Bureau International des Poids et Mesures

Consultative Committee for Amount of Substance: metrology in chemistry (CCQM)

Report of the 15th meeting
(22–24 April 2009)
to the International Committee for Weights and Measures

Comité international des poids et mesures

Note:

Following a decision of the International Committee for Weights and Measures at its 92nd meeting (October 2003), reports of meetings of the Consultative Committees are now published only on the BIPM website and in the form presented here.

Full bilingual versions in French and English are no longer published.

A.J. Wallard,
Director BIPM

LIST OF MEMBERS OF THE CONSULTATIVE COMMITTEE FOR AMOUNT OF SUBSTANCE: METROLOGY IN CHEMISTRY
as of 22 April 2009

President

Dr R. Kaarls, member of the International Committee for Weights and Measures.

Executive Secretary

Dr R. Wielgosz, International Bureau of Weights and Measures [BIPM], Sèvres.

Members

Centro Nacional de Metrología [CENAM], Querétaro.

D.I. Mendeleyev Institute for Metrology, Rostekhregulirovaniye of Russia [VNIIM], St Petersburg.

Danish Fundamental Metrology Ltd [DFM], Lyngby.

Federal Office of Metrology [METAS], Bern-Wabern.

Institute for Reference Materials and Measurements [IRMM].

International Atomic Energy Agency [IAEA].

International Federation of Clinical Chemistry and Laboratory Medicine [IFCC].

International Organization for Standardization, Committee on Reference Materials [ISO REMCO].

International Union of Pure and Applied Chemistry [IUPAC].

Korea Research Institute of Standards and Science [KRISS], Daejeon.

Laboratoire National de Métrologie et d'Essais [LNE], Paris.

National Institute of Metrology [NIM], Beijing.

National Institute of Standards and Technology [NIST], Gaithersburg.

National Measurement Institute, Australia [NMIA], Lindfield.

National Metrology Institute of Japan, National Institute of Advanced Industrial Science and Technology [NMIJ/AIST], Tsukuba.

National Metrology Institute of South Africa [NMISA], Pretoria.

National Physical Laboratory [NPL]/Laboratory of the Government Chemist [LGC], Teddington.

National Research Council of Canada, Institute for National Measurement Standards [NRC-INMS], Ottawa.

Physikalisch-Technische Bundesanstalt [PTB]/Bundesanstalt für Material-forschung und -prüfung [BAM]/Federal Institute for Materials Research and Testing, Braunschweig and Berlin.

Slovak Institute of Metrology/Slovenský Metrologický Ústav [SMU], Bratislava.

State Laboratory [SL], Co. Kildare.

Technical Research Institute of Sweden [SP], Borås.

VSL [VSL], Delft.

The Director of the International Bureau of Weights and Measures [BIPM], Sèvres.

Observers

Agency for Science, Technology and Research [A*STAR], Singapore.

Bulgarian Institute of Metrology, General Directorate "National Centre of Metrology" [BIM], Sofia.

Central Office of Measures/Glówny Urzad Miar [GUM], Warsaw.

Centro Español de Metrología [CEM], Madrid.

Cooperation on International Traceability in Analytical Chemistry [CITAC], Trappes.

Hungarian Trade Licensing Office [MKEH], Budapest.

Istituto Nazionale di Ricerca Metrologica [INRIM], Turin.

National Institute of Metrology, Standardization and Industrial Quality [INMETRO], Rio de Janeiro.

National Metrology Institute of Turkey/Ulusal Metroloji Enstitüsü [UME], Gebze-Kocaeli.

National Physical Laboratory of India [NPLI], New Delhi.

National Physical Laboratory of Israel [INPL], Jerusalem.

1 OPENING OF THE MEETING; APPOINTMENT OF RAPPORTEURS; APPROVAL OF THE AGENDA

The Consultative Committee for Amount of Substance: metrology in chemistry (CCQM)* held its 15th meeting at the International Bureau of Weights of Measures (BIPM), at Sèvres on 22–23 April 2009.

The following were present: H. Andres (METAS), R.J.C. Brown (NPL), G. Carroll (SL), P. Charlet (LNE), K. Chiba (NMIJ/AIST), P. Corbisier (IRMM), M. Cox (NPL), P. De Bièvre (IUPAC/CIAAW), S. Ellison (LGC), H. Emons (IRMM), A. Fajgelj (IAEA/IUPAC), M. Fernandes-Whaley (NMISA), B. Güttler (PTB), Q. Han (NIM), H.D. Jensen (DFM), R. Kaarls (President of the CCQM), K. Kato (NMIJ/AIST), Y. Kustikov (VNIIM), H. Li (NIM), L. Locascio (NIST), L. Ma (NIM), L. Mackay (NMIA), B. Magnusson (SP), M. Máriássy (SMU), W.E. May (NIST), M.J.T. Milton (NPL), Y. Mitani (CENAM), U. Panne (BAM), H. Parkes (LGC), M. Pérez-Urquiza (CENAM), S. Prins (NMISA), C. Quétel (IRMM), M. Sargent (LGC), L. Siekmann (IFCC), H.-Y. So (KRISS), R. Sturgeon (NRC), W. Unger (BAM), S. Vaslin-Reimann (LNE), A.J. Wallard (Director of the BIPM), S. Wise (NIST).

Observers: H.S. Brandi, O. Cankur (UME), H.A. Chua (A*STAR), V.S. Da Cunha (INMETRO), R. Daroda (INMETRO), P.K. Gupta (NPLI), I. Kuselman (INPL, CITAC), M.P. Sassi (INRIM), M. Sega (INRIM), Z.N. Szilágyi (MKEH).

Invited: M. Buzoianu (INM), J.S.H. Chan (HAS), C. Cherdchu (NIMT), S. Sik-Man Choi (GL), P. Chui (HSA), D.K. Dikshit (CDRI), T. Eklin (MIKES), J.N. Gikubu (KEBS), J. Kioko (KEBS), T.K. Lee (HSA), G. Massiff (Fundacion Chile), H.K. Rotich (KEBS), V.D. Sattigeri (CFTRI), K.-D. Sommer (PTB), P. Totarong (NIMT), G. Turk (NIST), S. Valkiers (IRMM).

Also present: A. Daireaux (BIPM), L. Érard (CIPM, LNE), E. Flores Jardines (BIPM), R. Josephs (BIPM), M. Kühne (BIPM Deputy Director),

* For the list of acronyms, click here.

S. Maniguet (BIPM), P. Moussay (BIPM), A. Picard (BIPM), T.J. Quinn (Director Emeritus of the BIPM), C. Thomas (Coordinator of the KCDB, BIPM), J. Viallon (BIPM), S. Westwood (BIPM), R. Wielgosz (Executive Secretary of the CCQM, BIPM).

Apologies: T. Fernández Vicente (CEM), E. Gray (NIBSC), A. Squirrell (SM, ILAC, NATA), T. Steiger (BAM), P. Taylor (IRMM), A. van der Veen (VSL, ISO REMCO).

Dr Kaarls, the President, welcomed members, observers and invitees to the 15th meeting of the CCQM, noting that an exceptionally lengthy agenda necessitated an earlier than usual start this year. Prof. Wallard, Director of the BIPM, also expressed a warm welcome to all, further adding that the CCQM is the largest of the Consultative Committees (CCs) and this in itself contributes to the lively and comprehensive discussions. Prof. Wallard then introduced and welcomed Prof. Michael Kühne, the newly appointed BIPM Deputy Director who had joined the organization several weeks previously and will succeed Prof. Wallard at the end of 2010. Prof. Kühne noted that he is himself a spectroscopist in synchrotron radiometry but also had experience in contact and ultra-low temperature thermometry and had served as chairman of EURAMET from June 2006 to April 2009. He stated that the overlap between Prof. Kühne and Prof. Wallard would be of significant benefit to him and he asked for and thanked participants for their help during this transition period.

Dr Kaarls proposed that Dr Sturgeon act as rapporteur for the meeting. Dr Sturgeon agreed. Dr Wielgosz would assist him.

The agenda was approved without change.

2 REPORT ON THE 14TH MEETING OF THE CCQM

The report of the 14th meeting was approved and Dr Sturgeon thanked for his efforts.

3 REPORTS FROM THE CCQM *AD HOC* WORKING GROUPS

3.1 *Ad hoc* Working Group on the KCRV

Dr M. Cox, chairman of the Key Comparison Reference Value Working Group (KCRVWG), outlined the terms of reference of his group (established by the President in April 2007), i.e. to examine the various approaches currently in use for the determination of the KCRV and its uncertainty, and to produce a set of guidelines that would be adopted by the CCQM for the calculation of these values. Once established, it would become feasible to link calibration and measurement capabilities (CMCs) with results of key comparisons (KCs) in a more transparent manner. He enumerated a set of thirteen data evaluation principles (document CCQM/09-03). The relationship between performance in a key comparison (KC) and a claimed CMC was at present limited to conditions under which a 1:1 correspondence between the matrix, the measurand and its amount fraction and the measurement procedure were clearly established. In cases where the deviation of a laboratory's result from the reference value was smaller than the uncertainty of the degree of equivalence, then an uncertainty no smaller than that achieved in the comparison could be in the CMC claim without further supporting evidence. Consequently, for a participant having results inconsistent with the KCRV, a justifiable value of any CMC uncertainty claim must be larger than its declared uncertainty in the KC. Future steps included the promotion of these principles within the CCQM in an effort to identify their limitations; the preparation of a tutorial on the most common estimators used for establishing the KCRV and their impact on degrees of equivalence for the November meeting; and an investigation into ways of addressing harmonization of claimed uncertainties in certified reference materials (CRMs) in relation to the claimed CMC uncertainties.

Dr May opened the discussions arising from the presentation, noting that the proposed 1:1 consideration is too limiting. Furthermore, he stated that the uncertainty associated with a CRM is based on different measurement capabilities from that tested with a KC and hence the linkage is tenuous. Dr Emons agreed with Dr May on both items and also objected to the bias from the reference value in a key comparison being added to the uncertainty of a CMC. Dr Ellison replied that in such a case the detected bias was not being used as a correction for the uncertainty of a CMC, but

rather as a criterion to judge whether the uncertainty of the CMC was consistent with the laboratory's performance. A number of delegates supported the 1:1 correspondence examples, as it was important to understand how to link measurements to claims in the most straightforward cases, but it was acknowledged that in general such conditions could only be rigorously met in the domain of gas analysis. Dr Sargent proposed that special circumstances should be noted for real sample matrices.

The President concluded by noting an urgent need for transparency and guidance so that there would be a clear understanding of what underpins a CMC. A tutorial with practical examples for discussion and harmonization was needed. Dr Cox reiterated that the principles were posted on the website and input to improve the language and understanding was welcome.

3.2 *Ad hoc* Working Group on Efficient and Effective Testing of CMC Claims

The *Ad hoc* Working Group on Efficient and Effective Testing of CMC Claims (EETWG) was established by the President in April 2007 and mandated to provide recommendations to the CCQM on how best to deal with the ever increasing problem of the need for more and more key comparisons arising from the current *modus operandi* by developing policies to minimize the number needed to underpin the CIPM MRA without compromising their effectiveness. Dr Turk, chairman of the WG, elaborated on a system to test CMC claims based on demonstrations through KCs of the "core capabilities" required to deliver the CMC. He outlined the current concepts independently adopted by the CCQM Working Groups on Organic Analysis (OAWG), Gas Analysis (GAWG) and Inorganic Analysis (IAWG). He then elaborated on the model developed by the IAWG, based on a consideration of instrumental methods of analysis, giving ID-ICP-MS as a detailed example. It was noted that P-118 (Toxic elements in algae) was to serve as a demonstration of the utility of the system. Unresolved issues include the relationship between the degree of equivalence and uncertainty of multiple KC results and the CMC uncertainty; and how to combine the uncertainties of several studies, and the implications of a poor KC result (which core capability failed and which CMCs are thereby affected). Implementation of the model requires adoption of a new format for the organization of information describing comparisons. This would make it

easier to decide if new comparisons should go ahead by taking into account related and planned CMCs, KCs and pilot studies (PSs).

Dr Quinn opened discussions by endorsing the approach as a practical way forward. There was general agreement that the model would assist in the selection of new KCs. Dr Mitani noted that differences remained in approaches used by the various WGs but no clear harmonization was evident. Dr Mackay stated that the approach taken by the EETWG did not raise any concerns for the KCWG and recognizes that it is an evolving model.

4 REPORT AND DISCUSSION ON REDEFINITION OF THE MOLE AND DRAFT *MISE EN PRATIQUE*

4.1 Overview of the Avogadro project

In view of the future redefinition of the kilogram, Mr Picard reviewed progress in the Avogadro project. An initial relative uncertainty of 15×10^{-8} was substantially higher than the targeted value of 2×10^{-8} for the Avogadro constant. From the relationships linking the Avogadro constant (N_A) to the molar mass, density and lattice parameter for the Si sphere, it was shown that the determination of N_A using the X-ray crystal density (XRCD) method was limited by the uncertainty associated with the determination of the molar mass of Si with natural isotopic composition. Dr Picard summarized the multi-institute efforts undertaken since April 2008 when two 1 kg ^{28}Si enriched spheres were polished at the former CSIRO and released for study. Parameters relating to roundness, volume, lattice parameter, mass, surface oxide identity and thickness, density and molar volume were all characterized using X-ray diffraction, mass spectrometry, interferometry, weighing and ellipsometry techniques. A relative uncertainty of 4.1×10^{-8} had been achieved. Dr Picard went on to discuss the relationship between N_A and the Planck constant, pointing out that there remains a -1.08×10^{-6} relative bias in h values derived from watt balance experiments and that derived from N_A. Recent availability of synthetic isotope primary measurement standards for silicon 28, 29 and 30 have permitted a more accurate calibration to be implemented for the molar mass such that a corresponding shift of $+1.2 \times 10^{-6}$ in N_A has brought the results of the XRCD method to within 0.1×10^{-6} of the 2006 CODATA value.

Some discussion ensued and concern was expressed about the stability of the sphere as it may oxidize over time, but Dr De Bièvre correctly pointed out that this was not an issue as the sphere was only a measurement artefact and once the measurement data were acquired, its stability would no longer be of importance.

4.2 Isotope number ratio measurements on natural and enriched silicon

Dr Valkiers summarized efforts at the IRMM to undertake isotope amount ratio measurements on natural and highly enriched ^{28}Si to establish its molar mass in support of the determination of N_A by XRCD methodology. The -1.08×10^{-6} discrepancy in the determination of h by watt balance experiments and XRCD data along with the fact that crystal imperfections can account for $<10^{-8}$ U_{rel} leaves molar mass as the final parameter that merits additional investigation. Further reduction in the uncertainty of N_A to $<10^{-8}$ (relative) could be accomplished by reducing the effect of ^{29}Si and ^{30}Si isotope fractions to the level of small corrections for the molar mass of ^{28}Si. This has been accomplished using an isotope ratio gas mass spectrometer with calibration based on gravimetrically blended solutions of enriched ^{29}Si and ^{30}Si. The newly determined molar mass for ^{nat}Si WASO 04 crystal brought the value of N_A into agreement with the 2006 CODATA value. Work was ongoing with further improvements to the experimental protocol designed to reduce the relative uncertainty in the molar mass of ^{28}Si to $<1 \times 10^{-8}$.

Discussions centred on technical aspects of the establishment of the mass discrimination factor (K) for the spectrometer and the need to control and accurately assess contamination from ^{nat}Si arising from the chemical processing steps.

4.3 IDMS method for the relative molecular mass of silicon

Dr Schiel outlined an alternative approach under way at the PTB based on classical solution nebulization sample introduction and multi-collector ICP-MS for the determination of amount content of ^{29}Si and ^{30}Si in enriched ^{28}Si by treating the system as a (virtual) two-isotope element impurity. A relative uncertainty in molar mass of $<1 \times 10^{-8}$ was targeted. This could be obtained by achieving a 10^{-3} uncertainty in the determined isotope ratios. Mass

discrimination/fractionation factors were accounted for using gravimetrically prepared blends of enriched ^{29}Si and ^{30}Si calibration standards. Preliminary results appear encouraging and the approach has the advantage of simplified chemistry and sample introduction without the need to measure ultra-low detector currents.

Concerns were expressed regarding the impact of impurities from contamination sources such as the sodium hydroxide used for sample dissolution, especially considering that these account for some 25 % of the gross signal intensities.

4.4 Draft *mise en pratique* for the realization of the mole

Dr Milton presented a historical overview of the origins of the mole and the definition of the term "amount of substance". The latter has evolved from its present definition, "a quantity proportional to the number of specified elementary entities N in a sample" to the proposed definition, "a quantity that measures the size of an ensemble of entities", noting that "it is proportional to the number of specified entities and the constant of proportionality is the same for all substances". A newly proposed redefinition refers to the mole as "the amount of substance of a system that contains exactly $6.022\,1415 \times 10^{23}$ elementary entities". This is equivalent to the statement that "the amount of substance of a system is such that N_A is exactly $6.022\,1415 \times 10^{23}$ per mole". In such cases, the exact number of entities in one mole could be specified but we would not know the exact mass of one mole. Fundamentally, there was no objection to moving to a definition of the mole based on a fixed value for the Avogadro constant as it is, in fact, an amount of substance. There is an urgent need to debate the issue widely and promote this through publications such as Dr Milton's recent paper in *Chemistry International* (Vol. **31**(2), 2009) and a future paper for *Metrologia*. The *Ad hoc* Working Group assembled and chaired by Dr Milton in April 2007 (comprising Drs Besley, De Bièvre, Kaarls, Milton, Salit and Wielgosz) will further develop these actions. A draft Recommendation (Q 1 (2009)) to be submitted by the CCQM to the CCU in May 2009 was distributed for comment and throughout the course of the meeting was subjected to several iterations, essentially recommending that "any decision on redefining the mole and the kilogram be deferred until the discrepancy between results from the watt balance and XRCD measurements be resolved; that agreement is demonstrated between values of N_A derived from independent measurements of the isotope amount ratios of Si on samples of both ^{nat}Si and ^{28}Si and, at that time, full consideration be given to

redefining the mole by fixing the value of N_A and to the interests of the chemical measurement community; and that the BIPM, NMIs and other official representatives in the CCs increase their efforts to spread awareness of the changes contemplated to the various scientific, industrial, and professional organizations and seek their views at an early stage". A previous recommendation from the CCQM had stated that any decision on redefining the kilogram should wait until the discrepancy in the value of the Planck constant arising from the watt balance and the X-ray crystal density/molar mass measurements had been resolved. The largest source of uncertainty in the molar mass measurement arose from the isotope amount ratio measurements, and even with the isotopically enriched samples the influence of contamination from naturally abundant silicon needed to be accounted for. Therefore, the decision on the redefinition of the kilogram indeed depended on ensuring accurate and reliable silicon amount ratio measurements and this was the reason for the CCQM's recommendation to refer to both the mole and the kilogram.

Significant discussion ensued, but with near unanimous support for favouring the proposed redefinition, as it appears that technological barriers to the resolution of the above-noted 1×10^{-6} discrepancy could soon be favourably resolved. Dr Anders (an author of the METAS article "There is no rationale for redefinition of the mole") argued that a more conservative approach should be adopted as neither definition would have impact on any practical work and the need for change could only be substantiated if the community began counting atoms or molecules. The President urged everyone to read the METAS article carefully but that in his opinion it was the only objection so far registered for not proceeding with the recommended change to the redefinition of the mole. Dr Wielgosz commented that whereas the concept of the amount of substance has been clarified, it may be necessary in the future to consider counting individual entities rather than an ensemble of entities. This sentiment was echoed by Dr De Bièvre, who mused on the possibility of a basic deficiency in the SI in that a base unit of one was missing, which would be applicable to counting entities. Such a concept could assume more importance as detection techniques are improved to allow routine measurement of single atoms or molecules. There was considerable discussion on whether the document would comment only on the mole or on the kilogram as well. Drs Wielgosz and De Bièvre stated that the Avogadro project work to refine the molar mass of Si had been undertaken to resolve the discrepancy in the value of the Planck constant arising from the watt balance and the X-ray crystal density/molar mass measurements, and the

resolution of this discrepancy was an important criterion for any decision to proceed with the redefinition of the kilogram. Furthermore, the earlier presentations had demonstrated that the resolution of the discrepancy would require accurate and validated chemical measurements, and the CCQM was the appropriate Consultative Committee to comment on these. The President summarized by saying that considering the importance of chemical measurements in considerations on the redefinition of the kilogram, the CCQM recommendation should address both the mole and the kilogram. He charged Drs Milton and De Bièvre to re-examine the final wording. The subsequent final iteration, examined by the members, states the CCQM's preference for a redefinition of the mole, the SI unit of amount of substance, based on a fixed value of the Avogadro constant.

The text of Recommendation Q 1 (2009) is reproduced at the end of this report.

Drs De Bièvre and Fajgelj stated that the IUPAC will support the proposal and table the information at the upcoming General Assembly meeting in Glasgow (United Kingdom). Concerns were then expressed over the level of consultation being achieved within the chemical community, noting that perhaps a specific BIPM webpage addressing the issue would be useful. Dr May agreed on the need for a proactive outreach to the community and noted that the President of the American Chemical Society will host a Presidential Event focused on the redefinition of the kilogram at one of the American Chemical Society's National Meetings next year. He further noted that Dr Richard Davis, Head of the BIPM Mass section, presented a well-received Plenary Lecture on the redefinition of the kilogram at this year's National Meeting of the National Organization for the Professional Advancement of Black Chemists and Chemical Engineers (NOBCChE).

5 METROLOGY FOR BIOFUELS

Dr Charlet summarized the outcomes of the first European meeting on metrology for biofuels (BioFuels Met 2008) organized by the PTB and LNE and convened in Strasbourg, November 2008. Some 70 participants exchanged ideas to better understand the metrology required to support production and use of biofuels. Most NMIs were represented. Among the

outputs were the recognition that metrology requires competence in diverse fields and that NMIs should focus on achieving traceability of important components of biofuels; produce the required reference materials; devise practical methods for tracking the provenance of biofuels ("trackability"); provide training; support proficiency testing (PT) exercises; and take a leadership role on the issue of biofuels.

Dr May expressed concern that the presentation appeared to indicate that NMIs should recommend that biofuels be supported more vigorously than other alternative energy sources, and this would be a mistake. Furthermore, NMIs are typically not expert in issues of sustainability. Dr Emons challenged the conclusion that NMIs should take the lead in this field, where they have limited experience. Dr May reiterated that the development of metrics to measure sustainability was important but NMIs may not be interested in this exercise. Dr Sommer added that NMIs should not be addressing sustainability *per se* but rather developing methods and traceability of measurements, which was a metrology *versus* political activity.

The President noted the activities of BIOREMA, an EU project coordinated by INMETRO, NIST and VSL which aims to assess the comparability of laboratories analysing biofuels. Dr May outlined the status of this project, which arose from a White Paper Report (http://www.nist.gov/public_affairs/biofuels_report.pdf) developed by Brazil, the European Community and the United States in 2007, highlighting that a bioethanol material would be delivered from INMETRO as well as a FAME biodiesel material from the IRMM. The use of the two biofuel materials as samples for key comparisons and pilot studies had been discussed in the EAWG, IAWG and OAWG. Dr May noted that the OAWG had agreed that the samples could be used in comparisons and had decided on a list of analytes that would be measured and could underpin CMC claims. Dr Sargent stated that there were five or six elemental analytes that could be measured in the materials. He noted that the level of impurities in the bioethanol sample were likely to be too low to be of interest for a comparison and that the lack of characterization information on these materials was likely to prevent many NMIs from engaging in comparison exercises, and if amount contents of measurands could be approximately specified this would substantially improve the situation. Dr Brown stated that additional information on these samples would be available by autumn 2009, in time for the November meeting in Rio de Janeiro. Dr Máriássy stated that the conductivity of the samples could be a measurand of interest for a comparison but that the pH of ethanol was

operationally defined and not of interest to the Working Group members. Dr Charlet pointed out that research programmes on biofuel analysis methods were currently being developed under the European Metrology Research Programme (EMRP), and it would be appropriate to use the samples for pilot studies rather than key comparisons at this stage. Dr Emons noted that the BIOREMA project had to respect its deadlines and that the samples would be available for only a limited time. The President urged that the issues be left in the hands of the EAWG, IAWG and OAWG for future consideration once additional information becomes available characterizing the samples.

6 REPORT FROM NIST OCTOBER 2008 BIOSCIENCE CONFERENCE

Dr May highlighted the aims and outcomes of the "Accelerating Innovation in 21st Century Biosciences, Identifying Measurements, Standards and Technological Challenges" meeting held at the NIST in October 2008. Designed to provide a listing of measurement, standards and technology needs to inform and guide research at the NIST and the measurement and standards community worldwide, outcomes could be used as a roadmap for planning future activities. Bioscience has emerged as a new and significant field and a strategic planning process was under way at the NIST in order to leverage funding to aid emerging industry and technology. The conference identified and prioritized measurement barriers to innovation, focusing on medicine, energy, manufacturing, agriculture and the environment. A significant number of experts from industry, academia and government sector laboratories participated. Much of the presentation targeted the NIST's current and future activities in healthcare with the idea that standards to support next-generation healthcare measurements will facilitate the transformation of medical practice from an art to a science.

Mrs Parkes noted the significant overlap of many NIST selected activities with existing NMI activities, asking how this would roll out in practice, to which Dr May replied that the NIST welcomed extended collaboration.

7 JOINT COMMITTEE ON TRACEABILITY IN LABORATORY MEDICINE (JCTLM)

7.1 JCTLM WG1

Dr Wielgosz reported on the current status of the JCTLM and its WG1 on Reference Materials and Measurement Methods. Its mandate was to review nominated higher-order reference materials and methods for publication in a quality-assured JCTLM database. The list of materials was divided into two parts – one for which SI traceability was available, and one for which it was not. Some 226 CRMs are currently listed along with 146 reference methods spanning 71 analytes. A members' and stakeholders' meeting held at the BIPM in December 2008 reviewed the past six years of activity and examined future challenges. Dr Wielgosz highlighted a number of the presentations made during the meeting, notably on the revision of ISO 15194, which was now consistent with the ISO REMCO guides; the support that the JCTLM provides to industry/manufacturers (procedures, CRMs and a network of reference laboratories); issues of variabilities in reference limits and the need to standardize these demonstrated by a study performed in Australia; and new IFCC initiatives relating to efforts being made to develop reference methods for selected currently non-SI-traceable analytes. Future activities included a new call for nominations for CRMs, Reference Measurement Methods (RMMs) and Reference Measurement Services (RMSs) (deadline 30 April 2009), a WG meeting scheduled for July in concert with the AACC conference in Chicago and a JCTLM Executive meeting at the BIPM in December 2009.

7.2 JCTLM WG2

Prof. Siekmann described the work of WG2 of the JCTLM, which is concerned with reference measurement laboratory services. He reported that 111 services from 19 laboratories are currently listed on the JCTLM database. Evidence of (annual) participation in PT schemes and a quality assessment of the laboratory in accordance with ISO/IEC 17025 and 15195 (or an NMI with relevant CMCs listed in Appendix C of the KCDB), and the use of a JCTLM-listed reference method are requirements for listing. In 2003, the IFCC created a proficiency testing system for use by reference

laboratories (EQAS); the number of measurands has risen to 195. Participating laboratories (there are now 38) are identified and must submit their values and expanded uncertainties as well as their methodology to the coordinator. At least five laboratories are needed to provide a reference value.

He presented the results of ring trials (Youden plots) among laboratories conducting measurements of glucose, cholesterol, thyroxin and the enzymes ALT and AST in human serum. The results of these comparisons were available from the DGKL website (http:/www.dgkl-rfb.de:81). Prof. Siekmann then discussed the calibration and measurement hierarchy in laboratory medicine with NMIs showing competence via the CIPM MRA with KCDB results positioned at the top, followed by reference or calibration laboratories demonstrating competence via ISO/IEC 17025 and 15195 that use JCTLM methods and participate in ring trials, followed by routine testing laboratories that are accredited to ISO/IEC 15189 and regularly participate in PT exercises. Thus a traceability link is established through the use of reference methods and reference materials. It was therefore important that NMIs list their reference measurement services in the JCTLM database.

Cycle 1 Reference Measurement Laboratory Services received 200 nominations with 89 being accepted, whereas cycle 2 comprised only 33 nominations with less than 50 % being accepted.

Prof. Kühne expressed his support of these activities, noting that at the highest levels of metrology we have good CMCs, but that NMIs have to ensure that there are mechanisms in place to transfer these values to routine test laboratories to provide high-quality data, otherwise the system simply would not work.

8 SUMMARY OF THE CCQM PHARMA AND BIO-PHARMA WORKSHOP

Dr Wielgosz summarized the main points of the “Measurement Traceability for Pharmaceutical and Bio-pharmaceutical Measurements” workshop convened at the BIPM in December 2008. Issues addressed by the workshop included developments and requirements for analytical measurements and measurement systems to ensure the comparability and consistency of

pharmaceutical and bio-pharmaceutical products; regulatory requirements for the characterization of pharmaceutical and bio-pharma products; the development of reference materials and methods; strategies for the value assignment of the properties of materials; trends in physico-chemical characterization of biological materials; identification and quantification of impurities in pharmaceutical and bio-pharma products; the challenge of defining the measured property in bio-assay measurements and the unit in which it is expressed, and the requirements and possibilities of establishing traceability to the SI. Key presentations from the United States Pharmacopeia USP, the European Pharmacopoeia and the Japanese Pharmacopoeia as well as industry were highlighted.

Dr Kaarls noted that a follow-on workshop may be organized to determine the contributions of NMIs to this field. Mrs Parkes recognized that CCQM WGs are making contributions to the development of reference procedures for some bio-pharma measurands and that there was now recognition that measurement tools are available that could augment or replace some of the bioassay characterization studies. Discussion of terminology in use by the pharmacopoeias arose, noting that attempts to suggest harmonization with the VIM may be difficult and certainly protracted.

Dr Mitani noted that the accreditation of the USP to ISO Guide 34 may bring more CRMs onto the market and aid in harmonization of this sector. Dr Wielgosz noted that such new CRMs would probably be introduced only gradually, and that the diagnostics industry certainly required CRMs for the active ingredients of pharmaceutical products that were monitored in patients.

9 REPORTS OF CCQM WORKING GROUPS

9.1 Organic analysis

Dr May reported on the progress made by the CCQM Working Group on Organic Analysis (OAWG), which had met twice since the last meeting of the CCQM, in November 2008 at NIMT, Bangkok, with 41 participants from 29 institutes and earlier that week at the BIPM, with 51 participants representing 29 institutes. He then presented an update on OAWG strategic planning to efficiently and effectively support CMCs in the organic CMC

"space"; reports on comparison studies; plans for a quantitative nuclear magnetic resonance (NMR) symposium; and comments on the CIPM/JCRB Traceability Document.

After outlining the OAWG terms of reference, he noted that over the past ten years some 20 KCs targeting 50 measurands and 41 pilot studies on 126 analyte-matrix combinations had been undertaken with eleven comparisons currently in progress and five in the queue. It was clear that this level of effort could not continue; a new strategic framework was needed to define a finite number of comparisons that would test the institutional knowledge and core competencies required to deliver services recognized under the CIPM MRA, rather than the techniques. Three types of comparison were proposed: (A) key comparisons that test core competencies for the delivery of measurement services to customers; (B) key comparisons that assess the equivalence of measurement services actually provided to customers; and (C) key comparison studies in emerging areas of global interest and importance with an accompanying pilot study. To move forward with these priorities, an internal task force was created to agree on set of core competencies; design a limited set of studies to test the core competencies required for carrying out organic analyses; and report back to the OAWG for ratification at the November meeting. Additionally, he proposed that all CRMs and PT samples for the same matrix/analyte combination listed as "Mechanisms for service delivery" should be tested to assess their comparability in order to assure the integrity of the CIPM MRA and to assess the effectiveness of the overall CMC and QS review processes. Concerning emerging topical areas of interest, each participating NMI would be required to identify and provide supporting information as to which of the core competencies were tested in the comparison based on the methodology they used.

A brief discussion of the merits of testing all CRMs ensued, with strenuous objections from Dr Emons who contended that the issue was not comparability of materials but correctness of the certificates. The President was strongly in favour of such a comparability study as it was a real test of the mechanisms used for service delivery to customers and already common practice in the CCQM GAWG. Dr Wielgosz commented that the GAWG studies had been very successful, but it was worth remembering that such studies were highly dependent on the laboratory carrying out measurements on all samples, the uncertainty they attributed to their measurements, and the matrix independence of the method used.

Dr May then outlined progress in key comparisons and pilot studies.

- CCQM-K55.a [in parallel with CCQM P117.a] (Purity assessment of high purity organic materials: 17β-Estradiol). This exercise was coordinated by the BIPM and involved 21 participants (12 KC labs and 9 PS labs). The majority of participants used the "mass balance" or "100 % minus impurities" concept, wherein the levels of related structure, water, residual solvent and non-volatile impurities are independently estimated. Subtraction of the sum of the mass fraction of each of these classes of impurities from the theoretical maximum provides the estimate for the mass fraction content of the main component. Most disagreement among participants' results arose from differences in the estimation of water content as well as assigning estradiol dimers that formed *in situ* as being true impurities. The KCRV will be calculated using a consensus best estimate of the levels of each class of impurity as the basis for a mass balance calculation of the 17β-estradiol content of the comparison sample.
- CCQM-K69 (Anabolic steroids in freeze-dried human urine: testosterone glucuronide) in parallel with CCQM-P115 (Anabolic steroids in urine: testosterone glucuronide and epitestosterone glucuronide) attracted three laboratories for each study. Results from four laboratories were in good agreement for testosterone glucuronide with a 1.8 % relative standard deviation (RSD) for three KC laboratories. A Draft B report will be submitted to the November meeting and an approach to the calculation of the KCRV decided at that time.
- CCQM-P91 (Pesticides in foods: pyrethroids in apple juice) was coordinated by the NIM. Good agreement among the majority of the ten participating laboratories was evident. The median of the results would be used, with an associated uncertainty being the robust estimate of the standard deviation (MADe). Preparation of a revised draft report would include the reference values and their uncertainties.
- CCQM-P114 (Selected PBDEs and PPBs in plastic) was coordinated by the IRMM with good agreement among participants whose capabilities for extraction, separation, calibration, quantification and analyte degradation due to thermal instability were tested. The revised draft summary report to be prepared will include reference values and uncertainties and a proposal will be forwarded for a subsequent KC.
- The status of other studies in progress was then briefly covered, including CCQM–K62 [in parallel with CCQM-P78.1] (Nutrients in infant/adult formula: vitamins); CCQM-P88 (Antifungals in food: malachite green in fish); a follow-up to CCQM-P90 (Chloramphenicol in

muscle tissue) and a proposal for CCQM-K55.b (Aldrin), the next measurands for purity study and a related calibration solution comparison. Requested studies that remain tabled include quinolones in pork, tetracyclines in poultry and acetylcholine in microdialysate.

The BIOREMA project, which is to make available homogeneous and stable samples of bioethanol and biodiesel (FAME), raises the possibility of imminent comparisons to test measurement capabilities for ethanol and water content in bio-ethanol as well as water, methanol, free glycerol, triolein and methyl esters of linolenic, linoleic, oleic and stearic acids.

Dr May outlined plans for a half-day symposium on quantitative NMR to be organized by the LGC for the November meeting, focusing on the use of NMR for direct purity assays as well as for quantitative trace analysis.

Minor amendments to the CIPM/JCRB Traceability Document were tabled.

Dr Kaarls again requested that all comments relating to the Traceability Document should be received by the end of the week. Dr Emons noted that the wording should ensure that traceability was to the SI and not to an institute.

9.2 Inorganic analysis

Dr Sargent reviewed the work of the CCQM Working Group on Inorganic Analysis (IAWG). The group had met twice (jointly with the EAWG) since the last CCQM, at the IAEA Vienna and Seibersdorf in October 2008, and workshop on method validation had just taken place at the BIPM. During the October meeting, a joint workshop with the IAEA on technical challenges of standards and CRM production was run in addition to a tour of the IAEA Seibersdorf laboratory. A Gantt chart of all current pilot and key comparisons was presented, allowing him to conclude that a healthy portfolio of activities was under way.

Dr Sargent subsequently reported on two key comparisons and four pilot studies completed since the last reporting period.

- CCQM-K60/P86.1 (Total Se and Se methionine in wheat flour) coordinated jointly by the LGC and NRC, attracted nine NMIs for the KC as well as four NMIs and ten universities or institutes for the pilot study. Participants were supplied with a solution of ^{78}Se-mehionine for ID-MS protocols. This exercise was more challenging than the preceding

pilot CCQM-P86 because the amount content of the SeMet was some 50-fold lower. Despite this, the four NMIs submitted data that were in good agreement, with a spread of the mean of under 2 % RSD.

- CCQM-K64 (Analysis of Cu alloy), coordinated by the BAM, involved analysis of a lead-containing brass sample for which Pb, Sn, Ni and Fe were impurity measurands and Cu was determined by electrogravimetry. Five NMIs returned excellent results for Cu whereas results which were considered to be outliers were observed for Pb (two outliers in the reported results) and Fe, Ni and Sn (one outlier for each of these analytes).
- CCQM-P96 (As and arsenobetaine (AB) content in marine fish), coordinated by the NMIJ, revealed unexpected and surprisingly discordant results among its five participants. The problem was traced to differences in the amount contents of (certified) calibrants, resulting in a major experimental investigation and identification of problems with this study. The ICP-MS technique was determined not to provide a response independent of the form of the measurands and, more importantly, BCR CRM 626 and NMIJ 7901-a were found to be respectively 15 % and 20 %, lower than their certified amount content. It was planned to rerun CCQM-P96 at a later date once these issues have been settled.

A second technical issue placed before the IAWG was a proposal from the UNIIM to consider detection of moisture in grain. It was noted that the IAWG does not consider moisture a measurands *per se* but it is frequently determined as a correction for calculation of dry mass. The issue will be discussed with CCT WG6 (Humidity) and other CCQM WGs.

Dr May noted that this same issue arose some three years ago within the OAWG, to which Dr Kaarls responded that the subject was clearly in need of some reflection.

Dr Sargent summarized future comparisons, which may include a KC for Pb in lead-free solder (coordinated by the NMIJ) as a possible successor to CCQM-P119; activities relating to biofuels, i.e. BIOREMA's proposal for two KCs/pilot studies, inorganic calibration solutions, and an isotope ratio determination.

He then summarized the development of the IAWG strategy for more efficient and effective testing of CMCs which fostered the development of core competencies to avoid the need for a 1:1 correspondence between KCs and CMCs. It was hoped that the performance of all NMIs can be

benchmarked once per year with participation in a small number of KCs. To date, two such exercises have achieved that aim, CCQM-K49/P85 and CCQM-P106; a third was now proposed, CCQM-K75/P118 (toxic metals in algae).

Dr Sargent addressed the need to respond to the CIPM/JCRB Traceability Document, stating that the JCRB draft (version 3) requires further revision with clearer wording and correct terminology; that there are serious problems, summarized by the IAWG, in implementing the guidelines and these also need to be addressed in the next revision; that if the CIPM and JCRB believe an appropriate revision of the generic document is not (fully) feasible, the CCQM should prepare an annex addressing any remaining issues raised by the IAWG, which could take the form of a statement such as: "CCQM believes the following specific current practices in metrology in chemistry are compliant with the guidance …".

Suggestions were also offered for revising the KCWG document "Guidelines on the review of chemistry CMCs", particularly noting that specific requirements be summarized in a separate document or annex (as opposed to a description of the overall review process) and that section 8 addresses only a specific case of a 1:1 correspondence which rarely occurs in the IAWG.

Future activities include meetings 4–6 November 2009 (INMETRO, Rio de Janeiro) and 12–13 April 2010 (BIPM) with proposals for future meetings from the SP, Borås (2010) and the NMIA, Sydney (2011). The third benchmarking exercise identified Ni and Pt as measurands for CCQM-K75/P118 (toxic metals in algae), which would test the core capability matrix concept. New KCs and/or pilot studies would be organized to fill identified gaps.

In conclusion, Dr Sargent stated that good progress had been made with comparisons, noting that the technical information received from participants had improved but that there were still delays in completing some reports. A strategy to link key comparisons with CMCs was now nearing implementation. Pilot studies continue to play a useful role in assisting newer NMIs with training and obtaining experience while providing a vehicle for the more experienced NMIs to address problems and new areas of interest. Two meetings per year were considered cost effective as they ensure that the work progresses steadily while allowing sufficient time for a wide range of technical presentations and discussion.

Dr Kuselman stated his belief that the determination of moisture was important for many measurements and should be considered to be within the purview of the CCQM.

9.3 Gas analysis

Dr Milton noted that the WG had met twice since the last meeting of the CCQM, at Bangkok in October 2008 during which a workshop was held to consider general strategies for future activities of the GAWG and "Gas metrology to support measurements of the atmosphere and ambient air quality", and a second at the BIPM earlier that week. He then turned to a brief discussion of the strategy used by the GAWG which was developed to help streamline the key comparison process by working on the concept of core species and their concentrations. He then highlighted the status of a number of current comparisons.

- CCQM-K51 (CO in nitrogen at 5 μmol/mol) was coordinated by the NMISA and attracted 25 participating laboratories of which only five did not achieve a degree of equivalence crossing the zero point.
- CCQM-K65 (methyl- and ethyl mercaptan at (20–30) μmol/mol in methane), piloted by the VNIIM, had four participants, all having satisfactory degrees of equivalence. This work was required to underpin CMCs for volatile sulfurous compounds in methane and nitrogen in the range (10–100) μmol/mol.
- CCQM-K68 (nitrous oxide), coordinated by the KRISS, demonstrated the comparability of measurements at the level of 320 nmol/mol, showing excellent degrees of equivalence and good agreement with the NOAA Global Monitoring Division Reference Laboratory. The target level is typical of current ambient N_2O levels arising from sources in the oceans, soils and anthropogenic emissions.
- CCQM-K71 (multi-component stack gas emissions), coordinated by the VSL, targeted work on a multi-component mixture relevant to monitoring of typical industrial stack-emission gases. Measurands targeted include μmol/mol levels of NO, SO_2, CO, CO_2 and C_3H_8, with nitrogen as the balance. Good agreement among participants was evident, and the effect of cross interferences had been discussed.
- CCQM-K46 (ammonia in nitrogen), coordinated by the VSL, brought together a group of seven NMIs using a variety of independent

measurement techniques that returned a suite of data characterizing ammonia at a nominal mole fraction of 30 µmol/mol with significant dispersion in the results (–6 % to +1.5 % relative deviation). Static gravimetric and dynamic methods based on permeation tubes had been used by laboratories, and further work by participants had not resolved the discrepancy in results. Thus, a consensus mean reference value would be considered. The results of the comparison were considered to be representative of the current state of the art for these measurement standards.

- CCQM-K53 (oxygen in nitrogen) was a preparative comparison coordinated by the KRISS which had developed a very high accuracy comparison method based on GC-TCD. Based on a developed regression approach, it was concluded that four laboratories should not be included in the calculation of the KCRV.
- CCQM-K54 (n-hexane in nitrogen) was coordinated by the VSL with seven of the eight participating laboratories submitting data that contributed to the KCRV, the outlier being excluded because of a contamination issue.

Ongoing activities included CCQM-K66 (Impurities in methane), coordinated by the NMIJ, and CCQM-K74/P110 (NO_2 in N_2), coordinated by the BIPM. This latter study was particularly interesting as it would allow the comparison of results obtained using Fourier-transform infrared (FTIR) with traceability to reference spectra, and allow NMIs to assess the uncertainty of this approach, particularly important for purity analysis in cases where calibration gas standards do not exist.

Progress was made in developing approaches to compare gas standards that could be generated using dynamic methods (mass loss through a permeable membrane). A questionnaire developed by the METAS identified interest among NMIS for five such measurands (NO_2, SO_2, NH_3, benzene, formaldehyde). The METAS will carry out some validation studies in preparation for a comparison based on one or more of these species.

Several proposals for new comparisons included methane in air (BIPM), which would be a preparative exercise that would also permit the testing of CRM production, SO_2 in nitrogen (NIST), which is important for trading in stack gas emissions and synthetic refinery gas (VSL), which presents an analytical challenge because of the presence of alkenes and hydrogen.

In an effort to harmonize CMCs in the gas area, guidelines for ozone were drafted and all laboratories asked to resubmit their claims based on the adopted criteria. For CRMs, all laboratories were asked to provide more information on their certification/preparation processes.

In connection with the need to plan for more efficient and effective testing of CMCs and to streamline the process used for their approval and review, the GAWG has been working on the idea of "core species and concentrations". Analysis of KCs has shown that performance for a range of species over a range of concentrations is largely constant and thus performance in a limited set of comparisons using key facilities and competencies can "shine the light" over all "core" species within specified ranges of concentration. In addition to comparisons of core species (those that utilize generic techniques for their analysis, are stable in cylinders and for which standards are prepared from gases), Dr Milton defined an additional comparison type, an "analytical challenge" (for which species may not be stable in cylinders and preparation of standards may be complex). Using these definitions, he proceeded to show that long-term NMI performance is stable and not a function of the concentration of the measurands for core species and certain species in natural gas. This raised several options for future approaches but favoured the option in which two statements of "How far the light shines" (HFTLS), i.e. the range of CMCs that could be underpinned from the results of a key comparison, could be made for each key comparison: one based on existing principles and a second, much wider statement of HFTLS, covering all the core species and concentrations.

Future challenges raised by the GAWG include nanoparticles and trace water vapour detection.

A brief discussion ensued, with Dr May pointing out that because the NOAA laboratories were not designated institutes under the CIPM MRA and had no quality system, they were not eligible to participate in KCs. Dr Fajgelj suggested that a term other than "species" should be used to group measurands, to which Dr Milton replied that this was the terminology in use and preferred by the industry.

9.4 Electrochemical analysis

Dr Máriássy reported on the work of the CCQM Working Group on Electrochemical Analysis (EAWG), which had met twice since the last

meeting of the CCQM (both joint meetings with the IAWG, at the IAEA Vienna and Seibersdorf in October 2008 and earlier that week at the BIPM. He presented the results of one key comparison and three pilot studies.

- CCQM-K34.2 (assay of KHP) was coordinated by the SMU and follows similar comparisons from previous years (CCQM-K34 and CCQM-K34.1). The participants, INMETRO and UNIIM, showed agreement with the earlier studies.
- CCQM-P83 (electrolytic conductivity) coordinated by the DFM sought to expand the demonstrated capabilities of participants to lower levels than previously assessed by the CCQM-P47 and CCQM-K36 comparisons. Generally, good agreement was achieved among the ten participants but the samples tested had 100-fold higher conductivity than ultra-pure water.
- CCQM-P111 (seawater salinity), coordinated by the PTB, attracted 24 participants (13 NMIs, 11 oceanographic laboratories) for measurement of conductivity. Determination of practical salinity is a significant parameter used by oceanographers and climatologists due to its impact on global temperatures. In addition, the composition of International Association for the Physical Sciences of the Oceans (IAPSO) reference seawater was also studied. This comparison was important as it provided a comparison of results that were traceable to the SI with results that were traceable to a conventional sea salinity scale. With the exception of Sr^{2+} and SO_4^{2-}, the composition of the standard was confirmed.
- CCQM-P112 (assay of EDTA) was coordinated by the BAM with support from the PTB and SMU. A protocol similar to that used for CCQM-P46 was followed, in that all samples were sent to one laboratory where the EDTA was titrated with a solution of zinc of known purity after 99 % of theoretical equivalence was chelated with Cu^{2+}. Samples had been prepared at pH 5 and pH 9; leading to identical results except in one case where alkaline earth impurities which are liberated from their complexes in more acidic solution were suspected. Titration curves revealed contamination by nitrilotriacetic acid, a common impurity in EDTA, in some samples. This and other issues relating to buoyancy corrections and even the molar mass of zinc were discussed. Results for some participants revealed apparent purities in excess of 100 % (resulting from the method of calculation and because of varying degrees of hydration of the sample material).

Dr Máriássy summarized the agreed and planned comparisons for 2009 and 2010, concluding that key comparisons targeting pH now span the range 1.5 to 10 and are considered complete and will only be readdressed in future years, at which point it is likely that a pH 4 study similar to CCQM-K17 might be rerun due to the importance of being able to undertake a difficult measurement such as this. Similarly, the suite of key and pilot studies undertaken for electrolyte conductivity spans nearly 5 orders of magnitude with seawater at one extreme but pure water not yet being accessed and lying 100-fold below the lowest conductivity comparisons undertaken to date by the EAWG (CCQM-P83). The next comparison assessing conductivity will probably retarget seawater (as CCQM-P111.1). Conductivity measurements in biofuels (in connection with BIOREMA) may be similar to those represented by CCQM-P83 but sample volumes in excess of those suggested available through BIOREMA would be required.

Dr Máriássy summarized two technical presentations hosted by the EAWG: impedance-frequency relationships and their effects on resistance extrapolations in conductivity cells (NIST) and the effect of structural design of Ag/AgCl electrodes on stability and response time (NPL). This was followed by brief presentations of several additional issues which included recommendations for the OIML, and a request that the BIPM place a statement on a publicly accessible website regarding the 2009 EAWG declaration on the primary method for pH measurement.

Dr Máriássy concluded by highlighting a few current problems requiring attention, including the need to conduct additional pilot studies in response to requests from some regions wishing to support CMC claims, the need to commence reviews of all CMC claims in the near future, and a policy for discerning HFTLS.

A lively discussion ensued over the report that purity of a measurand could possibly exceed 100 %. Dr Emons suggested that, in connection with CCQM-P112, the EAWG needed to define the EDTA measurand more precisely. Dr Máriássy defended his conclusion because the measurand was correctly defined as the complexing agent in the sample expressed as the mass fraction of EDTA, not as purity. If the real composition differed because of varying degrees of hydration, this could lead to the >100 % figure. Dr Emons recommended in this case that the definition of the measurand be redefined but Dr Máriássy noted that it was consistent with historical usage. Dr De Bièvre insisted that purity must always fall within the natural limits of 0 % to 100 % and it was only a matter of correctly defining the measurand to

overcome such anomalies. Dr Máriássy provided another common example of where this problem can arise – that of a mass fraction assay for sodium sulphate (a decahydrate, but in fact a mixture of various hydrates). A final comment from Dr Ma supported Dr Máriássy's claim wherein an example of undertaking a coulometric assay of KCl (containing some NaCl impurity) by measurement of chloride could lead to purity >100 % being reported, despite properly defined measurands. It was noted by Dr Máriássy that the presentation of mass fraction in this manner was for visualization purposes only.

Dr Máriássy concluded by stating the CCQM-EAWG opinion on pH measurements and the Harned cell, following a request from the NMIJ to support the planned change of the traceability chain in Japan. It was the CCQM-EAWG's opinion that the Harned cell

$$\mathrm{Pt} \mid \mathrm{H2} \mid \text{buffer}, \mathrm{Cl}^- \mid \mathrm{AgCl} \mid \mathrm{Ag}$$

can be operated in such a way that the measurement fulfils the conditions of a primary method of measurement, that it is the internationally agreed basis for the primary pH measurement method; pH values routinely do not include the uncertainty of the chloride ion activity coefficient but do include all other sources of uncertainty; and traceability to the SI can be achieved if the uncertainty of the chloride ion activity coefficient (as calculated using the Bates–Guggenheim convention or other convention that may replace it in the future) is also taken into account.

9.5 Surface analysis

Dr Unger reported on the progress of the CCQM Working Group on Surface Analysis (SAWG), which comprises seventeen institutes. He noted that the SAWG has applications spanning life sciences, electronics, chemistry, physics and materials science with a portfolio of techniques that span the 0.1 nm to 10 μm dimension which could play a role in the expanding field of nanotechnology, in particular the characterization of carbon nanotubes (CNTs). He outlined the business plan for ISO TC 229 WG2 regarding the measurement and characterization of CNTs and the complementary methodology available within the SAWG had prompted the Working Group to propose an extension of their scope of activities to include the development of metrological infrastructure in the chemical characterization of objects of nanotechnology. He then turned to a consideration of active comparisons.

- CCQM-K32/P84 (Gate oxide, SiO_2 on Si). The comparisons addressed the need for traceable determinations of the thickness of gate oxides in the semiconductor industry. A suite of nine samples of nominal thickness in the range 1.5 nm to 8 nm on (100) and (111) Si substrates were examined. Nine NMIs participated in CCQM-K32 (coordinated by the NPL) with an additional four in the parallel CCQM-P84 study. A discussion of the Draft B report was completed in November 2007 and a KCRV agreed at the 2008 meeting of the SAWG prior to the CCQM meeting, as the weighted mean of results with an inverse variance weighted uncertainty. As noted in earlier reports, the existence of an effect in some results due to the presence of hydrocarbons, dehydrogenated carbon and physisorbed/chemisorbed water on the samples precipitated a lack of agreement in results. In particular, because the layer was not detected by X-ray reflectometry, results contributed by the NMIJ were not included in the calculation of the KCRV. Eleven NMIs produced results that were in good agreement at 1.5 nm surface thickness with an expanded uncertainty of 1.5 % relative. A publication arising from the new science developed over the course of CCQM-K32 was accepted in *Surface and Interface Analysis*.

As a consequence of this study, the first SAWG CMCs have been claimed in cycle X submissions by the BAM, NIM, NMISA and PTB with more NMIs likely to make such claims in the future. A consensus had been reached within the SAWG following discussion on how such claims would be harmonized. A new measurement service category to include claims currently under 14.5 (thin films) was requested as category 15: Surfaces, Thin Films and Engineered Nanomaterials.

A short consideration of the CIPM/JCRB Document on Traceability suggested only minor concerns for the SAWG that could potentially arise linked with Note 1, Paragraph 1, wherein it may occur that new emerging NMIs will not have capabilities to analyse high-purity CRMs from sources other than NMIs.

- CCQM-K67/P108 (Amount of Fe and Ni in (200 nm) Fe-Ni alloy film on Si) was coordinated by the KRISS. Thin film alloy compositions are frequently characterized by micro-analytical techniques and a set of traceable Fe-Ni alloys prepared and certified by the KRISS (using ICP-MS) were used for this study. Five NMIs showed good measurement agreement, as summarized in Draft Report A distributed in January 2009.

The method of calculation of the KCRV was agreed and Draft Report B was to be distributed in June 2009.

- Dr Unger then reviewed follow-up work pertinent to CCQM-P80, P81 and P95 studies relating to the need to address calibration issues for electron probe microanalysis (EPMA). EPMA is extremely important for industrial applications and quantitative measurements are accomplished using a standards/matrix correction methodology whereby the unknown is measured under identical conditions relative to a suite of standards. Earlier pilot studies (CCQM-P80, 81 and 95) demonstrated that this method resulted in differences in measurements greater than the expanded uncertainties, highlighting the need for better standards and CRMs in this field. Modern instrumentation uses solid state detectors whose efficiency needs to be calibrated to permit standardless analysis to be undertaken in which only the unknown is measured. This approach will be attempted using the BESSY facilities at the BAM to calibrate absolute spectrometer efficiency via a transfer (multi-element) calibration standard which can be used to normalize response from future participants' instrumentation. Dr Unger illustrated the current situation with examples of the determination of carbon in TiC (CCQM-P80) and nitrogen in diamond-like carbon films (CCQM-P95) wherein scatter of the results was larger than the claimed expanded uncertainties. As such, it was concluded that the SAWG was not ready to proceed with a key comparison based on the use of EPMA, that the measurement of low *z* elements is very challenging and that the issue will be revisited when CRMs are available and after pilot studies on single-phase materials have been run.

The NMIJ was expected to propose a pilot study to address the RoHS Directive (Restriction on the use of certain hazardous substances in electrical and electronic equipment) issues relating to Cr(VI) as it was developing procedures for the determination of Cr(VI) in chromate thin films.

Dr Unger reported on two further developments for the SAWG that would expand its activities. A joint meeting was planned with the BAWG during the November 2009 meeting to address potential comparisons relevant to the life sciences in areas such as the determination of organic functional group surface density; the amount of functional groups at surfaces used for biochip technology; and the amount of DNA on surfaces. Additionally, the SAWG was interested in possible comparisons on the determination of functional groups on carbon nanotubes and on inorganic engineered nanoparticle surfaces.

Mrs Parkes enquired as to what matrices or what monolayers are of interest for life science applications, to which Dr Unger replied that this required a simple start but would eventually move to more complex systems.

Dr Cox noted that with respect to CCQM-K32, the KCRV uncertainty was no longer based on the weighted mean. Dr Unger replied that this was correct as the effect of the contamination layer had to be taken into account, which affected only XRR-based methods.

Dr Emons noted that whereas standardized methods were being proposed/developed by ISO TC229, he did not believe that severe measurement problems existed. Dr Unger replied that effort needs to be invested to determine whether this was true.

Mrs Parkes commented that many issues are cross-disciplinary and there was value in joint meetings, but discussion needed to be taken back to relevant institutes with the relevant expertise, thus cross-communication was important. Dr May replied that the CIPM MRA covered the competence of institutes and not just the subset of experts from a given institute within an individual committee. He therefore urged that Working Groups remained focused on measurement disciplines, i.e. surface analysis, rather than on technologies, e.g. nanotechnology, which would require involvement of many more disciplines and experts. Dr Kaarls agreed in principle but argued that most experts were in fact present in the CCQM.

Dr Kaarls proposed the creation of a new measurement service category 15; no objections were raised.

9.6 Bioanalysis

Mrs Parkes reported on the progress made by the CCQM Working Group on Bioanalysis (BAWG) which had met twice since the last CCQM. The 14th meeting took place in November 2008 at the NIMT, Bangkok, and was well attended with 53 participants from 24 organizations) and the 15th had just been held at the BIPM. It was noted that the first BAWG-related CMC claim had been made based on CCQM-K61 (Quantitative PCR calibration), and underpinned key DNA measurements. The report of the key comparison was being circulated to CCQM WG chairs for approval before submission to the KCDB. She went on to highlight activities of the past year.

- CCQM-P113 (Relative quantification of genomic DNA fragments) was coordinated by the IRMM and attracted fourteen laboratories. Four

samples were analysed, and a standard (candidate ERM®-AD418) for which the relative amount of DNA sequences had been characterized. Good agreement among the laboratories was achieved demonstrating that they were able to extract and quantify gDNA fragments present in low numbers. Results are traceable to the calibrant used and that should become traceable to the SI. The exercise underpins legal requirements for GM testing in Europe.

- CCQM-P59.1 (Protein structural measurements by circular dichroism) was coordinated by the NPL and involved seven laboratories with a focus on improving aspects of sample handling relative to the earlier CCQM-P59 study. Results were requested in the format of molar CD rather than ellipticity (as this corrects for path length and concentration, but not calibration). In both far and near UV, multivariate statistical analysis showed greatly improved comparability compared with the previous study. In addition, an uncertainty model for CD, and models for component uncertainties including calibration, concentration and path length were derived with the next step being to develop traceable CD capability so that a key comparison can be envisioned.
- CCQM-P101 (Protein glycosylation) was deemed to be of high bio-pharmaceutical importance as glycosylation plays a direct role in the modulation of biological activity and such an analysis constitutes an important aspect of the quality control of glycoprotein products. The study, designed to identify and determine relative quantities of glycan species in a mixture typical of that released from therapeutic glycoproteins, was coordinated by the NIBSC and the US Pharmacopoeia and attracted 48 participants from industry, academia, regulatory agencies and NMIs. Variations of up to 35 % relative in the results may be a cause for concern for regulators.

Mrs Parkes proceeded to briefly mention several ongoing studies, which included CCQM-P58.1 (Comparability of fluorescence in ELISA), CCQM-P94.1 (Quantification of DNA methylation), CCQM-P102 (Quantification of cells with specific phenotypic characteristics) and CCQM-P103 (Measurement of a multiplexed panel of RNA transcripts). This latter exercise would build on experience gained from CCQM-K61 with DNA and demonstrate capabilities for RNA. The LGC and NIST were coordinating the study involving eight laboratories. An outline of a route map from CCQM-K61 to CCQM-P103 biomarker which would strive to quantify an unknown amount (copy number) of an RNA transcript relative to a known calibrant made from the same material was then presented. The calibrant value would

be assessed by UV spec, RiboGreen, real-time RT-PCR and digital PCR with quantification of unknowns ideally performed by real-time RT-PCR or digital PCR. The test sample was selected to be External RNA Controls Consortium (ERCC) number 81 and would be supplied at two unknown concentrations together with a matched calibrant.

New study proposals were highlighted which included measurement of α-amylase activity, an important issue for food science but providing no added value to the clinical community (coordinated by the NIM in liaison with the Codex Alimentarius), and peptide mapping/profiling for impurity measurement in complex bio-pharmaceuticals (proposed by the LGC, NIBSC, NPL and USP). This study was triggered by requirements for procedural reference methods and standards by the bio-pharmaceutical community. A model system, probably rhGH (somatropin), would be selected for a study to identify and quantify degradation products and impurities.

Mrs Parkes then moved to a policy discussion concerning draft guidelines provided by the KCWG, stating that the document will be used to help align BAWG CMCs and this will require dialogue with the KCWG on an annual basis, particularly with respect to measurement uncertainties. The wording in the CIPM/JCRB Document on Traceability currently would seriously limit any BAWG CMC claims due to the need to be traceable to the SI or derive that traceability from other NMIs with CMCs. A CMC claim for hGH from the PTB referred to the BAWG for input was supported provided a change in the definition of the measurand was made and the uncertainty claims were revised.

Mrs Parkes then focused on cell measurements and imaging, presenting a brief overview on the various topics engaging NMIs and other organizations including, among other measurement issues, cell isolation and handling, imaging, culture and passport data, cell function measurements, tissue engineering, measuring DNA, RNA and protein content released from cells. It was noted that there was considerable expertise within NMIs undertaking comprehensive work programmes and that the BAWG was keen to develop pilot studies to support these capabilities. A detailed route map for a biomeasurement reference measurement system was presented.

Strategic planning groups were established to identify needs in at least six areas, addressing such questions as what applications each group needs to support, the measurement requirements, the measurement tools/building blocks needed in each area, the measurands and what the CCQM/BAWG

should do to establish traceability. Discussions will be facilitated via a WiKi blog. The 16th meeting of the BAWG to be held in Rio de Janeiro will further the development of these strategies, host a workshop on measurement uncertainty, and undertake a joint session with the SAWG to explore areas of mutual interest.

Prof. Wallard remarked on the need to engage in dialogue with regulators in connection with the study conclusions arising from CCQM-P101 (Protein glycosylation) to which Mrs Parkes commented that the regulators had been shown the data, which were no surprise to them, in part because no standards were available to support the measurements. Dr May noted that glycosylation measurements are important but it is not within the remit of the CCQM to address these problems but rather for the NMIs to engage in their individual programmes and then for the CCQM to make comparisons without going back to the regulators.

Dr Wielgosz mentioned that he had presented the BIPM plans to launch a study of "Measurement service and comparison needs for an international measurement infrastructure for the biosciences and biotechnology'' during the BAWG meeting. A call for tender to undertake this study would be issued shortly, with a written report of the outcome due in 2010.

9.7 Key comparisons and CMC quality

Dr Mackay reported on the work of the CCQM Key Comparison and CMC Quality Working Group (KCWG). This Working Group, comprising 21 members drawn from all the RMOs, had had an opportunity to meet in preceding days and now includes participants drawn from the SAWG and BAWG. She updated the meeting on the status of chemistry CMCs (4314 entries) in Appendix C of the KCDB. Between 200 and 300 submissions are considered each year, especially those of new NMIs coming on board for the first time. Cycle X statistics were summarized, as was the timetable for submissions outlined, noting that June and October provide two opportunities for the JCRB to meet for approvals. Three new claims relating to the SAWG activities were accepted in 2009, a new service category was created for engineered materials and the BAWG was being consulted for their needs for new categories.

Also noteworthy was the recent issue of a guidance document for CMCs, the aim of which is to document all aspects of the process of submission, review

and approval of CMCs. All CCQM WG chairs agreed to the current version in March 2009. It includes a flow chart outlining the role of the RMOs, KCWG and JCRB and describes in more detail the criteria used to assess chemistry CMCs and their hierarchy [including the "other" evidence which may be pilot studies (they must have a formal reference value) and publications, which must cover the "metrological" aspects of methodologies used] and expectations with respect to links to KCs. It clearly outlines how CMCs will be assessed where there is one-to-one correspondence with KCs, and how the uncertainty claim of a CMC can be assessed with respect to the degree of equivalence. The document will be updated annually. This more transparent process has resulted in a larger percentage of cycle X CMCs being fast-tracked through the RMO approval process than was the case for cycle IX.

Concern was expressed over the large number of CMCs that are older than five years (typically >60 %) and the fact that many have neither a KC nor PS underpinning them. A review of all CMCs was thus to be scheduled by category rather than simply by age and the CCQM KCWG would ask each RMO to coordinate this review and ask their NMIs to ensure that they are still offering the service as described, and to assess the CMC with respect to new KC/pilot study results available since the CMC was submitted. The process was to start with pH and electrolytic conductivity in 2009/2010, move to pure chemicals in 2010/2011 and then to water, inorganic and organic solutions in 2011/2012.

A need for additional KCs was expressed for inorganic solutions and isotope ratios. All comparisons were to be summarized and updated by the WG chairs twice per year (June and December) and the status of each comparison in its ability to support CMCs was to be posted on the KCDB.

Dr Mackay addressed the CIPM/JCRB Document on Traceability, noting that the practice of obtaining traceability from another NMI that does not list the needed CMC will have to be dealt with.

Dr Milton offered his congratulations to the KCWG for the smooth operation and success in fast-tracking CMCs through the latest cycle.

Dr Emons noted that the guidance document was to be a "living" document with respect to the process but that it also needs to be consistent with respect to technical requirements and asked if both concepts are to be "floated". Concerning traceability, serious concerns were also raised over the consequences of this document. Dr Kaarls did not hesitate to point out that

many comments had now been received and that in all probability an annex to the document would be drafted to accommodate as many as possible of the concerns of the chemical community.

Prof. Wallard remarked that the document was drafted one or two years ago in response to the question of whether NMIs could use accredited laboratories for the key parts of their uncertainty budgets. However, a clear policy under which all CCs can operate seems difficult to achieve, thus a supplementary document may have to be prepared to address specific problems. He agreed that more work was needed and that the issue would be readdressed.

Subsequent discussion centred on ensuring that the list of invitees for WG meetings was accurate and updated. Dr Wielgosz suggested that WG chairs update their lists every six months. It was noted that members names on such lists fluctuate from year to year because NMIs rotate staff and thus all the WG chair could do was send out a general letter of invitation. Dr Quetel suggested a quality manual covering the formal process. Dr Wielgosz replied that rules on attendance of all CCs and Working Groups were currently being prepared by the BIPM.

10 JCRB

Dr Mussio reported on the Joint Committee of the Regional Metrology Organizations and the BIPM (JCRB), reviewing membership and noting that AFRIMETS was approved as an expanded existing RMO (SADCMET) and accorded full rights. He proceeded to highlight actions and resolutions of the JCRB arising from meetings in May 2008 in Wellington (New Zealand), and in September and March at the BIPM targeting the status and review of CMCs, ILAC documentation for accreditation of NMIs, and CMC traceability issues, among others.

He noted that 20 May 2009 was designated World Metrology Day and invited all members to visit the website (www.worldmetrologyday.org/) for information on activities, notably the October Directors' meeting and the celebration of the 10th anniversary of the CIPM MRA on 8 October featuring an extensive agenda of speakers.

11 UPDATE ON THE BIPM KCDB

Dr Thomas updated the current status of the KCDB, noting that as of 15 April there were 21 436 CMCs published with 4314 in chemistry. Eleven of the 26 Associates to the CGPM do not yet have any CMCs published in the KCDB and no CMCs in chemistry are greyed-out because of the lack of an approved quality system. Regarding key and supplementary comparisons, there have been 112 KCs in chemistry: one run as a BIPM ongoing comparison, 99 from the CCQM and 12 from the RMOs (APMP, COOMET and EURAMET).

Several new features have recently been added to the KCDB, including information on traceability to the SI through the BIPM, links to BIPM calibration and measurement services in chemistry from the "QM" page, and a facility for exporting comparison information from the KCDB to an EXCEL file (operational on request to Dr Thomas) with the returned data being valid only at a specified date. Also in preparation are a set of FAQs (nine currently posted) and others are welcome. Answers must be approved by the JCRB prior to posting. EXCEL file summaries of CMCs published in the KCDB are available from the JCRB CMC website (restricted access, but guest password available on request to Dr Thomas) as are KCDB statistics (PDF version is available in open access, but a guest password is required for access to EXCEL file version).

Dr Thomas reminded members of the availability of the biannual *KCDB Newsletter* and announced the 14th International Congress of Metrology taking place in Paris 22–25 June, which will host a plenary session to celebrate the 10th anniversary of the CIPM MRA.

12 BIPM PROGRAMME ON METROLOGY IN CHEMISTRY

Dr Wielgosz reported on the work accomplished during the year by the BIPM Chemistry section, remarking that the BIPM operated with four-year funding cycles. He would summarize the impact of the completed programme (2005–

2008) and comment on the planned programme of activities to 2012 before considering future activities up to 2016. He thanked colleagues for their contribution to the work, noting that the group had benefited from the presence of one research fellow and two technical officers, in addition to two staff seconded from NMIs who had worked at the BIPM. He then summarize the activities undertaken in the fields of organic analysis and gas metrology being coordinated by the BIPM.

During the period 2005–2008, the BIPM had coordinated comparisons that engaged 124 NMI participants in the fields of gas metrology and organic compound purity. CCQM-P20.e (Theophylline: purity) and CCQM-20.f (Digoxin: purity) provided good models for primary calibrator comparisons and enhanced laboratory performance in supporting laboratory medicine. The BIPM-NIST programme to maintain the comparability of the worldwide network of ozone reference standards was supported by the results from BIPM.QM-K1 (Ozone ground-level) for which the spread of laboratory results was now consistent with reported measurement uncertainties in the order of 0.1 % relative. This was a considerable improvement in performance compared with previous comparisons such as EUROMET 414 (spread of results over 1 % of measured value) and the preceding CCQM-P28 (Ozone ground-level). This enabled long-term climate change target uncertainties of 0.1 % to be achieved as required by the WMO Global Atmospheric Watch (GAW) programme. CCQM-P73 (Nitrogen monoxide) examined the degree of equivalence of 24 standards using a dedicated measurement system and repeatability conditions at the BIPM. The measurement uncertainties obtained were considerably smaller than in previous studies such as CCQM-K1.c as well as EUROMET-K1.c. Differences from the reference value due to systematic effects in the preparation of six cylinders were confirmed by additional FTIR measurements performed by the BIPM and the quantification of N_2O and NO_2 impurities.

Dr Wielgosz then enumerated BIPM technical programmes underpinning international coordination activities with a number of agencies, including those with the WMO-GAW (surface ozone, CCL for VOCs and formaldehyde), ISO TC 146 (development of written standards for ozone and GPT), USP and other pharmacopoeias (participation in BIPM purity comparisons, December workshop, CRMs and SI traceability), ISO TC 212 and JCTLM (primary calibrators for monitored therapeutic drugs), Codex and food analysis and WADA (extension of primary calibrator programme) and WHO/ NIBSC.

With respect to its current programme of activity (2009-2012), activities in the BIPM Organic Programme are structured around themes similar to those delineated by the OAWG to assess core competencies in three areas to underpin the broadest range of CMC claims. In the area of primary calibrator comparisons, CCQM-K55.a/P117.a, an assessment of purity for 17β-estradiol was undertaken. The OAWG had previously developed a model based on "molecular weight versus polarity space'' as a way of classifying measurement capabilities for the characterization of pure organic materials. This measurand is a challenge due to medium structural complexity, intermediate polarity and the presence of related-structure impurities that could be quantified by capillary gas chromatography (GC) or high performance liquid chromatography (HPLC). The spread of results for this KC was determined to be primarily a consequence of moisture content correction and, in the case of the BIPM, artefact formation of dimers which were not corrected for when using reverse phase liquid chromatography (RPLC-UV). Homogeneity issues in the distributed sample were not significant. A KCRV will be proposed based on results from a select ensemble of non-biased methods. Subsequent KCs include CCQM-K55.b (purity of aldrin) and CCQM-K55.c (purity of tetracycline). They will present respectively a measurand of low to medium structural complexity and low polarity for which impurities are best quantified by GC, and a medium to high structural complexity measurand of high polarity wherein related structure impurities could be quantified by HPLC but not GC. The set of three purity comparisons coordinated by the BIPM has been designed to underpin all CMCs for pure organic calibrator materials.

Dr Wielgosz then summarized the 2009–2012 BIPM gas metrology programme, targeting analytical challenge species (a nomenclature developed by the GAWG) including activities and capabilities of the ozone reference standard comparison facility as well as international comparisons in support of greenhouse gas monitoring and air quality measurements. CCQM-K74/P110 (NO_2 in nitrogen, 10 µmol/mol) attracted 16 participants who will submit 32 results. The pilot study will investigate the accuracy of FTIR measurement methods with traceability either to gravimetric standards or to reference spectra, and has been organized in response to previous GAWG workshops on spectroscopic measurements. The ozone reference standard comparison BIPM.QM-K1 has continued, with the majority of SRPs now upgraded with corrections to systematic effects of cell design and temperature control, improving both the performance of the BIPM SRP27 and the degrees of equivalence of all SRPs.

A report on measurement infrastructure needs for biometrology was in progress and input was being solicited as to which measurement services are required or will be developed by NMIs, what the current industry requirements may be, which international comparisons may be required to demonstrate the degree of equivalence of measurement services and what research and development activities for higher metrological order measurement standards and methods for the biosciences should be considered.

Dr Wielgosz then turned to a consideration of future work covering the period 2013–2016, highlighting NMI responses surveyed in 2006 (CCQM/06-41) on the major drivers that would influence their activities. In the gas metrology programme, air quality and climate change (reference methods and calibration with traceable gas standards, VOCs), and the hydrogen economy (international product specifications) were identified. In the organic analysis area, clinical, food, forensics, pharma and the environment were mentioned as future areas in which NMIs would be developing measurement capabilities underpinned by OAWG core comparisons for primary calibrators and calibration solutions coordinated by the BIPM. In the bioanalysis arena, core comparisons within the BAWG were likely to be established, and it would be appropriate for possible future BIPM activities to be linked to these. Additionally, stakeholder support from communities such as the IFCC, Pharma, Codex, WADA, WHO and WMO would continue to be sought. The overall vision was outlined in a timeline encompassing 2005-2015, illustrating the evolution of the work programmes.

Dr De Bièvre noted the impressive level of activity and asked how traceability for ozone measurements was established. Dr Wielgosz referred to his presentation which summarized the traceability chains for ozone measurements, and noted that the optical techniques were dependent on values of the cross-section of ozone. The value and uncertainty to be used for ground-level surface ozone measurements had recently been published in a BIPM-NIST paper, and further work was under way at the BIPM to perform new measurements with improved uncertainty.

Mrs Parkes enthusiastically supported the report and roadmapping plans for metrology in the biosciences, and for the BIPM to explore how it could contribute to the BAWG programme, similar to its part in the OAWG and GAWG activities.

Dr Emons raised the point that an extensive legally mandated European network exists on food analysis which should be consulted for a gap analysis,

to which Dr Wielgosz replied that the CCQM and the BIPM could provide needed comparisons and traceability services. Dr Emons questioned that in view of the already existing interlaboratory comparison schemes and the mandate of the European Community Reference Laboratories (CRLs).

Dr Milton remarked that the current and older reports of the CCQM show that the activities of the BIPM complement NMI activities, fill existing gaps and benchmark CRM capabilities, for example by establishing a central facility for the comparison of standards for methane in air, which was the second most important greenhouse gas. He questioned whether there were enough staff to be able to continue such ambitious programmes. Dr Wielgosz stated that the secondment programme serves to ease this shortcoming, and in recent years the Chemistry section had been successful in attracting visiting researchers to contribute to its activities. Prof. Wallard added that it is the policy of the CIPM to expand the BIPM group, especially in chemistry, taking advantage of postdoctoral and seconded appointments. Dr May supported the BIPM programme, but challenged Dr Milton's assessment by stating that it was not that NMIs were unable to technically carry out the activities undertaken by the BIPM, but that they would not wish to do so, and thus community support for the BIPM programme was evident. Dr Quinn agreed that the work of the BIPM could be technically done by NMIs but the organization's political independence allowed unique activities to be undertaken.

13 REPORTS ON RMO ACTIVITIES

13.1 AFRIMETS

Ms Prins reviewed the history, structure, composition and activities of the relatively newly instituted Intra-Africa Metrology System (AFRIMETS). The first General Assembly took place in July 2007 and the 3rd was scheduled for July 2009. The structure mirrors SADCMET with five Technical Committees having scientific and industrial and legal metrology subsections. Members from CEMACMET, EAMET, MAGMET, SADCMET and SOAMET total 36 with an additional three Ordinary, two Associate and three Observer members. Regional comparisons constitute the most important activity,

coupled with review and approval of CMCs. Activities in 2008/2009 were highlighted with a focus on PT schemes covering water, wheat flour, salt and edible oil. AFRIMENTS submitted three new CMCs for inorganic chemistry and one for surface analysis in 2009 while engaging in seven KCs and three PSs under the auspices of the CCQM. CRMs for aqueous ethanol and sodium fluoride solutions are supported as well as a number of gas standards. Future plans include an AFRIMETS call for a PT based on aqueous ethanol, extension of the EAMET PTs to the rest of Africa, a Metrology in Chemistry Workshop at the General Assembly in July, training for new NMIs and preparation of new CRMs targeting trace elements and mycotoxins in maize, gas standards for VOCs, BTEX, H_2S and ethanol in nitrogen, and calibration solutions.

13.2 APMP

Dr Kato, chairman of the Technical Committee on Metrology in Chemistry (TCQM), reported on the activities of the APMP, which comprises 37 NMIs and DIs from 23 Economies and four Associate Member Economies. He summarized the 14 APMP KCs and PSs completed by 2009 and nine planned or under way for 2008–2009. Notably, APMP.QM-K1.c (NO in N_2) is a bilateral with the KRISS and NIM to permit a CMC claim for the NIM. The report was modified to include the degrees of equivalence for the link to CCQM-K1.c and was presented to the GAWG. APMP-QM-P10 (Cd and Pb in Herb) coordinated by HKGL parallels CCQM-P97 and was in Final Report form.

Dr Kato summarized a number of APMP meetings held during 2008, including the 24th General Assembly in Jakarta in November (the 25th to be held in Kuala Lumpur in December 2009), 4th APMP Workshop on MiC in Dhaka, Bangladesh (May 2008), 5th International MiC Workshop in Jakarta, Indonesia (October 2008), 6th APMP Gas CRM workshop at the KRISS in May with the planned 7th meeting at SIRIM in May 2009, the 8th meeting of the Asian Collaboration on Certified Reference Materials (China, Japan, Korea) in Yunnan (China) and the scheduled 9th meeting in Seoul in September. These meetings formulate metrology needs for food and the environment, gas analysis, RoHS and biological analysis.

13.3 COOMET

Dr Kustikov (Deputy chairman) reported on behalf of Prof. Konopelko, chairman of TC1.8, the Physical Chemistry Committee of COOMET. The seventeen members of COOMET have made contributions through KCs to all CCQM WGs with the exception of surface analysis and currently have 252 CMCs in Appendix C, the majority arising from the VNIIM. Eight KCs and ten PSs were undertaken in 2008–2009, along with five interregional comparisons.

He then detailed several COOMET coordinated comparisons, including CCQM-K65 (Mercaptans in methane) and involved four laboratories; COOMET.QM-K1.a (CO in nitrogen) and COOMET.QM-K23.b (Synthetic natural gas) linked to CCQM-K23.b and involving six participants. Good results were achieved in all cases.

Dr Kustikov concluded his presentation with a summary of meetings planned for 2009 which included the annual meeting of TC 1.8 "Physical chemistry", 12-13 May 2008 at the VNIIM, the 2nd All-Russian Conference on Certified Reference Materials in Measurements and Technologies, June 2008, St Petersburg, the 3rd International Conference on "Metrological assurance of physico-chemical measurements", November 2008, Kiev, the International Scientific and Practical conference on "Metrology – 2009", April 2009, Minsk, Belarus, the 3rd International Competition for the "The Best Young Metrologist of COOMET 2009", 14-15 April 2009, Minsk, Belarus, the 19th COOMET Committee meeting, May 2009, Baku, Azerbaijan, and the International Seminar "Mathematics, statistics and computation to support measurement quality", June 2009, VNIIM, noting that it was the 175th anniversary of the birth of Mendeleyev and mentioning some of the activities organized to celebrate this event.

13.4 EURAMET

Dr Güttler summarized the work of EURAMET (European Association of National Metrology Institutes) which currently comprises 33 European NMI members along with the IRMM and 69 DIs with liaisons with three NMIs beyond Europe, four RMOs and six corresponding organizations. With the departure of Prof. Kühne, Prof. Leslie Pendrill (SP, Sweden) was elected EURAMET chairman for 2009–2012.

A TC chemistry meeting was held in Bucharest in February 2009 to discuss EURAMET projects, cycle X CMC claims and iMERA-Plus projects (Implementing Metrology in the European Research Area) and the EMRP. At present, nineteen projects are active, covering both comparisons and research projects on issues of importance to the environment, nanoparticles, energy and general metrology. Four iMERA projects are under way on health, T2.J02, T2.J10 and T2.J11, as well as SI and Fundamentals (T1.J1.2, the Avogadro Project). Over time, EURAMET is to become a more autonomous organization that will not have to rely on seconded staff and subcontractors.

Dr Güttler outlined planned R&D calls for EMRP projects supporting energy, environment, health, metrology for industry and fundamental SI-related activities; decisions will be rendered in December 2009.

13.5 SIM

Dr May began his report on the SIM Chemical Metrology Working Group (CMWG) by noting that a May 2008 CMWG strategic planning meeting in St Lucia was followed by the CMWG business meeting in September in Honduras. A similar meeting would take place in May 2009 in Paraguay with an autumn meeting in Peru. He then briefly outlined the SIM programme in chemical metrology which is implemented through a cooperative arrangement between the OAS/SIM and the German Government. The principal activities of the group involve outreach and awareness, proficiency assessment activities and training in CMC preparation and review. Approximately twenty countries within SIM now have regular participants in SIM CMWG meetings with about fifteen NMIs or their designated laboratories being regular participants in SIM comparison studies (up from three in 2002). Six countries now participate in CCQM meetings and various activities [up from three (Canada, Mexico and the United States) in 2002]. NMIs from six countries (Argentina, Brazil, Canada, Chile, Mexico and the United States) have chemistry CMCs in the BIPM KCDB and Uruguay plans to submit CMCs within the next few years.

The operational structure within SIM was briefly summarized as it was recognized that the needs of metrology in chemistry programmes vary widely among SIM countries. These needs are consequently better met through WG sub-groups populated according to the level of development and requirements within each country, which stimulate and facilitate cooperation among countries of a similar level of development in a more direct manner

than can be achieved with one large group. Thus, sub-group I comprises NMIs or DIs participating in the CIPM MRA and having CMCs. Sub-group II comprises NMIs with no CMCs and sub-group III comprises those countries having no NMI for chemical metrology activities. The primary needs of sub-group I are key and supplementary comparisons to support their CMCs; those of sub-group II are awareness seminars, assistance with the preparation of CMCs and proficiency assessment studies; while those of sub-group III are awareness seminars, assistance in framing arguments for obtaining sustainable government support and for conducting needs assessments with relevant customers.

Dr May summarized activities, which included KCs and parallel PSs as well as workshops and awareness seminars, the latest being that held in St Lucia which attracted 45 participants from 15 countries representing 34 institutes. The agenda for the May 2009 meeting in Paraguay was presented, followed by a summary of published chemistry CMCs for each SIM country. He concluded his presentation by noting that a new chair would be tabling this report from 2010.

14 ISO TC 229 LIAISON AND BIPM WORKSHOP ON NANOMETROLOGY

Dr Viallon, appointed as BIPM liaison to ISO TC 229 in October 2008, reported on the committee's scope, meetings and activities as this was of interest to many CCs. ISO TC 229 was challenged with standardization in the field of nanotechnologies that includes either or both of the following: understanding and control of matter and processes at the nanoscale, typically, but not exclusively, below 100 nm in one or more dimensions where the onset of size-dependent phenomena usually enables novel applications, and utilizing the properties of nanoscale materials that differ from the properties of individual atoms, molecules and bulk matter, to create improved materials, devices and systems that exploit these new properties. Specifically, there are four WGs focusing on specific tasks which include developing standards for terminology and nomenclature (JWG1); measurement and characterization (JWG2); health, safety, and environmental aspects of nanotechnologies (JWG3); and material specifications (JWG4). Two meetings take place each

year; the November 2008 meeting in Shanghai attracted 223 delegates with approximately 30 from NMIs; the next meeting was scheduled for Seattle in June 2009. Two documents have now been published: the technical report ISO/TR 12885:2008 (Nanotechnologies – Health and safety practices in occupational settings relevant to nanotechnologies) and the technical study ISO/TS 27687:2008 (Nanotechnologies – Terminology and definitions for nano-objects – Nanoparticle, nanofibre and nanoplate) with some 30 others under development.

Measurement techniques identified as significant for nanotechnologies include many utilized in metrology in chemistry. This was highlighted in the last liaison report from the BIPM to ISO TC 229 with some examples of relevant activities in the CCQM, such as the recent CCQM-K32 in the SAWG, activities on nanoparticles being planned in the GAWG and the joint interest of the BAWG/SAWG for bio-analysis at surfaces. The liaison report also underlined activities in other CCs, such as the CCL (Length), CCEM (Electricity and Magnetism) and CCRI (Ionizing Radiation).

An inter-NMI workshop on "International Needs for Metrology at the Nanoscale" will be hosted by the BIPM from 18 to 19 February 2010.

15 WADA

Dr Westwood presented a brief report, noting that he was now serving on the WADA Laboratory Committee as an external member with expertise in metrology, replacing Dr Siekmann who had recently stepped down. The UNESCO International Convention against Doping in Sport was ratified in February 2007 and now represents 110 signatories. Four international standards exist, covering the Prohibited List, Therapeutic Use Exemptions, Testing and an International Standard for Laboratories. Dr Westwood elaborated on the last standard, noting that there are currently 34 accredited laboratories, two probationary laboratories and a waiting list of others. Strict accreditation criteria are adhered to, including ISO/IEC 17025 as well as successful participation in an External Quality Assessment Scheme (EQAS) – a PT exercise conducted four times per year with testing up to twenty blind and double-blind samples focusing on detection of prohibited and threshold substances. A single report of a false positive on a prohibited substance

results in suspension of accreditation. A new technical document, "Estimation of Measurement Uncertainty for the Quantification of Threshold Substances" was under development. US$44 million have been committed to the WADA scientific programme since 2001 for the development of new or improvement of existing detection methodologies, with 75 % of the funding being disbursed to research teams outside WADA-accredited laboratories. Additionally, a database (ADAMS – Anti-Doping Administration and Management System) was being developed to comprehensively look after laboratories and athletes registered in the system.

16 CODEX ALIMENTARIUS COMMISSION REPORT

Dr Josephs (BIPM) presented a brief report on the Codex Alimentarius Commission. The Commission was created in 1963 by the FAO and the WHO to develop food standards, guidelines and related texts such as codes of practice under the Joint FAO/WHO Food Standards Programme. The main purposes of this programme are protecting consumer health and ensuring fair practices in food trade, and promoting coordination of all food standards work undertaken by international governmental and non-governmental organizations. He then outlined the terms of reference of the Codex Committee on Methods of Analysis and Sampling which hosted its 30th meeting in March 2009 at Balatonalmadi, Hungary. The highlights included discontinuance of work on "Draft Guidelines for Evaluating Acceptable Methods of Analysis"; an agreement to forward amendments on "Draft Guidelines for Settling Disputes over Analytical (Test) Results", "Draft Guidelines on Analytical Terminology" and "Guidelines on Establishing Methods Criteria for the Identification of Relevant Analytical Methods" to the 32nd session of the Commission for adoption. However, further work on "Criteria for the Methods for the Detection and Identification of Foods Derived from Biotechnology", "Guidance on Measurement Uncertainty", and "Guidance on Sampling Uncertainty" was recommended, perhaps by combining the latter two documents. The BIPM will participate in electronic Working Groups concerning further work on Guidance on Measurement Uncertainty and Guidance on Sampling Uncertainty.

Dr Josephs then turned to a consideration of the activities of the Inter-Agency Meeting and MoniQA (the EU Network of Excellence funded project on the Monitoring and Quality Assurance in the Food Supply Chain) which seeks to establish long-lasting cooperation among leading research institutions, industrial partners, and the small- and medium-sized businesses that dominate European food manufacture and retail, in order to ensure food quality and safety for consumers. Their first workshop took place in March 2008, Budapest, focusing on "Method Performance and Analytical Uncertainty" and attracted 80 participants. A second workshop occurred in March 2009, Balatonalmadi, Hungary devoted to "Method Performance and the Criteria Approach: Truth and Consequences", which attracted 90 participants and generated interest in the need for subsequent workshops. The BIPM was involved in the organization and has contributed to both workshops.

17 WMO

Dr Milton provided an update on activities of the WMO Global Atmosphere Watch Programme (GAW). He focused on the GAW network for volatile organic compound (VOC) measurements, detailing the QA/QC strategy involving a central calibration laboratory and the dissemination of transfer/travelling standards. A proposition had been made several years ago that certain NMIs active in the GAWG would provide the central calibration laboratory for the GAW with calibration standards for (VOCs). The collaboration thus principally concerns the provision of standards for VOCs at the nmol/mol level, which are of interest to the GAW because of their role in the generation of ozone in the troposphere. EURAMET 886 (Comparison of multi-component VOC measurements at ambient levels) was completed in December 2008, showing, in general, excellent agreement with gravimetric values.

A proposal for a WMO-BIPM Workshop sometime in 2010 on "Measurement Challenges in Global Observation Systems for Climate Change Monitoring: traceability, stability and uncertainty" was sent to the WMO in Geneva for their agreement. Possible sessions would cut across many CCs with a number of anticipated outcomes, including alerting the

metrology community to the measurement needs of the meteorology community; increasing awareness of the measurement science capabilities of the metrology community; promoting a culture of metrological traceability to the SI in global observation projects, and furthering collaboration between the observation centres and the NMIs.

18 ISO REMCO

Dr Emons presented an ISO/REMCO status report on behalf of Dr van der Veen, briefly outlining the terms of reference and the committee structure. He summarized developments relating to the status of a number of ISO Guides, including Guide 34, which was subjected to limited revision to improve the text and provide more explanation based on accreditation assessors experience and feedback, was expected to be completed in July; the next draft version of the new Guide 80 on quality-control reference materials will also be ready in July and will address "in-house" preparation of RMs; Guide 33, which will provide a wider scope in covering applications of RMs as well as addressing calibration (formerly covered in Guide 32), was currently under revision; Guide 30, whose revision had just started, will include a thesaurus of terms to encompass the widest vocabulary usage, incorporating that from the VIM; and Guide 31, whose revision had been started and will encompass not only the contents of certificates but also labels and other RM documentation. Additionally, a new ISO Guide, "RMs for qualitative analysis – Testing of nominal properties", was to be discussed in July 2009 while a proposed "Guide to the Guides", would be developed as an ISO/REMCO paper, freely available after approval. An *ad hoc* WG on "CRMs and Metrological Traceability" has been re-established with the aim of expressing how to make statements on traceability.

Dr Emons concluded by noting that the next ISO/REMCO meeting was scheduled for Teddington in July 2009 and then made a reminder announcement on the next BERM conference in Oxford, July 2009.

Dr Kaarls noted that Dr Emons was chairing a WG on "Transportation" and hoped that it was addressing the specific needs of posting reference materials, to which Dr Emons replied that experiences with the specific customs tariff number for Certified Reference Materials will be included.

Dr Sargent raised the issue of the status of Guides 34 and 35, asking if they would become standards, to which Dr Emons replied that the last review cycle concluded that Guide 35, last issued in 2006, was still adequate. The subject of guide versus standard arises frequently and only an ISO TC can issue standards. REMCO is a policy committee and cannot do so. However, there was interest in REMCO to transform Guide 34 into a standard.

19 INTERNATIONAL ATOMIC ENERGY AGENCY

Dr Fajgelj delivered a short report, stating that the agency hosted the joint meeting of the IAWG and the EAWG in October in Vienna that included a tour of the laboratories at Seibersdorf and a workshop on uncertainty of measurement of stable and light element isotope ratios.

20 INTERNATIONAL UNION OF PURE AND APPLIED CHEMISTRY

Dr Fajgelj noted that the 42nd IUPAC World Congress and the 45th General Assembly would take place in Glasgow in July/August 2009 and that 2011 was approved in December 2009 by the 63rd General Assembly of the United Nations as the International Year of Chemistry to celebrate the achievements of chemistry and its contributions to the well-being of humankind. All stakeholders are invited to participate in this event and to visit the website at www.chemistry2011.org.

Copies of all issues of *Pure and Applied Chemistry* were freely available in electronic format, and whereas manuscripts are peer reviewed, there was no reason not to consider republishing significant papers of interest.

Dr De Bièvre reported on the status of the IUPAC Project on "Metrological Traceability of Measurement Results in Chemistry: Concepts and Implementation". Comments from individual scientists and organizations on the draft submitted in September 2007 (which was version 23) to fourteen

international organizations and bodies, including the CCQM, had been carefully examined, implemented when appropriate, and all replied to. A revised version (which will be version 30) will be submitted for information and final comments within a few weeks and will in parallel be subjected to a second internal IUPAC review. The final version was expected to be available at the time of the IUPAC General Assembly in Glasgow in August 2009. Nevertheless, comments from CCQM participants were still welcomed.

Dr De Bièvre also informed the CCQM that IUPAC intended to produce guidelines for practitioners on metrological traceability of measurement results in chemistry which will be based on the above document and will include more practical examples.

Dr May remarked that perhaps there was too much replication of documents by different organizations, and asked why the CCQM was not drafting rather than simply commenting on such issues, further raising the question of whether there was to be anything different from existing CITAC documents. Dr De Bièvre replied that this activity started in 2000/2001 and was supposed to be concluded much more quickly. Dr Wielgosz noted that the BIPM still had strong reservations about the "Metrological Traceability" document and wanted to comment on the second draft, expressing a formal request to do so. Although Dr De Bièvre expressed concern about the excessive delays this may involve, Dr Kaarls cautioned that there were very serious BIPM comments on the draft. Dr Fajgelj supported sending the revised version to the CCQM but reiterated that many organizations had been consulted for their input. Prof. Wallard remarked on the need for better coordination of these processes to avoid mutual rejection of each others' documents.

21 CITAC

The President welcomed Dr Kuselman, the current chairman of Cooperation on International Traceability in Analytical Chemistry (CITAC), who provided a brief overview of its structure and board, noting that it was created in 1993 and currently comprises 36 members (50 % from NMIs) from 26 Economies. The primary mission of CITAC is to globally improve traceability and comparability of the results of chemical measurements by dissemination of information accumulated by NMIs. Liaisons were noted

with international organizations that provide a multi-faceted strategy to foster publication of scientific papers addressing metrological principles, organization of seminars, symposia and workshops, assisting analytical laboratories with the required tools, clarifying concepts and disseminating them globally.

The 24th CITAC members meeting held at the LNE in April 2009 highlighted progress with the draft of a guide addressing "Selection and use of proficiency testing schemes for a limited number of participants – chemical analytical laboratories" which was undergoing external review, as well as development of a guide on "Out of specification test results based on metrological concepts" (IUPAC Project 2008-030-1). Authors of remarkable papers in receipt of 2008 CITAC awards were mentioned, as was the development of new procedures for planning and performing CITAC projects to increase effectiveness of CITAC activity in knowledge dissemination in Metrology in Chemistry. Dr Kuselman concluded with an announcement of the Isranalytica meeting scheduled for Tel Aviv in January 2010.

22 INTERNATIONAL LABORATORY ACCREDITATION COOPERATION (ILAC)

As Mr Squirrell was unable to attend the meeting, a brief presentation was made on his behalf by Dr Kuselman, who relayed Mr Squirrell's regrets as well as an expression of thanks to the CCQM and BIPM for their ongoing interaction and cooperation. A written general update for ILAC (CCQM/09-08) was provided and any questions were to be directed to Mr Squirrell at ilac@nata.asn.au.

It was noted that ILAC membership as at 9 April 2009 (total 138 bodies) consisted of 62 Full Members representing 48 Economies; 24 Associates representing 23 Economies; 20 Affiliates representing 18 Economies; four regional and one national cooperation bodies, and 27 stakeholders. Some 33 000 laboratories and over 6000 inspection bodies are involved.

23 CCQM WORKSHOPS

The President welcomed suggestions for workshops; none were raised.

24 CCQM RECOMMENDATIONS

The President summarized by stating that the recommendation regarding agreement from the CCQM on the proposed redefinition of the mole would be conveyed to the CCU, CIPM and IUPAC.

25 ANY OTHER BUSINESS

Dr Máriássy requested that the EAWG would like to formally introduce into the minutes the opinion of the EAWG with respect to pH measurements. Dr Kaarls agreed that this opinion would be included in the section of the report dealing with EAWG activities.

26 DATE OF NEXT MEETING

The next meeting of the CCQM was fixed for 14-16 April 2010 at the BIPM with the preceding days reserved for meetings of the CCQM WGs and a workshop.

27 COORDINATION OF CCQM WG MEETINGS

The President was grateful to INMETRO for extending an invitation to host a meeting of the CCQM WGs during the week of 3-6 November 2009 in Rio de Janeiro, Brazil. This will be the first time that the CCQM will meet in South America.

28 CLOSURE

The President closed the meeting at 16.30 by thanking all participants for their excellent contributions to a successful plenary and prior week of meetings, and the staff of the BIPM for their support, expressing best wishes for safe travel to all participants.

R.E. Sturgeon, Rapporteur 12/05/09

revised 25/06/09

RECOMMANDATION DU COMITE CONSULTATIF POUR LA QUANTITE DE MATIERE – METROLOGIE EN CHIMIE PRESENTEE AU COMITE INTERNATIONAL DES POIDS ET MESURES

RECOMMANDATION Q 1 (2009) : Sur les éventuelles redéfinitions de la mole et du kilogramme

Le Comité consultatif pour la quantité de matière – métrologie en chimie (CCQM),

considérant

- sa précédente Recommandation au CIPM sur les éventuelles redéfinitions de la mole et du kilogramme, CCQM Q1 (2007),
- que la définition actuelle de la mole fait référence au kilogramme,
- l'importance du kilogramme aussi bien que de la mole pour la communauté de la métrologie en chimie,

prenant acte

- des progrès des mesures expérimentales pour résoudre la différence relative de 1×10^{-6} entre la valeur de la constante de Planck obtenue à partir des mesures effectuées avec la balance du watt et celle fondée sur les mesures de masses molaire et volumique associées à l'interférométrie par rayons x d'un cristal,
- de la mise au point de méthodes indépendantes de spectrométrie de masse pour la détermination des rapports de teneur isotopique du silicium naturel et du silicium enrichi dans le cadre du programme de coordination internationale Avogadro,
- que les communautés concernées sont insuffisamment informées du projet de redéfinition de la mole,
- que la proposition de redéfinition de la mole ne reçoit pas encore un soutien unanime,

recommande que

- la décision de redéfinir la mole et le kilogramme soit différée jusqu'à ce que

  - la différence entre les résultats obtenus à partir des mesures effectuées avec la balance du watt et ceux fondés sur les mesures de masses molaire et volumique associées à l'interférométrie par rayons x d'un cristal soit résolue, et
  - l'on ait apporté la preuve d'un accord entre les valeurs de la constante d'Avogadro obtenues à partir de mesures indépendantes des rapports de teneur isotopique d'échantillons de silicium naturel et enrichi,
- l'on tienne pleinement compte des intérêts de la communauté de la métrologie en chimie,
- le Bureau international des poids et mesures (BIPM), les laboratoires nationaux de métrologie, et les autres représentants officiels des Comités consultatifs accroissent leurs efforts pour sensibiliser les diverses organisations scientifiques, industrielles et professionnelles aux propositions de changement, et pour connaître leur point de vue à un stade préliminaire,

déclare sa préférence pour une redéfinition de la mole, l'unité de quantité de matière du SI, fondée sur une valeur fixée de la constante d'Avogadro.

RECOMMENDATION OF THE CONSULTATIVE COMMITTEE FOR AMOUNT OF SUBSTANCE – METROLOGY IN CHEMISTRY SUBMITTED TO THE INTERNATIONAL COMMITTEE FOR WEIGHTS AND MEASURES

RECOMMENDATION Q 1 (2009): On the possible redefinition of the mole and the kilogram

The Consultative Committee for Amount of Substance – Metrology in Chemistry (CCQM),

considering

- its previous recommendation to the CIPM on the possible redefinition of the mole and the kilogram, CCQM1 (2007)
- that the present definition of the mole refers to the kilogram,
- the importance of both the kilogram and the mole to the chemical measurement community,

noting

- the progress with experimental efforts to resolve the discrepancy of about 1 part in 10^6 between the value of the Planck constant arising from the watt balance and the X-ray crystal density/molar mass measurements,
- the development of independent mass spectrometric methods for the determination of the isotope amount ratios of silicon both at natural and enriched isotopic abundances as part of the IAC programme,
- that the level of awareness of the proposal to redefine the mole is low in the relevant communities,
- that support for the proposal to redefine the mole is not yet unanimous,

recommends that

- any decision on redefining the mole and kilogram be deferred until:
  - the discrepancy between results from the watt balance and the X-ray crystal density/molar mass measurements has been resolved; and

- agreement is demonstrated between values for the Avogadro constant derived from independent measurements of the isotope amount ratios of silicon on samples at both natural and enriched isotopic composition,

- full consideration be given to the interests of the chemical measurement community,
- the BIPM, the National Metrology Institutes, and the other official representatives in the Consultative Committees increase their efforts to spread awareness of the proposals to the various scientific, industrial, and professional organizations, and seek their views at an early stage

states its preference for a redefinition of the mole, the SI unit of amount of substance, based on a fixed value of the Avogadro constant.

APPENDIX Q 1.
Working documents submitted to the CCQM at its 15th meeting

Working documents submitted to the CCQM at its 15th meeting are on restricted access.

Documents restricted to Committee Members can be accessed at the restricted website.