Bureau International des Poids et Mesures

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

Report of the 16th meeting
(15-16 April 2010)
to the International Committee for Weights and Measures

Comité international des poids et mesures

Note:

Following a decision made by the International Committee for Weights and Measures at its 92nd meeting in October 2003, reports of meetings of Consultative Committees will henceforth be published only on the BIPM website in the form presented here.

Full bilingual printed versions in French and English will no longer appear.

A.J. Wallard,
Director BIPM

LIST OF MEMBERS OF THE CONSULTATIVE COMMITTEE FOR AMOUNT OF SUBSTANCE: METROLOGY IN CHEMISTRY AS OF 15 APRIL 2010

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

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 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.

Instituto Português da Qualidade [IPQ], Caparica

Istituto Nazionale di Ricerca Metrologica [INRIM], Turin.

National Institute of Metrology [NIMT], Pathumthani

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

The Consultative Committee for Amount of Substance: metrology in chemistry (CCQM)* held its sixteenth meeting at the International Bureau of Weights of Measures (BIPM), at Sèvres on 15-16 April 2010.

The following were present: H. Andres (METAS), H.S. Brandi (INMETRO), R.J.C. Brown (NPL), G. Carroll (SL), P. Charlet (LNE), K. Chiba (NMIJ/AIST), M. Cox (NPL), D. Craston (LGC), P. De Bièvre (IUPAC), V.S. Da Cunha (INMETRO), H. Emons (IRMM, ISO REMCO), H. Ent (VSL), M. Fernandes-Whaley (NMISA), N. Gonzalez-Rojano (CENAM), B. Güttler (PTB), E. Hwang (KRISS), H.D. Jensen (DFM), R. Kaarls (President of the CCQM), K. Kato (NMIJ/AIST), Y. Kustikov (VNIIM), H. Li (NIM), L. Mackay (NMIA), B. Magnusson (SP), M. Máriássy (SMU), W.E. May (NIST), Z. Mester (NRC-INMS), M.J.T. Milton (NPL), Y. Mitani (CENAM), J. Murby (NMIA), U. Panne (BAM), H. Parkes (LGC), S. Prins (NMISA), M. Sargent (LGC), H.-Y. So (KRISS), J.A. Salas Téllez (CENAM), U. Sansone (IAEA), K.-D. Sommer (PTB), R. Sturgeon (NRC), W. Unger (BAM), S. Vaslin-Reimann (LNE), A.J. Wallard (Director of the BIPM), R.L. Watters (NIST), S. Wise (NIST), Z. Zeyi (NIM).

Observers: T. Fernández Vicente (CEM), O. Cankur (UME), C. Cherdchu (NIMT), H.A. Chua (A*STAR), F. Dias (IPQ), P.K. Gupta (NPLI), W. Kozlowski (GUM), I. Kuselman (INPL, CITAC), M.P. Sassi (INRIM), M. Sega (INRIM), P. Totarong (NIMT), Z.N. Szilágyi (MKEH), A. Zoń (GUM).

Invited: M. Buzoianu (INM), P. Chui (HSA), D. Schiel (PTB), D. Wai Mei Sin (GL), A. Squirrell (ILAC)

Also present: A. Daireaux (BIPM), E. Flores Jardines (BIPM), R. Josephs (BIPM), M. Kühne (BIPM Deputy Director), S. Maniguet (BIPM), P. Moussay, C. Thomas (Coordinator of the KCDB, BIPM), J. Viallon (BIPM), S. Westwood (BIPM), R. Wielgosz (Executive Secretary of the CCQM, BIPM).

Sent regrets: P. Banerjee (NPLI), A. Fajgelj (IAEA, IUPAC), M. Müller (IFCC)

*For the list of acronyms, click here.

Dr Kaarls, the President, welcomed participants and observers to the 16th meeting of the CCQM, noting that the past year was one of increased activities involving a larger number of Associate and Member States as well as enhanced participation from NMIs and Designated Institutes. He expressed, on behalf of all present, his regrets concerning the situation in Chile and wished them a speedy recovery following the devastation arising from the recent earthquake. Prof. Wallard, Director of the BIPM, also welcomed participants.

2. APPOINTMENT OF A *RAPPORTEUR*

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

3. APPROVAL OF THE AGENDA

No additional points were raised that needed to be addressed. The agenda was approved without change

4. REPORT ON THE FIFTEENTH MEETING OF THE CCQM

No comments were raised with respect to the report of the 15th meeting of the CCQM, which Dr Kaarls subsequently declared approved. Dr Sturgeon was thanked for his efforts.

5. RULES OF PROCEDURE FOR CCS, CCWGS AND CC WORKSHOPS

Dr Kaarls drew attention to a new document, finalized and approved by the CIPM in October 2009, entitled, “Rules of Procedure for the Consultative Committees (CCs) Created by the CIPM, Working Groups and CC Workshops” (CIPM-D-01) which was written for the CC Presidents, section and WG chairs and CC executive secretaries to ensure consistency, harmonization and smooth functioning of all the CCs, CC WGs and workshops. The document is available at http://www.bipm.org/en/committees/cc/.

5.1 REAPPOINTMENT OF CCQM WG CHAIRS

In accordance with the above document, WG Chairs are required to be reappointed every 4 years. Dr Kaarls reaffirmed his satisfaction with the work of the current Chairs and proposed their reappointment for another term. There was no opposition to the proposal and the current WG chairpersons were reappointed.

6. REPORT FROM THE CCQM *AD-HOC* WORKING GROUP ON THE KCRV

Dr Cox, chair 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 CMCs with results of KCs in a more transparent manner. Dr Cox briefly outlined earlier activities relating to document CCQM/09-03 wherein a set of thirteen data evaluation principles had been established, as well as document CCQM/09-15 containing criteria by which a CMC uncertainty can be judged in terms of a particular KC result (limited to conditions under which a 1:1 correspondence between the matrix, the measurand and its amount fraction and the measurement procedure applied were clearly established). Recent activities arising from the workshop in Rio de Janeiro in November 2009 focused on a half-day workshop, which was primarily a tutorial, and discussion of two further documents. The first of these, CCQM/10-03, provides guidance for KCRV and DoE calculations and a procedure for calculation of the KCRV through implementation of the earlier data evaluation principles. This is ongoing and comments have been solicited. The second, a procedures document complements the guidance document and contains worked examples of KC data evaluation which will be made available after further consultations and after the guidance document has been stabilized through additional discussions in the stakeholder community.

Future steps were then outlined which would include further promotion of the principles of the document so as to better discern its possible limitations, an invitation for comments on the guidance document so as to update it, and release of the procedures document and work with the KCWG on uses of KCRVs and DoEs for CMC approval.

Dr Cox's presentation was followed by an in-depth presentation by Dr Ellison which addressed document CCQM/10-03 ("Estimation of a consensus KCRV and associated Degrees of Equivalence"), a draft of which was circulated in March 2010. CCQM/10-03 serves as a bridge between chemists and statisticians and provides guidance on general statistical criteria, its application and the use of sound judgment for the WGs to follow when calculating a KCRV, estimating the KCRU and calculating the DoEs. He raised the point that calculation of uncertainty associated with the DoE should take into account correlation and gave examples of the impact of disregarding this advice. Dr Ellison outlined the next steps to be taken, which will address the few substantive comments obtained to date and addition of examples of data analysis. The next steps will also include approaches that are likely to be useful for individual WGs, in particular, the BAWG. Remaining issues included handling of anomalous values through development of better statistical models and the impact of more rigorous treatment of KCRV uncertainties and correlation effects on the

interpretation of DoEs, raising the question of whether the *ad hoc* WG should return to a consideration of the interpretation of KC results *vis-à-vis* CMCs.

Dr Kaarls opened the presentations for discussions, noting that document CCQM/10-03 had arrived too late for any appreciable reading/discussion prior to the meeting and thus proposed that the WGs examine it in more detail during the fall meetings.

Dr May felt that it was unwise to undertake more work until the WG chairs received input to determine exactly what KCs are and what they mean, suggesting that they are being interpreted differently by the different WGs.

Dr Wielgosz commended both Dr Cox and Dr Ellison for their efforts and noted his agreement with the concerns expressed by Dr May. There was a pressing need to resolve the problem of how to interpret results of KCs, and the uncertainty of the KCRV, and whether a laboratory could claim uncertainties on its CMCs that were smaller than that of the KCRV.

Dr Milton agreed on the need for interpretation, citing the necessity of examining both charts of the DoEs as well as the original data to gain more insightful information.

Prof. De Bièvre stressed that any decision to remove outliers to enable calculation of the KCRV could only be sanctioned on the basis of a sound debate relating to physico-chemical principles behind the measurement.

Dr Sargent reiterated Dr May's assertion regarding the need for clear interpretation of KC exercises, citing the significant distinction between measurements made on matrix materials versus those of calibrators.

Dr Sommer argued that there was an urgent need for better statistical models to cope with outliers.

Dr Mackay noted that there was a definite need for a document which would improve the consistency of approaches because WGs required worked examples in order to fully comprehend the meaning and usage of the proposed statistical calculations.

Dr Kaarls concluded discussions by noting that more input from the CCQM community was required and that a future meeting should address specifically what it is that KCs tell us and how outliers should be handled. He suggested that Dr Cox and Dr Ellison should await further feedback from the WGs before investing additional time into further development of the documents.

Dr Kaarls noted that the work of the *ad hoc* WG chaired by Dr Turk on efficient and effective testing of CMC claims (EETWG - established by the President in April 2007) 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 their number, needed to underpin the CIPM MRA without compromising their effectiveness. Dr Turk's work has resulted in much debate and provided direction and a gap analysis on where to go, especially for the three WGs where most comparisons have been made (GAWG, IAWG and OAWG) and the WGs are now on track to determine what needs to be done. As such, Dr Kaarls suggested that the EETWG had fulfilled its mandate and could now be dissolved. He will move to accomplish this after consultation with Dr Turk.

7. REPORT AND DISCUSSION ON THE REDEFINITION OF THE MOLE AND DRAFT *MISE-EN-PRATIQUE*

Dr Milton, chair of the CCQM Working Group on the redefinition of the mole, presented a brief historical overview of the origins and 1971 definition of the mole and then introduced several proposed wordings for a re-definition [*Metrologia*, 2009, **46**, 332], semantically somewhat different but essentially all based on the notion that it is "the amount of substance of a system that contains exactly 6 022 141 5 × 10^{23} elementary entities", equivalent to an "explicit constant" definition stating "the amount of substance of a system is such that N_A is exactly 6.022 141 5 × 10^{23} per mole". In such a case, the exact number of entities in one mole could be specified but the exact mass of one mole would not be known. Fundamentally, there is no objection to moving to a definition of the mole based on a fixed value for the Avogadro constant since it is, in fact, an amount of substance. Dr Milton then examined the few known views opposing redefinition as arising from Prof. Jeannin [*Chemistry International* 2009], Andreas *et al.* [METAS, *Chimia* 2009] and Prof. Leonard [*Metrologia*, in print 2010], providing simple rebuttals to each of the points raised. He then concluded, stating that redefinition would not be considered independently of a proposal to re-define the ampere and the Kelvin; that if a change is made, the *mise en pratique* for the mole would need to be rethought and reworded and that there must be broad-based agreement such that the change would be widely promoted within the community.

Dr Kaarls reminded the CCQM that last year a recommendation was made that in principle (with the exception of METAS) there was agreement that:

1. a redefinition of the mole was favoured,
2. that there should be no action taken without more measurements being available on the isotope abundance measurements of silicon and,
3. that prior to the change, a more widespread understanding of the concepts and its acceptance within the chemical community must be achieved.

7.1 IDMS METHOD FOR THE RELATIVE ATOMIC MASS OF SILICON

Dr Schiel presented an overview of the status of the molar measurements of silicon at PTB, summarizing progress with a novel approach to the use of isotope dilution to accurately and precisely determine the abundances of ^{28}Si, ^{29}Si and ^{30}Si in 99.9949 % enriched ^{28}Si substrate used for the Avogadro sphere; ^{29}Si and ^{30}Si would be treated as isotope element impurities. The target expanded uncertainty was $< 1 \times 10^{-8}$, deemed achievable as only the ^{30}Si/^{29}Si amount ratios needed to be measured following alkaline dissolution of the ^{28}Si sample. Results for the molar mass of ^{28}Si between PTB and IRMM were discrepant by 0.4 ppm and after an in-depth technical presentation of the measurement techniques used at PTB, it was concluded that this discrepancy could be accounted for by contamination of the ^{28}Si sample with natural abundance Si at IRMM during manipulations, as perfect agreement could have been achieved if the procedural blank value was changed from 509 ng/g to 309 ng/g of Si. For this reason, PTB will utilize ID-MS to determine an accurate blank content of the solutions used at IRMM. Currently, there exists a bias of 0.3 ppm between the molar mass of Si determined in this manner with that of the CODATA value which arises from the NIST watt balance.

Dr Güttler opened discussions by noting that when searching for an explanation for discrepancies in on-going work it is necessary to make an assumption and then to experimentally verify that assumption, which may involve a great deal of work, time and effort which needs to be appreciated by the community. Dr Kaarls agreed that such an approach is essential in order to confidently arrive at a correct decision.

Dr Kaarls then called upon Prof. De Bièvre as the representative of IUPAC to present the opinion of this valuable stakeholder organization. Prof. De Bièvre noted that the committee on nomenclature had met in Lisbon the previous week during which definitions of base quantities and base units were reviewed. It is noteworthy that although the seven base quantities are by convention regarded as independent, their respective base units are in a number of cases interdependent; for example, the definition of the mole incorporates the kilogram. He stated his concern that a definition of the mole based on a fixed number of base entities such that N_A is fixed will be difficult to teach. However, he recognized that in 2009, IUPAC did not oppose a definition of the mole based on the concept of fixing N_A as had been discussed in CCU. In light of the nomenclature committee meeting in Lisbon, IUPAC would support a redefinition subject to the following suggestions: the name of the base quantity needs to be changed and a note should accompany the new definition to explain that the molar mass of ^{12}C will be an experimental quantity, but was unable to confirm if these would be the final recommendations of IUPAC. In response to further questioning by Dr Milton and Dr Thomas (BIPM) that the CCU website does not cite the same definition as presented by Prof. De Bièvre, it became clear that there was some confusion as to the wording propagated through various IUPAC documents. Dr Wielgosz commented that there seemed now to be a supplementary recommendation from IUPAC (see document CCQM/10-22) to consider changing the name of the amount of substance, which has hitherto not been submitted. Dr Kaarls noted that due to the vagaries of the proposals tabled, discussions would have to be limited but that in any case the CCQM does not agree to any renaming of the amount of substance.

Dr Andres questioned how to proceed with community outreach. Dr Charlet announced that a recent meeting of the French Academy of Sciences failed to reach agreement on the proposals to redefine the mole and concluded that a series of meetings to voice opinions was needed and a document should be prepared which summarized all arguments so as to facilitate further discussions.

Dr Kaarls summarized the current discussions, noting the excellent work undertaken by IRMM and PTB on isotope measurements but also the need to await further work before a final decision could be taken. He agreed that most of the chemical community is unaware of these issues. He called for the CCQM to reconfirm to the CIPM that its recommendation to the CIPM was the same as last year. The position of IUPAC is not clear, especially in regard to the need for a new term for the amount of substance and, if this is the case, more in-depth discussions on the reasoning behind this proposal will be needed. In regard to Dr Charlet's suggestion of creating a document outlining the pros and cons of the situation, he charged the CCQM WG on the redefinition to undertake this task and invited others to join with Dr Milton in ensuring this was accomplished. The CCQM will thus not agree to proceed with a redefinition of the mole until these issues have been settled.

Prof. De Bièvre expressed his agreement with this summary, as well as the need for a redefinition of the mole, subject to the proviso of wider consultations. He noted there was a need to establish traceability of documents within the IUPAC hierarchy to track their movement from committee to committee and to understand how the wording of definitions may become altered in the process.

Prof. Wallard clarified that a new resolution is not being sought, only a confirmation in the minutes that the position of the CCQM has not changed from that offered in 2009. (Note that these recommendations are summarized in the Appendices at the end of this report).

8. SUMMARY OF THE CCQM WORKSHOP ON FORENSICS

Dr Wielgosz made a short presentation summarizing the activities of a one day workshop organized at the BIPM (abstracts available as CCQM/10-10) on 12 April 2010 devoted to "Metrology for Forensic Science – Chemical and Biochemical Analysis" which attracted international participation. Some of the major conclusions of this meeting were that:

(i) forensic laboratories now have ISO/IEC 17025 accreditation requirements;

(ii) that 70 % of all testing undertaken comprises those for controlled substances, toxicology and DNA work;

(iii) that annual reports from the European Network for Forensic Science Institutes (ENFSI) on CRM requirements will be distributed to NMIs;

(iv) that regional networks are being formed and;

(v) International cooperation is under discussion, which provides an opportunity for follow-up by the BIPM; that measurements of nominal properties and descriptors of probability require further discussion and cooperation, and that reference materials are required for designer drugs.

Dr Emons remarked that with respect to point (v) above, the issue of nominative properties was already being addressed by other committees and that this may not need to be a priority for the CCQM. Mrs Parkes further supported this assertion by stating that written standards for nominal properties were in preparation and duplication of effort should be avoided, to which Dr Kaarls replied that an open mind should be kept. Dr May asserted that such discussions are frequently undertaken at NIST, revolving around issues of what could be done versus what should be done or could be afforded to be done. He suggested that a scenario might soon arise whereby the CCQM may be overextended. Dr Kaarls argued that the CCQM should see what develops, that there was a need to serve the outside community, and that if it had a high priority the CCQM should look into it. Dr May noted that this may require having to stop doing something we are now doing. Dr Kaarls agreed that this point was well taken.

9. SUMMARY OF THE BIPM-WMO WORKSHOP ON MEASUREMENT CHALLENGES FOR GLOBAL OBSERVATION SYSTEMS FOR CLIMATE CHANGE MONITORING

Dr Kaarls noted that a highly successful three day workshop took place in Geneva, Switzerland, between 30 March–1 April 2010 during which the World Meteorological Organization (WMO) signed the CIPM MRA, nominating three reference laboratories: a radiation laboratory in Davos, Switzerland; one at the EMPA for ozone measurements; and a NOAA laboratory for greenhouse gases in the US, with the possibility for additional reference laboratories being added in the future. He then invited Dr Wielgosz to make a presentation.

Dr Wielgosz remarked that the meeting should be considered an important step in responding to the challenges set by climate change monitoring since long term measurements require stable reference standards and a calibration infrastructure. The aims of the workshop were to identify key measurement issues in climate science, the Numerical Weather Prediction (NWP) model and earth observation where there is a requirement for improved underpinning metrology; to foster closer links between the metrology and earth observation systems communities; to drive agenda-setting and road-mapping within NMIs to ensure that tools are developed to meet the needs of climate science, NWP and earth observation communities and to inform the earth observation system communities about the capabilities and plans for the NMIs. A draft written report summarizing the workshop is scheduled for release on the BIPM website by 1 June 2010. Eight technical breakout sessions on key topics occurred over three days to develop findings and recommendations on what the real metrology issues were and to discuss how they could be implemented. Issues relating to stable time series for key greenhouse gases (GHG) and other trace species, remote sensing of atmospheric composition and traceability issues in spectroscopic data, ocean salinity and aerosol composition and radiative properties were all addressed. A number of recommendations were made.

For the GHG studies, these included: that the WMO, BIPM and academic communities continue to work together to increase redundancy through development of independent approaches to the provision of standards and carry out necessary comparisons, with results being traceable to the SI where practical; that they continue to develop criteria for Central Calibration Laboratory performance and establish necessary external review of such performance; and that the WMO, BIPM and academic communities collaborate to make best use of established national and international infrastructure, capability and funding to meet the needs for standards.

In the area of remote sensing, it was recommended that research be conducted within the WMO, BIPM and academic communities to resolve identified issues; that all atmospheric measurements of GHGs should demonstrate traceability to established reference standards; quantified uncertainties be reported for all atmospheric processes and measurements of GHGs; spectroscopic reference standards be validated against both laboratory and atmospheric reference spectra; benchmark spectra recorded by different NMIs and laboratories be available to the research community for testing and verification; spectroscopic reference standards yield consistent results across applicable wavelength ranges and molecular spectroscopic reference standards should use validated standardized algorithms for line shapes, mixing, speed dependence etc.

In the domain of ocean salinity it was recommended that a new oceanographic salinity measure was required that was traceable to the SI and consistent with current oceanographic practice as well as with marine chemistry and biology. Commensurate with this was the need to develop the scientific basis and practical implementation of a density-based salinity standard and an advanced seawater standard (pH, density, stability) as well as implementation of standard equations of state for water, water vapour, ice, seawater and humid air.

With respect to aerosol composition and its radiative properties, areas in which the NMIs could contribute included provision of reference materials, development of standards for aerosol number concentration and sampling inlets and sample conditioning as well as supporting inter-comparisons and transferring knowledge to instrument manufacturers.

Dr Kaarls noted that there was significant work to be done within the WMO community and that the latter had an open mind with respect to the recommendations and he suggested that a recommendation be drafted on the subject of further cooperation.

Prof. Wallard supported this recommendation as these activities have been planned for a long time and the workshop opened with high expectations, which have evidently succeeded. He emphasized that there was a real opportunity to capitalize on this momentum since there appeared to be a significant willingness on the part of senior WMO staff as well as those from various institutes to work with the BIPM to undertake comparisons and help deliver on the recommendations. He made a passionate plea that the CCQM and its WGs become engaged in this endeavor so as to emphasize cross-representation.

Prof. De Bièvre noted that the CCQM should welcome this agreement warmly and that very significant progress has been made by the WMO concerning reference standards in the past four years.

Dr Milton reiterated that the workshop was a remarkable success with all the leading authorities in the world in attendance and that the WMO was fully apprised of the benefits of the CIPM MRA. He noted the large number of recommendations and findings which were both broad and detailed. Dr Milton raised the question as to how to proceed and monitor progress with regard to taking forward these recommendations and wondered if a small committee should be established to undertake this task.

Dr Squirrell commented that, from his perspective, this was an excellent initiative and he is delighted with the progress made as ILAC is also interested in promoting such activities.

Mrs Parkes asked whether the newer area of biodiversity was discussed as a result of climate change, considering our current abilities to gauge such effects by monitoring microbiology changes. Dr Wielgosz responded that this topic was not specifically discussed but was alluded to during the presentation of other topics.

Dr Kaarls summarized by noting that a recommendation needed to be formulated to present to the CIPM which would subsequently be conveyed to the meeting of the CGPM in 2011. He asked that Dr Milton and Dr Wielgosz develop a draft for consideration the following day. A second point, raised by Dr Milton, regarded the establishment of a means of gauging follow-up that needed to be considered and suggested, that at least the GAWG and EAWG (for salinity measurements) could follow-up.

Dr May pointed out that the required technical expertise was typically not included in the current WGs and that a broader selection of the "right" persons from each NMI may provide a better approach. Dr Milton suggested that the process should start with an approach to the WMO to form a small committee to oversee this progress. Dr Kaarls summarized that he would take these suggestions forward to the CIPM and ensure that a follow-up strategy was developed.

10. SUMMARY OF THE BIPM WORKSHOP ON METROLOGY FOR THE NANOSCIENCES

Dr Viallon presented a preliminary report on a workshop on Metrology for the Nanosciences, chaired by Dr Steele and which was held at the BIPM on 18-19 February 2010. The workshop was attended by 105 participants from 47 different institutes/companies but with the majority being from NMIs or ISO TC delegates (CCQM/10-09; http://www.bipm.org/en/events/nanoscale). Experts from industry as well as several CCs covered relevant topics of traceability, standardization and reference materials as well as contributing to 8 thematic breakout sessions devoted to thin films and coatings, surface analysis, electricity and magnetism, mechanical metrology, microscopy, aerosols, nanotechnologies and toxicological testing. The principal target question being addressed was "what activities are

required to establish an effective international infrastructure for metrology at the nanoscale". Industry is interested in metrology due to quality control issues whereas the health and safety community takes an interest due to the need for more accurate characterization. In this connection, it was noted that there are numerous technical issues as the measurand is frequently difficult to define, interactions occur with the environment altering the nature of the particles and the use of models and calculations do not yet generally account for uncertainties. Traceability to the SI was principally connected with dimensional metrology and amount of substance. Potential solutions to this area may arise from the activities under way in surface analysis and microscopy and it was apparent that method defined approaches and standard procedures need metrology to aid calibration and that more discussion with industry about metrological terms in general and traceability in particular are needed. Most of the discussion groups acknowledged an important need for reference materials in all fields, especially those relating to aerosols, surface analysis, nanobiotechnologies and toxicity testing, from which it was concluded that a significant amount of work remains to be done. Environmental health and safety issues are strong drivers for the development of normative standards to underpin existing and future legislation but there remains a need for a more scientific basis, translating to the need for more participation in ISO TCs, particularly ISO/TC 229 (nanotechnologies), to foster enhanced liaison with the BIPM as it is a vehicle for NMI participation and awareness of documentary standards activities. As the interdisciplinary nature of nanotechnologies envelopes related work in a number of CCs, notably the CCQM (SAWG and GAWG) as well as the CCL and CCEM, increased communications between CCs should be facilitated by the CIPM and the BIPM. It was concluded that there was a need for more workshops such as this one, as well as satellite workshops held in conjunction with larger conferences and regional programmes such as CO-NANOMET in Europe and VAMAS.

Dr Kaarls asked if there was to be any follow-up with experts from any specific ISO/TCs to which Dr Viallon replied that the diversity of fields requiring such a large number of experts has not been conducive to this. Dr Güttler reiterated the need for cooperation across different CCs and WGs as well as external experts because of the cross-cutting activities in question. Dr Kaarls noted that some elements of this were under way in the GAWG and SAWG and clear examples of how to foster closer collaboration amongst the CCs is needed. Prof. De Bièvre pointed out that the VIM allows for operationally defined measurands and procedures.

11. SUMMARY OF THE BIPM WORKSHOP ON PHYSIOLOGICAL QUANTITIES AND SI UNITS

Prof. Kühne summarized the activities and outcomes of the successful workshop on Physiological Quantities and SI Units held at the BIPM on 16-17 November 2009, which attracted 70 participants from 22 countries, principally from NMIs and those working on relevant international TCs. The summary document is posted on the BIPM website (CCQM/10-05). The aim of the workshop was to bring together those communities concerned with traceable, reliable and comparable measurements with those responsible for writing and applying specification standards and/or health and safety legislation in order to identify future challenges and the steps forward. The principal topic was Health and Safety for Humans, covering the areas of optical radiation, radio and microwaves, ionizing radiation, sound and ultrasound, magnetic fields and biological quantities. Physiological quantities and SI units encompasses diverse subjects in all disciplines and it was concluded that the aim of the workshop was best achieved by establishing direct links between the relevant CC or Joint Committee (e.g., JCTLM) and the relevant TCs of the standardization bodies. The most useful cooperation might

possibly involve the CCAUV, CCPR, CCEM, CCQM and CCRI. The next edition of the SI brochure should include more information on physiological quantities and SI units for which the CCU will need additional input from the CCs. Physiological quantities can generally be expressed in SI units but currently uncertainties are not well accounted for and the limits of application of "action" models are not well understood. The potential for harmonization of terms was emphasized as a draft of ISO/IEC 80003 (Quantities and units used in physiology) which will be circulated in 2010. A need for more uniform regulations was expressed and regulators were encouraged to inform NMIs and relevant TCs on how best to get involved. It was clear that additional workshops and forums may be useful to increase awareness at the national, regional and international levels. Two direct recommendations arose: contact should be established between the CCEM and the ICNIRP (International Committee for Non-Ionizing Radiation Protection) as effects induced by magnetic fields on the human body have not been considered within the framework of the CCEM; and it was advised that the CCEM and the CCRP consider in future the case of radiation at terahertz frequencies, possibly through the creation of a joint group arising from both CCs.

Dr Milton opened the discussion by commenting on the fact that this was a fascinating field of study and questioned how units were defined, to which Prof. Kühne replied that each community used its own definition to relate the physiological function to an SI measurement through a response function but that there were unfortunate "grey" areas between extremes of working models which remain to be explored. Dr Sommer stressed that conversion factors relating physiological response to SI units were extremely important and since there are many databases, NMIs should clearly be involved in the development of this work.

12. SUMMARY OF ACTIVITIES RELATED TO MATERIALS METROLOGY

Prof. Wallard spoke briefly about materials metrology with respect to VAMAS activities. Several years ago the CIPM commissioned a report on the potential opportunities for activities in the broad materials metrology area which provided, among others, a recommendation that a new CC was not needed in this domain as all issues could be handed within existing CCs. More importantly, the broad relationship that was expected to develop with VAMAS has not yet materialized; no representatives were nominated to serve as liaisons between VAMAS and relevant CCs and no proposed areas of need in which they would like to see comparisons organized have been put forward, despite repeated and recent requests for this information by the Director.

Dr Kaarls noted that this situation was a rather strange circumstance since the BIPM had been earlier approached by VAMAS to provide help on metrology matters. Dr Prins mentioned that VAMAS is now working on this matter and will prepare a list of needs and nominate the requested liaisons. Dr Milton noted that the advances in material metrology were summarized in *Metrologia*, 2010, **47**(2).

13. REPORTS OF CCQM WORKING GROUPS

13.1 Organic analysis

Dr May presented his report of progress made by the CCQM Working Group on Organic Analysis (OAWG), which had met twice since the last meeting of the CCQM. A meeting was held in Rio de

Janeiro, Brazil, on 4-5 November 2009, for all of the CCQM WGs and 51 participants from 27 institutes of 23 countries/economies attended the OAWG, during which a workshop was hosted at INMETRO on "Metrological use of NMR for organic purity analysis/assessment within the NMI community". Earlier in the week, 44 participants representing 28 institutes of 21 countries/economies met at the BIPM. After outlining the OAWG terms of reference, Dr May proceeded with an update on the OAWG strategic plan to efficiently and effectively support CMCs in the organic CMC "space"; report on results from comparison studies and highlight discussions of the OAWG pertaining to the CIPM Traceability document, a need for the establishment of a new OAWG mailing list and the need for stronger communications with RMOs regarding their Key, Pilot and Supplementary studies in the organic area.

Dr May noted that during the past year, CCQM-K50 (PAHs in Soils/Particulates) and CCQM-P114 (Selected PBDEs and PBBs in plastic) studies were issued as final reports; that KC Draft reports of CCQM-K62 (Nutrients in Infant/Adult Formula: Vitamins), CCQM-K63a, CCQM-K63b (Non-Peptide Hormones in Serum: Cortisol and Progesterone) and CCQM-K69 (Anabolic Steroids in Urine: Testosterone Gluconoride) were approved by the OAWG and sent to CCQM WG chairs for review and that Pilot Study summary reports covering CCQM-P20.f (Organic Purity Assessment Series: Digoxin), CCQM-P78.1 (Nutrients in Infant/Adult Formula: Vitamins), CCQM-P109 (Determination of Acrylamide in Cooked High-Carbohydrate Food) and CCQM-P115 (Anabolic Steroids in Urine: Testosterone Gluconoride and Epitestosterone Gluconoride) were approved by the OAWG to be archived on the restricted access OAWG website. To date, a total of 53 KCs have been conducted and their associated KCRVs have been agreed by the OAWG along with 41 Pilot Studies with a total of 126 analyte-material combinations. It is evident that the current approach, at the same level of effort, is not sustainable, and a finite number of comparisons that test the institutional knowledge and core competencies required to deliver services recognized under the CIPM MRA, rather than the techniques, is needed.

Dr May outlined a four-track strategic approach for comparison studies:

(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;

(C) Key comparison studies in emerging areas of global interest and importance with an accompanying pilot study; and

(D) capability assessment studies of measurement capabilities being established in new areas for NMIs/DIs.

Track A activities would establish approximately 10 KCs over the next 5 years to test core capabilities which would be repeated with different analyte/matrix combinations at 5 year intervals; these were summarized in tabular form, identifying generic measurands. A current example of such a study was that of CCQM-K55.a/CCQM-P117.a (Purity assessment of high purity organic materials: 17 β-Estradiol) whose Draft B report is now under review. Examples of Track B studies which would support services listed in the database submitted by more than one NMI, included CCQM-K80 (Creatinine in serum) and CCQM-K79 (Ethanol in aqueous matrix) which are in Draft B and Draft A report status, respectively. The CCQM-K80 study examined 17 materials from 6 NMIs using repeatability tests conducted at NIST and found excellent DoEs for all NMIs, illustrating their ability

to make such measurements and to provide CRMs. For CCQM-K79, 27 materials from 9 NMIs examined by BAM at ethanol levels ranging from forensic to commodity were in excellent agreement. Dr May argued that results from studies such as these should also be written for submission to peer reviewed journals which are widely read by the chemical measurements community. This would further demonstrate the practical value of the CCQM activities to the scientific community and reinforce the integrity of the CIPM MRA. Track C studies were exemplified by CCQM-K81 / CCQM-P122 (Chloramphenicol in pig muscle), currently in draft A report phase, for which 6 laboratories participated in the KC and 4 in the pilot with all laboratories demonstrating good agreement at measurand amount contents lower than action levels for the KC, but with less satisfactory agreement amongst participants in the pilot study.

Dr May outlined additional studies planned for each of these tracks. For the testing of core capabilities (Track A), CCQM-K55.b with parallel CCQM-P117b – purity assessment of high purity organic materials: Aldrin; CCQM-K55.c – chloramphenicol or a measurand with similar properties; and CCQM-K78 – mass fraction composition of a calibration solution: Aldrin, would be undertaken. For Track B, the OAWG is awaiting feedback from the KCWG as to what additional KCs are needed. For Track C, comparisons in emerging areas, CCQM-K85 – antifungals in food: malachite green in fish tissue was being considered. For Track D, capability assessment studies for new areas, a pilot comparison would be considered targeting the determination of ethanol and water in bioethanol derived from sugar cane.

Additional samples available for potential Key and Pilot comparisons could be derived from the BIOREMA project, which is to make available homogeneous and stable samples of bioethanol and biodiesel (FAME) for testing capabilities for determination of ethanol and water content in bio-ethanol as well as methanol, free glycerol, triolein and methyl esters of linolenic, linoleic, oleic and stearic acids. This study has now been postponed for at least three years because the ability of the OAWG to do the work is not compatible with the needs of BIOREMA, in that an imminent use of the materials for a PT cycle precludes its usefulness for a CCQM study. Noteworthy, however, is that a number of NMIs will participate in the PT scheme. With regard to a potential purity comparison of the ethanol and water content in bioethanol, it was decided that the homogeneity of the material with respect to water would be insufficient for testing capabilities of NMIs because heterogeneity at the level of several tenths of a percent was determined for the water content. Additionally, ethanol does not occupy any unique space in the proposed purity assessment strategy to be of significant interest to many NMIs. Thus, a Pilot study may proceed, but no plan is under way for a KC.

Several relevant RMO studies were then summarized, including APMP.QM-P19 (melamine in milk); APMP.QM-Pxx (illicit drugs in hair) and APMP.QM-Pxx (pesticides in tea) and Dr May again stressed the need to minimize the growing number of comparisons that needed to be brought under control.

Dr May mentioned that he was working with the BIPM in order to correct the mailing lists that are used to solicit interest in comparisons amongst OAWG participants. He asked for help to reduce the list to a minimum number of the most appropriate people in each Institute to receive mailing information before April 2011. Other recommendations arising from discussion were the need for an official template to cover Key Comparison reports and publications, and the development of guidelines on publication of Pilot studies (decision to publish, authorship, where published). It was suggested that NMIs should publish new measurement methods developed for delivery services to customers when and where they wish, providing an acknowledgement to the CCQM for use of the samples and data was made.

Dr May concluded by announcing that meetings of the OAWG, BAWG and GAWG would take place during 2-5 November 2010 in Singapore.

Dr Cox remarked that KCs test for measurement capabilities, but that the reported uncertainty will also reflect a component of stability of the measurand in the sample and it is important that the stated uncertainty be clearly defined as to what it represents. Dr Magnusson noted that in regard to CCQM-K80 (Creatinine in serum) where the effective reference value is zero, it suggests that different CRMs each with different uncertainties were equivalent, but asked to what degree. Dr May replied that most were equivalent and that a user could select an appropriate one from any of the producers with a view to fulfilling fit for purpose needs for decision making such that there would be no adverse impact on the quality of measurement. Dr Emons returned to the discussion point raised by Dr Cox, stating that there was no direct relationship between an NMI's ability demonstrating measurement capability on a material during a comparison and their claims on a calibration certificate. Additionally, he stressed the need to get targeted readership and the increased number of publications arising from CCQM studies into the more open literature. Dr Wielgosz praised the CCQM-K80 results as being very valuable and noted that a similar approach had been used in CCQM-P73 coordinated by the BIPM and organized through the GAWG, in which proper treatment of the DoE's required correlations to be taken into account, and there was a need for the statistical treatment of such studies to be more formalized. As regards the e-mailing lists of CCQM WG participants, the situation was more complicated for CCQM than other CCs in that normally the membership of the WGs was a subset of the membership of the CC. Since the CCQM had not followed this model, the BIPM had no lists of Membership for the WGs. Lists of participants in WG meetings were maintained by each of the WG Chairs. Dr May reiterated his frustration with the large list and the inability to identify persons within NMIs/DIs actually responsible for decision making and suggested that the BIPM be responsible for mailing future invitations for comparisons. Dr Ellison pointed out that CCQM-K80 did not take into account correlation and in this manner was not equivalent to the approach taken by the GAWG.

Dr Güttler commented that the results of the chloramphenicol study demonstrated a significant improvement in agreement amongst laboratories compared to typical results within this measurement community at large, which were frequently characterized by a greater than 30 % spread in values. Dr Cox returned to the earlier issue of the need to clarify his point about whether KCs test measurement capability of an NMI or their ability to deliver a service to customers. Dr Kaarls pointed out that they were meant to test the claimed CMCs, as these are the services that NMIs regularly provide to their customers. He emphasized his agreement that the system used to test capabilities needed to be changed due to the large volume of work. He asked that the representatives of all RMOs make available information on planned KCs and Pilot studies as they may be of use/interest to laboratories in other RMOs and duplication of efforts could be minimized. Dr Wielgosz emphasized that the current discussions relating to mailing lists were tantamount to decisions of membership regarding who could participate/attend meetings and if current practice was to be changed these issues would need to be clarified.

13.2 Inorganic analysis

Dr Sargent presented his review of the activities of the CCQM Working Group on Inorganic Analysis (IAWG). The group had met twice (jointly with the EAWG) since the last CCQM, at the joint WG meeting in Rio de Janeiro in November, 2009 as well as earlier in this week during which a workshop

on the analysis of HCl was jointly held with the EAWG, along with discussions on strategy development. Eight KCs have been approved for equivalence during 2009-2010, with 5 KCs and 13 Pilot studies currently in progress. He noted that CCQM-K30.1 (Lead in wine) was on hold as a consequence of the recent earthquake in Chile and the damage to the CMQ-F laboratories; that CCQM-K72 (Purity of zinc) was delayed because of a difficulty in sourcing the test material and that the majority of the Pilot studies were finished but reports needed to be completed. He then highlighted details of these studies.

CCQM-K70/P100.3 (Mercury in natural water) was organized by PTB, BAM and LNE and targeted a concentration level required by the European Environmental Quality Standard (< 50 ng/L). A spiked natural water served as the test matrix and a gravimetric reference value was ultimately established following a metrological assessment of the endogenous background mercury content. The results of the comparison, involving 10 NMIs were determined to be fit for purpose and complemented a Euramet 924 parallel study.

CCQM-K75/P118 (Toxic metals in algae) served a "benchmarking" exercise for the determination of Pt and Ni in that all NMIs were encouraged to participate, resulting in contributions from 18 NMIs. IAEA, the coordinating laboratory, suggested a KCRV based on the median for each measurand.

Pilot study CCQM-P119 (Lead in lead-free solder) was coordinated by NMIJ, NIM and KRISS and served to address issues relating to the RoHS directive. Eight NMIs and 2 expert laboratories participated, with good agreement and it was concluded that a follow-up KC would be organized and a core capability matrix completed, the structure of which he outlined. NMIJ was proceeding with the certification of the test material as a future CRM.

Recently approved KC and Pilot studies were noted: a KC and a Pilot Study Comparison (PS) for elemental calibration solutions (PTB); for lead in lead-free solder (NMIJ); for trace and essential elements in a herb (HKGL) as well as Pilot studies for arsenic and arsenobetaine in calibration solution and Japanese sea bass samples (NMIJ) and for Pb, As and Hg in a cosmetic material (NIM). A possible KC/Pilot study on bio-ethanol (same sample as BIOREMA) and a proposed first KC following 3 earlier Pilot studies on isotope ratio determinations, based on a request from the KCWG, was being planned if a coordinating laboratory could be identified.

Dr Sargent then summarized activities undertaken by the RMOs, citing 4 comparisons: a completed APMP.QM-K24/P12 linked to CCQM-K24 (Cd in rice); a proposed APMP KC to be linked with CCQM-K56 (essential elements in soybean powder); a proposed SIM supplementary comparison on trace elements in water (SIM.QM-S2) and a COOMET proposed supplementary comparison on moisture in grain. With respect to APMP.QM-K24/P12, CCQM-K24 is already 10 years old and although KRISS, NIM and NMIJ participated in both comparisons, discussions centered on how to link these two KCs and whether it may be better to have a stand-alone Supplementary comparison. KRISS agreed to contact the participants to seek their approval to change it to a KC, prepare a report on how a linkage could be established and arrange to send it to suitable statisticians for their input and guidance. Similar concerns were raised with respect to linking CCQM-K56 to APMP.QM-KC/PS for trace elements in soybean powder. For SIM.QM-S2, it remains to scope out this comparison to define what CMCs it will support as concerns were expressed that it would not be a significant enough challenge to provide for any depth with respect to how far the light shines regarding core capabilities. With respect to the COOMET proposal for determination of moisture in grain, it was concluded that there was no expertise within the IAWG to support such an activity but it was noted that the CCT WG on humidity reported that Euramet Project 1061 was engaged in the evaluation of current status of work in NMIs on measurement of moisture in materials and was

gathering opinions on future directions. It was concluded that a measurement of weight loss on drying (i.e., moisture) relies on industry standard protocols.

Dr Sargent then turned to a discussion of strategy development within the IAWG aimed at enabling more efficient and effective testing of CMCs. This is to be achieved by developing the concept of core capabilities in an effort to minimize the number of KCs and to annually engage all members in benchmarking studies and provide evidence that a small number of comparisons can reliably underpin a wide variety of CMCs. To date, 3 such benchmarking exercises have been completed: CCQM-K49/P85 (using Fe and Zn in bovine liver), CCQM-P106 (Cd and Cr in polypropylene) and, most recently, CCQM-K75/P118 (using Pt and Ni in algae). For the latter study, participants were requested to complete a core capabilities template, designed by Dr Turk, which will be appended to the study report. As CMCs will rarely have a 1:1 match to an existing KC, CMCs can be tested based on clearly defined core capabilities and an NMI's performance in several relevant KCs, which together would comprise a valuation of their claimed delivery service. Additionally, the IAWG proposes that a core capability matrix be added to earlier studies as either an insertion into unfinished reports or as an annex to previous reports; the final decision will depend on feedback from the KCWG and the RMOs.

A 5-year plan for the IAWG was then outlined by accounting for inorganic CMC categories, IAWG techniques and measurands, from which an analysis indicated that 10 major groups of sample matrices, 10-15 major groups of measurands and 10 distinct measurement techniques needed to be addressed. Dr Sargent suggested that 3 KCs (with attendant Pilot study) per year would be targeted for the next 5 years to cover all 10 CMC categories and periodic benchmarking with participation by all NMIs. An NMI with CMC claims would be expected to undertake at least one KC every 1-2 years. Real sample matrices would be used where possible and would address cycles of elemental analyses, anions/inorganic compounds, isotope ratios and speciation. The next meeting of the IAWG / EAWG was scheduled for 29 September–1 October 2010 at SP, Boras, Sweden, with proposals from NMIA and NIMT for 2011 fall meetings.

Dr Kaarls expressed his pleasure at seeing the development of ideas and plans designed to handle the number of comparisons needed. He recommended that everyone read pertinent documents posted on the BIPM website as the documents provide answers to many questions, such as how to deal with supplementary comparisons and link results to earlier KCs.

Dr Kustikov expressed thanks for efforts made by CCQM to raise interest in the determination of moisture in grain, but noted that the CCT had been unresponsive in this matter and thus such comparisons are currently orphaned. There are no reference materials available for dew point measurements. A number of COOMET laboratories are attempting to organize a moisture comparison study but solid decisions are needed and, as a consequence, he was tabling a complaint that CMC claims have been made but no one in the CCQM or CCT is prepared to run a relevant comparison to support them. Dr Kaarls asked if other WG chairs had any ideas to express on this issue. Dr Milton noted that trace moisture in gases was undertaken in the GAWG and a CCQM comparison will soon be under way, but testing is not usually traceable to the SI and thus does not fit into the CIPM MRA structure. Dr Kaarls agreed with this argument and that there was a gap between NMI work and that undertaken in testing laboratories, but questioned whether NMIs should contribute to solving this problem. Dr May emphasized that the answer to this issue was already presented following a joint meeting of the OAWG with the CCT in Korea along with a member of an ISO committee wherein it was concluded that moisture is a method defined measurand and

traceability is to the method. The method only measures weight loss, which will include all volatile species. Thus, does the CCQM provide traceability to a method or to the SI, and within the OAWG there is already too much work. Dr Emons welcomed these comments and argued that this issue is faced daily by all NMIs producing dry-powder CRMs as there is a need to determine moisture correction factors for dry mass. NMIs currently specify method defined approaches for this measurement and thus these issues are not completely clear. Dr Máriássy raised a question about whether NMIs should deal with operationally defined measurands – are NMIs able to carry out such procedures better than testing laboratories and what benefit is achieved by having NMIs make such measurements? Prof. Kühne noted that some NMIs have this capability arising from legal impacts of international trade; he thus questioned why such measurements would not be applicable to this testing problem and therefore supported the organization of a comparison exercise. Dr Güttler was in agreement with this as long as all the methods used were identical. Dr May reiterated that the CCQM is attempting to define traceability primarily to the SI and not to a method and questioned why the testing community could not take care of this issue itself. What are the unique capabilities of NMIs – the measurand is not water. Dr Kaarls suggested that a unique solution could not be immediately found and that the question should be referred back to the CIPM for guidance as perhaps such capabilities should not be classed as CMCs as they may not fit into the scope of the CIPM MRA. Prof. De Bièvre suggested that the solution to the problem lay with the definition of traceability and reference measurement procedures and referred to these concepts in the VIM, specifically the section "Metrological Traceability (Note 1), Measurement Procedure (note 3) and Reference measurement procedure".

13.3 Gas analysis

Dr Milton noted that the WG had met twice since the last meeting of the CCQM, in Rio de Janeiro in November 2010 and at the BIPM earlier in the week. He then presented a brief summary of activities during the past year, including those related to Key Comparisons CCQM-K51, CCQM-K53 and CCQM-K65 for which reports were delivered to the KCDB; newly proposed KCs, all targeting atmospheric components, which comprised CCQM-K82 (Methane in air, coordinated by the BIPM), CCQM-K83 (Halocarbons in air, coordinated by the NIST) and CCQM-K84 (Carbon monoxide in air, coordinated by the KRISS), species for which the WMO had developed stringent data quality objectives. He noted an enhanced interaction with the global atmosphere watch (GAW) programme and with regards to the latter three KC proposals, presented recent trends in the global concentrations of these measurands to justify the needed comparisons.

CCQM-K66 (Purity of methane, coordinated by NMIJ) was discussed in some detail. It was concluded that a nitrogen filling error had occurred in some of the distributed tanks and thus determination of the KCRV was postponed until the issue could be resolved. CCQM-K46 (Ammonia in nitrogen, coordinated by 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). Expected biases due to the individual measurement methods used (ND-IR, photo acoustic IR, chemiluminescence and UV absorption) were identified and the data were statistically modeled to yield a KCRV (based on a weighted mean). Dr Milton stressed that there was a need for consideration of both the charts for participant's results as well as that for DoEs in order to generate a comprehensive picture of performance, for which CCQM-K46 was a prime example.

Dr Milton then turned to a consideration of the strategy used by the GAWG which was developed to streamline the key comparison process by working on the concept of core species and their concentrations. Analysis of earlier 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 be expanded to cover all "core" species within specified ranges of concentrations. 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 two additional comparison types, i.e., "analytical challenge" (for which species may not be stable in cylinders and preparation of standards may be complex) and "natural gas" comparisons (which contain both core and analytical challenge species). Based on these definitions, 29 % of CMCs arising from the GAWG activities can be classified as core compounds, 17 % are natural gas and 54 % are categorized as analytical challenges for which about half have some kind of supporting comparisons, 25 % have no comparison evidence and 18 % are in need of comparisons. He presented a table of needed comparisons, highlighting carbon monoxide (which is now under proposal), hydrogen, which needs to be revisited, methane, water and a number of species which can be considered as impurities in nitrogen.

Dr Milton then examined the criteria for having CMCs for core compounds/concentrations underpinned by a limited set of CMCs, noting that a laboratory must have participated in at least 3 KCs relevant to the concentration range claimed; participate in one additional KC every 2 years when available through the GAWG; that CMCs would be judged against the most recently demonstrated performance; and that there must be agreement on the establishment of a quantitative link between the CMCs and the KCs. The GAWG would thus continue with the existing approach on HFTLS based on these principles, extending the approach to cover all of the core for those NMIs meeting the criteria, produce a list of comparisons that cover the core and use it to implement cycle XII CMC submissions.

Dr Milton turned to the activities for which the GAWG was engaged in, with respect to the global atmosphere watch programme (GAW) of the WMO, noting that certain GAWG members have been providing VOC standards for GAW stations. During a workshop in Hohenpeissenberg in September 2009, the WMO requested further support for the provision of standards covering NO, NO_2 and additional oxides of nitrogen. There was generally stronger engagement of the GAWG with the global monitoring communities and Dr Milton noted that NOAA/ESRL has joined the CIPM MRA as a DI.

Dr Milton spoke of two major challenges ahead arising from the enhanced interactions with GAWG-WMO activities. The first challenge is maintaining traceability of measurement results to the SI in contrast to environmental monitoring agencies which typically provide measurements that are traceable to another scale. There are many benefits to using the scale, in that the results are generally highly precise, but in order to achieve this precision there is often only one reference laboratory and one scientist producing the data, limiting flexibility and possibly overlooking drift. The SI traceable approach provides for more independence but "suffers" from the fact that its more comprehensive uncertainty budget, comprising verification and stability components (which are frequently lacking from the scale data), results in larger uncertainties in the result. The monitoring community remains to be convinced that the SI traceable approach does produce acceptable values. The second challenge is a technical one, arising from either dynamic or static approaches being used by NMIs to effect calibration, which has created biases amongst participants (e.g., CCQM-K26a, CCQM-K26b) that need to be resolved.

Prof. De Bièvre commended Dr Milton and his GAWG team for their excellent and interesting work. He asked if the scale approach was traceable itself to the SI, to which Dr Milton replied that this was in fact possible to achieve, but current ones are not, since all uncertainties are not accounted for. Dr Wielgosz noted that this was an interesting point and that the scale approach sometimes relies on $u_{\text{stability}} = 0$. Thus, whereas the value is derived to be SI traceable, the reliance is on the stability of the standard. It is not just a question of retrospectively adding appropriately larger uncertainties and it is too late to introduce step functions into long-term measurements; thus the challenge is how to implement traceability while providing stability in the measurement scales over a long period of time.

13.4 Electrochemical analysis

Dr Máriássy presented his report of the work of the CCQM Working Group on Electrochemical Analysis (EAWG), which had met twice since the last meeting of the CCQM: during 4-5 November 2009 in Rio de Janeiro which attracted 12 participants from 10 countries, and the past week which hosted 21 participants from 17 countries. Technical presentations included highlights of recent activities in electrochemistry at INMETRO as well as a presentation by DFM on the influence of the thermostatting liquid on electrical conductivity measurements. Results for several Key Comparisons were presented. CCQM-K19.1 (pH of borate buffer, coordinated by PTB) underpinned measurement capabilities to primary pH measurements in borate buffer and was successfully undertaken subsequent to comparison CCQM-K19 at the request of INMETRO to document their new measurement capabilities. CCQM-K73/P19.2 (Assay of HCl, coordinated by NIST) served as a follow-up to earlier CCQM-P19 and CCQM-P19.2 studies but at higher concentration. Although agreement amongst participants appears good, closer inspection of the results submitted by a group of "high precision laboratories" reveals disagreement for the CCQM-K73 participants. A subsequent statistical analysis by Dr Ellison suggests the study should be abandoned. A half-day workshop on coulometry at the BIPM on 14 April 2010, hosted to resolve or identify problems, was largely unsuccessful but included participation from CENAM, INMETRO, NIM, NIST, NMIJ, SMU and UkrMetrTestStandart. Potential sources of disagreement included sample inhomogeneity, instrumental changes and the influence of carbon dioxide. Concern was expressed that if the comparison is abandoned, support of any CMCs is in peril and thus it would be preferable to salvage something useful. This will be agreed upon in the coming months, possibly through the use of additional bi-lateral or multi-lateral comparisons.

During the CMC submission process an error was discovered in the report for CCQM-K36 (Electrolytic conductivity at 0.5 S/m and 5 mS/m) in that the uncertainty of one participant was incorrectly transcribed, resulting in a need for recalculation of the KCRV. No changes would occur in the calculated DoEs.

CCQM-P37.1 (Ag/AgCl electrode survey) was aimed at identifying factors pertinent to the preparation of Ag/AgCl electrodes and the corresponding effects on measurement results. A report will be prepared in the near future.

Dr Máriássy briefly revisited CCQM-P112 (Assay for EDTA, coordinated by the BAM) to note that on-going efforts to identify the sources of bias amongst participants suggested that one sample had been contaminated with metal impurities and a high concentration of ammonia had been used by another participant, which could account for the disagreement.

CCQM-P83 (Electrolytic conductivity, coordinated by the DFM) sought to expand the demonstrated capabilities of participants to lower levels (0.5 mS/m) than earlier assessed by CCQM-P47 and CCQM-K36 comparisons. Generally, good agreement was achieved amongst the 10 participants but

the samples tested had 100-fold higher conductivity than that for ultrapure water. Measurements at DFM show no significant difference between conductivity determined using paraffin oil or water as the thermostatting medium but water results in a larger cell capacitance.

Dr Máriássy discussed the EAWG strategy and its impact on CMC claims, noting that harmonization of claims with respect to format and evaluation was needed so as to be able to identify relevant new KCs. In the area of pH, phthalate and carbonate buffers were more difficult to work with than others and additional comparisons are needed. He proposed one difficult and one easy comparison in alternate cycles with no more than one every 2 years to support CMCs. For electrolytic conductivity, the degree of difficulty encountered with comparisons depends on an institute's cell design and the focus for needed work lies in the range of log (*k*) from -2 to 0. Planned studies up to 2013 to support CMCs included CCQM-P93 (pH preparation study), CCQM-P111.1 (seawater), CCQM-K14.1 (pH phthalate), CCQM-Kxx/Pxx (conductivity and pH phosphate). He noted that the complete set of CMCs had been submitted and reviewed by the KCWG, revealing issues relating to format, claims made which were far outside any supporting comparison ranges, questions about the lifetime of KCs and their participation and how to deal with cases when more than one KC relates to a CMC when results for all KCs are not consistently good.

Other issues raised by Dr Máriássy included the help being given to the OIML with a draft of IR54 and the work being done with IUPAC on the formation of a new subcommittee on pH which was setup to address problems in pH and its traceability.

Prof. Wallard asked what the participants have done with respect to CCQM-K73 and its use in supporting CMCs, considering the problems that have been so far unresolved. Dr Máriássy replied that no conclusions have yet been reached, but it is intended that this will be achieved in the coming months. In the interim, SMU has stopped certifications of high accuracy until issues are resolved.

13.5 Surface analysis

Dr Unger presented his report on progress of the CCQM Working Group on Surface Analysis (SAWG). He was pleased to note that the SAWG now comprises 13 active members, with Italy (INRIM) recently joining along with an expert laboratory from France (Chimie Paris Tech, ENSCP). Dr Unger noted that the SAWG has broadened applications to include the life sciences and nanotechnology with a diverse portfolio of surface and nanoanalysis techniques that span the 0.1 nm to 10 µm dimension. He announced the first successful CMC claims (cycle X) in this area arise from NIM, NMISA, PTB and BAM as a consequence of CCQM-K32 (Gate oxide, SiO_2 on Si).

Dr Unger proceeded to detail results for CCQM-K67/P108 (Amount of Fe and Ni in (200 nm) Fe-Ni alloy film on Si) which was coordinated by KRISS; a draft B report was submitted in April 2009 and a report in *Metrologia* appeared in February 2010. Noteworthy was that participants developed protocols for XPS, AES and EPMA techniques, enabling the analysis of chemical composition of a nano-scaled alloy layer with uncertainties of approximately 5 % relative and that matrix effects are reduced by calibration with alloy reference samples as opposed to pure elements. CMC claims are expected to be submitted for the next cycle under category 15 with traceability arising from a KRISS CRM.

Dr Unger then turned to a consideration of pilot studies scheduled for 2010, i.e., CCQM-P80, CCQM-P81 and CCQM-P95, studies relating to the need to address calibration issues for electron

probe microanalysis (EPMA) which is extremely important for industrial applications. 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 use of this method resulted in differences in measurements larger than the reported expanded uncertainties, highlighting the need for better standards and CRMs in this field. As such, Dr Unger concluded that the SAWG was not ready to go forward with a key comparison based on the use of EPMA. Measurements will thus be undertaken on single-phase materials in the absence of low *z* elements. The first pilot study will examine a set of four NIST CuAu alloy SRMs with calibration against pure Au and Cu standards. The second pilot study will determine the composition of a thin film solar cell material ($CuInGaSe_2$) using XPS, AES, SIMS and RBS techniques. Calibration will be achieved using a thin film CRM to be produced by KRISS.

SAWG is also considering development of comparisons on surface composition of engineered nanoparticles (ENPs). A proposal for a pilot study on TiO_2 from NRC has been put forward. The ENPs would have to be immobilized on a substrate before analysis and additional experts from outside of SAWG need to be involved to develop a measurement protocol.

Dr Unger then turned to a consideration of a foresight document or template that would be developed to guide the future activities of the SAWG. To accomplish this, a survey on the existing most relevant working areas for metrology undertaken by participating NMIs/DIs was initiated. It was due to be completed by June 2010. Future road mapping would be based on the survey results.

A joint meeting with the BAWG during November 2009 in Rio de Janeiro explored overlap of common areas of interest and potential use of shared reference materials, but highlighted the diversity of the techniques used by the two WGs. Representatives from SAWG were invited to join the BAWG nanobio strategy group and the BAWG WiKi pages.

Dr Emons expressed concern that an activity such as the nanoparticle project should focus on what the needed or relevant measurands are and not simply the question of measuring what can be done with existing techniques. Dr Unger responded that earlier discussions with experts suggested that this was relevant but conceded the need to identify objects for study which are relevant to measurement needs. Dr Wielgosz commented on the use of CRMs for calibration of EMPA needed for CCQM-P80 and asked about their pedigree, to which Dr Unger reiterated the need for such materials for accurate calibration but did not comment on the issue of traceability of those used in that study. Dr Güttler questioned the prevalence of use of EPMA and asked why other primary methods were not considered, to which Dr Unger replied that this was a result of the current state of technology in that the majority of testing laboratories use EPMA. Dr May emphasized that it was time for the WG chairs to meet and discuss their terms of reference and have a collective discussion to see where there was agreement and disagreement. The President noted that this was in the planning process for sometime during 2010. Dr Milton asked what techniques could be used to interrogate curved surfaces such as would be encountered when examining ENPs to which Dr Unger replied that XPS, AES and SIMS were appropriate for this purpose with XPS likely providing the more direct route to quantitation.

13.6 Bioanalysis

Mrs Parkes presented her report of the progress made by the CCQM Working Group on Bioanalysis (BAWG), noting an extremely active year with increased interest and participation from NMIs and

other expert laboratories. The BAWG met twice since the last CCQM. The 15th meeting was held in November 2009 in Rio de Janeiro during which a workshop on bio/surface and bio/nanotechnology took place. The 16th meeting was held in April 2010, which attracted 50 participants from 24 organizations. NRC and UME (Turkey) were welcomed as new members at the meeting.

Mrs Parkes highlighted progress in on-going studies that included: CCQM-P55.1 (Peptide quantification, coordinated by NIST, LGC and PTB), for which concerns arise due to a lack of traceable amino acid standards; CCQM-P58.1 (Comparability of fluorescence in ELISA, coordinated by NIST and NPL) which is being conducted in cooperation with the IFCC; CCQM-P59.1 (Protein structural measurements by circular dichroism, coordinated by NPL) and involving seven laboratories with a focus on improving aspects of sample handling relative to the earlier CCQM-P59 study; CCQM-P94.1 (Quantification of DNA methylation, coordinated by KRISS), which has been delayed; CCQM-P102 (Quantification of cells with specific phenotypic characteristics, coordinated by NIBSC, NIST and PTB) which has recently started and CCQM-P103 (Measurement of multiplexed panel of RNA transcripts, coordinated by LGC and NIST) which is in the report stage. An outline of a route map from CCQM-K61 to CCQM-P103 biomarker which quantified 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 was assessed by UV spec, RiboGreen, real-time RT-PCR and digital PCR with quantification of unknowns performed by real-time RT-PCR and digital RT-PCR. The test sample was selected to be External RNA Controls Consortium (ERCC) number 81, supplied at two unknown concentrations together with a matched calibrant. Results were characterized by a <50 % coefficient of variation (the expectation was a factor of 10!) and discussions for the reasons for outliers suggested these were operational issues; continued discussion was expected to occur during the meeting in Singapore in fall 2010. It was concluded that the future of this area lay with detection and quantitation of multiple RNA transcripts using multiplexed measurements.

Mrs Parkes then turned to a consideration of new study proposals. Based on the earlier success of CCQM-P113 (Relative quantification of genomic DNA fragments, coordinated by IRMM) a Key Comparison (with a parallel pilot study) would be arranged to target relative quantification of genomic DNA fragments extracted from a biological tissue. The measurands would comprise a copy number ratio of two DNA fragments present in MON810, possibly using ERM®-AD418 as the calibrant. Two unknown powders would be subjected to analysis by an expected 80-100 expert laboratories. The exercise would be of interest to laboratories concerned with GM food regulations and thus aligns itself well with activities at IRMM.

Demands from industry (cell therapeutics and biopharma regulators amongst others) for assessing measurement uncertainty for cell bioassays prompted further interest in this area. CCQM-P102 was an earlier successful study supporting bioassay and diagnostic marker measurements that examined cells in suspension by flow cytometry and fluorescence targeting a CD4+ biomarker. A new pilot study proposal will focus on number and geometrical property of cells adhered to a solid substrate, as the morphological characteristics and cell number in suspension or on a solid are key components of any bioassay. Six laboratories are initially participating (INRIM, LGC, NIST, VNIIM, USP and PTB).

Mrs Parkes then considered CMC issues, noting the need to establish guidelines and agreement within RMOs on criteria for their review. There are currently no RMOs with bio-working groups. Experience in this area is limited and thus the KCWG defers all CMCs to the BAWG. Mrs Parkes called for more co-operation between the BAWG and the OAWG, highlighting the need for traceable

standards, as exemplified by amino acids for peptide and protein quantitation. A second issue concerned the relevance of KCs and Pilot studies in supporting CMC claims. A current test case example is the submission of CMCs for quantitative PCR and GM food testing based on CCQM-P113. Difficulties in making decisions on what information to enter into the CMC templates range from HFTLS on the matrix, to exactly what is the measurand and what is the measurement unit – since a copy number ratio (ratio of modified to endogenous material) is actually measured. Additionally, for the dissemination range, CCQM-P113 examined only 0.05 % and 0.5 % but requests covering the entire GM range are desired. A further example, CCQM-K60, was very specific for PCR quantitation and the calibrants used were not traceable, as was the case for CCQM-K60. These problems raised the question of whether any of the claims are actually worth having, since it is likely that the service would never be offered to customers.

A strategic plan was then outlined for the BAWG, focusing on protein and nucleic acid measurements encompassing nano-biotechnology and epigenomics. These topics would be further developed through discussions planned for the 18th meeting of the BAWG in Singapore in the fall of 2010 at which a measurement uncertainty workshop and bio-CMC review group would be convened. Mrs Parkes concluded by drawing attention to an upcoming Biopolis bioscience symposium to be held in Asia which would address bio-measurements underpinning requirements of the biopharmaceutical and medical genetics industries.

A lively discussion of these issues ensued. Dr Kaarls was pleased to hear that a strategic approach to the way forward was being adopted as for the other WGs. Dr May expressed his opinion that KCs are not fundamentally designed to permit new CMCs to be claimed, otherwise NMI staff would be coming to the CCQM to determine what comparisons they could undertake in order to expand the services NMIs offer or to obtain advice on how to serve customers. Rather, it is the opposite situation; NMIs participate in benchmarking activities relevant to the services they already provide. More discussions are needed as to what the WGs should be doing. Mrs Parkes replied that discussions had been held on all activities and the desire is to benchmark and demonstrate competence with existing capabilities but the KCWG guidelines are difficult to achieve. The BAWG has addressed some very difficult challenges which have necessitated some basic research. Dr May strongly felt that it was not the role of any WG to set the research agendas of NMIs, but rather only address their existing capabilities. Dr Kaarls expressed his view that in very new areas some research exercises might be needed and this presents no conflict with Dr May's concerns. Dr Güttler expressed the view that responsible persons need to make a decision on whether to commit money and people to a comparison exercise following discussion of its potential use and impact and that such decisions are risk taking with respect to whether the exercise will be ultimately successful in testing a CMC claim. The studies are designed as carefully as possible with input from many experts but unfortunately criticisms, which may be valid, are raised after the study. Any criticisms should have been discussed at the beginning of the study. Dr Kaarls reiterated that what NMIs deliver to customers should dictate what should be tested by comparisons but in the BAWG one of the problems lies with the review of the CMCs. At the RMO level this does not function properly because there are no RMO bioanalysis WG committees and thus the work falls to the BAWG itself. Mrs Parkes pointed out that some RMOs accept specific CMCs whereas others reject these same claims. There is need for more harmonization and specific criteria to be developed. Dr Emons returned to the discussion of DNA/GMO quantification, noting that it was unfortunate that most customers do not know what it is that they are asking for in a measurement and thus it is difficult to adapt to their needs. NMIs are in the process of understanding the fundamental limitations of PCR with respect to traceability, precision, etc. There are no real alternative measurement techniques that can be applied, with the consequence that the level of maturity of the underlying science and measurement protocols, as noted by Mrs Parkes, is not

sufficient to formulate sufficiently general tests. More studies are needed. Prof. Kühne clarified that the CIPM MRA requires KC exercises to be conducted to compare the DoEs of national measurement standards for those NMIs that are delivering measurement services. An NMI engages in a KC in order to obtain international recognition through the CIPM MRA for that service. Pilot studies are typically reserved for developing methods which may then progress to KCs. The core reason for carrying out KCs is to determine the DoEs of national measurement standards on which services are based. Dr Kaarls agreed, stating that there are research aspects that periodically need to be investigated by the CCs which lead to the development of international measurement standards. Dr Emons disagreed with the assumption that there are always national measurement standards that need to be demonstrated, citing the GMO area as an example where there will never be various national measurement standards (for the same GM event) but rather commonly accepted global standards. Dr May asked the rhetorical question "is anyone delivering or preparing to deliver services in this area". NIST is and has a need to compare this service capability with others and that must drive the establishment of KCs rather than progressing with the philosophy of undertaking a KC and then determining what CMCs can be derived from it. Mrs Parkes responded that the end desire is to demonstrate DoEs but believes this does not require a KC for every measurand/matrix combination but rather a focus on general capability and thus the question comes down to HFTLS for any KC.

13.7 Key comparisons and CMC quality

Dr Mackay presented her report on the work of CCQM Key Comparison and CMC Quality Working Group (KCWG). This Working Group, comprising 21 members drawn from all of the RMOs, had an opportunity to meet during the days preceding the CCQM meeting. Dr Mackay updated the meeting on the status of chemistry CMCs (4558 entries) in the Appendix C of the KCDB, noting the need for new categories or sub-categories for bioanalysis and the fact that most of them are over 5 years old. Some 448 claims were submitted for Cycle XI covering 13 measurement service categories. The first formal review of existing CMC was included in 2010, bringing the total number of CMCs which had to be covered to 751. Gas, pH and electrolytic conductivity were comprehensively reviewed which led to improved consistency and formatting and effected a single-time equivalency check. During the next cycle, metals, alloys and fuels will be selected for this process. The overall approach will be to undertake a review by category rather than age and each RMO will be requested to coordinate this review with their NMIs to ensure that specific services are still being offered as described. The approach will also assess the CMCs with respect to new KC/pilot study results available since the CMC was originally submitted. Three NMIs (PTB, LGC and NIM) submitted claims relating to BAWG activities which generated significant discussion with respect to traceability issues, large extrapolations and narrow HFTLS statements. It was concluded that future BAWG claims would be reviewed by the WG in their mid-year meeting as the RMOs have very limited ability to expertly review them during the intra-regional review process.

Dr Mackay drew attention to the guidance document for KCRV and DoE Estimation (CCQM/10-03) which was briefly discussed and noted that while both the KC results and the DoE chart need to be examined simultaneously, further guidance on the practical application of these principles was still required. The KCWG is now carefully examining the traceability of submitted claims, particularly calibrants, during their review process and raised a reminder of the CIPM traceability requirements (CIPM 2009-24).

Dr Mackay stressed that the Guidance Document for CMCs within the CCQM, which is intended to document all aspects of the process of submission, review and approval of CMCs, may require an additional year to finalize, as it is awaiting progress on the above two documents, on which it depends. The document includes a flow chart outlining the role of the RMOs, KCWG and the 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 (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 a one-to-one correspondence with KCs, and how the uncertainty claim of a CMC can be assessed with respect to the DoE. The document will be updated annually and will be widely distributed to all RMO TC chairs for distribution and use in the intra-regional review process once it is completed.

Dr Sargent suggested that it would be very useful to obtain a simpler summary of only the submission portion of the process for CMCs. Dr Milton stated that he concurs with the need to review CMCs, but questioned the cost of the effectiveness of this process, noting that Euramet, in the last year, only reviewed approximately 10 % in full detail, with the outcome that nothing changed. He suggested that perhaps a lighter review, based on the likelihood that there would be no changes, would be more appropriate. Dr Mackay explained that the GAWG presented a rather unique case study with few concerns but other areas are more problematic. Dr Mitani felt that if the reliability of the decision making process was enhanced with such a detailed review, then it was cost effective and worth the investment. Dr May suggested that it would be even more cost effective to place attention on only approximately 20 % of those CMC claims that actually support measurement services rather than a simple capability. Dr Mitani felt that the issues were broader than that because testing laboratories perform the services and NMIs must support them. Dr Squirrell noted that those NMIs that deliver real services are subjected to an additional review process during accreditation because CMCs are again reviewed in detail. There are on-going discussions between the BIPM and ILAC on how to make better use of such accreditation assessments and CMC reviews.

14. MEASUREMENT AND STANDARD NEEDS TO SUPPORT SUSTAINABILITY ASSESSMENT: THE ROLE OF NMIs

Dr Brandi, presenting on behalf of Dr Jornada, spoke about issues of sustainability (the responsible use and conservation of our environment), noting that the quality of a delivered product must include consideration of this parameter. It was argued that the pillars of sustainability include standardization, conformity assessment and metrology and that chemistry was the basis of technologies needed for sustainable development. These included measurements relevant to climate change, management of industrial wastes and air/soil/water pollution monitoring. Within this context, the roles of NMIs include development of methodologies, their validation and CRMs to provide traceability and international harmonization and acceptance. A SWOT analysis on the relationship of NMIs to the issue of sustainability was presented and climate change was identified as the single most important subject area for further examination. This naturally led to a consideration of use of biofuels and a summary of the positive experiences at INMETRO with the BIOREMA project involving IRMM, NIST, NPL and VSL. He then examined aspects of CRM production, and development of techniques for monitoring the impact of carbon release and sequestration in the atmosphere, soils and crops or biomass which, taken together, comprise a cycle of energy and mass (life cycle analysis - LCA).

Dr Brandi then presented several suggestions for moving forward, including the creation of a task force to prepare a report on chemical metrology needs for sustainability and the role of NMIs, which

would be similar in format and impact to the report issued earlier by Dr Kaarls; presentation of the report at the next Directors meeting with discussions on the implementation of the recommendations and finally, articulation to and engagement of representative international fora involved in the subject, such as the International Biofuels Organization, ISO, and others. He noted that a committee on sustainable bio-energy would meet in Rio de Janeiro in May 2011.

Dr Milton stated that standards for LCA would involve ISO but questioned whether there was a role for NMIs to play, to which Dr Brandi replied that there is a need for good data. Calculations are now based on measurement data from 1994 inventories and thus use average parameters, yielding possibly erroneous results for climate change. Also, most LCAs use no uncertainties. Dr May agreed on the attendant advantages offered by a metrological programme in LCA but questioned which community or government organizations are asking for this information. Dr Brandi replied that the US EPA is developing new LCA models. Dr May suggested that NIST may support EPA in the future when asked, but on a global scale is there an intergovernmental body that has that responsibility. Dr Sommer noted that there were no governmental bodies within the EU acting on this, but only private agencies which are interested in learning from NMIs how to include uncertainties in model calculations. Dr Brandi added that there was a need to move ahead with LCAs based on a solid inventory with which to demonstrate the utility of an LCA approach. Dr Emons added that there is a clear structure within the EU for identified bodies responsible for risk assessment and risk management and from a research viewpoint the JRC is modifying its priorities for the next 10 years to look at a low carbon society which will demand more resources to coordinate diversified activities. Prof. Kühne expressed the notion that LCA is important but questioned whether NMIs are the most suitable resource for this, agreeing that they may have an important role on input into measurement questions but are they really able to take a leading role. Dr May expressed the reservation that NMIs may not be able to provide input on how to undertake modelling and statistically evaluate data and also wondered whether experiments and actual measurements need to be designed and run by NMIs to test some of the current assumptions. Although NMIs can do such things, who would authorize them to do it on their behalf? Dr Brandi replied that LCAs do not need to be undertaken by the NMIs, but the latter can provide good data and methods for pollution measurements. Dr Kaarls stated that such ideas could be worked into the recommendations which could be integrated with the WMO issues discussed earlier. Prof. Wallard remarked that the little contact that he has had with various governments suggests that great difficulty lies ahead on any progress with trying to engage in any such measurement aspects because it has been made using only measurements of inputs and it stalls with the modelling relating input to output. Although the regulatory agencies appear to be persuaded that something can be done, he is doubtful of investing significant effort into such projects when the real question is what comes out of it.

Dr Kaarls thanked Prof. Brandi for his interesting presentation. He then proposed a change in the agenda with a presentation by Dr Wielgosz on the BIPM programme on metrology in chemistry since the CCQM had been asked to express an opinion on this subject.

15. BIPM PROGRAMME ON METROLOGY IN CHEMISTRY (CURRENT AND FUTURE)

Dr Wielgosz presented an overview of the BIPM chemistry programme (document CCQM/10-12, 26 March 2010) covering the period 2009–2012, followed by proposals for the period 2013-2016. He highlighted current dedicated measurement capabilities and infrastructure coordination of comparisons in gas metrology, organic analyses and for the engagement of new stakeholder communities, such as those related to the JCTLM and biotechnology. In the gas metrology field, this focused on ozone, methane and nitrogen dioxide, whereas primary calibrator comparisons for purity assessment comprised the organic analysis field and support of CRMs for laboratory medicine was offered through the JCTLM database. For 2013-2016, the BIPM Metrology in Chemistry Programme would develop along four major themes, which Dr Wielgosz proceeded to map out.

(i) international equivalence of gas standards for air quality and global climate change monitoring for which the BIPM will coordinate comparisons of surface ozone, nitrogen oxides, formaldehyde and support the development of reference methods for key greenhouse gases and air quality. This will enable national air quality monitoring networks and pollution control strategies for high priority pollutants to be based on accurate internationally recognized standards and to provide measurements that are fit for assessing air quality and monitoring the effects of control measures. The activities will also ensure the stability and reliability of measurements for the long term monitoring of greenhouse gases and their use in radiative and climate change models, and for monitoring the effectiveness of mitigation activities. The activities will facilitate the establishment of WMO-GAW Central Calibration Laboratories for VOCs and NOxy for the global monitoring of these species.

(ii) international equivalence of organic primary calibrators for clinical chemistry and laboratory medicine, food analysis, environmental analysis, forensics and pharma. In this arena, the BIPM will coordinate three key comparisons demonstrating the capabilities of NMIs to deliver the primary calibration reference services required to underpin their provision of SI-traceable measurements in organic analysis, and support NMI calibration and measurement capability claims to provide “small organic molecule” primary calibrants (MW < 500), both as pure substances and calibration solutions. The activity will facilitate the demonstration of equivalence of national capabilities for the value assignment of primary calibrators/calibration solutions in support of reference measurement systems for healthcare, food, environmental analysis, pharmaceuticals and forensics.

(iii) international equivalence of large molecule standards for diagnostics and therapeutics, which will extend the BIPM’s Organic analysis facility to enable the characterization and comparisons of high molecular weight organic molecule purity determinations, notably for peptides and small proteins in order to support the development of reference measurement systems for these entities and improvements in the quality assurance of diagnostic measurements and therapeutic products such as insulin, insulin like growth factor (IGF-1), parathyroid hormone (PTH), Human growth hormone (hGh) and transferrin. The activity will enable the development and use of higher order reference materials, methods/procedures and services by NMIs both for large molecules and their use by the IVD industry, leading to accurate diagnostic systems, reduced costs from retesting and improved patient care. It will promote the development of reference measurement systems for therapeutics, for large molecule analytes where physicochemical characterization is required and value assignment of properties in SI units is envisioned, allowing improved accuracy in therapeutic product manufacture.

(iv) support of CCQM and JCTLM and international liaison activities for metrology in chemistry, which are linked to the BIPM's role in establishing and supporting international metrology projects and liaisons with other international organizations which benefit from an international infrastructure for chemical metrology. This ensures awareness of the metrology infrastructure available at the international and national level, promotes the activities under the Metre Convention, and facilitates the establishment of activities at the national level.

A final brief summary of where the BIPM metrology in chemistry programme was in 2005 and where it plans to be in 2015 was presented.

Dr Kaarls noted that the CCQM has been asked to express an opinion on this future work programme and to make it available to the CIPM in time for their scheduled meeting in October 2010, during which they will make a decision to send a recommendation to the CGPM with respect to the total work programme of the BIPM. The CGPM, in its meeting in October 2011, will decide on the BIPM's budget for the 2013-2016 period. In this regard, pending discussions, Dr Sargent, the *Rapporteur* for the CCQM *ad hoc* Advisory Group (consisting of the chairs of the CCQM WGs and chairs of the RMO technical committees on metrology in chemistry as well as several additional global members) convened by the President to discuss in depth this programme proposal, was invited to present the recommendations of their study (CCQM/10-23).

16. REPORT FROM THE CCQM *AD-HOC* ADVISORY GROUP ON THE BIPM PROGRAMME

Based on the examined draft CCQM/10-12, Dr Sargent noted that the Advisory Group welcomed and supported the BIPM programme with its focus on the international equivalence of standards in support of greenhouse gas and air quality monitoring, primary calibrators for clinical chemistry, pharma, forensics, environmental, and food analyses, organic large molecule calibrators for therapeutics and diagnostics, and noted the high quality and technical competence demonstrated in the delivery of the programmes to date.

The Advisory Group considered the proposals for the 2013-2016 programme in light of several criteria:

(a) the BIPM needs to develop and maintain a high level of scientific expertise in order to deliver its mission;

(b) the BIPM science programme should focus on specific needs of international metrology and demonstrate clear added value to the world-wide measurement community and

(c) activities of the BIPM should occupy an area which addresses high level metrology issues and reflects BIPM's global status, and complements the activities of NMIs.

The 2009-2012 chemistry programme comprises activities on international equivalence of gas standards for air quality and climate change monitoring, organic primary calibrators including method development for large molecules and support for the CCQM together with the JCTLM and other international liaison activities in the field of chemistry. An important aspect of the current work on gas and organic analysis is the development and coordination of international comparisons on behalf of the CCQM participants working in these fields. The BIPM has proposed to continue and

strengthen these activities in the 2013-2016 programme by extending the range of measurands and developing additional facilities which this requires.

The Advisory Group welcomed the BIPM's proposals for its 2013-2016 chemistry programme as a challenging and beneficial extension of its present activities and supports them. The BIPM is requested to update the proposals, taking into account the suggestions of the Advisory Group, including a summary of the wider benefits of the programme for Member States. The full text of the Advisory Group's recommendation (CCQM/10-23) is reproduced in the Appendix Q2.

Prof. Wallard and Prof. Kühne welcomed the Advisory Group's remarks, and agreed that the proposed programme of work relating to large molecule calibrators for therapeutics and diagnostics was a logical extension of the BIPM activities on primary organic calibrators. Dr Emons felt that since the activity was not focussed on addressing the bio-relevance of a molecule but rather its analytical detection and quantitation it was indeed logical to include such large molecules. Dr Emons suggested that concrete examples should not be presented since some molecules of current interest may no longer be so by 2015 and thus there was a need to show flexibility or cuts may occur to the budget. Prof. Kühne replied that the problem was that members want to see details, otherwise they will not support the proposals, leading to a "catch 22" situation. Dr Emons further suggested that the existence of prioritization criteria may provide the needed long-term flexibility. Dr May stated that there was a need to make clear statement of support for the gas metrology and organic measurement needs of NMIs and not just the WGs that were being considered.

Dr Kaarls moved that the CCQM approve the report as presented by Dr Sargent; there were none opposed, the report was approved.

17. JCRB

Dr Mussio presented his report on the Joint Committee of the Regional Metrology Organizations and the BIPM (JCRB, document CCQM/10-41). He briefly summarized actions and resolutions of the 23rd 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.

The following highlights were of note. The CIPM policy for traceability was being implemented by the JCRB. The chair of the JCRB will contact the IAEA to establish a date for re-approval of their QS and to encourage the IAEA to regularly attend RMO quality systems review committees. The JCRB recommends that periodic presentations of the QS of DIs to the corresponding QS review panel of the RMOs must be submitted direct from the DI and not through its NMI. Similarly, the QS annual reports must be prepared and submitted directly by the DI. NMIs must include the status and actions related to greyed out CMCs in their annual reports and those that have been greyed out for more than 5 years will be permanently deleted from the KCDB. The JCRB is moving towards a common position relating to accreditation or self declaration following peer review. It is recommended that accreditation bodies follow guidelines for the selection and dissemination of information on individuals selected as assessors.

Dr May opened the discussion with comments relating to the issue of the responsibilities of DIs. Although agreeing that the DIs should be submitting their own QS, governments have not given them that responsibility. Further, there are situations in which the NMI maintains a close administrative

role on the DI, as in the case of a reactor or large NMR facility. Because the laboratory undertakes some measurements on behalf of the NMI, it essentially operates under the QS of the NMI. He asked if any consideration had been given to these two different situations. Dr Kaarls noted that it was necessary to realize that the global situation is likely a combination of these extreme situations. Prof. Kühne stated that he was not aware of any case describing the second kind of DI situation outlined by Dr May in that to date, all DIs are legally designated entities with their own QS and responsibility, and as such, they should participate in RMO meetings to profit from the process. Such DIs as described by Dr May are more likely to be subcontractors. Dr May exemplified his position by citing the case of Canon Instruments serving as the DI to service viscosity measurements for NIST customers wherein this service is undertaken within the overall QS of the appropriate NIST division. He noted that as the CIPM MRA does not distinguish between these two situations, who should take the responsibility – the DI or the NMI? Dr Kaarls felt this was an entirely different situation. Prof. Kühne interpreted this situation as being one that was clear; the work was done under the QS of NIST and thus NIST is the responsible entity. Prof. Wallard asked whether Canon represented themselves at the SIM QSTF, to which Dr May replied that they did.

Dr Emons raised the subject of what a review of a QS was supposed to be about, arguing that the JCRB ILAC proposal revealed a lack of harmonization. Technical issues are handled through Key Comparisons and the CMC process whereas the quality management review should not require questioning by the accreditors to examine the qualifications and technical competence of the assessors. On a second note, he turned to the issue of the CIPM traceability document which he felt was severely limiting the work of the CCs since the majority of the measurands can not fulfil the requirements of traceable calibration. Dr Kaarls stated that the WGs and all CCs have been asked to provide suggestions for exceptions that will be considered but that the basis of the traceability document itself stands as it is, with the exception of agreed upon exceptions which will be addressed during the autumn meetings. Prof. Kühne explained that the origin of the request for assessor qualifications stemmed from an incident some 2 years ago between a European NMI and its accreditation body wherein the question arose as to whether it was possible for ILAC to make better use of information collected by the accreditation body to enhance confidence amongst all NMIs of their accredited capabilities. Mr Squirrell noted that NMIs selecting accreditation as their option feel that they should accrue some benefit in having met the requirements of the CIPM MRA. He noted that where it states quality system in the text of the CIPM MRA, it always includes technical and management systems. Accreditation implies both, and must include technical aspects otherwise ISO/IEC 9000 would be sufficient. Thus, if the NMI wishes to be accredited, the ILAC document is suggesting that benefits will accrue and progress is being made in this respect such that in the future a more harmonized approach will be evident.

18. UPDATE ON THE BIPM KCDB

Dr Thomas presented an update on the current status of the KCDB, noting that new software was installed on 1 January 2009 and that during the past year there were 90 000 visits to the BIPM website, opening 821 000 pages and constituting 5 600 – 10 000 monthly visits. Nearly all pages were equally visited, including CMCs, and only 25 % of such visits were accessed through NMI website links. She noted that the BIPM encourages NMI links to its website.

As of 1 March 2010, a new look to the website had been implemented with restructuring of the "related links" now offered from all of the KCDB website pages, as it was previously underutilized. A single unique "box" on all KCDB pages now provides links to the CIPM MRA page, the JCRB page, the KCDB statistics page, a new page entitled "KCDB FAQ" (with answers to 10 such questions), "Find my NMI" and *Metrologia*. Dr Thomas gave a live demonstration of the website to illustrate the options and flexibility of the new format and the power of the related links, along with general navigation features throughout the KCDB website. Comments were invited regarding the need to add any additional features that may be of use.

Dr Milton expressed his thanks to Dr Thomas for her work in simplifying the retrieval of information from the KCDB. Dr Sargent commented that, at present, it is necessary to access all of the CMCs or all of the KCs from a single institute in a sequential fashion and asked if in the future it might be possible to select such information for multiple NMIs and retrieve the information in one printout. Dr Thomas agreed that access to CMCs must be simplified and this aspect will be addressed in the future as the work is being contracted out.

Prof. Wallard announced that Dr Mussio's secondment to the BIPM will expire on 1 March 2011 and invited proposals from colleagues for a 2 year secondment as JCRB secretary; the announcement can be found on the BIPM home page. Dr Kaarls also expressed his on-going thanks on behalf of everyone to Dr Mussio for his work in making all the rules/procedures/documents much more accessible.

19. AUTHORSHIP OF REPORTS AND PUBLICATIONS OF RESULTS OF CC COMPARISONS

Dr Kaarls stated that there was nothing significant to report at the moment other than the JCRB was preparing a guidance document relating to authorship because a wide variety of approaches to this are being used, as is evident from an examination of reports in the KCDB. The document is expected to be available later in 2010 for consideration by the CIPM.

Dr Wielgosz noted, for the record, that a previous decision by the CCQM that all participants' names should be on the reports for all comparisons has evidently not been followed in practice. Dr May clarified this by stating that he believed this earlier decision related to the reports of KCs and not pilot studies, to which Dr Wielgosz reiterated that even in this case the agreed upon protocol is not being followed.

20. COMMENTS ON WRITTEN REPORTS OF RMO ACTIVITIES

Dr Kaarls noted that in 2010 the RMOs and other international organizations that liaise with the CCQM were requested to send in written reports that could be read in advance and obviate the need for a long series of short presentations. He opened the floor for questions.

Dr Charlet asked if there were plans to set up a bio-working group within the RMOs to which Dr Güttler replied that there was little need for such a separate WG in Euramet. Dr Sin noted that this subject will be discussed by APMP during the meeting in Thailand. Dr Kaarls believed there was no

interest in this expressed by COOMET or AFRIMETS and, in general, the concept did not make much sense. He concluded by imploring all RMO chairs to make notes on plans for future comparisons and to communicate and share such plans in advance in order to maximize their use / impact.

21. THE JOINT COMMITTEE ON TRACEABILITY IN LABORATORY MEDICINE (JCTLM)

Dr Wielgosz presented a brief summary of the current status of the JCTLM database. He drew attention to the updates made in January 2009 and 2010 to note that there were now 227 CRMs, 146 reference measurement procedures and 135 reference measurement services listed. There are currently some 1 000 hits per month on the database. Some of the changes recently implemented were highlighted, including revision to ISO/IEC 15194:2009 which allows for harmonization of the standard for reference materials in the diagnostic area of the ISO REMCO guide. This necessitated a change to the nomination procedure, which has been completed. A new version of the review process will be in effect for February 2010.

Two reference materials have been de-listed from the database either because the original was out of stock or was being replaced by new material. He then moved on to discuss the Global Harmonization Task Force, which developed a harmonization document on health and safety of IVDs and the JCTLM has submitted its comments on traceability.

Dr Kaarls noted that normally Dr Siekmann would be present, but he was unfortunately unable to attend. There was no discussion of this report by the CCQM participants.

22. COMMENTS ON WRITTEN REPORTS FROM INTERNATIONAL ORGANIZATIONS IN LIAISON WITH THE CCQM

Dr Kaarls asked if the chairs or representatives of the organizations had anything to add to the reports from International Organizations in liaison with the CCQM to bring them forward. Prof. De Bièvre reminded everyone that 2011 is designated the International Year of Chemistry, as agreed upon by UNESCO. He also took the opportunity to announce that Dr Dybkaer, a long-time colleague and CCQM attendee, was presented with a doctorate in Copenhagen a few years earlier for his work on terminology which will now be placed in its totality on the IUPAC website.

23. CCQM WORKSHOPS (MICRO-BIOLOGY)

The President announced that a workshop on microbiology would be organized during the first half of December 2010 at the BIPM; the programme would be formulated during April / May to address

the needs and possibilities for this measurement area and noted that he would be in contact with individuals for input.

No further suggestions for other workshops were raised.

24. CCQM RESOLUTIONS

Dr Milton presented a draft resolution on monitoring the global climate and collaboration with the WMO, as outlined in an Appendix below. Prof. De Bièvre requested that terminology be consistent with that recommended in the VIM (3rd Edition). Dr Kaarls noted that this would be considered by the BIPM but cautioned on being too "pure" as there is a need to maintain a clear understanding with the rest of the world. In the absence of additional comments, the President concluded that the CCQM agreed with the recommendation and that there would be a reference placed in the document by Dr Milton (as a footnote) that a full report was available.

25. ANY OTHER BUSINESS

Prof. Wallard noted that 20 May 2010 is designated World Metrology Day and a poster could be downloaded from www.worldmetologyday.org for subsequent use. In 2011, during the International Year of Chemistry, World Metrology Day 2011 will have a special significance with emphasis on chemistry. Activities relating to its celebration will fall to the purview of his successor, Prof. Kühne. He noted that when he became Director of the BIPM, one of his personal priorities was to expand the BIPM's activities in chemistry and that could not have happened without the support of the CCQM. He expressed his thanks for this support. Dr Kaarls thanked Prof. Wallard for his personal support of the CCQM. Prof. Kühne assured the CCQM that during his tenure as Director, starting 2011, chemistry will continue to receive the recognition that it deserves and in the tradition that Prof. Wallard had started.

26. DATE OF NEXT MEETING

The next meeting of the CCQM was fixed for 14-16 April 2011 at the BIPM with the preceding days reserved for meetings of the CCQM WGs and a workshop.

26.1 COORDINATION OF CCQM WG MEETINGS

Dr Kaarls noted that the fall meetings for the IAWG and the EAWG would take place in Boras, Sweden, while those for the BAWG, GAWG and OAWG would occur in Singapore.

27. CLOSURE

The President closed the meeting at 15:15, thanking everyone for their reports, feedback, active participation and suggestions which help make for more effective support for our customers. He thanked the staff of the BIPM for their support and expressed best wishes for safe travel to all participants.

R.E. Sturgeon, *rapporteur* 07/06/10

revised 16/09/10

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 EVENTUELLES REDEFINITIONS 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

- 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.

RECOMMANDATION DU COMITÉ CONSULTATIF POUR LA QUANTITÉ DE MATIÈRE – MÉTROLOGIE EN CHIMIE PRÉSENTÉE AU COMITÉ INTERNATIONAL DES POIDS ET MESURES

RECOMMANDATION Q 1 (2010) : sur la surveillance du climat mondial et la collaboration avec l'Organisation météorologique mondiale

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

considérant

la signature récente du CIPM MRA par l'Organisation météorologique mondiale (OMM),

les recommandations et conclusions[1] de l'atelier commun à l'OMM et au BIPM, intitulé « Measurement Challenges for Global Observation Systems for Climate Change Monitoring: Traceability, Stability and Uncertainty »,

notant

que la comparabilité des résultats provenant de différentes stations de réseaux mondiaux d'observation, dont le programme Integrated Global Atmospheric Chemistry Observation Strategy est un exemple, a un impact scientifique considérable,

[1] L'atelier « Measurement Challenges for Global Observation Systems for Climate Change Monitoring: Traceability, Stability and Uncertainty », organisé conjointement par l'OMM et le BIPM, s'est tenu au siège de l'OMM à Genève, du 30 mars au 1er avril 2010. Le rapport de cet atelier est disponible sur le site Web du BIPM.

que la stabilité à long terme et la reproductibilité des matériaux de référence traçables au SI, ainsi que les échelles d'étalonnage explicitement définies, sont essentielles à l'étude du changement climatique,

que les données mondiales concernant les gaz à effet de serre pourraient faire l'objet d'un examen minutieux lors de la préparation du cinquième Rapport d'évaluation du Groupe d'experts intergouvernemental sur l'évolution du climat,

qu'il est fondamental que les résultats de mesures concernant la durabilité environnementale soient de grande qualité,

recommande

que le CIPM prenne les mesures nécessaires pour porter les recommandations et les conclusions de l'atelier à l'attention des laboratoires nationaux de métrologie,

que le CIPM et le BIPM réfléchissent au moyen le plus efficace pour mettre en œuvre les recommandations de l'atelier, en collaboration avec la communauté des météorologistes, afin d'utiliser au mieux les infrastructures nationales et internationales existantes pour répondre aux besoins en étalons pour la surveillance du changement climatique dans le monde,

que le CIPM évalue les besoins en matière de traçabilité métrologique des mesures effectuées dans le cadre d'études concernant la durabilité environnementale.

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

RECOMMENDATION Q 1 (2010): ON MONITORING THE GLOBAL CLIMATE AND COLLABORATION WITH THE WMO

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

considering

the signature of the CIPM MRA by the WMO,

the recommendations and findings[2] of the workshop held between the WMO and the BIPM on "Measurement Challenges for Global Observation Systems for Climate Change Monitoring: Traceability, Stability and Uncertainty",

noting

[2] The WMO-BIPM Workshop on 'Measurement Challenges for Global Observation Systems for Climate Change Monitoring: Traceability, Stability and Uncertainty' was held at the WMO Headquarters, Geneva on 30 March - 1 April 2010. The report of the workshop is available on the BIPM website.

that enormous scientific value arises from the comparability of data from different stations in global monitoring networks as exemplified by the Integrated Global Atmospheric Chemistry Observation Strategy,

that the long-term stability and reproducibility of SI traceable reference materials, and explicitly defined calibration scales, are critical to the study of climate change,

that the global records of GHGs may come under great scrutiny during the preparation of the 5th Assessment Report of the IPCC,

the importance of high quality data in measurements of environmental sustainability,

recommends that

the CIPM takes steps to bring the recommendations and findings of the workshop to the attention of the NMIs,

the CIPM and the BIPM consider the most effective mechanism to take the recommendations forward in collaboration with the WMO community in order to make best use of established national and international infrastructure to meet the requirements for standards for monitoring global climate change,

the CIPM considers the requirements for metrological traceability of data used in studies of environmental sustainability.

APPENDIX Q1.
WORKING DOCUMENTS SUBMITTED TO THE CCQM AT ITS 16TH MEETING

Working documents submitted to the CCQM at its 16th meeting are on restricted access. The text of document CCQM/10-23 is reproduced in full below.

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

Document CCQM/	
10-01	Draft agenda for the 2010 CCQM meeting, 1 pp.
10-02	Draft Timetable of CCQM meetings, 1 pp.
10-03	Draft CCQM guidance note: Estimation of a consensus KCRV and associated degrees of Equivalence (Version 6), 33 pp.
10-04	CITAC report to CCQM 2010, I. Kuselman, 13 pp.
10-05	Report on the BIPM Workshop on Physiological Quantities and SI units (CCU/10-8), 5 pp
10-06	EURAMET METCHEM Report, B. Güttler, 2 pp.
10-07	Report of recent activities of the SIM Chemical Metrology Working Group, W.E. May, 3 pp.
10-08	MC Traceability Exceptions for QM
10-09	ISO TC 229 Liaison Report, J. Viallon, 3 pp.
10-10	Abstracts from the CCQM Workshop on Metrology for Forensic Science, 18 pp.
10-11	Pharmaceutical Industry Request to Implement a New International Standard for Biosynthetic Insulin, 4 pp.
10-12	BIPM Metrology in Chemistry Programme, Proposed Programme of Work (2013-2016), R.I. Wielgosz, 26 pp.
10-13	Review of Roadmaps and Strategies for Bio-measurement (Deliverable P2-D1 of BIPM Commissioned Study), 34 pp.
10-14	COOMET report to CCQM, 5 pp.
10-15	Codex CCMAS report to CCQM, R. Josephs, 1 pp.
10-16	IAM report to CCQM, R. Josephs, 2 pp.
10-17	AFRIMETS report to CCQM, 3 pp.
10-18	ISO/REMCO report to CCQM, H. Emons, 2 pp.
10-19	ILAC update for CCQM, A. Squirrell, 5 pp.
10-20	Mass metrology and the new SI kilogram, R. Davis, 32 pp.

10-21 APMP report to CCQM, D. Wai Mei Sin, 18 pp.

10-22 ICTNS IUPAC resolution on a new definition of the Mole, 2 pp.

10-23 CCQM Advisory Group's Opinion on the BIPM Chemistry Programme 2013-2016, M. Sargent, 3 pp.

10-24 CCQM Recommendation Q1 (2010) - On monitoring the global climate and collaboration with the WMO, BIPM, 1 pp.

10-25 Amount of Substance and the proposed redefinition of the mole, M. Milton, 7 pp.

10-26 Report from the CCQM ad hoc WG on the KCRV, Maurice Cox, 7 pp.

10-27 Status of the molar measurements at PTB, Detlef Schiel, 32 pp.

10-28 Back to Basics (BtB) with the 2008 VIM, Prof. Dr Paul de Bièvre, 27 pp.

10-29 Amount of substance and the mole, Report to the CCQM, Martin Milton, 18 pp.

10-30 CCQM Forensics Workshop Outcomes, Robert I. Wielgosz, 1 pp.

10-31 Summary of the WMO-BIPM Workshop 30 March- 01 April 2010, Robert I. Wielgosz, 49 pp.

10-32 Summary of the BIPM Workshop on Metrology at the Nanoscale, Joële Viallon, 11 pp.

10-33 Report of the Organic Analysis Working Group to CCQM, Willie E. May, 50 pp.

10-34 Report of the Inorganic Analysis Working Group to CCQM, Mike Sargent, 60 pp.

10-35 Report of the Gas Analysis Working Group to CCQM, Martin Milton, 29 pp.

10-36 Report of the Electrochemical Analysis Working Group to CCQM, Michal Máriássy, 35 pp.

10-37 Report of the Surface Analysis Working Group to CCQM, Wolfgang Unger, 28 pp.

10-38 Report of the Bioanalysis Working Group to CCQM, Helen Parkes

10-39 Report of the Key Comparison and CMC Quality Working group to CCQM, Lindsey Mackay, 23 pp.

10-40 Measurement and Standards Needs to support Sustainability Assessment: the Role of NMIs, João Jordana, 30 pp.

10-41 JCRB, Luis Mussio

10-42 Update on the BIPM KCDB, Claudine Thomas, 3 pp.

10-43 BIPM Chemistry Department Programme Proposals (2013-2016), Robert I. Wielgosz, 64 pp.

10-44 JCTLM Database, Robert I. Wielgosz, 5 pp.

10-45 2009-05-12 ACQUAL Simple principles for metrology in chemistry identifying and counting, Gary Price-Paul de Bièvre, 11 pp.

10-46 BIPM Chemistry Department (2013-2016) Programme Proposals Version 0.4, Robert I. Wielgosz, 27 pp.

APPENDIX Q2.

CCQM/10-23

BIPM Metrology in Chemistry Programme

Project Proposals (2013-2016)

Opinion of the CCQM Advisory Group

Background

The Advisory Group members were invited to comment on Version 0.3 of the BIPM proposals, dated 26 March 2010 (CCQM/10-12). Their comments were discussed at a meeting held at the BIPM on 12 April 2010, following a presentation by the BIPM setting out the main features of its proposals and the benefits of the planned activities. The BIPM also gave an overview of progress with delivery of the 2009-2012 chemistry programme.

The Advisory Group welcomed and supported the BIPM programme with its focus on the international equivalence of standards in support of greenhouse gas and air quality monitoring, primary calibrators for clinical chemistry, pharma, forensics, environmental, and food analyses, organic large molecule calibrators for therapeutics and diagnostics, and noted the high quality and technical competence demonstrated in the delivery of the programmes to date.

The Advisory Group considered the proposals for the 2013-2016 programme having regard to the following criteria:

- The BIPM needs to develop and maintain a high level of scientific expertise in order to deliver its mission.
- The BIPM science programme should focus on specific needs of international metrology and demonstrate clear added value to the world-wide measurement community.
- Activities of the BIPM should occupy an area which addresses high level metrology issues and reflects BIPM's global status, and complements the activities of NMIs.

The 2009-2012 chemistry programme comprises activities on international equivalence of gas standards for air quality and climate change monitoring, organic primary calibrators including method development for large molecules and support for the CCQM together with the JCTLM and other international liaison activities in the field of chemistry. An important aspect of the current work on gas and organic analysis is the development and coordination of international comparisons on behalf of the CCQM participants working in these fields. The BIPM has proposed to continue and strengthen these activities in the 2013-2016 programme by extending the range of measurands and developing additional facilities which this requires.

The current level of resource (for the 2009-2012 programme) is 5.5 scientists, 3.5 technicians, Research Fellows (4 man-years), and 1 man-year from NMI scientists on secondment.

Gas Standards for Air Quality and Climate Change Monitoring

The proposed programme was strongly supported and considered to be an important extension of the current activities, which are well-regarded internationally. They are linked to the strategy developed by the CCQM Gas Analysis Working Group (GAWG) and the collaboration between the BIPM and the WMO. The projects address some of the scientific issues encountered in linking between the capabilities of the NMIs, largely based on synthetic standards, and the capabilities of the WMO and its reference laboratories which largely depend on "whole air" standards.

All projects demonstrated that they underpinned measurement needs for key global issues such as monitoring greenhouse gases and compounds identified as essential for monitoring air quality around the world. It was noted that the strong scientific focus on coordinating key comparisons and pilot studies by the BIPM projects brings substantial value to participating NMIs. The Advisory Group noted that some tasks identified for 2013-2016 may require revision following the developing collaboration with the WMO after the recent WMO-BIPM Workshop in Geneva. The BIPM will identify tasks with the consultation and co-operation of the CCQM-GAWG at the appropriate time, particularly the evolving priorities for comparisons of greenhouse gases.

Organic Primary Calibrants

The programme was fully supported, noting that it placed the main emphasis on organic primary calibrator comparisons identified by the CCQM Organic Analysis Working Group (OAWG) in support of NMI measurement capabilities in key sectors, notably clinical, environmental, food analyses, forensics and pharma. This work entails a significant resource for the critical evaluation of methods, and the dissemination of this information to NMIs working in the field. The BIPM was also requested to continue to work closely with participants in the CCQM to facilitate the availability of the model compounds as calibrants for NMIs, and thereby enhance the value derived from the comparison studies it coordinated, noting however that the major benefit of comparisons was to underpin NMI measurement capabilities.

Large Molecule Standards

This area of the 2013-2016 programme was discussed at some length by the Advisory Group. The Group agreed that the work proposed is a logical and feasible extension of the BIPM activities on primary organic calibrators which includes feasibility studies on peptide (angiotensin) and insulin calibrator material purity characterization, and noted the requirement for SI traceability for insulin standards as expressed by the Pharmaceutical Industry. The BIPM will continue to work with the CCQM Working groups in prioritizing materials for comparisons and executing the programme of method development and comparisons.

Support of CCQM and JCTLM and International Liaison Activities

The support for international liaisons was accepted as an important task to be continued.

Additional Resources to deliver 2013-2016 Programme

The additional resources required to deliver the 2013-2016 programme are: 1 Scientist and 1.5 technicians. The BIPM's mass spectrometric and gas analysis instrumentation and handling facilities will be expanded to deliver the programme.

Recommendation

The Advisory Group welcomes the BIPM's proposals for its 2013-2016 chemistry programme as a challenging and beneficial extension of its present activities and supports them. The BIPM is requested to update the proposals taking into account the suggestions of the Advisory Group, including a summary of the wider benefits of the programme for Member States.

Mike Sargent

Rapporteur, CCQM Advisory Group

14 April 2010