex matrices, although the state-of-art allows adequate control of false positive and negative results;
- The decision to assess product conformity without taking the measurement uncertainty into account to make laboratory assessment easier, although this decision does not allow proper control of the risk of false decisions;
- The investment of huge economic resources in monitoring chemical parameters in food and environmental samples that are not adequately interpreted considering the studied system and the efficiency of analytical work.

Analysts must strive for greater self-demand because if not, bad analytical work will flourish and affect those who depend on the results from chemical analysis reports — in fact, all of us. Furthermore, those outside the analytical community — the so-called direct and indirect clients of laboratory work — must become more knowledgeable and demanding. Their increased awareness and scrutiny can help drive the improvements that the analytical field has been too slow to implement.

Image generated by ChatGPT

In today's world, where ethical lapses are more visible and potentially more harmful, we must remember: analysts have their own set of ethical responsibilities to uphold. It's time we take those seriously—at the top of the metrological chain and beyond.

MESSAGE FROM THE CITAC SECRETARY

ANALYTICAL QUALITY BY DESIGN: A STRATEGIC APPROACH FOR MODERN CHEMICAL MEASUREMENTS

Felipe Rebello Lourenço // University of São Paulo, Brazil

Introduction to AQbD

The world of analytical science continues to evolve, driven by increasing demands for accuracy, reliability, and regulatory compliance across a wide spectrum of applications. Within this dynamic environment, Analytical Quality by Design (AQbD) has emerged as a forward-looking and structured framework that applies the principles of Quality by Design (QbD) to analytical procedures.

AQbD is defined as a systematic, science- and risk-based approach to the development of analytical procedures, emphasizing the understanding of method variables, identification of critical analytical parameters, and the establishment of a Method Operable Design Region (MODR). The central principle of AQbD is to ensure that the analytical procedure remains fit-for-purpose throughout its lifecycle by aligning it with the Analytical Target Profile (ATP).

Rather than relying on empirical one factor at a time (OFAT) development, AQbD is based on statistical and risk management tools to develop analytical procedures that are robust and flexible. It ensures consistency in analytical performance, increases regulatory confidence, and facilitates method transferability.

Steps of AQbD Implementation

AQbD can be divided into several interrelated steps that provide a roadmap for analytical development and lifecycle management:

Analytical Target Profile (ATP): The ATP defines the objective and performance criteria of the analytical method. It is the cornerstone of the AQbD strategy and typically includes required sensitivity, selectivity, accuracy, precision, and robustness. The ATP guides decision-making throughout the development process, ensuring that every step contributes to achieving the predefined goals. For example, an ATP may state: "The method should quantify Drug X in plasma with a limit of detection below 1 ng/mL and relative standard uncertainty below 5%."

Risk Assessment: Risk assessment identifies and prioritizes the method variables (e.g., sample preparation steps, instrument settings) that may affect the method's performance. Both qualitative and quantitative tools can be used:

a) Qualitative Tools: cause-effect diagrams and brainstorming sessions help visualize potential sources of variation; and
b) Quantitative Tools: Failure Mode and Effects Analysis (FMEA) and risk ranking matrices allow for numerical estimation of risk, helping to focus efforts on the most critical factors. By understanding which variables pose the highest risk, developers can prioritize experimental efforts more efficiently.

Design of Experiments (DoE): DoE is used to investigate the influence of multiple variables simultaneously, enabling a deep understanding of method behavior. DoE designs are divided into two types:

a) Screening Designs (e.g., Plackett-Burman, Fractionate Factorial Designs): used to identify critical method parameters (CMPs) from a large pool of variables; and
b) Optimization Designs (e.g., Box-Behnken, Central Composite Designs): used to determine optimal values of CMPs and assess interactions between them. DoE ensures that method development is data-driven, facilitating reproducibility and minimizing variability.

Method Operable Design Region (MODR): MODR is the multidimensional combination of method parameters that ensures the method meets the ATP. It is a proven space within which the method delivers acceptable performance. Operating within the MODR allows for flexibility in analytical procedure execution without revalidation, which is a significant regulatory advantage. The MODR can be visualized as a design space where changes within the defined limits do not compromise method performance. This concept supports lifecycle management by enabling method adjustments without compromising quality.

Control Strategy: Control Strategy refers to a planned set of controls, derived from understanding the method and its risks, to ensure consistent method performance throughout its lifecycle. It includes controls on critical method parameters (CMPs), system suitability tests, and sample preparation conditions. By implementing a robust control strategy, the analytical procedure remains within the MODR and continues to meet the ATP under routine conditions, ensuring data reliability and regulatory compliance.

Conclusions

The Analytical Quality by Design (AQbD) approach presents several compelling advantages that make it a valuable framework for modern analytical method development, including enhanced method robustness, regulatory flexibility, and simplified method transfer process. Furthermore, AQbD supports effective lifecycle management by enabling ongoing monitoring and continuous improvement of the analytical method, ensuring that it remains reliable and fit for purpose over time.

Analytical Quality by Design is a scientifically sound, regulatory-aligned, and value-driven approach to developing and maintaining high-quality analytical methods. As CITAC continues to promote global comparability and reliability in chemical measurements, the principles of AQbD resonate strongly with our mission.

References

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https://database.ich.org/sites/default/files/ICH_Q14_Document_Step2_Guideline_2022_0324.pdf

M.C. Breitkreitz & H.C. Goicoechea (Eds) Introduction to Quality by Design in Pharmaceutical Manufacturing and Analytical Development. 2023, AAPS / Springer. https://doi.org/10.1007/978-3-031-31505-3

I.M. Fukuda, C.F.F. Pinto, C.S. Moreira, A.M. Saviano & F.R. Lourenço. Design of Experiments (DoE) applied to Pharmaceutical and Analytical Quality by Design (QbD). 2018, Brazilian Journal of Pharmaceutical Sciences.

https://doi.org/10.1590/s2175-97902018000001006

LIAISON REPORTS 2024 OF THE SISTER INTERNATIONAL ORGANIZATIONS

AFRIMETS REPORT

Angelique Botha // National Metrology Institute of South Africa (NMISA), South Africa

APMP REPORT

Tang Lin Teo // Health Sciences Authority, Singapore

COOMET REPORT

Narine Oganyan // VNIIFTRI, Russia

EURAMET REPORT

Michela Sega // INRiM, Italy

IUPAC REPORT

Stephen Ellison // LGC, UK

IMEKO REPORT

Michela Sega // INRiM, Italy, IMEKO TC8 Chair

REPORT FROM ISO/TC 334 REFERENCE MATERIALS

Angelique Botha // NMISA, South Africa, ISO/TC 334 Chair

LIAISON REPORTS

AFRIMETS ACTIVITIES

Angelique Botha // NMISA, South Africa

SUMMARY OF GENERAL ISSUES

AFRIMETS (the African regional metrology organisation) held its 17th General Assembly and related meetings from 29 July to 2 August 2024 hosted by Kenya Bureau of Standards (KEBS) in Nairobi, Kenya. Most of the technical committees including the Technical Committee for Quality Systems (TC-QS) met before the General Assembly.

***Figure 1**: AFRIMETS is collaborating with PAQI on capacity building within the region to meet the Agenda 2063 aspirations also in terms of the African Continental Free Trade Area (AfCFTA)*

In terms of the election of executive officers, no new members were elected during 2024. Dr Henry Rotich from the Kenya Bureau of Standards (KEBS) is currently the Chairperson of AFRIMETS until 2025. Mr John-Paul Musimami from the Uganda National Bureau of Standards (UNBS) is the Vice-chair responsible for Legal Metrology and Mr Matthew Ranganai from SIRDC-NMI in Zimbabwe is the Vice-chair for Scientific and Industrial Metrology. Mr Donald Masuku from the NMISA in South Africa is the current Interim Head of the AFRIMETS Secretariat.

The 18th General Assembly of AFRIMETS will be hosted by Ghana in July 2025.

CURRENT TC AND WORKING GROUP CHAIRS AND CONTACT DETAILS

The AFRIMETS structure includes working groups to mirror the international consultative committee working groups (CC-WGs) and are identified as TC-(parameter).

The contact details of the TC Chairs important to Chemistry and Biology are listed below:

Function	Name	Details
TC-QM Vice-Chair (Bio analysis)	Dr Angelique Botha Ms Désirée Prevoo-Franszen	National Metrology Institute of South Africa (NMISA), Private Bag X34, Lynnwood Ridge, 0040, RSA Tel: +27 12 947 2705 e-mail: abotha@nmisa.org Tel: +27 12 947 2738 e-mail: dprevoo@nmisa.org
TC-Mass and Related Quantities	Mr Thomas Mautjana	National Metrology Institute of South Africa, Private Bag X34, Lynnwood Ridge, 0040, RSA Tel: +27 12 947 2880 e-mail: tmautjana@nmisa.org
TC-QS	Dr Noha Emad Khaled	National Institute for Standards (NIS), Tersa Street, El Haram, Giza, 12211 Egypt Tel: +(202)33862322 e-mail: nemadnis@yahoo.co.uk or nemadnis@netscape.net

AFRIMETS CMCs

As of 20 March 2025, there were a total of 769 AFRIMETS CMCs accepted in Appendix C of the KCDB.

The CMCs originate from:

South Africa (NMISA) = 523
(124 CMCs in Chemistry and Biology)

Egypt (NIS) = 112
(2 CMCs in Chemistry)

Zimbabwe (SIRDC) = 19

Kenya (KEBS) = 37
(2 CMCs in Chemistry)

Tunisia (DEFNAT) = 25

Ethiopia (EMI) = 12

Zambia (ZMA) = 11

Namibia (NSI) = 7

Morocco (LPEE) = 20

Botswana (BOBS) = 3

DEVELOPMENT WORK IN CHEMISTRY AND BIOLOGY

LNMc-INRAP, who is the designated institute for metrology in chemistry in Tunisia is now ready to release the first draft of The Tunisian National Strategy of Metrology in chemistry with the support of BIPM and AFRIMETS. The strategy aims to achieve comparability of measurement results to meet the national needs in terms of production of certified reference materials (CRMs) and organisation of proficiency testing (PT) schemes in the MAGMET/AFRIMETS framework.

Figure 2: *INRAP gravimetric unit for the preparation of certified calibration solutions*

In the beginning of 2024, LNMc-INRAP has organised a bilateral comparison with PTB in Germany: LNMc-BC-01: Copper Standard Solution in Nitric Acid: SRM-Cu Bilateral Comparison. Pending the approval of the AFRIMETS TC-QM, the LNMc is planning to pilot an AFRIMETS key comparison with the same copper calibration solution produced by LNMc-INRAP. As a capacity building and knowledge transfer (CBKT) participant, LNMc-INRAP will be participating in the key comparison study CCQM-K154.e: Organic solvent calibration solution - gravimetric preparation and value assignment of Ochratoxin A (OTA) in acetonitrile (ACN).

LNMc-INRAP in Tunisia has submitted their first CMCs for certified calibration solutions for patulin and aflatoxins. The institute has registered to participate in the supplementary comparison, AFRIMETS.QM-S1 - Pesticides in yellow plum. This comparison will allow INRAP to support the planned CMC claims for analysing pesticides in vegetable and fruit matrices. In the framework of this activity INRAP is currently planning a training activity with the NMISA in South Africa to continue building on core competencies and knowledge transfer.

The AFRIMETS supplementary comparison for pesticides in fruit (AFRIMETS.QM-S1) has been delayed. Upon request from the participants, the NMISA is trying to source calibrators from NIM China to distribute with the comparison samples to the participants. The comparison will enable AFRIMETS NMIs, such as LNMc-INRAP in Tunisia, who has an advanced capability in this area, to claim new CMCs. Much interest has also been shown by developing NMIs from the SIM region, such as Columbia, Bolivia, and Argentina as well as from other NMIs active in the Organic Analysis Working Group (OAWG) of the CCQM.

NIS in Egypt has submitted new CMC claims in the field of Chemistry and Biology for 2025 that includes their measurement capabilities for anions in a range of water matrices (seawater, contaminated water, drinking water, fresh water and ground water), certified calibration solutions for patulin and heavy metals in rice.

AFRIMETS is also supporting Phase 2 of the Pan African Quality Infrastructure (PAQI) project *Harnessing quality infrastructure in the cassava value chain* with the organisation of a proficiency testing (PT) scheme for cassava. The PT scheme was planned for four (4) PT rounds for the determination of the moisture and protein content in cassava; toxic and nutritional elements in cassava flour; pesticides in cassava and hydrocyanic acid (HCN) in cassava. All four PT rounds have been completed, and an on-line workshop was held in February 2024 to discuss the results with the participants.

The participants were invited to present their methods including how they established metrological traceability, validated their methods, and performed quality assurance. Based on the outcomes of the PT scheme and the feedback from the participants, PTB in Germany agreed to support two repeat rounds of the PT scheme that were run from April to June 2024. These two repeat PT rounds were for the determination of toxic and nutritional elements in cassava and the determination of mycotoxins in cassava.

At the end of 2024, the NMISA Training Centre was contracted to provide training on ISO 17034 for the competent production of reference materials for the British Standards Institute (BSI) Standards Partnership programme to the Rwanda Standards Board (RSB) in Rwanda and the Kenya Bureau of Standards (KEBS) in Kenya. The training also had a practical competent to introduce trainees to the practical aspects of reference material preparation.

Figure 3: *Gap analysis session with colleagues from Rwanda Standards Board (RSB) in preparation for ISO/IEC 17043 accreditation*

In February 2025, ten (10) participants from RSB were assisted with training and consultation to perform a gap analysis on their readiness for accreditation to ISO/IEC 17043 for the provision of PT schemes. Also in February 2025, fifteen (15) trainees from KEBS received training in ISO 17034 and ISO 33405. The NMISA was also contracted by the International Trade Centre MARKUP II program to provide ISO 17034 training for eight (8) colleagues of the East African Community (EAC).

Figure 4: *Colleagues from KEBS receive practical training in RM preparation*

For any further information on the activities in AFRIMETS or the activities of the TC-QM for Chemistry, please contact:

AFRIMETS Chair: Dr Henry Rotich (KEBS, Kenya): Rotich Henry: rotichh@kebs.org

TC-QM Chair; Dr Angelique Botha (NMISA, South Africa): abotha@nmisa.org

TC-QM Vice-Chair; Ms Désirée Prevoo-Franszen (NMISA, South Africa): dprevoo@nmisa.org

LIAISON REPORTS

APMP ACTIVITIES

Tang Lin Teo // Health Sciences Authority, Singapore

INTRODUCTION

The 24th Asia Pacific Metrology Programme (APMP) Technical Committee for Amount of Substance (TCQM) Meeting was held on November 25–26, 2024, in New Delhi, India, and online *via* Zoom. The meeting took place during the 40th APMP General Assembly & Related Meetings from 22 to 29 Nov 2024. Chaired by Dr. Byungjoo Kim from Korea Research Institute on Standards and Science (KRISS), the meeting brought together representatives from APMP member institutes to discuss key activities related to metrology in chemistry and biology, including comparisons, proficiency testing (PT), capacity-building efforts, and strategic initiatives on public health.

KEY COMPARISONS AND MEASUREMENT CAPABILITIES

CMCs Review

APMP TCQM accounted for 24.8% of the total quality management (QM) CMCs in the Key Comparison Database (KCDB) as of January 2024. Notable contributions were in High Purity Chemicals (59%) and Food (66%).

Inter-RMO Review

In 2024, 107 CMCs were reviewed, and 99 were submitted for inter-RMO review, all of which were published.

Comparisons and Studies

Since 1999, APMP TCQM has conducted 16 key comparisons (KCs), 25 supplementary comparisons (SCs), and 38 pilot studies (PSs). Ongoing studies include histamine in fish co-organised by the Government Laboratory (GL, Hong Kong, China) and Health Sciences Authority (HSA, Singapore), pesticide residues in water co-organised by the National Institute of Metrology (NIM, China) and HSA, and adulterants in herbal products co-organised by the Department of Chemistry (KIMIA, Malaysia) and HSA.

Peer Reviews

In 2024, three institutes, the National Physical Laboratory (India), National Institute of

Metrology of Thailand (NIMT), Industrial Technology Development Institute (Philippines) underwent peer reviews.

CAPACITY-BUILDING AND WORKSHOPS

- MEDEA 3.0 Training: Workshops focused on elemental analysis in water, conductivity measurement, and pH measurement using advanced techniques organised by NIMT.

- APMP-APAC Joint PT Programmes: Programmes included toxic elements in cosmetics organised by HSA, pesticide residues in water proposed by NIM and HSA, genetically modified organisms in soybean proposed by NIM, SARS-CoV-2 in wastewater proposed by NIM.

- ISO/IEC 17043 Workshop: A hybrid workshop on ISO/IEC 17043:2023 and ISO 13528:2022, co-organised by HSA, National Metrology Centre A*STAR, and National Standardisation Agency of Indonesia, attracted 230 online participants and 32 onsite attendees.

STRATEGIC INITIATIVES AND FUTURE DIRECTIONS

Bio-Metrology and Public Health

APMP TCQM is transitioning from COVID-19-related metrology to broader public health challenges and is establishing a bio-metrology platform to support this shift.

Formation of Sub-committees

Sub-committees were established for Gas Analysis, Bio-Analysis, Organic Analysis, and Inorganic & Electrochemical Analysis to enhance technical expertise and collaboration. The sub-committees are chaired by

Dr Hai Wu from NIM, Dr Eugene Lee representing KRISS, Dr Xiuqin Li from NIM and Dr Alvin Fung from GL.

CMCs Submission and Review

Guidelines for CMCs submission and inter-RMO review were refined to improve the process and prevent delays.

NEXT STEPS AND CONCLUSIONS

The meeting concluded with plans for the 2025 APMP TCQM meeting in Incheon, South Korea, along with continued efforts to strengthen regional and international collaborations in metrology. Participants emphasized the importance of capacity-building, strategic partnerships, and advancing metrological capabilities to support industry and regulatory needs across APMP member economies.

Dr Byungjoo Kim passed the leadership baton to Dr Tang Lin Teo from HSA who assumed the 3-year role after the 40th APMP General Assembly.

LIAISON REPORTS

COOMET ACTIVITIES

Narine Oganyan // VNIIFTRI, Russia

COOMET, as the Organization for Cooperation of State Metrology Institutions of Central and Eastern Europe, was established on June 12, 1991, on the initiative of metrologists from Bulgaria, Poland, Romania, the USSR and Czechoslovakia. In May 2000, it was renamed to **"Euro-Asian Cooperation of State Metrology Institutions"**.

Today, COOMET is an officially recognized as both **regional metrology organization** (**RMO**) and **regional organization for legal metrology** (**ROLM**) and unites **17 countries**. The organization is open to metrology institutions from other regions, including as associate members.

COOMET mission: to advance metrology, support and expand the integrated measurement infrastructure for the countries of the Euro-Asian region and other interested countries, which will enable each national metrology institute to promote innovation, competitiveness, trade, consumer safety, sustainable development and ensure international recognition.

The aims of COOMET are:

- to promote effective solutions for the task of ensuring the uniformity of measurements;
- to increase the level of development of metrology in the countries of the Eurasian region and other interested countries;
- to support and expand the integrated measurement infrastructure for the countries of the Eurasian region and other interested countries; and
- to be a forum for cooperation in the field of scientific and legal metrology.

The official languages are **Russian** and **English**.

The supreme body of COOMET is the **COOMET Committee** consisting of Heads of national metrology institutions from COOMET Member Countries, or their representatives. The Committee meets at least once a year.

The Committee elects the **COOMET President** from among its members for three years with the possibility of one repeat period. The national body, from which the President is elected, appoints the **Head of COOMET Secretariat** and support the work of the Secretariat.

The President, Vice-Presidents and Head of the COOMET Secretariat constitute the **COOMET President Council**, which decides upon the COOMET policy, interacts with international and regional metrology organizations, coordinates cooperation in the period between the Committee meetings and identifies problems to be discussed at these meetings.

In 2024, extraordinary presidential elections were held, as a result of which the Director of the Uzbek National Institute of Metrology **Lazizbek Saidoripov** (**Uzbekistan**) became the President of COOMET.

Nominated specialists by COOMET member countries are awarded annually the **Honorary Title "COOMET Honored Metrologist"** and **a breastplate** for a great personal contribution to the work and activities of COOMET.

Once in every two years, the international competition **"The Best Young Metrologist of COOMET"** is held.

Metrologists younger than 35 years of age take part in it. The competition is also open to representatives of other RMOs.

The last time it was held in 2023 in a hybrid format (Yekaterinburg, Russia) at the **II International Scientific and Practical Conference of Young Scientists and Specialists** "**The Future is Ours**", which brought together more than 150 young specialists in the field of standardization and metrology. The next such competition will be held in 2025.

The structure of COOMET includes: the **Joint Committee on Standards** (**JCS**), which includes 12 technical committees for different measurement directions (**TC 1.1-TC 1.12**), **TC 2 "Legal Metrology"**, **TC 3.1 "Quality Forum"**, **TC 4 "Information and Training"**, **TC 5 "Joint Scientific Research"**.

Two of the twelve technical committees of the JCS are related to the activities of Consultative Committee of Amount of Substance: Metrology in Chemistry and Biology (**CCQM**): **TC 1.8 "Physics - Chemistry"** and **TC 1.12 "Reference Materials"**. The Chairperson of TC 1.8 "Physics - Chemistry" is **Yuri Kustikov (VNIIM, Russia)**, the Chairperson of TC 1.12 "Reference Materials" is **Egor Sobina (UNIIM-VNIIM, Russia)**.

In turn, **8 subcommittees** have been established at TC 1.8 "**Physics - Chemistry**":
PC 1.8.1 "Electrochemistry",
PC 1.8.2 "Metals and alloys",
PC 1.8.3 "Inorganic analysis",
PC 1.8.4 "Gas analysis",
PC 1.8.5 "Organic analysis",
PC 1.8.6 "Bioanalysis",
PC 1.8.7 "Particle characterization",
PC 1.8.8 "Isotope analysis".

The last year was especially rich of the various metrological events associated with anniversary dates.

On April 9-10, 2024, **the International Scientific and Technical Conference "Metrology-2024"** was dedicated to the **100th anniversary** of the **Belarusian Chamber of Weights and Measures** (the National Metrology Institute of Belarus **(BelGIM), Minsk, Belarus**).

The conference included more than 200 representatives of metrological institutions from nine countries, including Armenia, Azerbaijan, Belarus, Kazakhstan, China, Kyrgyzstan, the Russian Federation, Uzbekistan, and Tajikistan. During the conference, the main achievements and trends in the development of metrology at the national and international levels were outlined. An open dialogue was also held in the format of sectional meetings. The choice of topics was focused on modern challenges for metrology. These are the issues of increasing human and scientific potential, the role of metrology in healthcare and new directions for the development of metrology in general.

On 4-5 June 2024, the **36th COOMET Committee meeting** was held in a hybrid format in Tashkent (Uzbek National Institute of Metrology **(UzNIM), Uzbekistan**).

According to the established tradition at the COOMET Committee meetings, the Committee Members exchanged information on the state of metrological activities and the most significant events in the field of metrology that took place in their respective countries. On 6 June 2024, as part of the COOMET Committee meeting, an **International Scientific and Technical Conference**, dedicated to the **100th anniversary of metrological activities in Uzbekistan** was organized in a hybrid format. Representatives of the national metrology institutes of Azerbaijan, Armenia, Belarus, Kazakhstan, Kyrgyzstan, Russia, Tajikistan and Uzbekistan, as well as representatives of International Bureau of Weights and Measures (**BIPM**), International Organization/Bureau of Legal Metrology (**OIML/BIML**) and regional metrology organizations (**APMP, AFRIMETS, SIM and IGU**) took part in the events with reports on their activities in 2023 – 2024.

On September 2, 2024, the **29th meeting of TC 1.12 "Reference Materials"** was held in a hybrid format at **UNIIM-VNIIM** (Yekaterinburg, **Russia**). During the meeting, issues related to the harmonization of TC 1.12 "Reference Materials" activities with CCQM activities on reference materials and training programs, joint development of COOMET reference materials, digitalization of the COOMET reference material database and other current issues were discussed. The meeting was attended by representatives of COOMET member countries: Azerbaijan, Belarus, Bosnia and Herzegovina, Kazakhstan, Russia, Tajikistan, Uzbekistan. Also, the members of **TC 1.8 "Physics-Chemistry"**, including **Chairperson Yuri Kustikov**, participated at the meeting.

The meeting was timed to coincide with the holding of the **VI International Scientific Conference "Standard Materials in Measurements and Technologies"** in a hybrid format, dedicated to the **190th anniversary of the birthday of D.I. Mendeleyev**. Organizers: **ROSSTANDART**, **VNIIM** and the **Academy of Standardization, Metrology and Certification"** (educational). The event was attended by over than 200 experts from Russia, neighboring countries, representatives of national regulatory bodies, national metrology institutes, state centers for standardization, metrology and testing, as well as industrial enterprises and others. During the conference program, the participants discussed matters related to the development, production, distribution and application of reference materials, metrological support of measurements in various areas of the economy, primary reference and reference measurement methods, metrological traceability of measurements, interlaboratory comparisons and international cooperation in the field of reference materials. In total, the event included 9 thematic sections related to such areas as healthcare, energy, mining, support of a wide range of physical and chemical measurements, etc.

The **next meeting of TC 1.12 «Reference Materials»** will be combined with the **VII International Scientific and Technical Conference «Metrology of Physical and Chemical Measurements»**, which will be held on **September 2-5, 2025** in the historical city of **Yaroslavl**, located near the **Volga River** and part of the tourist «**Golden Ring»** of Russia. The conference is organized by **VNIIFTRI** (**ROSSTANDART, Russia**).

On November 28-29, 2024, the meeting of **TC 1.8 "Physics - Chemistry"** was held in a hybrid format at **VNIIFTRI** (**Russia**). Representatives of 17 COOMET member countries took part in it. The reports of the Chairpersons of TC 1.8 and TC 1.12, as well as Heads of the subcommittees, on the work done over the last year were heard. Current and proposed projects on comparisons and pilot studies, participation of representatives of COOMET member countries in working bodies of relevant international organizations and RMOs, further harmonization of TC 1.8 and CCQM activities, appointment of new COOMET technical experts, etc. were discussed. Also, within the framework of the meeting, a training seminar was organized on the topic "**World experience in the production of reference materials of pure inorganic substances**" (the speaker was the Chairperson of TC 1.12 "Reference Materials" **Egor Sobina**, **Russia**).

In general, based on the reports of the TC 1.8 "Physics - Chemistry" Secretariat, the number of **COOMET CMCs** (Calibration and measurements capabilities of national metrology institutes) in the **KCDB** (Key comparison database) of **BIPM** related to the **CCQM** were:

- **2023 – 51 CMCs** have been revised and **12 CMCs** have been recognized as new;
- **2024 – 37 CMCs** have been revised and **12 CMCs** have been recognized as new.

The next meeting is planned to be held in autumn 2025 at **VNIIM** (**St. Petersburg**, **Russia**).

This is far from a complete list of events organized by metrological organizations of COOMET member countries.

In general, over the past two years, COOMET has become more active, the number of comparisons and pilot studies, joint developments, seminars and conferences conducted have increased with the aim of disseminating ideas on ensuring the uniformity of measurements both within a single country and establishing international traceability of measurements. Active work is being carried out with stakeholders from the fields of ecology, healthcare, production of medicines and cosmetics, food products, water treatment, energy, etc.

LIAISON REPORTS

EURAMET ACTIVITIES

Michela Sega // INRiM, Italy

EURAMET is the Regional Metrology Organization (RMO) of Europe. It coordinates the cooperation of National Metrology Institutes (NMI) in Europe in different fields of metrology, such as traceability of measurements to the SI units, international recognition of national measurement standards and related Calibration and Measurement Capabilities (CMC), research projects, etc. Through the knowledge transfer and cooperation among its members, EURAMET facilitates the development of the national metrology infrastructures.

EURAMET is currently chaired by Dolores Del Campo (CEM, Spain) who succeeded to Jörn Stenger (PTB, Germany); the two Vice-Chairpersons are Miruna Dobre (SMD, Belgium) for the General Assembly (GA) and Maguelonne Chambon (LNE, France) for the *European Metrology Programme for Innovation and Research* (*EMPIR*) and the European Partnership on Metrology related matters. EURAMET welcomed in 2024 the full membership of GEOSTM (Georgia) and has at present 38 full members, plus the European Commission which has the role of associate with observer status at the GA and 79 associates. In 2024 EURAMET published the white paper "Metrology for a stronger Europe – A European Metrology Agenda for the next decade" (https://www.euramet.org/about-euramet/metrology-for-a-stronger-europe-white-paper). It calls for a coordinated European metrology response building on the needs arising from the transition of the energy supply, protection of the environment, a strong, sovereign, and competitive European industry, digital transformation, circular economy, developments of the health system, and resilience of European infrastructures. The 18th EURAMET GA took place as a hybrid meeting from 30th May to 2nd June 2024, in Teddington, UK, hosted by NPL, the British NMI. The 19th EURAMET GA will take place from 2nd to 5th June 2025, in Berlin, DE, hosted by PTB, the German NMI.

European Partnerships are a key implementation tool of the European Commission's Horizon Europe; an ambitious research and innovation programme, running from 2021 to 2027. Among these, the European Partnership on Metrology (Metrology Partnership, EPM) aims at bringing together the measurement science community and stakeholders to deliver on global challenges including health and digital transformation, support the European Green Deal, and underpin innovation and competitiveness in industry through collaborative research. The EPM is co-funded by the Member States and the European Union with an expected budget of around 690 million euro. The expected impact of the European Partnership on Metrology is manifold, as it will support a wide range of policies, commerce and advancement of key European challenges. It will comprise seven call cycles between 2021 to 2027, covering topics such as Green Deal, Health, Digital Transformation, Fundamental Metrology, Integrated European Metrology, Industry needs, Pre-normative and Knowledge transfer and capacity building measures. The Partnership builds on the progress achieved under the previous European Metrology Research Programmes and aims to break new ground by contributing to the development of self-sustaining, coordinated metrology infrastructures, with the capacity to continue joint research and innovation after 2030.

In 2024 a call within the EPM framework was launched, via the usual two stage process, on the following major topics, addressing specific challenges: Green Deal, digital transformation, normative, metrology support for research potential, communication and impact coordination. Stage 1, opening on the 10th January, aimed at offering stakeholders from any country the opportunity to influence the projects undertaken by the European Community by identifying potential research topics. The highest priority topics received at Stage 1 provided the basis for Stage 2 which opened on 26th June 2024. In November 2024 the review conference was held, after which the call ranked list was published, thus establishing the projects to be financed within the call. An additional call for Coordination and Support Actions (CSAs) for Coordination of Impact and Communication supporting metrology opened on 26th June 2024. In 2025 another call within the EPM framework was launched on the following major topics: health, integrated European metrology, regulation, research potential. Stage 1 opened on 8th January 2025 and closed on 17th February 2025. The highest priority topics received at Stage 1 will provide the basis for Stage 2 which will open on 24th June 2025. Major information on the calls can be found at https://www.metpart.eu/.

The creation of European Metrology Networks (EMNs) represents one of the most important initiatives undertaken by EURAMET for the promotion of cooperation conceived in a broader scope towards better partnership, communication, and harmonization. The EMNs are collaborative structures which go beyond joint research to increase the coordination of measurement science across Europe, addressing scientific and societal challenges, infrastructure and services in response to European and global metrology needs. Currently there are twelve EMNs: Advanced Manufacturing, Clean Energy, Climate and Ocean Observation, Energy Gases, Laboratory Medicine, Mathematics and Statistics, Pollution Monitoring, Quantum Technologies, Radiation Protection, Safe and Sustainable Food, Smart Electricity Grids and Smart Specialization in Northern Europe. Each EMN, by providing a single point of contact, aims at underpinning regulation and standardization by establishing a comprehensive and longer-term infrastructure, promoting best practice and disseminating knowledge in its respective fields. Further EMNs are in preparation or under consideration. More information on EMNs can be found at the following link: https://www.euramet.org/european-metrology-networks/.

Technical collaboration in EURAMET is organized within ten Technical Committees (TCs), focusing on specific areas which represent the forum for scientific and technical cooperation in the respective fields. In addition, two Committees deal with the overall topics Quality and Interdisciplinary Metrology (https://www.euramet.org/technical-committees). The TCs are responsible for the execution of the activities required by EURAMET as RMO for the fulfilment of the Mutual Recognition Arrangement of the International Committee of Weights and Measures (CIPM-MRA). The types of technical cooperation carried out within the TCs are: cooperation in research, comparison of measurement standards, metrological traceability, and consultation on facilities.

One of the ten TCs is devoted to Metrology in Chemistry (Technical Committee for Metrology in Chemistry, TC-MC), which is concerned with primary methods and reference materials for chemical measurements and research in metrology to support different sectors in the amount of substance fields (https://www.euramet.org/technical-committees/tc-mc).

NEWS FROM EURAMET TECHNICAL COMMITTEE IN METROLOGY IN CHEMISTRY (TC-MC)

TC-MC is chaired by Teemu Näykki (SYKE, FI). 31 EURAMET member countries are represented in TC-MC plus the European Commission. BIPM has the status of observers.

The technical activities are carried out within the four technical Sub-committees dealing with gas analysis (SC-GA), inorganic analysis (SC-IA), electrochemical analysis (SC-EA), bio and organic analysis (SC-BOA). The convenors of the subcommittees are: Janneke van Wjik (VSL, NL) for SC-GA, Rainer Stosch (PTB, DE) for SC-IA, Gavin O'Connor (PTB, DE) who took over in 2025 the responsibility of SC-BOA convenorship from Mine Bilsel (UME, TR) and Anton Petrenko (SE Ukrmetrteststandard) for SC-EA who replaced Matilda Roziková (CMI, CZ) for the year 2025. In addition, a strategy working group, chaired by the TC-Chair, is also active on the following tasks: advice to TC-Chair and subcommittee convenors, strategic planning of comparisons, support actions, coordination, organization of workshops.

The TC-MC members are actively participating in the European Programmes on Metrology, being involved in all the targeted programmes; in addition, they cooperate within various EMN, among which Climate and Ocean Observation, Energy Gases, Mathematics and Statistics, Pollution Monitoring, Safe and Sustainable Food, Traceability in Laboratory Medicine, thus indicating the cross-disciplinary nature of the TC itself.

TC-MC meeting in 2024

The annual meeting of the TC-MC was held in Delft, the Netherlands, hosted by VSL from 6th to 8th February 2024. The Strategy WG meeting had an online meeting on 2nd February 2024.

The four technical subcommittees reconvened, as usual, ahead of the annual TC-MC plenary meeting on 6th February 2024. A review of new claims as well as the obligatory re-review of a range of existing claims were carried out. Running and new projects and comparisons in the framework of EURAMET, EMPIR, EMP and also proposals for the upcoming EMP call were discussed in detail in all sub-committees.

The plenary meeting took place on 7th and 8th February 2024. Some highlights on EURAMET, BIPM/CIPM, CCQM strategy and activities within its main working groups were given. Presentations on the activities carried out within liaison organisations like Eurachem and Eurolab were also given. The conveners of the subcommittees gave an overview of the activities of each subcommittee and of the main outcomes of the meetings carried out in the previous day. A section of the meeting was devoted to EMNs dealing with topics related to the amount of substance field. An overview was given on the EMN for Climate and Ocean Observation (coordinated by NPL), EMN for Energy Gases (coordinated by VSL), EMN for Laboratory Medicine (coordinated by LNE), EMN for Environment Pollution (coordinated by LNE) and EMN for Safe and Sustainable Food (coordinated by INRiM). On 7th February a specific workshop in preparation for the 2024 EMP call was held, addressing all the different targeted programmes.

TC-MC meeting in 2025

The annual meeting of the TC-MC was held in Borås, Sweden, hosted by RISE from 4th to 6th February 2025. The Strategy WG meeting had an online meeting on 29th January 2025.

The four technical subcommittees reconvened, as usual, ahead of the annual TC-MC plenary meeting on 4th February 2025. A review of new claims as well as the obligatory re-review of a range of existing claims were carried out.

Running and new projects and comparisons in the framework of EURAMET, EMPIR, EMP and also proposals for the upcoming EMP call were discussed in detail in all sub-committees.

The plenary meeting took place on 5th and 6th February 2025. An update on EURAMET and BIPM/CIPM activities, including a focus on progress on recommendations and new initiatives since the BIPM-WMO Workshop on Metrology for Climate action and the outcomes of the 1st CIPM STG-CENV stakeholder meeting was given. Some highlights on CCQM strategy and activities within its main working groups were also given. The activities carried out within the liaison organization Eurachem were presented, including a proposal of a MoU between EURAMET and Eurachem. The conveners of the subcommittees gave an overview of the activities of each subcommittee and of the main outcomes of the meetings carried out in the previous day. The convener of SC-BOA gave some information on the proposal of a summer school on Bioanalysis, scheduled for 2026 and organized together with the university of Braunschweig. A section of the meeting was devoted to EMNs dealing with topics related to the amount of substance field. An overview was given on the EMN for Climate and Ocean Observation (coordinated by NPL), EMN for Energy Gases (coordinated by VSL), EMN for Laboratory Medicine (coordinated by LNE), EMN for Pollution Monitoring (coordinated by LNE) and EMN for Safe and Sustainable Food (coordinated by INRiM). On 5th February 2025 a specific workshop in preparation for the 2025 EMP call was held, addressing all the different targeted programmes.

The next TC-MC meeting will be held from 2nd to 5th February 2026 in Ljubljana, Slovenia, hosted by MIRS.

LIAISON REPORTS

IUPAC ACTIVITIES

Stephen Ellison // LGC, UK

GENERAL IUPAC NEWS

GENERAL ASSEMBLY AND WORLD CHEMISTRY CONGRESS

The last Biennial Meeting took place in 2023. The 53rd IUPAC General Assembly will be in Kuala Lumpur, Malaysia 12-16 July 2025, in parallel to the IUPAC World Chemistry Congress 2025

IUPAC GOVERNANCE

Following the replacement of the IUPAC Bureau with the Executive Board and Science Board, IUPAC has begun the process of refreshing its scientific priorities and objectives for the next 4-6 years and is reflecting on how it is best structured to deliver these. This process is on-going and should report by the end of 2025.

KEY ACTIVITIES

2025 IUPAC Global Women's Breakfast - "Accelerating Equity In Science"

The most recent IUPAC Global Women's Breakfast was held on February 11, 2025. Held in conjunction with the International Day of Women and Girls in Science, the goal of "The Breakfast" is to establish an active network of people to overcome the barriers to gender equality in science. Over the last seven years, nearly 2500 GWB have been held in 100 countries. In 2025 the theme was "Accelerating Equity In Science"

GWB2026 will be held on 10 February 2026. Further details will be made available at https://iupac.org/gwb/

TOP TEN TECHNOLOGIES

IUPAC has released its call for proposals to identify the top ten emerging technologies in chemistry with the results to be announced later in 2025.

This is an activity that IUPAC has been running annually since 2019, and many of the technologies identified have had strong links with measurement science such as nanosensors, rapid diagnostics for testing, chemiluminescence for biological use, single cell metabolomics and wearable sensors.

A full list of technologies captured by this initiative can be found at the following web address (https://iupac.org/what-we-do/top-ten/) . In 2024, the top ten finalists were Active Adsorption, Aptamers, Bioinspired Nanofluidic Iontronics, the Electrochemical Nitrogen Cycle, Frustrated Lewis Pairs, Hydration Lubrication, KRAS Inhibitors, MXenes, Neural Network Potentials, and Triboelectric Nanogenerators. Review papers can be found at the links above.

COMMISSION ON ISOTOPIC ABUNDANCES AND ATOMIC WEIGHTS

The Commission on Isotopic Abundances and Atomic Weights (CIAAW) is an international scientific committee of IUPAC under its Inorganic Chemistry Division. Since 1899, CIAAW has been entrusted with periodic critical evaluation of atomic weights of elements and other cognate data, such as the isotopic composition of elements. CIAAW has been a part of IUPAC since the first General Assembly of IUPAC in 1920. Its most recent activity has been the amendment of the 13C/12C isotope ratio in Vienna Peedee belemnite (VPDB). The latest recommended value is

$R(^{13}C/^{12}C, VPDB) = 0.011\,113 \pm 0.000\,022$

where the quoted uncertainty is at 95 % confidence level. This value represents a consensus estimate from ten studies published between 1990 and 2024 from numerous independent measurement techniques.

ANALYTICAL CHEMISTRY DIVISION OF IUPAC

Division Details - IUPAC | International Union of Pure and Applied Chemistry

DIVISION MEMBERSHIP

The composition of the division for the 2024-2025 biennium is as follows:

Division President – Derek Craston

Division Past President – David Shaw

Division Secretary – Luisa Torsi

Titular Members – Aura Tintaru, Vasilisa B. Baranovskaia; Jiri Barek; Takae Takeuchi; Franziska Emmerling; Hongmei Li;

Associate Members – Erico Marlon de Moraes Flores; Ivo Leito;

National Representatives - D. Brynn Hibbert, Mariela Pistón, Rufus H. Sha'Ato, Resat Apak; Ilya Kuselman; Susanne Wiedmer

ANALYTICAL CHEMISTRY AWARDS

In 2020 the Analytical Chemistry Division of IUPAC established two awards:

– The Emerging Innovator Award in Analytical Chemistry – an award to recognize outstanding work undertaken by an emerging analytical scientist that corresponds to the aims of the Analytical Chemistry Division.

– The IUPAC Analytical Chemistry Medal – an award to recognize significant lifetime contribution to the aims of the Analytical Chemistry Division.

In 2025, the awards were to:-

Graham Cooks, Henry B. Hass Distinguished Professor of Chemistry, Purdue University, USA has been named the IUPAC Analytical Chemistry Medalist for 2025. The award was given recognition of his lifetime research achievements in the field of mass spectrometry including fundamental phenomena, instrumentation and analytical applications, and his mentoring of 156 PhD graduate students in addition to approximately 200 Postdoctoral Fellows and long-term scientific visitors.

Anna Regoutz, Associate Professor of Experimental Inorganic Chemistry, University of Oxford, UK is the recipient of the IUPAC Emerging Innovator Award in Analytical Chemistry for 2025. The award was given for her leadership of an interdisciplinary team of researchers exploring the structure-electronic structure relationship in inorganic solids with a goal of integrating such materials into opto-electronic devices.

Nominations for the 2026 Awards will be launched in the Autumn of 2025.

DIVISIONAL PROJECTS

A full list of projects currently running within the Analytical Division can be found at:

https://iupac.org/body/500/#:~:text=The%20Analytical%20Chemistry%20Division%20is,of%20the%20chemical%20measurement%20process.

The following projects were initiated in the last 18 months.

2023-028-1-500

IUPAC Brief Guide to Metrological Terms in Chemistry

The aim of this project is to provide a clear description of selected terms used in metrology in the chemical sciences. An IUPAC Brief Guide will be published in Pure and Applied Chemistry (PAC), and possibly as a stand-alone brochure. The target audience comprises researchers, educators, chemistry students, and working chemists who make measurements but might not have had much training in metrology. The focus will be on measurements that chemists make, and the goal is to provide easy-to-understand description of the major terms which, in turn, could be used to update the International Vocabulary of Metrology (VIM) and fill gaps in its coverage of contemporary measurements of chemical properties.

2023-016-1-500

IUPAC/CITAC GUIDE FOR INTERLABORATORY COMPARISON OF NOMINAL (QUALITATIVE) AND ORDINAL (SEMI-QUANTITATIVE) CHARACTERISTICS OF A SUBSTANCE OR MATERIAL

This project will produce a joint IUPAC/CITAC Guide for interlaboratory comparison of nominal (qualitative) and ordinal (semi-quantitative) characteristics of a substance or material. The guide will be helpful for application of relevant metrological and mathematical methods for statistical design of interlaboratory comparison of nominal and ordinal characteristics of a substance or material, and analysis of the obtained data.

2022-008-4-500

Introducing the IUPAC Seal of Approval for a wider adoption of IUPAC recommended symbols, terminology and nomenclature: Stage 1 – Symbols

IUPAC makes great efforts to standardize chemical nomenclature, symbols, and terms, but it is evident that the authors of textbooks and scientific publications have no motivation for adopting these recommendations. The present project represents the first step to introduce a "IUPAC Seal" to be conferred by IUPAC on books, textbooks or other scientific publications that adopt IUPAC recommendations. The Seal will add value to the work that receives it. This initial project will focus on IUPAC expert review of human-readable depictions of IUPAC symbols in analytical and physical chemistry publications.

Selected IUPAC Analytical Chemistry Publications in 2024

Glossary of terms for mass and volume in analytical chemistry (IUPAC Recommendations 2024)

Maria F. Camões , Gary D. Christian and David Brynn Hibbert

https://doi.org/10.1515/pac-2023-0903

A glossary of terms and definitions for concepts in the use of mass and volume in analytical chemistry is presented. These include definitions for analytical methods of measurement (gravimetry, volumetry, and titrimetry) and supporting terms. Terms are updates of earlier recommendations or Orange Book entries.

IUPAC Technical Report: A brief guide to measurement uncertainty (IUPAC Technical Report)

Antonio Possolo, David Brynn Hibbert, Jürgen Stohner, Olha Bodnar, and Juris Meija

https://doi.org/10.1515/pac-2022-1203

This Brief Guide reintroduces readers to the main concepts and technical tools used for the evaluation and expression of measurement uncertainty, including both classical and Bayesian statistical methods. The general approach is the same that was adopted by the Guide to the Expression of Uncertainty in Measurement (GUM). All the methods presented are illustrated with examples involving real measurement results from a wide range of fields of chemistry and related sciences, ranging from classical analytical chemistry as practiced at the beginning to the 20th century, to contemporary studies of isotopic compositions of the elements and clinical trials.

LIAISON REPORTS

IMEKO ACTIVITIES

Michela Sega // INRiM, Italy, IMEKO TC8 Chair

IMEKO, the International Measurement Confederation, founded in 1958, is a non-governmental federation of 42 Member Organizations individually concerned with the advancement of measurement technology. It has a consultative status with UNESCO and UNIDO. Its fundamental objectives are the promotion of international interchange of scientific and technical information in the field of measurement and instrumentation and the enhancement of international co-operation among scientists and engineers from research and industry.

In 2024, Prof. Paolo Carbone (Italy) took over the responsibility of President of IMEKO from Prof. Frank Härtig (Germany) who, as past President, is currently the Advisory President and Chair of the Advisory Board. Dr. Barbara Goldstein (USA) is the President Elect and Chair of the Technical Board and Prof. Pasquale Daponte (Italy) is the vice President in charge of the XXV IMEKO World Congress.

IMEKO Secretariat is located in Budapest (Hungary). More information about IMEKO and its structure can be found on the IMEKO website (www.imeko.org).

The absolute highlight of 2024 is the XXIV IMEKO World Congress (https://www.imeko2024.org/home), excellently organized by Prof. Frank Härtig and PTB, the German Member Organization of IMEKO, with Thomas Wiedenhoefer as congress chair. The Congress took place at the new Congress Center of Hamburg (Germany) on 26th-29th August 2024. The event attracted more than 1200 registered participants from 78 countries, reinforcing IMEKO's commitment to advancing the frontiers of measurement and instrumentation. The program was extremely rich with 89 sessions organized by 25 IMEKO Technical Committees, with 470 oral contributions and 153 posters. During the plenary sessions, notable key-note speakers gave fascinating lectures, including two Nobel Prize Laureates, Prof. Klaus von Klitzing (Nobel Prize in Physics in 1985) and Prof. Stefan Hell (Nobel Prize in Chemistry in 2014), as well as the President of the International Committee for Weights and Measures (CIPM), Dr. Wynand Louw, and the former President of the CIPM Consultative Committee for Units and former President of PTB, Prof. Joachim Ullrich. Specific insights into cutting-edge developments were offered with workshops on digitalization and quantum technology. A big exhibition area with 39 booths was also visited by the attendees in the congress venue. The Congress had also an interesting social programme, which included The Senate Reception in the historic City Hall of Hamburg. The congress contributions will be published in a special issue of the journal *Measurement: Sensors* as open access conference proceedings. Authors of papers selected by the Congress International Program Committee will be invited to submit an extended full paper for potential publication in the journal *Measurement*.

Additional contributions to IMEKO events can be also published in the IMEKO Online Journal *ACTA IMEKO*, which published its four issues in 2024 (https://acta.imeko.org/index.php/acta-imeko). It is indexed in, Scopus, and DOAJ and publishes also scientific articles on measurement and instrumentation not related to specific IMEKO events. In addition, two more specific open access journals are also available, in line with the rapid growth of studies on the relevant topics: *Measurement: Food*, and *Measurement: Energy*. Periodic newsletters are prepared by IMEKO Secretariat and can be accessed through IMEKO webpage.

The preparation for the XXV IMEKO World Congress is ongoing. It will take place at the Palacongressi di Rimini (Italy) from 30th September to 3rd August 2027. More information and updates can be found on the dedicated website (https://imeko2027.org/).

LIAISON REPORTS

ISO/TC 334 REFERENCE MATERIALS ACTIVITIES

Angelique Botha // NMISA, South Africa, ISO/TC 334 Chair

ANNUAL MEETING OF ISO/TC 334 IN JUNE 2024

The fifth meeting of ISO/TC 334 was held as an on-line meeting during the first week of June 2024. The number of participants who joined the meeting ranged between 60 to 70 with representatives from 16 member bodies (57% of the P-members) and 2 O-members also participated in the meeting. Representatives from 10 international organisations attended the meeting representing 56% of the external liaisons, and 9 of the ISO internal liaisons (other technical committees) representing 69% of the ISO/TC 334 membership attended the meeting. Currently, ISO/TC 334 has 13 internal (ISO) liaisons in force, 14 category A liaisons and 4 category C liaisons with external organisations. There are also three liaisons managed at ISO level (JCGM/WG1 responsible for the Guide to the Expression of Uncertainty in Measurement (GUM), JCGM/WG2 responsible for the International Vocabulary for Metrology (VIM) and JCTLM responsible for measurement traceability in laboratory medicine).

Some specific liaison activities of ISO/TC 334 during 2024 included working with ISO/TC 212 on the revision of ISO 15193 for the requirements for reference measurement procedures for in vitro diagnostic medical devices and ISO 15194 for the requirements of reference materials (RMs) for in vitro diagnostic medical devices. The 2nd committee draft of the fourth edition of the VIM (VIM4) was launched for balloting during 2023 and ISO/TC 334 commented extensively on the terminology related to RMs. ISO/TC 334 also wrote a letter to JCGM/WG2 about the developments related to terminology for reference materials in working group 10 (WG10) that is working on the revision of ISO 33400 – Reference materials - Vocabulary.

PROGRAM OF WORK OF ISO/TC 334

The program of work of ISO/TC 334 have now been updated based on the decisions taken at the fifth meeting and are listed below. The program includes the transformation of the existing ISO/REMCO Guides as well as the new work items of the committee that have been transformed into international standards. ISO/TC 334 currently has two (2) remaining projects in hand in the development of the ISO 33400 series of standards. During the fifth meeting of the committee, a decision was taken to launch committee internal ballot (CIB) questions to enquire from the committee members what the future of the technical reports published by ISO/REMCO and, now, fall within the scope of work of ISO/TC 334, should be. The outcomes of the CIB ballots will be discussed during the sixth meeting of ISO/TC 334.

Figure 1 below depicts the relationships between the different documents previously developed and currently being developed by ISO/TC 334.

ISO/DIS 33400 (previously ISO Guide 30) – Reference materials – Vocabulary

- ISO 33401 (previously ISO Guide 31) – Reference materials – Contents of certificates, labels and accompanying documentation
- ISO/TR 33402 (previously ISO Guide 80) – Good practice in reference materials preparation
- ISO 33403 (previously ISO Guide 33) – Reference materials – Requirements and recommendations for use
- ISO 33405 (previously ISO Guide 35) – Reference materials – Guidance for the characterization and assessment of homogeneity and stability
- ISO 33406 (previously WD/ISO Guide 85) – Guidance for the production of reference materials having one or more assigned qualitative property values
- ISO 33407 (previously WD/ISO Guide 86) – Guidance for the production of pure organic substance certified reference materials
- ISO/DIS 33408 (previously AWI/ISO Guide 87) – Guidance for 'pure' reference materials for metals and metalloids

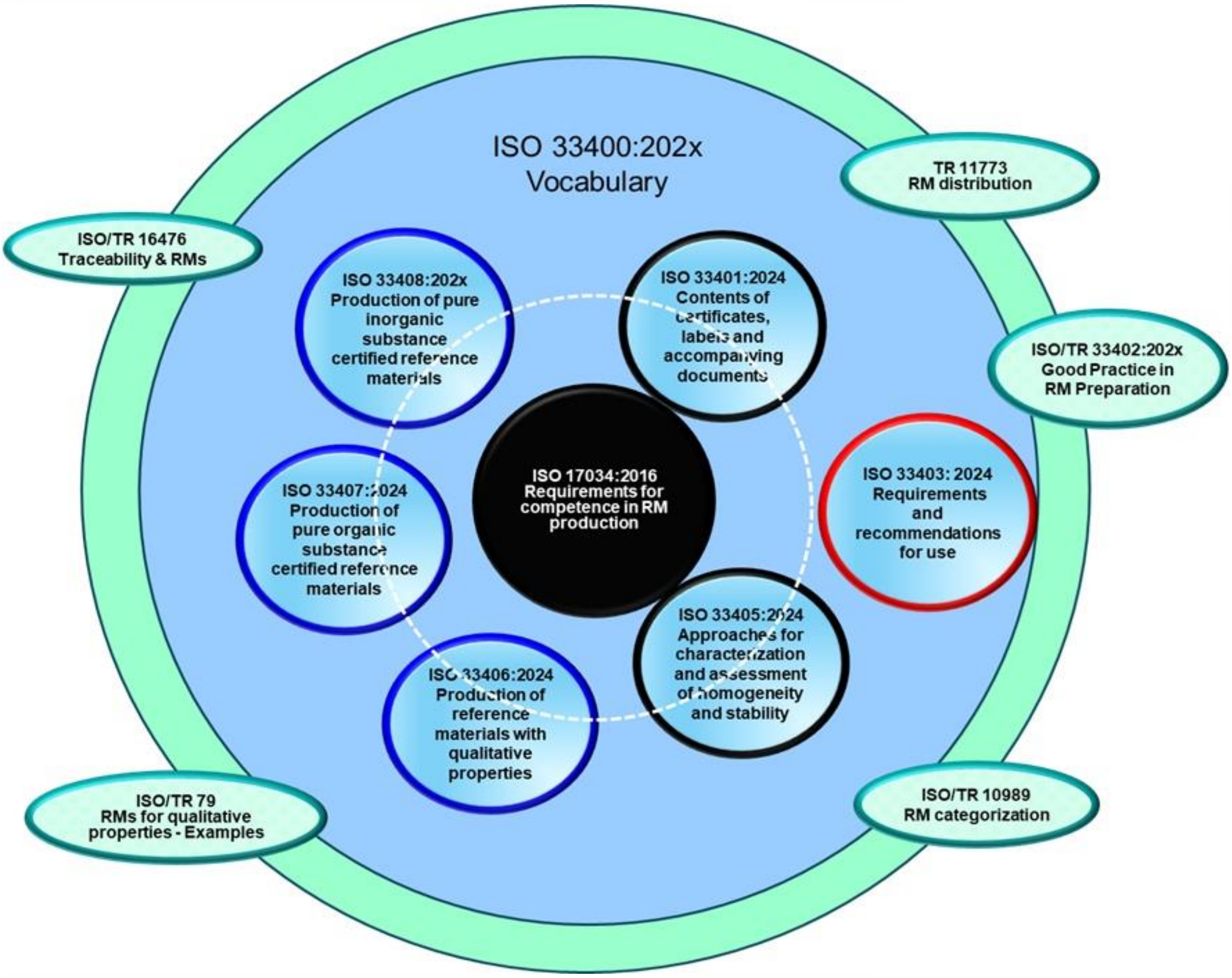

***Figure 1**: Schematic diagram of the ISO 33400 series of standards for the transformation of the ISO/REMCO Guides 30 to 35 to international standards as well as the other projects of ISO/TC 334*

Looking more closely at Fig. 1, ISO 33401:2024 and ISO 33405:2024 touch ISO 17034:2016 and are similarly circled in black because they are indispensable for implementation of ISO 17034:2016. ISO 33406:2024, ISO 33407:2024, and ISO 33408:2024 are circled in blue and intersect the dashed white circle that is concentric with ISO 17034:2016 because they are focused implementations of ISO 17034:2016 for their respective fields of application. ISO 33403:2024 is circled in red and sits outside the dashed white circle but still near the middle of Fig. 1 because it is of potentially great interest to readers of the other documents already described, but not indispensable for application of ISO 17034:2016. All these ISO deliverables lie in a field representing ISO 33400:202x because they are all tied together by, and depend upon, reference material vocabulary. The five ISO Technical Reports (TRs) sit around the perimeter of the figure to reflect their reduced importance to the implementation of ISO 17034:2016, even though they can still be helpful to producers and users of reference materials.

UPDATE ON THE PROGRESS WITH THE TRANSFORMATION OF THE ISO/REMCO GUIDES INTO THE ISO 33400 SERIES OF STANDARDS

Five of the new standards of the ISO 33400 series were published in 2024. ISO 33400 for the terms and definitions related to reference materials has progressed to the draft international standard (DIS) stage. Within the working group there have been some discussions about including new terms in the standard, such as operationally defined measurand. There is also strong support in the working group to refer to a certified reference material as a reference material with one or more certified property value and to specify the characteristics of a certified value, such as the requirement for a value with an associated uncertainty and established metrological traceability in the definition of certified value.

The strong liaison relationship between ISO/TC 334 and JCGM/WG2 working on the revision of the International Vocabulary for Metrology (VIM) also continues. The 2nd committee draft of the VIM4 was launched for balloting during 2023 and the ISO/TC 334 members provided comments on the draft. ISO/TC 334 also sent a letter to JCGM/WG2 to keep the group abreast of the developments related to the vocabulary for reference materials in ISO 33400.

ISO 33408 for the production of high purity reference materials of metals and metalloids is at the draft international standard (DIS) stage. ISO 33406 for the production of qualitative reference materials has been received very well by the chemical and biological testing communities. In September 2024, a very successful joint workshop between EURACHEM and CITAC was held to introduce the standard. The programme of the workshop included a brief introduction to the standard. Several producers of qualitative reference materials were invited to present how the guidance from ISO 33406 was incorporated in the production of their qualitative reference materials.

The next meeting of ISO/TC 334 will be held in conjunction with the International Symposium for Biological and Environmental Reference Materials (BERM-16) in Halifax, Canada, during the week of 1 to 6 June 2025.

EURACHEM/CITAC Online Workshop

Production of qualitative Reference Materials

Date: 19 September 2024 12:00 – 16:00 CEST

CITAC BEST PAPER AWARD 2024

INDEPENDENT CERTIFICATION OF A SUITE OF qNMR INTERNAL STANDARDS: ENSURING METROLOGICAL TRACEABILITY IN A WIDE RANGE OF APPLICATIONS

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NAVIGATING THE COMPLEXITY: MANAGING MULTIVARIATE ERROR AND UNCERTAINTIES IN SPECTROSCOPIC DATA MODELLING

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Introduction

Quantitative nuclear magnetic resonance (qNMR) is widely used in research and metrology, and is increasingly adopted by industries, especially pharmaceuticals, for drug quality control despite high NMR costs. Its growing popularity is due to its potential as a primary reference procedure (1, 2), allowing measurements without the need for a certified reference material (CRM) of the same analyte while still ensuring traceability to the International System of Units (SI). This is particularly valuable when no CRM exists or when determining the purity of a substance at the highest traceability level.

A qNMR procedure behaves as a primary method provided that an SI-traceable internal standard (IS) is used in the analysis; that is why IS qNMR is referred as a "ratio" primary measurement procedure (3, 4). The mass fraction of sample (w_A) determined by IS qNMR is calculated by (5):

$$w_A = \frac{I_A}{I_{IS}} \times \frac{N_{IS}}{N_A} \times \frac{M_A}{M_{IS}} \times \frac{m_{IS}}{m_A} \times w_{IS} \qquad (1)$$

Where I is the integral of the peak; N is the number of equivalent nuclei contributing to each quantified signal; M is the molar mass; m is the mass weighed for the preparation of the solution subject to the qNMR measurement; and w_{IS} is the purity of the IS expressed as mass fraction (mg g^{-1}). The subscripts A and IS refer to analyte and internal standard, respectively.

This study explored the use of orthogonal primary measurement procedures for reliable and independent chemical purity assessment. Alongside IS qNMR, traditional primary methods like mass balance, coulometric titration, and freezing-point depression were evaluated, along with strategies for assessing homogeneity and stability. Four candidate CRMs — potassium hydrogen phthalate (KHP); dimethyl terephthalate (DMTP); maleic acid (MA); and dimethyl sulfone ($DMSO_2$) — were selected from the BIPM list of potential ISs (6), covering a wide range of solubility and NMR shifts for diverse organic chemistry applications.

EXPERIMENTAL SECTION

Analytical methods

^{1}H-NMR Characterization and long-term stability studies were performed in a Bruker 400 MHz Avance III spectrometer (Switzerland) with a 5-mm broadband inverse probe (BBI) at 298 K. For stability monitoring, a Bruker 500 MHz Avance III HD spectrometer (Switzerland) with a 5-mm CryoProbe Prodigy TCI at 298 K was used. TopSpin software from Bruker was used for data acquisition. MestReNova 6.0.2 software (Mestrelab Research SL, Spain) was used for data processing.

HPLC-PDA Certification studies were conducted by high-performance liquid chromatography with photodiode array detection (HPLC-PDA) on Thermo Scientific Dionex UltiMate 3000 system (USA) and Waters UPLC Acquity I-Class system (USA). Columns: Acclaim Trinity P1 100 × 3.0 mm, 3 µm (Dionex, USA), HSS T3 50 × 2.1 mm, 1.8 µm (Waters, USA), Luna C18(2) 250 × 4.6 mm, 5 µm (Phenomenex, USA), and Primesep 100, 150 × 4.6 mm, 5 µm (Sielc Technologies, USA).

GC-FID Studies were conducted by gas chromatography with flame ionization detector (GC-FID) on Agilent 6890N system with injector 7683B (USA). Columns: HP-FFAP 50 m × 0.2 mm × 0.3 µm (Agilent, EUA), VF-17ms 30 m × 0.25 mm × 0.25 µm (Varian Factor Four, USA), and DB-5 ms 30 m × 0.25 mm × 0.25 µm (J&W Scientific, USA). For identification purposes, mass spectrometry (MS) detection was carried out with detector 5975B Inert XL MSD (Agilent, USA).

Headspace-GC-MS Residual organic solvents (ROSs) were determined by headspace-GC-MS on Agilent GC 7890A, headspace injector 7697A and Inert XL EI/CI MSD 5975C (USA), column DB-624 30 m × 0.32 mm × 1.8 µm (Agilent, EUA).

Karl Fischer Water was determined in Metrohm 831 KF Coulometer (Switzerland) with direct addition of samples to the titration vessel, and Metrohm 852 Titrando with 874 oven, titrator 800 Dosino (Switzerland) with either direct addition or oven transfer at 140 °C.

ICP-OES Inorganic elements were analyzed by inductively coupled plasma optical emission spectroscopy (ICP-OES) in Optima 8300 Perkin Elmer (USA) with standard addition procedure using IS and inverse calibration.

TGA Thermogravimetric analysis (TGA) was carried out in TGA/DSC 1 Star System Mettler Toledo (Switzerland) to determine mass loss at 120 °C (volatiles) and the residue after heating at 850 °C.

Freezing-point depression Differential scanning calorimetry (DSC) was used to determine purity of $DMSO_2$ and DMTP by the freezing-point depression method. Aliquots were weighed in a DSC Q2000 TA Instrument (USA).

Coulometric titration KHP was analyzed in the primary system of coulometry from Inmetro. Mass fraction of total acid (replaceable H^+) was expressed as KHP using Faraday constant of 96485.3365 C mol^{-1}, molar mass of 204.2236 g mol^{-1}, and density of 1640 kg m^{-3}.

Production and certification of candidate batches

Batch preparation MA, KHP, $DMSO_2$, and DMTP were sourced from Sigma-Aldrich. KHP was used as received, while MA and $DMSO_2$ were sieved to ensure uniform particle size. DMTP was ground (Retsch Grindomix GM200, Germany) and sieved for easier handling. All materials were homogenized and packed in 4 g portions into sealed amber glass bottles, with 200 units prepared per material.

Homogeneity Ten units were selected for the between-unit study, with three 20 mg subsamples from each. One unit was used to assess within-unit homogeneity and minimum sample size using six 5 mg subsamples. After adding IS to enhance method repeatability, samples were analyzed in triplicate via HPLC-PDA (MA, KHP, DMTP) or GC-FID ($DMSO_2$) using a randomized injection sequence.

Transport stability Short-term stability was evaluated using an isochronous design. Two units were stored at reference temperature (20–25 °C) and eight at 50 °C. Every 15 days, two units were removed from 50 °C and stored at reference temperature until analysis. Sample preparation and analysis followed the same procedure as the between-unit homogeneity study, with three subsamples per unit analyzed in triplicate.

Storage stability Long-term stability was assessed over 6 months at 20–25 °C. Every two months, two units were analyzed in triplicate by ^{1}H-qNMR. Time zero units were recapped after initial testing and reanalyzed alongside new units to check for subsampling effects.

Characterization Mass balance and IS qNMR were used for characterization studies of all the candidates. Coulometric titration was also used for KHP, and freezing-point depression for $DMSO_2$ and DMTP. Three units were analyzed in triplicate for each determination.

RESULTS AND DISCUSSION

Bulk reagent analysis

NMR spectra and GC-MS data confirmed the identity of the sourced reagents. Moisture analysis by Karl Fischer coulometric titration showed no detectable moisture or hygroscopic behavior under the tested conditions.

Homogeneity assessment

Approaches based on chromatographic response of analytes were used for the homogeneity assessment according to:

$$A_{corrected} = \frac{A_A}{w_S} \quad (2)$$

$$AR_{corrected} = \frac{A_A/A_{IS}}{w_S} \quad (3)$$

where A_A is the analyte area, A_{IS} is the IS area, and w_S is the mass fraction (mg g^{-1}) of the sample in the solution; IS was added to the diluent, so its mass fraction is the same in all samples and does not need to be considered in calculations.

The use of IS improved method repeatability for MA, $DMSO_2$ and DMTP (Table 1), with an expected larger effect on GC-FID analysis ($DMSO_2$) because injection system is usually less repeatable in GC than HPLC. Compared to that, KHP presented higher dispersion when IS was considered in calculation. This was attributed to a variation on IS area due to the proximity of its peak from void volume on KHP chromatographic analysis. Visual inspection of the plots of between-unit homogeneity based on $AR_{corrected}$ for MA, $DMSO_2$ and DMTP; and $A_{corrected}$ for KHP, showed that data did not present trends in experimental run order, neither in bottling order (data not shown).

Table 1 Influence of IS on homogeneity evaluation based on direct chromatographic area

Candidate	RSD (%) (*n*=30)		Technique
	$A_{corrected}$	$AR_{corrected}$	
MA	0.36	0.34	HPLC-PDA
KHP	0.38	0.55	HPLC-PDA
$DMSO_2$	6.16	0.33	GC-FID
DMTP	0.81	0.77	HPLC-PDA

Chromatographic methods based on direct area evaluation showed repeatability standard deviations (s_r) above the ISO 33405 precision recommendation for homogeneity studies [7]. Method precision ranged from 0.02% to 0.44%

(HPLC) and ~0.67% (GC), indicating that observed variability was more influenced by method repeatability and weighing than by true batch heterogeneity. This highlights the challenge of achieving low uncertainties for high-purity materials. Mass balance methods generally offer lower uncertainties. For DMTP, a partial mass balance was applied using HPLC-PDA impurity area normalization, which was acceptable due to its high purity. Acetic acid, which was the only other impurity detected in DMTP, was quantified by ^{1}H-qNMR for the batch characterization, but its low concentration suggested minimal impact on the homogeneity assessment. The partial mass balance approach showed excellent repeatability (0.0002% RSD) and was considered suitable for assessing the homogeneity of high-purity materials with a target uncertainty of 1 mg g^{-1} (0.1%). In contrast, the same batch evaluated using DMTP $AR_{corrected}$ showed a much higher RSD (0.77%) (Table 2). The lower variability of the mass balance method is mainly due to the absence of gravimetric sample preparation. The magnitude of this difference becomes clear from Fig. 1.

Table 2 Precision evaluation of the measurement methods used for homogeneity studies

Material	Parameter/ unit	Average	M_{within}	s_r	$s_r/\sqrt{3}$	$u_{trg}/3$
MA	$AR_{corrected}$	1.50×10^{1}	1.89×10^{-3}	4.34×10^{-2}	2.51×10^{-2}	5.00×10^{-3}
KHP	$A_{corrected}$	4.27×10^{8}	1.68×10^{12}	1.29×10^{6}	7.48×10^{5}	1.42×10^{4}
$DMSO_2$	$AR_{corrected}$	1.16×10^{1}	1.49×10^{-3}	3.86×10^{-2}	2.23×10^{-2}	3.86×10^{-3}
DMTP	$AR_{corrected}$	3.97×10^{0}	9.34×10^{-4}	3.06×10^{-2}	1.76×10^{-2}	1.33×10^{-3}
DMTP	mg g^{-1} *	1.00×10^{3}	2.86×10^{-6}	1.69×10^{-3}	9.76×10^{-4}	3.33×10^{-1}

*mass fraction considering the largest (approximately 0.056 mg g^{-1}) from two impurities detected during characterization; the smallest one presented signal-to-noise ratio < 3 in the method used for homogeneity

Within-unit homogeneity was evaluated using six 5 mg aliquots, each analyzed in triplicate. Standard deviations ranged from 0.06% to 0.16% across the materials, values consistent with method repeatability and the balance's uncertainty. No heterogeneity was detected, confirming 5 mg as an appropriate minimum sample size for certification.

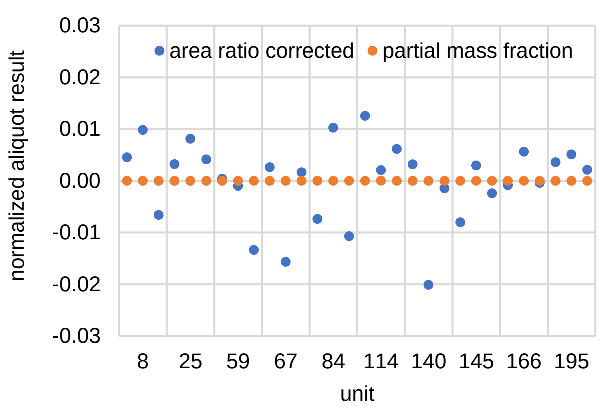

Fig. 1 Approaches to assess between-unit homogeneity for DMTP; both sets of data were intentionally plotted in the same scale to highlight the magnitude difference of dispersion.

Stability assessment

Transport stability of MA, KHP and $DMSO_2$ were evaluated by $AR_{corrected}$ results, while DMTP was assessed using the partial mass fraction approach to improve precision of results, as mentioned in the homogeneity study, at the cost of a more laborious data processing work. Data from stability at 50 °C presented no significant trend (see regression analysis results in Table 3 where $t_{b_1} < t_{crit}$), endorsing the suitability of transportation at room temperature for the candidate materials. Even without finding a significant trend, an uncertainty component associated with possible instability during transportation (u_{sts}) was estimated [7]:

$$u_{sts} = s(b_1) \times t_{max} \tag{4}$$

where $s(b_1)$ is the standard error for the estimated slope; and t_{max} is the maximum time stipulated for transporting the material.

Despite the study being conducted for 60 days at 50 °C, it is unlikely that transportation of the materials will exceed 30 days. Therefore, transportation time was limited to 30 days for the candidates with higher $s(b_1)$ (i.e. MA and $DMSO_2$) to reduce overall uncertainty of the CRM.

Table 3 Transport stability assessment data (as a function of time in days) and uncertainty estimation

Material	Parameter/ unit	$s(b_1)$	t_{b_1}*	Time	u_{sts}	$u_{sts\ rel}$ (%)
MA	$AR_{corrected}$	0.00034	1.7927	30	0.01037	0.06
KHP	$AR_{corrected}$	0.00009	0.4143	60	0.00563	0.09
$DMSO_2$	$AR_{corrected}$	0.00029	1.8929	30	0.00859	0.07
DMTP	mg g^{-1}	0.00002	1.4726	60	0.00107	<0.01

*t_{crit}=1.987 (0.95; v=88)

Storage stability was evaluated by qNMR. The units that were opened and analyzed in the first time point (time zero) were closed and re-sampled along with new units in the subsequent points to assess stability for repeated use. Regression analysis of datasets presented no significant trend (Table 4, where $t_{b_1} < t_{crit}$), proving batch stability under storage conditions and repeated use.

Table 4 Linear regression of mass fraction (mg g^{-1}) as a function of time (days) to assess stability under storage at room temperature and repeated use

Stability	Material	$s(b_1)$	t_{b_1}	t_{crit} (0.95)	v
Repeated use	MA	0.00127	0.9925	2.0322	34
	KHP	0.00068	0.0126	2.0369	32
	$DMSO_2$	0.00158	0.2587	2.0322	34
	DMTP	0.00144	1.1083	2.0423	30
Storage	MA	0.00054	0.0585	2.0057	53
	KHP	0.00123	0.0126	2.0106	48
	$DMSO_2$	0.00053	0.4531	2.0057	53
	DMTP	0.00175	0.5045	1.9977	64

Although no trend was observed in the long-term stability studies, the instability effect on CRM was conservatively considered and an uncertainty component (u_{lts}) was estimated by the equation [7]:

$$u_{lts} = s(b_1) \times (t_{m1} + t_{cert}) \qquad (5)$$

where t_{m1} is the time interval between value assignment and the initial stability monitoring point and t_{cert} is the period of validity of a certificate issued during that time.

Uncertainty due to long-term stability (u_{lts}) was estimated based on a 2-year validity period, resulting in 0.468 mg g^{-1} for MA, 1.073 mg g^{-1} for KHP, and 0.521 mg g^{-1} for $DMSO_2$. For DMTP, u_{lts} based on storage study data was 1.677 mg g^{-1}, but transport stability — assessed via partial mass balance — showed much lower variability, leading to a revised u_{lts} of 0.017 mg g^{-1} when transport stability $s(b_1)$ was used in u_{lts} estimation. Given the harsher conditions of the transport study and its higher precision, this lower value was adopted for DMTP. The magnitude of this difference is illustrated in Fig. 2, which shows the stability "triangles" as defined by Linsinger *et al.* [8]. The base of the triangles represents the uncertainty on the chosen shelf-life.

Fig. 2 highlights the advantage of using independent certification methods, as relying solely on qNMR would significantly increase the material's overall uncertainty. While higher uncertainty might be acceptable for general use, it could limit the CRM's suitability as a primary purity calibrator.

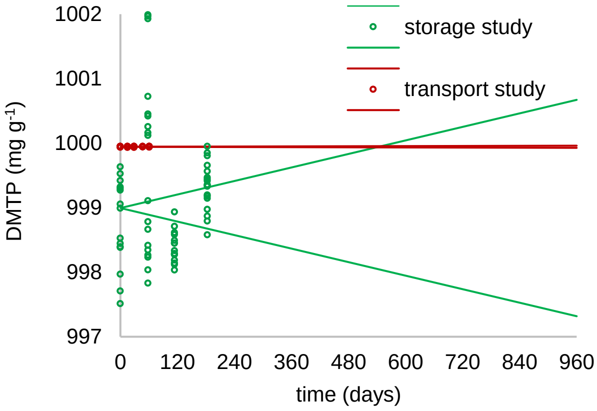

Fig. 2 Stability results for DMTP from transport study at 50 °C (obtained by partial mass balance) and from storage study at room temperature (obtained by qNMR); ascending and descending extrapolated lines were calculated by the intercept b_0 of each linear regression ± $s(b_1)$ × time.

Characterization

At least two independent measurement procedures were used for each CRM candidate to ensure a robust characterization methodology. All candidates were submitted to mass balance and qNMR determinations. Total acids in KHP were determined by coulometric titration, and $DMSO_2$ and DMTP were also analyzed by DSC using the freezing-point depression procedure according to ASTM E928-08 Standard Test Method for Purity by Differential Scanning Calorimetry [9]. All those methods are either primary or ratio primary measurement procedures (Table 5).

In the mass balance approach, when a group of impurities (i.e. structurally related impurities, water, ROSs, and inorganics) was not detected in the material, its mass fraction was assumed to be zero and an uncertainty allowance was estimated to account for residual impurity below the LoQ of the method. Besides the impurity categories usually investigated in the mass balance approach for organic materials (10), DMTP presented traces of acetic acid, which was detected and quantified by ^{1}H-qNMR. The occurrence of this impurity brings out the importance of a careful examination of analytical data when performing purity studies as acetic acid would not be detected in ordinary purity analyses by GC and reversed-phase HPLC.

Mass balance result (w_{MB}) was calculated by:

$$w_{MB} = \left(1000 - \sum w_{imp}\right) \times A_{norm} \tag{6}$$

where w_{imp} is the mass fraction of impurities and A_{norm} is the chromatographic area of the analyte normalized to the total area of the chromatogram.

Table 5 Characterization of CRM candidate batches

Measurement	MA		KHP		DMSO$_2$		DMTP	
	Value	*u*	Value	*u*	Value	*u*	Value	*u*
Water (mg g^{-1})	0.00	0.30	0.00	0.30	0.00	0.30	0.00	0.30
ROS (mg g^{-1})	0.0000	0.0043	0.0000	0.0043	0.0000	0.0043	0.0000 / 0.053^a	0.0043 / 0.012^a
Inorganics (mg g^{-1})	0.0017	0.0002	0.0230	0.0033	0.0027	0.0003	0.00000	0.00045
Area normalization	0.999932	0.000010	1.00000	0.00012	1.00000	0.00029	0.9999336	0.0000083
Mass balance (mg g^{-1})^b	999.93	0.30	999.98	0.32	1000.00	0.42	999.88	0.30
qNMR (mg g^{-1})^c	999.1	1.7	1000.0	1.3	1000.07	0.98	999.8	1.4
Coulometric titration (mg g^{-1})^d	NA	NA	1000.06	0.04	NA	NA	NA	NA
Freezing-point depression (mmol mol^{-1})	NA	NA	NA	NA	999.9	2.0	1000.0	2.0

^{a}Acetic acid as quantified by ^{1}H-qNMR

^{b}Used for value assignment of CRM batches

^{c}qNMR ISs: DMSO$_2$ CRM Inmetro 8783.0001, KHP CRM Inmetro 8943.0001, and KHP NIST 84L for MA; DMSO$_2$ CRM Inmetro 8783.0001, MA CRM Inmetro 8792.0001, and DMSO$_2$ TraceCert® BCBQ8884V for KHP; DMTP CRM Inmetro 8784.0001 for DMSO$_2$; and DMSO$_2$ CRM Inmetro 8783.0001 for DMTP

^{d}Total acid (replaceable H$^+$) expressed as KHP

Uncertainties were calculated following the GUM approach (11), resulting in values between 0.30 and 0.43 mg g^{-1} — slightly below the typical range reported by Westwood *et al.* (10), due to the high purity of the materials. While IS ^{1}H-qNMR yielded consistent results, its uncertainties were 2.3 to 5.7 times higher than those from the mass balance method, confirming the latter's superior precision for purity determination.

Coulometric titration and DSC offer direct mole fraction measurements but are limited to highly pure substances, with titration also requiring exchangeable protons. When applicable, both techniques were used to explore primary purity measurement methods. For KHP, coulometric titration measured total acid content and, since no acid impurities were present, the result directly reflected purity. The value agreed with mass balance and qNMR, with an uncertainty of 0.04 mg g^{-1}, matching previous reports (12). Freezing-point depression via DSC has limitations, requiring purity above 98.5 mol% and specific impurity solubility behavior (9, 13). Only DMSO$_2$ and DMTP met these criteria. While DSC results are in mol%, most CRM applications require mass fraction values. For DMSO$_2$, the impurity level was negligible, so no conversion was needed, and the DSC result (999.0 ± 2.0

mmol mol⁻¹) agreed with mass balance. For DMTP, the mass balance result was converted to mol%, and the DSC value (1000.0 ± 2.0 mmol mol⁻¹) was consistent with it.

Certified values

Characterization values were determined by mass balance and independently confirmed by qNMR using at least one National Metrology Institute (NMI)-certified CRM as IS, along with coulometric titration and DSC when applicable. This approach meets BIPM criteria and contrasts with studies relying solely on qNMR, which limits traceability. Combining mass balance and qNMR helps minimize potential biases. Final certified values (Table 6) included uncertainty contributions from purity assessment, transport, and storage. Uncertainty estimate attributed to heterogeneity was either overpowered by method repeatability (in the case of assessment through direct chromatographic area), and was hence disregarded; or was insignificant (in the case of partial mass balance approach for DMTP).

For most candidates, stability-related factors were the main contributors to combined uncertainty (Fig. 3), reflecting the precision limits of direct analyte determination methods (either chromatographic area for short-term study, or qNMR for storage study). DMTP was the exception, as its certification was based entirely on impurity determination via mass balance, resulting in minimal instability influence and a combined uncertainty 3 to 5 times lower than for the other materials.

CRM approval and use

After completing all laboratory work and data analysis, the candidate CRM batches underwent a two-stage review: an internal evaluation by Inmetro staff and a peer review by an expert from another NMI, ensuring compliance with certification requirements.

Table 6 Value assignment of CRM candidate batches

Study	MA		KHP		$DMSO_2$		DMTP	
	Value	*u*	Value	*u*	Value	*u*	Value	*u*
Characterization (mg g^{-1})	999.93	0.30	999.98	0.32	1000.00	0.42	999.88	0.30
Transport stability*	16.838	0.010	6.1186	0.0056	11.6681	0.0086	999.8805	0.0011
Storage stability (mg g^{-1})	999.62	0.47	998.5	1.1	1000.83	0.52	999.881	0.017
Certified value (mg g^{-1})^b	Value	*U* (*k*=2)	Value	*U* (*k*=2)	Value	*U* (*k*=2)	Value	*U* (*k*=2)
	999.9	1.7	1000	3	1000	2	999.88	0.60

*Units were $AR_{corrected}$ for MA, KHP and $DMSO_2$; and mg g^{-1} for DMTP

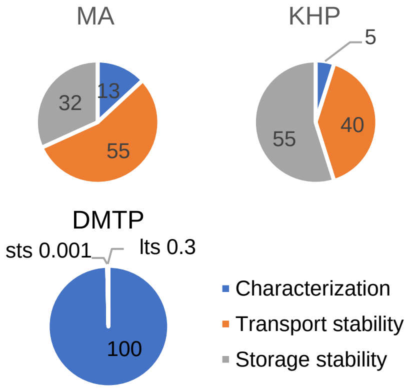

Fig. 3 Contribution (%) of uncertainty sources for the certified value uncertainty (square relative uncertainty components); *sts* refers to transport stability, *lts* refers to storage stability.

Successful peer review and Inmetro's acceptable performance in CCQM comparisons enabled the CRMs to be listed in the BIPM Key Comparison Database (KCDB). MA and $DMSO_2$ were covered by Inmetro's broad-scope CMCs, while KHP and DMTP received individual CMCs due to their specific characteristics.

The suite four CRMs demonstrate ideal characteristics for application in qNMR analysis, namely, chemical shifts across a typical ^{1}H-NMR spectrum (Fig. 4) and solubility in a range of polar to non-polar solvents. Demonstrated stability over a four-year period further confirms their suitability as CRMs.

These CRMs have supported the traceability chain for various Inmetro CRMs and proficiency test items, including drugs and pesticides analyzed by IS qNMR. They've been applied in studies involving substances like cocaine, methamphetamine, caffeine, acephate, carbofuran, and lambda-cyhalothrin. To foster qNMR use in metrology, CRM units were also sent to BIPM for distribution to other NMIs. Additionally, DMTP and MA were used in international key comparisons, such as CCQM-K148 and CCQM-K179.

The development of these CRMs led to the publication of a Portuguese-language guide on obtaining SI-traceable results using IS qNMR, freely available on Inmetro's website. Following its release, Brazilian universities and research institutes began adopting IS CRMs to advance the use of NMR for quantitative analysis.

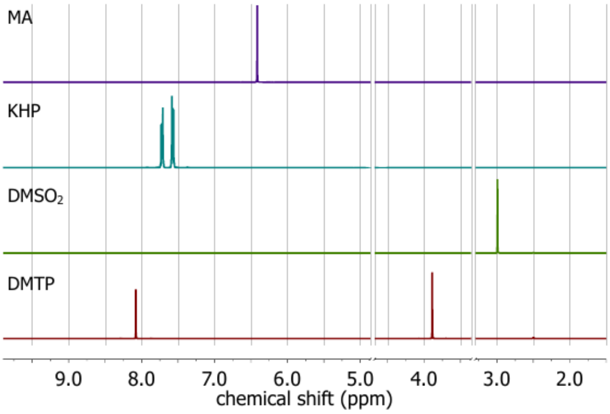

Fig. 4 ^{1}H-NMR spectra of MA and KHP in D_2O, and $DMSO_2$ and DMTP in DMSO-D_6; residual solvent signal regions (4.8 and 3.3 ppm) were excluded for ease of viewing.

Conclusion

A set of four high-purity CRMs was developed to support SI-traceable qNMR measurements, with mass fractions above 999.8 mg g^{-1} and expanded uncertainties between 0.6 and 3 mg g^{-1}. The study highlighted the advantages and limitations of primary and ratio primary methods for purity characterization, emphasizing the use of the mass balance approach to ensure traceability without relying solely on qNMR, in line with ISO 17034's risk-based recommendations. Simplified methods based on chromatographic area and area ratio were applied for homogeneity and stability studies, while mass balance procedures provided lower uncertainties, especially for DMTP (0.60 mg g^{-1}). The CRMs have been successfully applied to purity determinations of various analytes, strengthening metrological traceability in Brazil and beyond. Additionally, the work led to the publication of a practical guide on SI-traceable qNMR for the regional NMR community, fostering the use of high-accuracy measurements in research and industry.

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CITAC BEST PAPERS AWARD 2024

DEVELOPMENT AND APPLICATION OF REFERENCE AND ROUTINE ANALYTICAL METHODS PROVIDING SI-TRACEABLE RESULTS FOR THE DETERMINATION OF TECHNOLOGY-CRITICAL ELEMENTS IN PCB FROM WEEE

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Summary

The elements of the periodic table referred to as technology-critical elements (TCEs) are those elements that are subject to supply criticalities and are essential for the production of technological devices extensively used by the population. Supply criticalities could increase in the coming years based on the possible consequences of the changed international geopolitical and economic conditions. Moreover, there are also environmental criticalities linked to the impact of waste electrical and electronic equipment (WEEE) constituted by technological devices at the end of their life cycle whose quantity is constantly growing.

To address issues of supply shortage of TCEs and environmental impact of WEEE, the European Union has introduced the directives WEEE 2012/19/EU and RoHS 2011/65/EU. The first establishes collection and recycling targets for WEEE while the latter limits the use of hazardous materials in the production of electronic and electrical equipment. In this framework, the recovery and reprocess of TCEs from WEEE plays a fundamental role to provide materials for the production of new technological devices and to cover the management costs of subsequent collection and recycling.

Although the quantification of TCEs in WEEE is of primary importance for deciding recovery strategies, no specific and harmonized analytical methods are available. In addition, there are no certified matrix reference materials (CRMs) to validate or improve the analytical methods and/or establish traceability of the results to the International System of Units (SI). This has direct effects, e.g. difficulties in evaluating the economic values of WEEE and in comparing the efficiency of TCEs recovery processes.

Within the joint research project MetroCycleEU (20IND01, EMPIR programme co-financed by the Participating States and from the European Union's Horizon 2020 research and innovation programme), laboratories of National Metrology Institutes or Designated Institutes, Universities and Research Institutes aimed at the development and application of reference analytical methods based on inductively coupled plasma mass spectrometry (ICP-MS) with standard addition calibrations and Instrumental Neutron Activation Analysis (INAA) with k_0- and relative calibrations suitable to characterize WEEE matrix reference materials. In addition, as a further aim of the study for the benefit of recycling industries performing routine TCEs measurements, was the development of analytical methods based on more commonly adopted and easily implemented ICP-MS external standard calibrations.

Twenty elements (Ag, Au, Co, Cu, Dy, Ga, Gd, Ge, In, La, Li, Nd, Ni, Pd, Pr, Pt, Rh, Sm, Ta and Ti) were selected as the target analytes. A batch of powdered printed circuit boards (PCB) with 200 µm maximum particle size used as measurement material was obtained from shredding and milling of boards manually extracted from audio and video devices, toys, personal care products and domestic appliances.

The major problem addressed in quantitative elemental analysis performed with ICP-MS was the complete dissolution of the sample matrix in acid solution due to the complex matrix of the PCB material containing metals, plastics, organic substances and even ceramic silicates. Since no CRMs made from PCB were available to check the recovery, the digestions were optimized in each laboratory based on experience and in-house protocols. Overall, digestions in most cases were

carried out after a pre-ashing step and involved several acidic mixtures, and microwave assisted heating under elevated pressure. In addition, tetrafluoroboric acid (HBF4) was used as a substitute for hydrofluoric acid. Alkali fusion was also tested in order to achieve complete dissolution of the material.

In the light of the extraordinary complexity of the PCB matrix, the application of ICP-MS standard addition techniques following complete digestion was a suitable choice to develop reference methods, because the calibration within the matrix itself eliminates virtually completely biases arising from different sensitivities of the analytes between the sample matrix and the calibration standards. The same applies to matrix-matched standard addition techniques when sample matrix effects are fully compensated for. Simple external calibrations were adopted and applied as well to provide measurement methods for routine TCEs measurements.

INAA was also a suitable choice to develop candidate reference methods because measurements can be performed in the solid state without difficulties associated with PCB digestions. Samples were prepared by pressing tablets or using aliquots of the powdered material directly. Measurement procedures were developed by adjusting neutron irradiation, radioactivity decay and gamma counting times to reduce or, in some cases, to cancel out matrix interferences. The k_0 single comparator and the relative direct comparator calibrations were adopted.

All target analytes except Rh were quantified in the range within mass fractions of μg g^{-1} (Pt) and 0.1 g g^{-1} (Cu). Reference methods produced data in full mutual agreement for Ag, Au, Co, Cu, La, Li, Pr, Sm and Ta with relative expanded uncertainties ranging within 0.8% (La) and 7.2% (Ag), while some disagreement was observed for Dy and La. In addition, all but one of the routine methods produced data in agreement with the reference value or within ± 20% of it.

In conclusion, the developed ICP-MS standard addition and INAA k_0 and relative methods have proven to be fit for purpose of characterizing the matrix reference materials made from end-of-life PCB. Moreover, analytical methods used in industries working in WEEE recycling can be harmonized starting from the developed ICP-MS external calibration methods whose performance is sufficient to determine the economic and strategic value of PCB through TCEs quantification.

CITAC BEST PAPER AWARD 2024

NAVIGATING THE COMPLEXITY: MANAGING MULTIVARIATE ERROR AND UNCERTAINTIES IN SPECTROSCOPIC DATA MODELLING

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In recent years, I've often found myself returning to a recurring question: *How can we trust what we don't fully understand?* This thought became especially pressing in the context of spectroscopy and chemometrics, where we build increasingly complex multivariate models—but often neglect the foundational question of data quality.

This was the motivation behind our latest review. Although numerous papers have explored individual facets of error analysis and uncertainty estimation in spectroscopic data, the literature felt fragmented. Together with the coauthors, we decided it was time to consolidate the scattered knowledge into a single, structured resource—a guide that could support chemists and spectroscopists navigating these issues in their daily work.

What We See and What We Often Miss

Infrared spectroscopy, combined with chemometrics and powered by modern computing, has enabled extraordinary advances in analytical chemistry. We can classify, quantify, and explore complex systems with tools that are now even portable and widely deployed. Yet, despite these advances, one important aspect still tends to receive less attention than it deserves: the analysis of measurement errors.

In our review, we emphasize that spectroscopic data is inherently noisy and complex. Errors don't behave in simple ways—they're often heteroscedastic or correlated, stemming from various sources including instrumental limitations, sample variability, and environmental conditions. Ignoring this complexity can severely compromise model performance and interpretability.

A Collective Effort, A Shared Message

This work was a collaborative effort in the truest sense. Each coauthor brought unique perspectives and technical depth that enriched the review beyond what any of us could have done individually. Our shared conclusion was clear: if chemometrics is to be taken seriously as an analytical discipline, it must adopt the same rigor as traditional analytical chemistry.

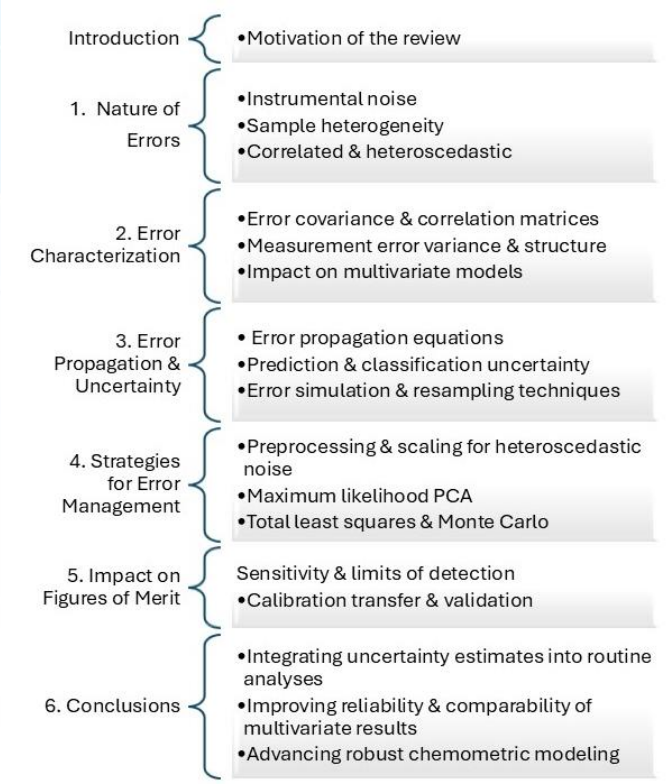
In several cases, chemometric models are shared without comprehensive information on aspects such as bias, uncertainty, or figures of merit. This can make it more challenging—particularly in industrial or regulatory settings—to fully evaluate a model's reliability. In traditional univariate methods, these elements are standard practice, and bringing chemometrics to the same level of methodological transparency will support its broader acceptance and application.

What We Propose

In this review, we outline concrete ways researchers can approach multivariate error analysis, adapt preprocessing methods to the nature of their data, and compute uncertainty with appropriate statistical tools. We discuss error propagation in the context of classification and prediction, and we highlight the impact that error structure has on figures of merit like Q-residuals or Hotelling's T^2.

Ultimately, we hope to encourage a broader shift in perspective within the field:

- First, by promoting a more systematic evaluation of measurement errors in both predictor (X) and predicted (Y) variables, to better inform data treatment strategies and model selection;
- Second, by highlighting the value of reporting uncertainty estimates alongside results obtained through multivariate models, to enhance their interpretability and comparability.



Introduction
- •Motivation of the review

1. Nature of Errors
- •Instrumental noise
- •Sample heterogeneity
- •Correlated & heteroscedastic

2. Error Characterization
- •Error covariance & correlation matrices
- •Measurement error variance & structure
- •Impact on multivariate models

3. Error Propagation & Uncertainty
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- •Prediction & classification uncertainty
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4. Strategies for Error Management
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5. Impact on Figures of Merit
- Sensitivity & limits of detection
- •Calibration transfer & validation

6. Conclusions
- •Integrating uncertainty estimates into routine analyses
- •Improving reliability & comparability of multivariate results
- •Advancing robust chemometric modeling

A Final Word

To quote Paul de Bièvre, 'A result without a reliability statement is not (yet) a result.' That statement resonated with us throughout the writing of this review. We hope our contribution serves not just as a resource, but also as an invitation to reflect more deeply on the quality and robustness of spectroscopic data analysis.

ABOUT CITAC

CITAC - Cooperation on International Traceability in Analytical Chemistry - arose out of an international workshop held in association with the Pittsburgh Conference in Atlanta in March 1993. The aim of this workshop was to discuss how analytical activities could be developed to meet the needs of the 21st century, and it identified a wide variety of issues to be addressed to ensure that analytical measurements made in different countries or at different times are comparable. These range from the development of traceable reference materials and methods to the harmonisation of analytical quality practices.

The CITAC initiative aims to foster collaboration between existing organisation to improve the international comparability of chemical measurement. A Working Group takes matters forward and its initial activities have centred on a few specific high priority activities. The first tasks included the compilation of a directory of certified reference materials under development; preparation of quality system guidelines for the production of reference materials; preparation of a directory of international chemical metrology activities; defining criteria for establishing traceability to the mole; and the preparation of an international guide to quality in analytical chemistry.

Many of these activities are of a strategic nature, laying the ground for the improvement of international analytical measurement. This reflects the added geographical complexities associated with a world-wide organisation, such as greater diversity in culture and in technical approach, and frequently long timescales associated with its activities. Nevertheless, if the full benefits of improved analytical measurement are to be realised internationally, a truly global approach is needed, and there is a clear role for CITAC to play in this respect.

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CITAC
Cooperation on International
Traceability in Analytical Chemistry

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