

Chemistry

Comparison on CO₂ isotope ratio standards

Preparation for a comparison on CO₂ isotope ratio standards (CCQM-P204), coordinated jointly by the BIPM and the IAEA continued, with the protocol prepared and distributed and twenty institutes expressing interest in participation. Development of isotope ratio measurement and gas preparation facilities continued with support from visiting scientists from VNIIM (Russian Federation) and NPL (UK). A paper describing the optimized optical measurement facility has been submitted for publication. Preparations for the comparison of nitrous oxide in air at background levels coordinated by the BIPM together with KRISS (CCQM-K68.2019) continued, with comparative measurements performed at the BIPM at the end of 2019 and completed into the first quarter of 2020.

FTIR Measurements on Gas Standards course

Two visiting scientists from the KEBS (Kenya) and KazInMetr (Kazakhstan) undertook the Metrology for Clean Air course on FTIR Measurements on Gas Standards in the BIPM laboratories. They received training in the use of B-FOS software for use with FTIR in gas metrology applications.



Left to right: E. Flores (BIPM), R. Gunn, NPL (UK), C. Solo, KEBS (Kenya) undertaking a six month training programme at the BIPM on FTIR for Gas Standards and P. Thompson, NPL (UK). The NPL sponsored visiting scientists to participate in the Metrology For Clean Air CBKT programme at the BIPM

Air quality measurement standards

The BIPM comparison programme in support of air quality measurement standards continued, focusing on ozone, nitrogen oxides and formaldehyde. The BIPM continued to contribute to the CCQM GAWG Ozone Cross Section Task Group, including the publication of a paper summarizing the recommended best value and uncertainty for the ozone cross-section to be used in the key comparison BIPM.QM-K1^[18]. Organization of a workshop on the implementation of the new cross-section value with stakeholder communities in September 2020 at the BIPM has started. Seven NMIs: ISCIII (Spain), KRISS (Republic of Korea), NIST (USA), JRC (EC), EEA (Austria), UBA (Germany) and

SE Ukrmetrteststandard (Ukraine) sent their ozone standards to the BIPM and participated in BIPM.QM K1, as well as a comparison for NSW (Australia). The Final report of CCQM-K137 (NO in N₂ at 30 μmol/mol and 70 μmol/mol) was completed and published in the BIPM key comparison database^[19]. Measurements at the BIPM for the comparison of NO₂ in N₂ standards at 10 μmol/mol (CCQM-K74.2018) were completed. Additional measurements for the pilot study of HNO₃ measurements in such standards (CCQM-P172) have been performed on a new set of standards. The work that was carried out on the stability of formaldehyde standards as part of the preparation for CCQM-K90 has been published^[20].

International comparison demonstrates accuracy of the global atmospheric CO₂ measurement scale

The results of the CCQM comparisons on CO₂ in air standards (CCQM-K120 and CCQM-P188) were presented at the 20th WMO/IAEA Meeting on Carbon Dioxide, Other Greenhouse Gases, and Related Measurement Techniques (GGMT-2019), held in Jeju (Republic of Korea) from 2-5 September 2019. This coincided with the presentation of an updated CO₂ scale (WMO-CO₂-X2019), the primary reference for the WMO-GAW monitoring network on which all global background observations of CO₂ are based.

The scale will be used by the WMO Global Atmosphere Watch programme in its global monitoring network for tracking trends in the background CO₂ amount fraction in the atmosphere. The accuracy of the scale was demonstrated in the key comparison (CCQM-K120), organized by the CCQM Working Group on Gas Analysis (CCQM-GAWG) with comparative measurements performed at the BIPM. The comparison involved the analysis of the composition of 46 gas reference materials from 13 NMIs and The National Oceanic and Atmospheric Administration (NOAA), the institute designated by the WMO.

Progress in reducing uncertainties in SI traceable standards has led to a key comparison reference value for CO₂ in air concentrations with state-of-the-art uncertainties of parts in 10⁴. This has enabled benchmarking of international comparability and provided support for an update to the global scale.

The developments to the global scale recognize the substantial collaborative progress made between the metrology and atmospheric monitoring communities since the WMO signed the CIPM MRA in 2010.

Organic and protein primary calibrators

In the area of organic and protein primary calibrators, the BIPM programme of comparisons for ensuring global equivalence of standards for small organic molecule and protein calibrators continued. The final report of CCQM-K78.a, a comparison on multi-component amino acid calibration solutions, was completed and published^[21]. Measurements for the CCQM-K148.a comparison (bisphenol A calibrator purity) were completed, and results presented to the meeting of the CCQM Working Group on Organic Analysis (CCQM-OAWG).

Internal Standards for qNMR

Support for laboratories using Quantitative NMR to value assign primary reference materials was provided through the development of four reference data documents on 'Internal Standards for qNMR' for Dimethyl Terephthalate, 3,5-Bis(trifluoromethyl) benzoic Acid, 1,4-Bis(trimethylsilyl)benzene and 4,4-Dimethyl-4-silapentane-1-sulfonic acid-d6. These documents were published on the BIPM website as BIPM Rappports 2019/01^[22], 2019/02^[23], 2019/03^[24] and 2019/04^[25]. A paper^[26] describing the universal calibrator programme for qNMR at the BIPM, an activity initiated together with the NMIJ, and supported by visiting scientists was published, together with a review article on the applications of qNMR^[27]. A visiting scientist from BAM (Germany) was seconded to the BIPM to further develop qNMR techniques for ¹⁹F NMR.

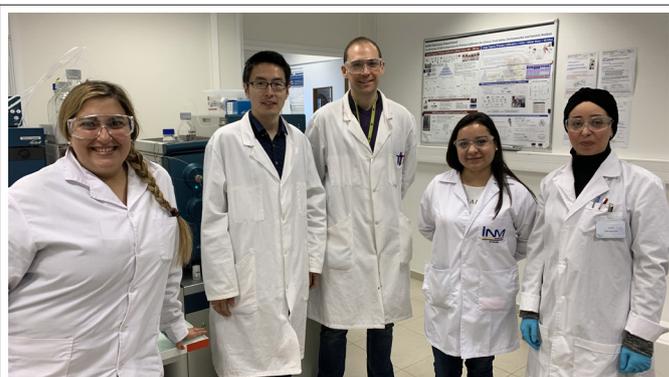
Peptide calibrator materials

Measurements for the comparison on oxytocin (peptide calibrator material) coordinated by the BIPM (CCQM-K155.b) were completed, and the results and Draft A report were presented at a meeting of the CCQM Working Group on Protein Analysis (CCQM PAWG). Samples for comparisons on hexapeptide calibrators for HbA1c glycated hexapeptide (GE), for CCQM-K115.c, and non-glycated hexapeptide (VE), for CCQM-K115.2019, were distributed by the BIPM to participating NMIs. A review article on purity assessment of peptide calibrators was published^[28]. Visiting scientists from NIMT (Thailand), LNE (France) and NPL (UK) were seconded to the BIPM to develop methods for the characterization of candidate peptides for comparisons, and notably hexapeptides for HbA1c and HbA0, parathyroid hormone with ion mobility mass spectrometry, and triskelion peptides.

Metrology for Safe Food and Feed

In April 2019, the fourth meeting for the CBKT programme on "Metrology for Safe Food and Feed" was held at the BIPM, focusing on mycotoxin metrology and standards. The BIPM laboratory programme on mycotoxin standards was supported by a visiting

scientist from NIM (China) working on aflatoxin B1, deoxynivalenol and patulin materials. Three visiting scientists from LATU (Uruguay), INM (Columbia) and INRAP (Tunisia) undertook three-month training secondments on mycotoxin calibration solution and characterization, focusing on Aflatoxin B1 calibrants. Stock solutions of AFB1 have been provided to the ten NMIs that have participated in the programme to date (KEBS, INTI, NMISA, NIMT, INMETRO, UME, LATU, INM, INRAP, NIM), in preparation for the second key comparison (CCQM-K154.b) on mycotoxin calibration solutions. Measurements for the key comparison on Zearalenone calibration solutions (CCQM-K154.a) were completed at the BIPM, and the Draft A report was presented at the meeting of the CCQM Working Group on Organic Analysis (CCQM-OAWG). A paper and guidelines describing the characterization of AFB1 calibrants was published^[29,30], as well as guidelines on the evaluation of purity of ZEN primary reference materials^[31]. An agreement with NRC (Canada) to extend and provide materials for a comparison on Ochratoxin A calibrants, which is foreseen for 2023, was concluded. A three-day workshop held from 4-6 November 2019 in Riyadh (Saudi Arabia) on "Metrology for Safe Food and Feed - Organic Analysis and Standards" was organized for GULFMET as part of the BIPM 'Metrology for Safe Food and Feed Programme' Capacity Building and Knowledge Transfer programme.



Visiting scientists L. Casas, LATU (Uruguay), L. Morales Erazo, INM (Columbia) and R. Torkhani, INRAP (Tunisia) undertaking three-month training secondments on mycotoxin calibration solution production and characterization, focusing on Aflatoxin B1 calibrants, together with Xianjiang Li, NIM (China) and G. Martos (BIPM)

25th anniversary of the CCQM

A CCQM Workshop on "Advances in Metrology in Chemistry and Biology", marking the 25th anniversary of the CCQM, was organized at the BIPM in April 2019, and resulted in twenty publications in the related *Metrologia* special issue, including a paper on Amount of substance and the mole in the SI^[32].