

Bureau International des Poids et Mesures

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

Report of the 18th meeting  
(19-20 April 2012)  
to the International Committee for Weights and Measures



Comité international des poids et mesures

Note:

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

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

M. Kühne,  
Director BIPM

**LIST OF MEMBERS OF THE  
CONSULTATIVE COMMITTEE FOR  
AMOUNT OF SUBSTANCE:  
METROLOGY IN CHEMISTRY  
AS OF 19 APRIL 2012**

**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. Mendeleev Institute for Metrology, Rosstandart [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].

Istituto Nazionale di Ricerca Metrologica [INRIM], Turin.

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 Ltd], Teddington.

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

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

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

State Laboratory [SL], Co. Kildare.

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/Główny Urząd 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

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 eighteenth meeting at the International Bureau of Weights and Measures (BIPM), at Sèvres on 19-20 April 2012.

The following were present: H. Andres (METAS), G. Carroll (SL), V.S. Da Cunha (INMETRO), B.-J. de Vos (NMISA), S. Ellison (LGC Ltd), H. Emons (IRMM, ISO REMCO), A. Fajgelj (IAEA, IUPAC), P. Fisicaro (LNE), T. Fujimoto (NMIJ/AIST), J.A. Guardado-Pérez (CENAM), B. Güttler (PTB), H.D. Jensen (DFM), R. Kaarls (President of the CCQM), K. Kato (NMIJ/AIST), M. Kühne (BIPM Director), Y. Kustikov (VNIIM), H. Li (NIM), L. Mackay (NMIA), B. Magnusson (SP), M. Máriássy (SMU), J. Marques Rodrigues Caxeiro (INMETRO), W.E. May (NIST), Z. Mester (NRC), M.J.T. Milton (NPL), S.-R. Park (KRISS), H. Parkes (LGC Ltd), M. Sargent (LGC Ltd), M.P. Sassi (INRIM), M. Sega (INRIM), H.-Y. So (KRISS), R. Sturgeon (NRC), W. Unger (BAM), A. van der Veen (VSL), S. Vaslin-Reimann (LNE), J. Vogl (BAM), S. Wise (NIST MML), Z. Zeyi (NIM).

Observers: F. Dias (IPQ), A.C. Gören (UME), P.K. Gupta (NPLI), W. Kozłowski (GUM), I. Kuselman (INPL), Z. N. Szilágyi (MKEH), A. Zoń (GUM).

Invited: M. Buzoianu (INM), J. Kang'iri Njeri (KEBS), G. Muriira Karau (KEBS), T.K. Lee (HSA), G. Massiff (Fundacion Chile), R. Parris (NIST), S. Sandoval (ISPCH), D. Wai Mei Sin (GLHK), A. Squirrell (ILAC), I. Triviño Angulo (ISPCH), A. Villa (Fundacion Chile, INTA, Universidad de Chile), O. Zakaria (NML-SIRIM).

Also present: A. Daireaux (BIPM), E. Flores Jardines (BIPM), R. Josephs (BIPM), S. Maniguet (BIPM), P. Moussay, (BIPM), N. Stoppacher (BIPM), C. Thomas (BIPM), J. Viallon (BIPM), S. Westwood (BIPM), R. Wielgosz (Executive Secretary of the CCQM, BIPM).

Sent regrets: A. Brewin (NPL), M. Cox (NPL), M. Fernández Vicente (CEM), M. Hennecke (BAM), L. Locascio (NIST), L. Siekmann (IFCC), U. Panne (BAM), A. Rosso (INTI).

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\* For the list of acronyms, [click here](#).

Dr R. Kaarls, the President of the CCQM, officially opened the 18th meeting of the CCQM on the morning of 14 April 2012, noting that a CCQM workshop on “The Re-definition of the Mole – A new era for chemical metrology” had concluded successfully the previous day. He passed the floor to Prof. M. Kühne, Director of the BIPM, who extended a general welcome to all participants and observers. Prof. M. Kühne highlighted the importance of metrology in chemistry and the successful operation of these activities at the BIPM. Notwithstanding, however, was the desire to address new challenges, including the redefinition of the mole. Following some brief housekeeping announcements, a round table self-introduction by all participants and observers was completed.

## **2. APPOINTMENT OF A RAPPORTEUR**

Dr R. Kaarls proposed Dr R. Sturgeon as rapporteur for the meeting; Dr R. Sturgeon agreed.

## **3. APPROVAL OF THE AGENDA**

Dr R. Kaarls noted that two additional items were to be added to the proposed agenda; 10.4 (Dr R. Kaarls to discuss strategic planning requirements arising from the 24th meeting of the General Conference on Weights and Measures (CGPM)) and 11.8 (Dr S. Ellison to discuss calculation models for the key comparison reference value (KCRV)). The edited agenda was approved.

## **4. REPORT ON THE SEVENTEENTH MEETING OF THE CCQM**

No verbal comments were raised with respect to the report of the 17th meeting of the CCQM, which Dr R. Kaarls subsequently declared approved; Dr R. Sturgeon was thanked for his efforts in preparing this report.

## 5. SUMMARY OF THE CCQM WORKSHOP ON THE MOLE

The President summarized the technical presentations that were delivered the previous day and progress made towards a redefinition of the mole. He passed the chair to Dr M. Milton (NPL) to report on progress made towards a *mise en pratique*.

## 6. REPORT FROM AD HOC WORKING GROUP ON THE MOLE AND DISCUSSION ON THE REDEFINITION OF THE MOLE AND DRAFT *MISE EN PRATIQUE*

Dr M. Milton (NPL) summarized the mandate of this *ad hoc* Working Group (WG), emphasizing that its role was to coordinate activities, and not to redraft the proposed definition. The proposed definition appears in the draft of the 9th edition of the SI Guide. He described the work of the *ad hoc* WG, which is to focus on providing examples of the practical realization of the mole and to address the issue of public consultation on the proposed changes. As the proposed definition specifies a fixed number of entities, practical realizations can be achieved using primary methods of measurement, as summarized in document CCQM/12-14. Therein, three specific examples are given: one makes the link to weighing substances of known purity; the second uses the equation of state for an ideal gas; and the third makes the link to the measurement of charge and the Faraday constant.

The *ad hoc* WG has reviewed what is known about public consultation on the proposed definition. It was noted that major international organizations, including the Royal Society of Chemistry, and eminent scholars in the UK and the USA, had been consulted. It was further noted that some alternative scientific and technical arguments had been promoted and had been responded to where feasible. Notwithstanding these considerations, two documents (available on the BIPM website as CCQM/12-16 and CCQM/12-17) were tabled to illustrate some counter proposals.

The CCQM workshop held on 18 April 2012 highlighted a desire for a more extensive list of “frequently asked questions” of more direct interest to chemists (all suggestions should be sent to Dr M. Milton or Prof. I. Mills). Also recommended was the preparation of an easy to understand paper on the “redefinition and realization of the mole”, to help CCQM representatives carry out consultations and communicate the proposed change and its impact to user communities. Dr M. Milton was asked to draft the paper. Draft document CCQM/12-14 will be posted for open access on the BIPM website.

Dr R. Kaarls reiterated that there had been thorough discussion during the workshop held on 18 April 2012 and that the International Union of Pure and Applied Chemistry (IUPAC) had reconfirmed their support for the new definition. Dr H. Andres (METAS) noted that talks and publicity had not resulted in any new issues being raised by the community, which was taken to indicate the presence of a non-responsive “silent majority”. This was most likely because there will be no impact on the accuracy and uncertainty of practical measurements from the new definition of the mole.

Dr R. Wielgosz (BIPM) reminded delegates that one further action had been agreed as a consequence of the workshop; this was that IUPAC had proposed a paper to be published in one of IUPAC’s journals,

such as *Chemistry International*, highlighting efforts to determine the Avogadro Constant. Dr M. Máriássy (SMU) added that it had been proposed during the workshop that an “article” in *Wikipedia* would be posted and that Dr M. Milton had agreed to investigate this possibility. Dr R. Wielgosz pointed out that only one year remained to influence the wider community on this issue and therefore it would be beneficial to have a short document explaining the proposals for distribution in the near future. Dr B. Güttler (PTB) reiterated that such a scientific paper would be helpful but it must be a joint communication from all of the laboratories participating in the International Avogadro Coordination (IAC) project and that had made isotope ratio measurements (i.e. PTB, NIST, NRC, NIM, IRMM) for publication in *Chemistry International*. Dr M. Milton noted that he was not in a position to undertake this work but that instead it should be coordinated among these laboratories. Ms B.-J. de Vos (NMISA) was concerned that the definition should be teachable with such a publication aimed more towards the public to provide some understandable examples. Dr Z. Mester (NRC) noted that one of the largest divisions of IUPAC is chemical education and should thus be approached. Prof. M. Kühne (BIPM) remarked that the limited resources of the BIPM meant that additional support from National Metrology Institutes (NMIs) was needed to publicize the proposed changes, to which Dr Z. Mester replied that a large number of supportive scientific unions exist that could be enlisted for such actions.

Dr A. Fajgelj (IAEA, IUPAC) expressed his satisfaction with the workshop, noting that it clearly revealed that the community is in a position to move forward with information to the outside agencies but that there is a need to identify which ones these should be. Dr R. Kaarls felt that this topic had been adequately discussed and was supported by Dr N. Moreau (IUPAC). Dr R. Wielgosz expressed the opinion that there is limited public relations capability within the BIPM but, irrespective of this, there is a pressing timeline to move forward with a press release, which could include some practical examples, but we must not lose the sense of urgency that clearly arose from the workshop. Dr B. Güttler cautioned that full agreement on the draft should be assured before any public statement is issued. Dr R. Kaarls noted that the “silent majority” must be assured that full agreement has been reached and asked that the *ad hoc* WG coordinate all communications activities, setting a deadline of eight weeks from now for receipt of all final internal comments on the draft *mise en pratique*, after which the draft would be posted to the open access pages of the BIPM website. Thus, all comments should be sent to Dr M. Milton by the end of May 2012 when the final draft will be released, although it was noted that the document will continue to remain open for additional comments.

## 7. REPORT ON ACTIVITIES OF THE *AD HOC* STEERING GROUP ON MICROBIAL MEASUREMENTS

Dr R. Kaarls announced that the chair of the *ad hoc* steering group on microbial measurements, Dr L. Locascio (NIST), was unable to attend the meeting and present her report. He noted that following the stakeholder workshop “Metrology and the need for traceable microbiological measurements to ensure food quality and safety” which was held at the BIPM headquarters in 2011, more cooperation among stakeholders, including the BIPM, was called for. There was also a call for the formation of two sub-working groups to consider issues relating to quantitation and identification of bacterial strains. Issues of importance included how to establish traceability and measurement uncertainty. It was



concluded that a metrological framework to address comparability of measurement techniques does not yet exist. Ms H. Parkes (LGC) was scheduled to give a more detailed presentation later in the CCQM meeting but in the interests of continuity, the presentation and associated discussion follows here.

Ms H. Parkes summarized the mandate of the *ad hoc* steering group on microbial measurements on behalf of Dr L. Locascio (NIST), including its composition and the role of the CCQM in this effort. The current status of a number of ongoing activities in microbial measurements, including what to measure, how to measure and how to evaluate measurement uncertainty, the availability of reference materials and the role of quantitation were highlighted. The steering group has had three quarterly calls supported by webinar connections facilitated through the NIST and the NMIA. The BIPM provided access to its website files from the April 2011 workshop.

Dr W. May initiated discussions by enquiring as to whether or not the steering group was a separate entity from the CCQM Working Group on Bioanalysis (BAWG). Dr R. Kaarls replied that the steering group was established by the CCQM plenary meeting, reported directly back to the CCQM plenary meeting and could become a full working group. Dr Z. Mester enquired about the nature of the measurements that were envisioned. Ms H. Parkes clarified that two projects were to be established to demonstrate to the stakeholders the value of metrology, one on quantification led by a sub-group and one on identification to be dealt with by another sub-group, but to date, these were undefined. The principle thrust was to tighten up the uncertainty associated with identification and quantification. Dr H. Emons suggested that the mandate of this group lies outside of metrology with respect to the efforts of the identity sub-group. He noted that issues relating to identity are not addressed by the VIM. Dr R. Kaarls clarified that the International Vocabulary of Metrology (VIM) is only a vocabulary and as such it should not be used to determine what the CCQM is doing. Dr H. Emons added that the identity group still needs a measurand to be clearly defined. Dr R. Wielgosz commented that although the steering group was set up in 2011 and identified two tasks, a work plan had not yet been developed; he asked where we would be in another year? Dr R. Kaarls clarified that a time line had indeed been established and a focused report was expected to be delivered within one year. Dr A. Fajgelj noted that the question is whether this issue belongs to the CCQM or not; a reflection of the complexity of chemical measurements. It was considered that it may not belong within the mandate of the CCQM, but if it is to move forward then care is needed to ensure that clearly defined issues are being addressed. Dr R. Kaarls reminded the plenary that following the 2011 workshop there was a desire to move forward collaboratively. Dr Z. Mester asked for clarification on how the WG mandate will be separated from that of the BAWG since the same measurement systems were involved in both. Ms H. Parkes replied that the Association of Analytical Communities (AOAC) and the International Organization for Standardization (ISO) require comparability of the different bio-methodologies to improve harmonization between new and older techniques, because most of the new methods are instrumental. Interaction between the two stakeholder communities representing new and traditional methods is needed and the composition of the group should address this problem. Overall, there will also be a benefit arising from the current BAWG activities and the results may crystallize in the form of a key comparison (KC). Mr A. Squirrell noted that proficiency testing (PT) providers are concerned with both identity and quantitation and this resource could be very useful. Ms H. Parkes replied that the LGC operates PT schemes and that these experts are present in the meetings of the sub-groups on identity and quantification..

## **8. CC DIRECTORY AND MEMBERSHIP OF CCQM WORKING GROUPS**

Dr R. Wielgosz reported that at the end of 2011 it was agreed to adopt a comprehensive system of contacts. This is now publicly available on the BIPM website as a Consultative Committee (CC) Directory and will be upgraded as of April 2012 to contain all WG members. Thereafter, it will be updated annually.

Dr S. Ellison (LGC) expressed concern over the open availability of email addresses. Dr R. Wielgosz noted that for other Consultative Committees and their Working Groups these contacts had been available for the last 12 years in pdf format, to which Dr S. Ellison remarked that permission to hold the data in this format should have been sought. Dr W. May expressed concern that passwords were freely distributed during WG sessions and not all participants in these sessions were members of the WG. Passwords need to be protected because the pilot studies accessible via these passwords are not intended to be public information. Dr R. Wielgosz noted that the current policy was to allow all participants to access documents and perhaps this practice should be reconsidered. Dr R. Kaarls agreed that additional reflection on the issue of passwords is required. Dr R. Wielgosz confirmed that a review of the policy for these two areas would be undertaken.

## **9. SUMMARY OF ACTIVITIES RELATED TO MATERIALS METROLOGY**

### **9.1. Moisture in grain**

Dr R. Kaarls briefly summarized matters relating to the determination of moisture in grain. The issue was tabled in 2010 by the CCQM because a number of NMIs were interested in making Calibration and Measurement Capability (CMC) claims in this area but faced problems because the determination is defined by method not measurand. During the past year the matter was referred to the International Organization of Legal Metrology (OIML) to determine whether global agreement on a method could be achieved such that traceability could be assured. The issue is not straightforward because close cooperation with other stakeholders is required, and this has meant that work at the OIML on a new reference method for moisture in grain has not yet started. Dr M. Sargent (LGC) noted that UNIIM made a technical presentation to the CCQM Working Group on Inorganic Analysis (IAWG) earlier in the week of the current CCQM meeting on technical aspects of the methodology and were advised to consult the OIML. Dr Y. Kustikov (VNIIM) expressed his pleasure that this issue was discussed at the IAWG and hopes that the OIML would be successful and the work will lead to a future comparison of this property by the CCQM.

## 10. APPOINTMENT OF CCQM PRESIDENT AND WORKING GROUP CHAIRS

### 10.1. CCQM President

Dr R. Kaarls formally announced that he would be stepping down as President of the CCQM as of 31 December 2012 after more than 20 years in the role. However, he will continue to serve as Secretary and a member of the CIPM as well as being available to support the new President. In this regard, he was pleased to note that the CIPM had appointed Dr W. May (NIST) to succeed him as President of the CCQM as of 1 January 2013.

### 10.2. CCQM Working Group on Organic Analysis (OAWG) chair

Dr W. May's appointment to President of the CCQM has resulted in a vacancy to fill his former position as chair of the OAWG. Dr R. Kaarls reported that Dr L. Mackay (NMIA) has agreed to assume this task on a provisional basis. In accordance with the rules of the CCQM, a formal proposal to the CCQM was thus made to approve Dr L. Mackay as chair of the OAWG; no objections were raised, and Dr L. Mackay was appointed chair of the OAWG.

### 10.3. CCQM Working Group on Key Comparisons and CMC Quality (KCWG) chair

Dr L. Mackay will be resigning as chair of the KCWG and Dr D. Sin (GLHK) has agreed to assume the provisional appointment as chair of the KCWG. Formal approval of the proposal by the CCQM is pending the decision of the CIPM to modify their rules to permit NMI representatives from Associates of the General Conference to act as CC working group chairs; no objections were raised.

### 10.4. Strategic Planning

Dr R. Kaarls reviewed points of interest arising from the 24th meeting of the CGPM (2011) and Session II of the 100th meeting of the CIPM, which were held in Paris during October 2011. An overview of the current membership status, CMCs, KC and Supplementary Comparisons was presented. Emphasis was placed on the need for Designated Institutes (DIs) and NMIs to ensure their Quality Systems were in conformance with ISO/IEC 17025 calibration (and not testing) requirements along with ISO Guide 34 where appropriate. He then detailed a number of Resolutions adopted by the CGPM. These related to future revision of the SI, development of SI traceable measurements related to climate change and radiometric standards; the dotation to the BIPM (which was declared for the three years 2013-2015) and associated financial matters; the need for a revision of the *mise en pratique* for the metre and development of new optical frequency standards; the adoption of the International Terrestrial Reference

System for all metrological applications; and, most significantly, the role, mission, objectives, long-term strategy and governance of the BIPM. In connection with the role and governance of the BIPM, the President noted that a CIPM *ad hoc* Working Group (WG) had been created, chaired by Dr B. Inglis (President of the CIPM), with a mandate to consider these matters. Recommendations made by the *ad hoc* WG and associated notes had been recently published. Members of the *ad hoc* WG are Government representatives of the Member States and NMI directors. Included in their recommendations was that the CGPM meet every two years, and for the BIPM budget to be fixed for four year periods. Also recommended was that transparency in the activities of the BIPM should be increased, including the planning processes and activities of the Consultative Committees (CCs) and the BIPM programme of work. It was therefore proposed that each CC should develop documented strategic mid- and long-term planning of Key, Supplementary and Pilot study comparisons including activities coordinated by the BIPM by the end of April 2013 for consolidation, consideration and decision by the CIPM in 2013 and further discussion with the Government representatives and NMI directors in a meeting to be held in the third quarter of 2013.

There followed a short discussion among delegates; lengthier discussion was not needed because these matters had been raised earlier during the WG sessions. Dr H. Emons expressed concern about the short lead time with which these requests had been presented to the WGs, i.e., only ten days prior to the CCQM meeting. Additionally, he noted that the information requested was not consistent from group to group. Moreover, the nature of the requests, such as the NMI resources to be made available to the CCQM, is beyond the mandate of the attendees to provide as they are not empowered to make such decisions. Dr H. Emons suggested that a new method to disseminate such requests was needed. Dr R. Kaarls agreed but noted that the current route was followed because of time constraints, and the fact that the *ad hoc* WG had met in March 2012 and their recommendations were only published two weeks before the CCQM meeting. Moreover, he expected that NMI Directors attending the October meeting would have already provided input into higher level decisions regarding the contributed resources. Dr B. Güttler cautioned that it was difficult for NMIs to predict their activities one year in advance, let alone to predict their work several years into the future. Dr R. Kaarls replied that strategies were required to enable an understanding of what comparison activities were required to sustain the CIPM MRA, and that this topic had already been a permanent discussion topic for the CCQM WGs. Documenting this would be achievable. Dr W. May was of the opinion that the *ad hoc* committee had expressed a clear request for dialogue and reasonable responses from the NMIs were required with understanding from both sides.

Dr R. Kaarls summarized the timetable of meetings proposed for the second half of 2012 (IAWG – October at UME; GAWG – October at VNIIM; BAWG/OAWG – November at GLHK) and confirmed that a discussion on future strategies for comparisons should be an agenda point for each of the working groups.

## 11. REPORTS OF CCQM WORKING GROUPS

### 11.1. CCQM Working Group on Gas Analysis (GAWG)

Dr M. Milton (NPL) commenced with a summary of activities of the Gas Analysis Working Group (GAWG) since April 2011. The 26th meeting, hosted by the National Oceanic and Atmospheric Administration (NOAA), was held in Boulder, CO, USA, in September 2011 with the 27th meeting completed during the week of the current CCQM meeting at the BIPM headquarters. During the last year, five Key Comparison reports had been submitted to the KCDB and three new KCs had been agreed ([CCQM-K93](#) – ethanol/nitrogen; [CCQM-K94](#) – dimethylsulfide; [CCQM-K101](#) – oxygen/nitrogen).

The results of CCQM-K93 were briefly reviewed. This study was an example of a preparative model wherein all measurements were undertaken by the coordinating laboratory (NPL) to which participants sent their prepared mixtures, reference values and uncertainties. The study revealed some inconsistencies, despite the expectation that all NMIs would be using very similar methods. The uncertainties on purity estimates varied by a factor of up to three orders of magnitude, while those for the added mass covered a factor of twenty. When combined, these two sources of uncertainty did not always reflect the certified uncertainty, and in some cases, the difference between the measured value and the certified value was larger than the expanded uncertainty. The GAWG is discussing the reference value.

Dr M. Milton then presented in more detail the progress made at the meeting on atmospheric issues held in Boulder, CO, USA, hosted by NOAA. The meeting provided an opportunity to engage with NOAA scientists who maintain the scales for many of the greenhouse gases and who share a common commitment to the accuracy and stability of data. A visit was made to the NOAA air sampling station at Niwot ridge. The meeting provided an opportunity to make practical progress on some of the agreements made at the 2010 joint WMO/BIPM workshop on “Measurement challenges for global observation systems for climate change monitoring” held in Geneva, Switzerland. Dr M. Milton highlighted a number of actions arising from the workshop including: progress that is being made in assessing the stability of reference materials through a series of CCQM KCs; moves by NOAA laboratories to implement a quality system; harmonization of methodology for preparation and dissemination of standards by both WMO Global Atmosphere Watch (GAW) and GAWG groups is progressing; data quality objectives were discussed for improved definition; and practical issues relating to the transport of gas cylinders were being examined through the World Customs Organization. Dr M. Milton noted that the GAW is interested in developing a standard for nitrogen oxides.

Dr M. Milton discussed the status of GAWG CMCs and the recent review held during Cycle XIII. The work was considered to be labour intensive and the process needs to be streamlined. As a result of this re-review, he noted that CCQM-K1 is 16 years old and while the core mixture approach to CMC support will be continued, it was noted that the process cannot address every CMC from every NMI. Consequently, NMIs must take the initiative to fill any gaps and if a current KC does not cover an anticipated CMC claim, it is the responsibility of the NMI to take action and seek out collaborators for a bilateral or other suitable comparison. Dr M. Milton concluded that the issue of the relationship between the measurement uncertainty stated for a measurement capability and that for a corresponding certified

reference material (CRM) within CMCs was still under discussion within the GAWG, and that future refinement of the approach could include input from ISO TC 158 (Analysis of gases).

Early thoughts on the GAWG strategy were highlighted with respect to three sectors: environmental (atmospheric, air quality and indoor air), energy gases (natural and refinery gas and other fuels) and emission/core gases (emission and forensic gases and stable binaries and multi-components) but these had been only briefly discussed. A summary chart of current CCQM and Regional Metrology Organization (RMO) comparison activities was presented highlighting activities in these various sectors. With respect to atmospheric monitoring, future requirements may include toxic gases in the workplace. Concerning energy gases, for which most NMIs have CMC claims, future challenges will be identified. In the area of emission gases and other applications, no significant progress has been made despite individual NMI activities. Some good progress has been made in benchmarking capabilities in the area of core mixtures.

A draft outline of a programme of activities covering 2012 to 2019 was presented that summarized proposed comparisons in each of the areas of interest. Dr M. Milton suggested that during the coming months he would supply more detailed information including a list of possible comparisons for which NMIs and the BIPM were identified as comparison coordinators.

Dr M. Milton summarized by reiterating the success of the Boulder, CO, meeting on atmospheric issues; and added that the preparative model would continue to be used for key comparisons; that NMIs must take responsibility themselves to plan evidence to underpin their CMC claims; and that a draft strategy for the future had been discussed and would be further developed at the next GAWG meeting planned for October 2012 in St. Petersburg, Russian Federation.

Discussions were brief and focused on the need for NMIs to provide evidence for their CMC claims. Dr B. Güttler asked whether NMIs would be asked to organize core comparisons. Dr M. Milton responded that some NMIs continue to rely on CCQM-K1 which the KCWG had begun to question and thus individual NMIs should consider action within their RMO to remedy this situation. Dr R. Kaarls noted that the problem is of a much more general nature and applies to all WGs which must take measures to ensure support for their core competencies without engaging in an “endless list” of comparisons. Prof. M. Kühne pointed out that this is a good example of strategic documentation that serves its purpose to highlight long-term needs, including planned KCs and identifies “who will do what” which will be useful to the BIPM. Mr A. Squirrell (ILAC) noted that any strategy proposed to support CMC claims in gases was critical and should rely on the RMOs. He reasoned that if working laboratories are to obtain their traceability from NMIs and there are no CMC claims in the database to support them, then these gaps have to be filled somehow. Mr A. Squirrell supported the philosophy.

## **11.2. CCQM Working Group on Inorganic Analysis (IAWG)**

Dr M. Sargent (LGC) presented an overview of the activities of the Inorganic Analysis Working Group (IAWG) throughout the past year. Interim meetings were hosted by NMIA in Sydney, Australia, on 1-4 November 2011, and at the BIPM headquarters during the week of the current CCQM meeting, each of which included joint meetings with the CCQM Working Group on Electrochemical Analysis (EAWG). Both meetings included technical workshops. In Sydney, these addressed the Avogadro Project and applications of mass spectrometry in clinical and bio-analysis. In the meeting at the BIPM headquarters,

the focus was on the European Metrology Research Programme (EMRP) suite of relevant activities. Additionally, technical presentations at the BIPM headquarters were delivered by: Dr R. Feistel of the Leibniz-Institute for Baltic Sea Research on the activities of the International Association for the Properties of Water and Steam (IAPWS) covering traceability of salinity measurements; Dr K. Pratt (NIST) on coulometry as a primary technique for purity assessment; and by Dr M. Medvedevskikh (UNIIM) on uncertainty of measurement results for moisture in grains. Core competency approaches to the development of KCs and support of CMCs were delivered by Dr G. Turk (NIST) on both occasions.

Dr M. Sargent presented a summary of IAWG KCs from 1998 to 2012, noting that 35 had been undertaken with 27 reports published and a further two in progress. Three new KCs are currently under way with three further KCs planned. Pilot studies in progress were highlighted as was the APMP.QM-S5 RMO comparison on essential and toxic elements in seafood.

[CCQM-K30.1](#) / -P12.2 (Pb in wine, coordinated by CMQ, Chile) was noteworthy in that one initial KC participant, Eurofins (Denmark), was removed from the data set as it was determined that it was not an official designated institute. A KCRV based on the median of Isotope Dilution Mass Spectrometry (ID-MS) results was selected. Outliers were identified following technical discussions by the participants and by using statistical techniques.

[CCQM-K87](#) /-P124 (Elemental calibration solutions, coordinated by PTB) is significant because a large number of NMIs have CMCs for this activity. Nine solutions containing Cr, Co and Pb were prepared by PTB, allowing a calculated KCRV based on gravimetry to be undertaken in each case. The study indicated that high precision, high accuracy results from individual NMIs could be extracted from the KC results without burdening one central laboratory by following a “preparative model” approach.

[CCQM-K89](#) /-P126 (Trace and essential elements in *Herba Ecliptae*, coordinated by HKGL) provided a test case because it required the participation of all NMIs to undertake determination of at least As and Ca in the matrix material. Outliers were based on a full technical discussion with the participants and a tentative proposal for the KCRV, calculated using a mixture model median approach, will be re-examined during the next IAWG meeting.

[CCQM-K100](#) / -P127 (Copper in ethanol, coordinated by INMETRO) is based on the distribution of a spiked fuel ethanol, as the endogenous level of copper was deemed to be too low for this study. A KCRV was calculated using various estimators and it was agreed that a MM-median estimate will be adopted.

[APMP.QM-S5](#) (Essential and toxic elements in seafood, coordinated by GLHK) focused on the measurement of Fe, Zn, As and Cd with half the participants coming from the APMP RMO and half from elsewhere. This study ran in parallel with APMP PT11-01 and APLAC PT082. A variety of estimators was applied for the calculation of the KCRV and the supplementary comparison reference value (SCRV) with the MM-median recommended by GLHK.

Dr M. Sargent briefly discussed the KC and pilot studies (PSs) agreed during 2011-2012. A KC and/or pilot study will follow-up the earlier CCQM-P128 comparison for Hg, Pb and As in cosmetic materials (NIM) in addition to a KC and PS for Se species in serum (LGC) and a KC or PS for analysis of As species in brown rice flower (NMIJ). Two KCs that were agreed earlier will be restarted ([CCQM-K72](#) and [CCQM-K98](#)).

Key aspects of the IAWG strategy were discussed, these being based on an assessment of core competencies addressed with each planned KC. Development of a “white paper” (Dr G. Turk, NIST) is maturing and a rolling five-year plan for KCs was adopted. Dr C. Quétel (IRMM) developed a spreadsheet template to summarize material availability from 13 NMIs. This information can be conveniently searched and updated to realize a source of matrix materials for future comparison exercises.

Issues arising from CIPM MRA requirements on traceability were discussed. The Joint Committee of the Regional Metrology Organizations and the BIPM (JCRB) traceability document ([CIPM 2009-24](#)) currently constrains users of Certified and Standard Reference Materials to ensure that the suppliers have corresponding CMCs in the database to support the corresponding measurement capabilities. This requirement is frequently not the case for high purity materials, isotope ratio standards (both absolute and delta-scale materials) and for some of the older calibration standards, which are still available or are in circulation. SI traceability derived through use of such materials therefore contravenes the requirements of the CIPM MRA. A long-term IAWG strategy is required to address these issues, and an exemption to the JCRB document has been requested to cover these areas of concern. In the interim, the IAWG will continue to work towards finding solutions to these problems, via the high purity elemental calibrant workshop planned for the October 2012 meeting in Istanbul, Turkey, as well as through joint discussions on current EMRP projects. Identification of available/future NMI resources and the possible need for an extension of the BIPM programme in chemistry in the inorganic field could be considered. Exception from the CIPM MRA traceability requirements is likely to be essential for a number of years for isotopic standards.

Dr M. Sargent suggested a strategy for implementation of the CIPM/CCQM requirements for WG strategy documents. He proposed the drafting of a document for submission to the BIPM with the help of a small *ad hoc* group. However, this would not be undertaken until the CIPM/CCQM requirements are more precisely known. The survey of sample availability will be updated and NMIs will be contacted to determine their potential commitments to future KCs. A draft document will be circulated for comment and further discussion will take place at the October 2012 meeting of the IAWG. An updated 5-year plan for supporting CMCs was presented and future activities were summarized. It was noted that the vision for the 5-year and 10-year projects was unlikely to differ but that new pilot studies may arise to target emerging areas.

Dr A. Fajgelj (IAEA) commented that for isotopic reference materials the traceability claim is not to the SI but to the delta scale, and therefore reference materials for the measurement community (at least for seawater) are now secure for the next 100 years. Dr R. Wielgosz noted that the major link of the IAEA in the field of chemical measurands is via the IAWG. He asked if the well-established and practical delta scales, with their accompanying available materials, would be brought into the scope of comparison studies in the IAWG. Dr M. Sargent replied that future issues will deal with compound specific applications for which requirements may increase. Dr Z. Mester (NRC) questioned whether the CCQM was the correct forum for discussion of issues related to non-SI traceable measurements. Dr M. Milton argued that issues were being confused with respect to the perceived limitations; the current reference materials will indeed run out, but the perceived benefits of forcing these measurements to be traceable to the SI is unclear. Dr L. Mackay (NMIA) noted that the carbon ratio artefacts are associated with claimed CMCs but they are not traceable to the NIST, for example, and this issue is yet to be resolved.



Dr A. Fajgelj argued that availability of stable isotope ratio materials are not a critical issue for the user community, and while there is a need to work on traceability, this should not limit CMC claims.

Dr H. Emons questioned the efficacy of the planned KC on arsenic species in brown rice, stating that it would be very difficult to separately quantify As(III) and As(V) species without uncontrolled transformations and suggested that the species are combined. Dr M. Sargent replied that this had been considered at the recent IAWG meeting and a request will go out for participants to register for total inorganic arsenic measurements.

Dr R. Sturgeon (NRC) noted that this is the first request for an exemption to the JCRB document on traceability and asked for clarification on how the process is expected to unfold. Dr R. Kaarls agreed that this would indeed be the first request and asked Dr R. Wielgosz to comment on the situation. Dr R. Wielgosz replied that ownership of this task now lies with the KCWG and that Dr D. Sin (GLHK), as chair of the KCWG, will work with Dr M. Sargent and the IAWG to complete a form that was developed two years ago to describe the proposed exception. This will be submitted to the BIPM/CIPM for consideration.

Dr W. May suggested that a high level philosophical discussion of what really constitutes a CMC is required because the database is rapidly filling with CMCs and will soon reach an unmanageable level which precludes its periodic review; it clearly cannot be analyte/matrix specific.

Dr S. Ellison (LGC) questioned whether current KCRV calculations are based on data which have been subjected to outlier rejection. He noted that the mixture model median approach is insufficiently characterized and is therefore not recommended for use in calculation of a KCRV but was only to be used as an aid to the identification of outliers. This is not particularly clear in the guide. The median can be the preferred statistic for the KCRV because of its simplicity and transparency over statistical rigour. Dr M. Sargent noted that this is in line with the practices of the IAWG but noted that it is difficult to rely on the median when the data set is limited to 3 or 4 participants. Outliers should only be removed for technical reasons and if a datum cannot be eliminated on these grounds then the median is a better estimate because it is much less affected by inclusion of such data. Additionally, it is interesting to evaluate the robustness of the KCRV when it is calculated by different estimators.

### **11.3. CCQM Working Group on Electrochemical Analysis (EAWG)**

Dr M. Máriássy (SMU) presented the work of the CCQM Working Group on Electrochemical Analysis (EAWG). Since the previous CCQM meeting held on 13-15 April 2011 the EAWG has met on two occasions in Sydney, Australia, during the week beginning 1 November 2011 attended by 5 participants from 5 countries and at the BIPM headquarters in April 2012 as part of the current CCQM meeting, with 26 participants from 18 countries. The next EAWG meeting will be held in Paris in April 2013. A joint meeting with the IAWG which was due to be held in October 2012 will no longer take place.

Dr M. Máriássy presented the results of two recent EAWG comparisons and two comparisons conducted jointly with the IAWG.

[CCQM-K91](#) (pH of phthalate, coordinated by PTB), the first repeat comparison, was a follow-up of the 2001 [CCQM-K17](#) study and indicated improved performance for measurements conducted at 25 °C. However, agreement among 16 participants (15 of whom used a primary method) deteriorated at other test temperatures.

[CCQM-K92](#) (Electrolytic conductivity, coordinated by SMU) was designed to fill the gap in completed comparisons (two samples with targets of 0.05 S/m and 20 S/m) and to extend the conductivity range to cover current CMCs (some claiming up to 50 S/m, 100-fold higher than the last Key Comparison target). Sixteen participants from 15 countries are engaged in the study but problems have been experienced with customs handling of the samples and affecting their potential stability. Data provided by two participants fell outside the mean, which were attributed to calibration issues.

[CCQM-K96](#) (Assay of dichromate, coordinated by SMU and KRISS) was a joint study with the IAWG. An extended deadline was required due to difficulties with sample distribution (e.g., UNIM only received its sample after many of the other reports had already been submitted), highlighting a problem that also affected CCQM studies [CCQM-K73](#), and [CCQM-K92](#). All results will be based on use of coulometry for the evaluation of the assay and the study is on hold pending receipt of results from three participants.

Dr M. Máriássy then discussed several technical presentations made to the WG and highlighted some of the salient points of each. These included: what to beware of when measuring electrical conductivity (Dr P. Jakobsen); traceability in electrical conductivity (Dr W. Kozłowski); ISE activity measurements for clinical applications (Dr B. Guettler); the IAPWS proposal for work on traceability of salinity measurements of seawater (Dr R. Feistel) and coulometry as an alternate route to traceability (Dr K. Pratt).

A brief presentation of the EAWG draft strategy focused on the issue of there being a small number of relevant staff at many NMIs, effectively limiting the number of comparisons that can be conducted. This lack of manpower necessitates a very efficient approach to KC and Pilot studies to support CMC claims. New studies and comparisons designed to overcome these issues were summarized. These included CCQM-Kxx (coordinated by PTB and INRIM, 2012) for conductivity in seawater at 5.3 S/m which will be a follow-on study to CCQM-P111 but will now use a multi-component aqueous salt solution; [CCQM-K99](#) (coordinated by PTB, 2013) for pH 4.7 phosphate buffer which will present handling challenges due to low buffer capacity; CCQM-P37.2 (coordinated by NPL, 2012) which will focus on the behavioural characteristics of different Ag/AgCl electrodes used by various institutes; CCQM-P93 (for 2013+), a pH preparation study with measurements performed at one laboratory and CCQM-Pxx (2013+) for an acidimetric suite study which will follow-up on the problems identified in [CCQM-K73](#), and may target both weak and strong acid samples.

Dr M. Máriássy identified two issues relevant for discussion of CMCs. The first was related to a communications error which prevented Chile from participating in a KC, which as a result now requires a follow-up bilateral to resolve. The second arises from a request for opinion on CMCs on determination of chemical oxygen demand (COD) from the KCWG. This is believed to lie outside the purview of the EAWG, nevertheless the group agreed that the measurand is operationally defined, as for moisture in grain or  $pH_e$ , so should be treated similarly. Dr M. Máriássy also noted that further discussion on the traceability of measurements of electrical conductivity, which arise because some institutes use published values of conductivity – raises the issue of whether this should be considered a viable traceability route. A

conclusion was reached that a viable traceability route is not achieved unless uncertainty contributions from all experimental factors, that may not be under full control, are taken into account.

Dr M. Máriássy concluded with a strategy plan to progress the recent need to provide input to the *ad hoc* WG that is addressing the Consultative Committee planning requirements covering the period 2012-2019, targeting studies of pH, electrical conductivity, coulometry and other methodologies.

Dr R. Wielgosz opened this discussion by questioning whether the planned comparisons relating to seawater were to use real seawater or synthetic solutions. Dr M. Máriássy replied that studies of pH will not be based on real samples but that seawater could be used for conductivity measurements, as was done for CCQM-P111. Dr R. Kaarls raised the issue of the small uncertainties associated with data pertaining to the various temperature regimes studied as part of [CCQM-K91](#). Dr M. Máriássy suggested that problems may yet be identified arising from this study and that a follow-up study should be considered. Dr R. Kaarls asked if any progress had been made to resolve problems with customs officials when sending test samples to avoid delays in receipt of samples by participants. Dr H. Emons noted that ISO/REMCO is to publish a report on the transport of test materials; it is hoped that delays in the distribution of comparison materials can be avoided. Dr R. Kaarls enquired as to whether full cooperation with the IAPWS was now established; this was affirmed by Dr M. Máriássy. Dr M. Milton asked whether the EAWG had experimented with the concept of a single coordinating laboratory serving as the central service laboratory to which all participants' samples could be sent for measurement. Dr M. Máriássy replied that although this was possible it had not yet been undertaken due to the onerous work load involved, but that such studies are planned. Dr W. May commented that it was unlikely that there would be any additional work entailed if simple service samples were collected from each of the NMIs since fewer comparisons would be conducted as time goes on. Dr M. Milton suggested that a significant portion of the potential workload could be avoided if fewer test temperatures were investigated; Dr M. Máriássy agreed.

#### 11.4. CCQM Working Group on Organic Analysis (OAWG)

Dr L. Mackay (NMIA) presented progress made by the Working Group on Organic Analysis (OAWG), which had met twice since the previous meeting of the CCQM held on 13-15 April 2011; 1-2 November 2011 in Sydney, Australia, attended by 39 participants from 22 different organizations, and in April 2012 at the BIPM headquarters during the week of the current CCQM meeting, attended by 41 participants from 29 different organizations. Dr L. Mackay gave a brief overview of the thematic four-track strategic approach adopted by the OAWG for comparison studies: (A) Key Comparisons that test core competencies for the delivery of measurement services to customers which are planned to be limited in number in which NMIs with relevant claims must participate; (B) Key Comparisons that assess the equivalence of measurement services actually provided to customers, the needs for such being determined by the KCWG; (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 which will not be used for the assessment of CMCs. Dr L. Mackay suggested that some merging of these comparison categories may be considered in future in the interests of efficiency.

An initial 5-year plan which was comprised of a large input from the BIPM activities covering low- and high-polarity materials underpinning performance in delivering services for primary calibrators and accuracy control measurement services was reviewed. Activities relevant to measurement service categories 1-10 were proposed up to 2019. Six reports have been completed since the previous meeting of the CCQM in April 2011, including [CCQM-K55.a](#)/-P117.a (Purity assessment of high purity materials: 17 $\beta$ -estradiol); [CCQM-K55.b](#)/-P117.b (Purity assessment of high purity materials: aldrin); [CCQM-K79](#)/-P123 (value assignment of CRMs and proficiency testing of materials for ethanol in aqueous matrix); [CCQM-K80](#) (value assignment of CRMs and proficiency testing of materials for creatinine in serum); [CCQM-K81](#)/-P122 (Chloramphenicol in pig muscle) and CCQM-P129 (Ethanol and water in a bioethanol material derived from sugar cane). One Draft B report on [CCQM-K85](#) (Antifungals in food: malachite green in fish tissue) will be finalized following the recent discussions during the current CCQM meeting in April 2012 at the BIPM headquarters. One new study proposal was suggested – [CCQM-K95](#) (Mid-polarity analytes in food: pesticides in tea).

Dr L. Mackay highlighted the outcomes of a joint OAWG/BAWG meeting devoted to a session on purity assessment held on 17 April 2012. The importance of purity assessment and approaches used to achieve traceability to the SI was discussed by Dr. S. Ellison and Dr G. O'Connor. The status of CCQM-K55c/-P117c (Purity assessment of high purity organic materials: L-valine) was reviewed by Dr S. Westwood. The BIPM organic primary calibrator programme and the mass balance approach to purity assessment presented by Dr R. Josephs included the differences between indirect mass balance estimates and direct assessment of the measurand by quantitative NMR; a concept paper on SI value assignment of purity is in preparation. The paper will summarize the performance of this approach for application to: theophylline, digoxin, estradiol and aldrin pure materials. Purity assessment of pharmaceutical proteins, with the drivers for purity measurements for larger molecules being delineated, including the fact that there are currently few CRMs available in this area, was considered by C. Jones. The session was concluded with a panel discussion on current experience, limitations and future approaches. Track A future comparisons relevant to purity (measurement service categories 1-4) and those for matrix materials (measurement service categories 5-10) were presented with activities in these areas forecast to 2019.

[CCQM-K95](#)/-P136 (Mid-polarity analytes in food matrix: pesticides in tea, coordinated by GLHK and NIM) was reviewed. A variety of methodologies were employed for the determination of  $\beta$ -endosulfan and endosulfan sulphate with results obtained ranging over nearly 2-orders of magnitude. It was concluded that this was not due to a calibrant problem, whereas water was deemed essential for efficient extraction. The issue remains as to how the KCRV will be calculated.

[CCQM-K102](#)/-P138 (Brominated flame retardants in sediment, coordinated by IRMM) was briefly highlighted as a 2012 Track A study material for low-polarity analytes in an abiotic matrix. Some details of the sample preparation were given and the proposed PBDE analytes defined.

Folate in serum (coordinated by NIST) was introduced as a 2013 Track A high-polarity analyte in a biological matrix and immunosuppressants in blood (coordinated by LGC) is under consideration for a future study.

Track C comparisons for 2012 included CCQM-K103 and APMP.QM-P19.1 (Melamine in milk powder, coordinated by NIM and GLHK), phthalates in PVC (coordinated by KRISS) and [CCQM-K6.2](#),

[CCQM-K11.2](#) and [CCQM-K12.2](#) (cholesterol, glucose and creatinine in human serum, coordinated by NIST).

Dr L. Mackay turned to a consideration of core competency tables for the OAWG, these comprise a set of competencies similar to those used by the IAWG. Its application to [CCQM-K55.c](#) was illustrated.

The OAWG Guidelines document, covering topics relating to types, selection and participation in comparisons, expected content of proposals and reports as well as the roles and responsibilities of the coordinators and participants is under final revision by Dr R. Parris (NIST). The document is expected to be finalized by the end of 2012.

Dr L. Mackay concluded by announcing that the 5-9 November 2012 interim meeting of the OAWG (in conjunction with a meeting of the BAWG) will be hosted by GLHK in Hong Kong (China).

Dr W. May suggested that in future, the term ‘competencies used’, rather than the term ‘competencies demonstrated’, be adopted for use. Dr H. Emons also commented on the use of terminology relating to analyte, suggesting that measurand would be more appropriate. Dr R. Wielgosz remarked that there was a request from the JCTLM that some comparisons be organized in the clinical area which would be better coordinated with some of the ring trials organized for the International Federation of Clinical Chemistry and Laboratory Medicine (IFCC). Ms H. Parkes noted that LGC had been approached by Abbott Laboratories to consider the development of reference methods and materials for immunosuppressants and agreed that there was a need to form better linkages with other groups. This suggestion was fully supported by the President, Dr R. Kaarls. Dr B. Güttler remarked that a Euramet comparison on creatinine was under way and that it may be useful to link to it. Mr A. Squirrell asked for clarification on how to link clinical laboratories to such comparisons if non-NMIs were unable to participate. Dr R. Wielgosz clarified that the link was via the test sample material itself. Dr W. May cautioned that NMIs delivering services in these areas will not have the time to run samples as frequently as is done in clinical laboratories. Dr R. Wielgosz clarified that when there is a requirement in the CCQM to run a comparison the sample test material could be supplied from the IFCC RELA scheme and thus enable compatibility of measurement results between NMIs and reference laboratories to be demonstrated. Dr R. Kaarls returned to the issue how to distinguish between the terms analyte and measurand, noting that the VIM does not use the term analyte; Dr R. Wielgosz clarified the terms.

#### **11.5. CCQM Working Group on Bioanalysis (BAWG)**

Ms H. Parkes presented the progress made by the CCQM Working Group on Bioanalysis (BAWG), highlighting another extremely active year with increased interest and participation from NMIs and other expert laboratories. The BAWG has met twice since the previous CCQM meeting in April 2011. The 20th meeting of the BAWG was hosted by CENAM in Queretaro in October 2011 and the 21st meeting was held in April 2012 during the current CCQM session. Fifty participants from 25 organizations attended the 21st meeting. Ms H. Parkes noted the considerable bioscience expertise in nucleic acid, protein and cell measurement capabilities with a diversity of measurement technologies is now assembled within the BAWG. Break-out sessions are used to optimize further study discussion opportunities. BAWG contributed to the organization and presentations of a workshop on “Role for reliable traceable microbial measurements to ensure food quality and safety” convened in April 2011 at the BIPM

headquarters and a symposium on “Biomeasurement in support of the clinical diagnostics community” held in Queretaro in October 2011, the letter served to engage bioscience networks within Mexico.

The strategic activity of the BAWG was then outlined. A desire for fewer Pilot studies and more KCs to underpin more generic CMCs was expressed and three essential activities were described:

- the standard Key and Pilot study comparisons undertaken for benchmarking purposes (to better establish measurement procedures);
- pre-Pilot and/or -Key (investigative) collaborative studies to develop and assess our ability to compare measurement results, evaluate approaches to improve measurement quality to enable comparisons (with clearly defined measurand, metrological traceability, measurement uncertainty, reference value);
- concept papers which will be explored as a way to discuss and record metrology issues, requirements and challenges in biomeasurement areas along with proposed solutions to achieve comparability.

Ms H. Parkes highlighted results from key comparison [CCQM-K86](#)/-P113.1 (Relative quantification of genomic DNA fragments extracted from a biological tissue, coordinated by IRMM) which supported competence in both DNA extraction and quantitative PCR techniques and was approved for equivalence. A KC proposal (CCQM-Kxx/-P113.2) received from NIM (Relative quantification of Bt63 in genetically modified (GM) rice matrix) arose from a planned APMP study that may also involve the GLHK and IRMM.

Ms H. Parkes then discussed the progress made in 2012 with Pilot studies. CCQM-P55.1 (Peptide/protein quantification, coordinated by NIST, LGC and PTB) which aimed to determine the concentration (mol/g) of four peptides in solution through amino acid analysis using valine, proline, isoleucine and phenylalanine with calibration against NIST SRM 2389a amino acid and in-house standards. The study did not address the question of peptide purity, therefore limitations were identified in a laboratory’s capability to assess the purity of a peptide in aqueous solution or to quantify peptides of known purity. This issue will be addressed in a concept paper. Before progressing to a KC in 2013, hydrolysis methods will be discussed at an interim meeting to be held in Hong Kong (China) in November 2012.

CCQM-P58.1 (Development of a reference immunoassay for cardiac troponin cTnI, coordinated by NPL and NIST) is linked to an IFCC initiative for standardization and traceability of cTnI measurements. Although comparable results were obtained by study participants, uncertainty estimates need clarification.

CCQM-P103.1 (Gene expression biomarker profiling, coordinated by LGC) follows up on the 2011 P103 study using a single RNA transcript to underpin work to distinguish normal from diseased cells. In study P103.1, multiple RNA transcripts (6) were quantitated by looking at a copy number of each target transcript in two unknown samples. Preliminary data analysis of the results showed generally good concordance on all samples despite the use of different techniques, including PCR, dPCR and NGS (next generation sequencing).

Ms H. Parkes then presented a number of pre-Key and -Pilot investigative studies that were in progress. Pre-001 (Study on absolute quantification of DNA, coordinated by KRIS) aims to assess cross-validation of direct counting and dPCR techniques as a route to traceability of nucleic acid measurements. A second investigative study, Pre-002 (coordinated by LGC), will examine cell viability measurements,

an important issue for biological therapeutics, which will discern how to distinguish live and dead cells based on fluorescence or colorimetric techniques. A labelled cell material will be used to benchmark measurements in different laboratories.

In the area of Concept Papers, Ms H. Parkes described two issues: Con 001 (Metrology for nominal properties, coordinated by NIST), and Con 002 (High throughput sequencing in reference measurement systems, coordinated by LGC). The latter will focus on issues for high throughput sequencing measurement e.g. sequencing a microbial gene (e.g., *E. Coli*) to examine the impact of transitions from PCR to other techniques such as NGS. Concept Paper Con-003 (Routes to macromolecular purity-approaches to the assignment of mass fraction purity, coordinated by BIPM) is the result of a joint BAWG/OAWG purity assessment workshop held at the BIPM headquarters in April 2012 (see section 11.4 for the relevant discussion). The workshop identified a lack of metrologically sound purity characterization methods for large biomolecules as a major barrier to the achievement of SI traceability in biological measurements. Ms H. Parkes noted that the issues of purity measurements had been submitted as a potential research topic in a recent EMRP call.

Ms H. Parkes concluded by announcing the venue and date of the next meeting of the BAWG as 6-9 November 2012 in Hong Kong (China) hosted by GLHK. The theme of the symposium will be “Challenges in food safety and biopharmaceutical testing”.

A brief discussion followed on the need to define the measurand vis-à-vis the impact of the measurement protocol. Dr H. Emons cautioned that, with respect to targets or measurands, careful consideration should be given to the fact that different techniques assess different properties. An example is the comparison of dPCR and direct counting techniques wherein the measurand is the amount of viable microorganism. The same situation is encountered when assessing protein concentration by mass spectrometry versus immunoassay, i.e. they are method dependent. Ms H. Parkes agreed but added that the aim was to achieve measurement comparability and dPCR reduces uncertainties. Dr S. Park (KRISS) stated that the organizers were aware of this shortcoming and it was for this precise reason that the study was being undertaken. Dr S. Ellison supported the need for this distinction and the need to first define what is intended to be measured and to subsequently determine if what was intended to be measured was in fact measured.

## 11.6. CCQM Working Group on Surface Analysis (SAWG)

Dr W. Unger (BAM) presented his report of progress in the CCQM Working Group on Surface Analysis (SAWG) which had convened for its 10th meeting, during the week of the 18th meeting of the CCQM at the BIPM headquarters. He was pleased to note that the SAWG now comprises 13 active members from a total of 18 participants. The scope of the SAWG was reviewed and included a brief description of the portfolio of instrumental techniques most commonly used to illustrate the relationships between spatial resolution (spanning the 0.1 nm to 10  $\mu\text{m}$  dimension) and information capabilities (elemental analysis, chemical state analysis and analysis with some structure) used to achieve surface and micro/nanoanalysis at the interface.

Dr W. Unger emphasized the critical role played by electron microprobe techniques and reviewed the past Pilot studies CCQM-P80, CCQM-P81 and CCQM-P95, results of which suggest that a KC in this area

would be premature. A taskforce comprising participants from NIST and BAM was formed in 2010 to design a working protocol to move this forward.

Pilot study CCQM-P130 (WD and ED electron probe micro-analysis on AuCu alloys, coordinated by BAM and NIST) was launched in 2011. Protocols for EDS (BAM) and WDS (NIST) had previously been established and four samples of AuCu alloys along with traceable pure Cu and pure Au calibrants were available (from NIST). A total of nine NMIs/DIs utilizing 17 instruments as well as two companies with three different instruments for EDS and WDS approaches were involved. Good results with consistent data were achieved, prompting the intended launch of a relevant key comparison, to be planned during the next SAWG meeting in 2013. The KC is likely to be based on a nickel super alloy material.

KRISS proposed a new Pilot study on the characterization of a CuInGaSe<sub>2</sub> 2 µm thin-film which is currently used in solar cell production. A variety of methods will be used to assess composition, with calibration based on a traceable CRM to be provided by KRISS. A homogeneity check of the samples has been completed and samples will be distributed to a substantial number of NMIs which expressed an interest in participating in June 2012. Results are expected to be reported in April 2013.

Dr W. Unger then considered the impact of the EURAMET EMRP on SAWG activities. A joint research project (SurfChem) was launched in October 2011 with a remit that included generation of new Pilot studies. He outlined the tasks associated with Work Package 1, which impacted inorganic reference materials for surface analysis and those of Work Package 2 which was focused on reference materials and methods for the analysis of organic moieties on surfaces, including the labelling of surface species that may be of interest to BAWG activities. Additional EMRP projects to be launched in 2012 include BioSurf and InfectMed. The former focuses on metrology for biosensing based on surface immobilized biomolecules; the latter targets metrology for real time quantitative PCR in which surface analysis would play a role in assessing the adsorption losses of biomolecules on PCR devices.

A discussion on the feasibility of a Pilot study to address the chemical composition of engineered nanoparticles (ENPs), including mixed source and core shell ENPs that were linked to the Organisation for Economic Co-operation and Development's (OECD) list of NPs of immediate relevance has been undertaken by the SAWG. The SAWG hosted a presentation by S Vaslin-Reimann (LNE) on "Nanoparticle populations" which summarized achievements of the Versailles Project on Advanced Materials and Standards (VAMAS) TWA 34 project.

Dr W. Unger noted that CMC claims by KRISS, BAM, NMISA and NIM arose from [CCQM-K67](#) (Amount of Fe and Ni in (200 nm) Fe-Ni alloy film on Si, coordinated by KRISS) which was completed in 2008. He concluded his presentation by noting that the SAWG has had an impact on the international standardization community in that members of several ISO TCs were SAWG participants and that the outcomes from [CCQM-K32](#) were disseminated through an ISO standard published by ISO TC 201 'Surface Chemical Analysis' in 2011.

Dr R. Kaarls expressed his pleasure in the positive outcome of [CCQM-K32](#) and Dr W. May opened discussions by asking if interest in engineered nanoparticles (ENPs) was based on bulk or surface characterization and that interest may depend on the nature of the measurement service delivered by the NMI, specifically because the CMC database is populated by only surface analytical capabilities. Thus, a clearer definition of the measurand is needed. Dr W. Unger noted that different oxidation states of elements in a nanoparticle and the core shell character of engineered nanoparticles are only accessible by



surface techniques but agreed there was a need for careful definition. Dr R. Wielgosz asked how the landscape of the SAWG, with regards to the number of comparisons currently planned, was related to the number and type of measurement services of the NMIs – what are the measurement services and how well are they covered by the proposed key comparisons. Dr W. Unger replied that a core capability approach was being adopted to minimize the number of potential comparisons. Dr M. Milton commented that most NMIs operated many instruments and asked how the situation will be handled when only one result could be submitted for a comparison. Dr W. Unger suggested that the approach developed was the broadest to allow an NMI to select the technique that was their method of choice. Dr W. May stated that a laboratory should use the technique used to deliver that service to its customers, and not just the technique which may be the most precise.

#### **11.7. CCQM Working Group on Key Comparisons and CMC Quality (KCWG)**

Dr L. Mackay (NMIA) presented the work of CCQM Key Comparison and CMC Quality Working Group (KCWG) which convenes once per year. The KCWG has 22 members (plus a Rapporteur) drawn from all the RMOs. The members met in the days preceding the 18th meeting of the CCQM and addressed concerns with submitted CMCs and procedural matters. Noteworthy is the appointment of Dr D. Sin (GLHK) to succeed Dr L. Mackay as the Chair of the KCWG. During the past week, over 1 200 CMCs, covering inorganics in solution/water, fuels and a subset of gas claims were examined for acceptance in the KCDB, constituting the third year of formal review of all existing CMCs. There are now very few CMCs that are not underpinned by either a KC or pilot study. A review of 270 claims for food and a subset of gases are planned for 2013. In 2012, 400 new CMCs were submitted, with the first claims coming from SP (Sweden) and BVL (Germany). A review of the bioanalysis related CMCs is undertaken by the BAWG during its mid-year meetings. This process was briefly described because it is required for integration into the general submission process as described in the Guidance Document for CMCs within the CCQM.

Dr L. Mackay concluded by briefly identifying emerging areas where new comparisons are required. It was suggested that these will address capabilities connected with rare earth metals, sulphur in fuel, seawater, anion calibration solutions, gas purity and isotope ratio measurements.

Dr R. Kaarls thanked Dr L. Mackay for her services and leadership in the KCWG over the past years, Dr W. May for his in the OAWG, and Dr R. Parris for her extensive work with CMCs.

#### **11.8. CCQM *ad hoc* Working Group on the KCRV**

Dr S. Ellison (LGC) spoke on behalf Dr M. Cox (NPL, chair of the Key Comparison Reference Value Working Group, KCRVWG). Dr S. Ellison reminded all participants that document CCQM/12-21, which is a report that he and Dr M. Cox had drafted and which arose from the 2009 principles earlier accepted in draft CCQM/10-03 (determining consensus KCRV and DoEs) which outlined methods for calculation of the KCRV, is in circulation. A model-based approach (excess variance) is based on the realization that inconsistencies exist in comparisons and an inter-laboratory variance is present (so-called excess

variance) which should account for unexplained effects. Details of this approach were elucidated at the April 2011 meeting of the CCQM wherein Dr M. Cox illustrated some examples using a DerSimonian-Laird technique for estimation of the excess variance. Further implementation work by the WG is required to enhance feedback and recent examples from the IAWG suggested that it was not as viable or universal as had been hoped. A more detailed look at the CCQM/10-03 document is therefore required, with the next steps to include the use of a DerSimonian-Laird estimator in the guidance document and the document updated for re-issue.

In the longer term, it is evident that estimators impact on the degrees of equivalence and there is a need to discern how far a given laboratory participant is from the true value. The methodology to undertake this task does not yet exist and some effort will be devoted to this calculation and the uncertainty associated with this question. Additionally, it is necessary to examine the relationship between DoEs and CMCs in more detail and the impact of homogeneity and stability testing of the material used for comparisons on the KCRV and its uncertainty remain to be assessed.

Dr R. Kaarls agreed that there is a need to re-examine and expand document CCQM/10-03 and wondered if the question of what constitutes a CMC should be addressed before looking into further development of the document so that the relationship between the KCRV and DoE versus claimed CMCs could become clearer. These issues could be addressed during the coming year through a workshop. Dr M. Milton agreed that it was important to wait for a better understanding of the concept of a CMC before further work on this document is undertaken. Dr R. Wielgosz asked for clarification on whether the *ad hoc* working group, which was established with a specific list of tasks, many of which were to be terminated in 2013, would be retained or would its terms of reference be revised. Dr R. Kaarls replied that the *ad hoc* group should complete its current set of tasks and report on these at the 19th meeting of the CCQM together with any further proposed activities which would be considered by the CCQM. Dr S. Ellison agreed to this, noting that one more iteration of the document would be beneficial.

## 12. JOINT COMMITTEE OF THE REGIONAL METROLOGY ORGANIZATIONS AND THE BIPM (JCRB) REPORT

The Joint Committee of the Regional Metrology Organizations and the BIPM (JCRB) Chairman and Director of the BIPM, Prof. M. Kühne, presented the report on the JCRB. Output of the 27th meeting of the JCRB held in Vienna, Austria, in September 2011 was briefly reviewed. This included: rules for modifying existing CMCs (as specified in document [CIPM MRA-D-04](#)); deletion of CMCs belonging to former DIs (Resolution 27/2); designated institutes (what is expected of them as well as their registration in Appendix A and the adoption of resolutions 28/1 and 28/2 requiring DIs to be responsible for holding national standards and the provision of traceability along with a quality system in conformance with ISO/IEC 17025 and/or ISO Guide 34); draft authorship guidelines for comparison reports (posted as [CIPM MRA-G-04](#) and to include anyone participating in the preparation of, contributing measurements to, undertaking data analysis for, or writing a comparison report) and the need for a workshop on best practices in CMC reviews to be held in March 2013 in conjunction with the 30th meeting of the JCRB with participation by all CCs and RMOs. Prof. M. Kühne concluded by noting that four new signatories

have been added to the CIPM MRA from institutes in Saudi Arabia, Bosnia and Herzegovina, Pakistan and Montenegro.

Significant discussion of these issues ensued. Dr H. Emons raised the issue of the required responsibility of the DIs for national standards, requesting clarification of whether they would be required to hold the national standard for the signatory country from which traceability is provided to the users. Dr W. May remarked that within the context of the CIPM MRA, the term 'national measurement standard' was used and had been interpreted to also include 'national measurement capability', and in this case the activities in Chemical Metrology were covered by the statement.

Prof. M. Kühne addressed the participation of Directors of DIs in the meeting of NMI Directors at the BIPM headquarters, which was of particular interest to the CCQM since many DIs were involved in Metrology in Chemistry. He noted that current practice was for the BIPM to send invitations to NMIs that are signatories of the CIPM MRA and that the policy is for the latter to pass them to DIs. After some discussion it was suggested that the invitation process should be examined by the CIPM, to ensure representation of institutes responsible for national activities in the field of Metrology in Chemistry at the NMI Directors' Meeting.

### **13. TRACEABILITY IN THE CIPM MRA (AND CCQM LIST OF EXCEPTIONS)**

Dr R. Kaarls announced that he had no further comments to add, noting that Dr L. Mackay had presented the topic during her discussion of the KCWG. With regard to exceptions, these must be tabled to the CCQM and then tabled to the CIPM. If the exception is agreed then it will be published in a list prepared by the BIPM. Dr R. Kaarls confirmed that action would be taken on the exemptions requested by the IAWG.

### **14. UPDATE ON THE BIPM KCDB**

Dr C. Thomas (BIPM) presented the current status (as of mid-April 2012) of the key comparison database (KCDB). The KCDB now contains 24 800 published CMCs, an increase of ~1 000 during the last twelve months. Some 5 009 CMCs pertain to chemistry and less than 200 chemistry CMCs are currently greyed out. A new open access page on the website (under CIPM MRA) provides information on the KCDB, including a tutorial on its use, which will be updated each year. The KCDB Newsletter (comprising 16 issues) ceased publication in December 2011 and has been merged with the BIPM Bulletin (6 issues) into a new newsletter called [BIPM e-news](#). BIPM e-news will contain general interest items, including scientific issues. The first issue is scheduled for completion in June 2012.

The overall process for posting data to the KCDB appears to be more efficient and a first set of CMCs has now been published by Ecuador. Some 1 081 Key and Supplementary comparisons are now available on-line and this is growing at the rate of 40 KCs and 25 SCs per year. Approximately 65 % of the reports are now published.

Currently, there are 33 Associates of the CGPM and the KCDB but of these Associates, only 14 have CMCs in the database. However, this is increasing (11 % over the last year). In conjunction with a refresh of the BIPM's main website, the KCDB website will also be refreshed. Dr C. Thomas reminded participants that a questionnaire is being circulated requesting feedback on the "useability" of the KCDB website. The questionnaire will be available online

Dr M. Milton commented that there were some shortcomings in the format of the questionnaire, particularly that there was no place to capture negative comments. Dr H. Emons suggested that each group should design its own questionnaire and circulate it. Dr C. Thomas replied that she would be pleased to receive feedback in whatever format it was delivered, and emphasized that she would like to be made aware of any problems.

Dr M. Milton raised the issue of availability of information of the scopes of DIs, noting that it was difficult to get information from each country. Dr W. May noted that such information should be available and clearly delineated in the relevant NMI Quality Manual. Dr R. Kaarls stated that this is more complicated due to the fact that in some countries it is not the NMI but the government that may designate the DI. Information was generally difficult to obtain because there may be more than one DI in an individual country, each responsible for its own area of expertise and more precise information was needed as to what each DI does. A process of clarifying this information has begun.

Dr R. Wielgosz clarified that the CIPM MRA currently prevents two CMCs from the same country claiming exactly the same measurement capability but it does not prevent two CMCs covering slightly different measurand ranges.

## 15. BIPM PROGRAMME ON METROLOGY IN CHEMISTRY

Dr R. Wielgosz (BIPM) presented an overview of the BIPM chemistry programme and discussed the proposed activities for 2013-2015 as well as future strategic planning. He noted that the original 2013-2016 proposals described in CCQM/10-46 had to be restructured due to recent CIPM decisions and were presented in CCQM/12-07 including the strong link between the CCQM strategy and the BIPM programme of work.

During the period 2009-2012 the BIPM coordinated a significant number of CCQM comparisons of relevance to NMIs as well as undertaking development work in support of their comparison capabilities, notably in the area of ozone absorption cross-section measurements, the results of which were published in the *Journal of Geophysical Research-Atmospheres*. In addition, a close collaboration with NIST on purity method development was pursued on large molecule purity using two approaches – the traditional mass balance approach for impurity detection and via amino acid analysis with correction for peptide impurities. Dr R. Wielgosz illustrated these using the characterization of angiotensin as an example and highlighted some of the pitfalls associated with the latter approach.

Dr R. Wielgosz then discussed the prioritization of activities already under way for the period 2013-2015 which had been developed after extensive consultation with various CCQM working groups. These activities are grouped into three main areas: international equivalence of gas standards for air quality and

climate change monitoring; international equivalence for organic primary calibrators; and support to the CCQM, JCTLM and other international liaison activities. As a consequence of the decisions of the 24th meeting of the CGPM, the BIPM programme of work was reduced from four years to three years and the laboratory budget was reduced by 25 %. The level of activity in all three main areas of interest was reduced in scope and no new permanent staff positions will be created. Despite these shortcomings, work planned with CCQM GAWG would proceed on [BIPM.QM-K1](#) (ozone), [CCQM-K90](#) (formaldehyde), CCQM-Kxx (a repeat of CCQM-P73 on NO), and nitric acid in support of a repeat of [CCQM-K74](#) (nitrogen dioxide) in 2016-2019 along with the CCQM-Kxx study of methane and carbon dioxide. BIPM activities in the area of international equivalence for organic primary calibrators would be linked to support Track A comparisons within the OAWG. These include CCQM-K55.d (folic acid, planned for 2014), [CCQM-K78](#) (multi-component amino acid calibration solution, planned for 2015) and a follow on to [CCQM-K55.a](#) (based on a different material). In the field of large organic molecule primary calibrator characterization the BIPM will complete method validation studies, pursue an extension of the collaboration with NIST on purity of human C-peptide and, as an outcome of the OAWG/BAWG workshop, contribute to the drafting of a concept paper on purity for larger molecules. The request of the CCQM BAWG for the BIPM to coordinate a comparison on purity methods for large organic molecules (1-10 kDa) cannot be met in the 2013-2015 programme of work without additional resources because this would require an additional researcher in the Chemistry Department. The activity has been postponed until the 2016-2019 programme of work.

Dr R. Wielgosz then discussed the 2016-2019 future strategic plans, which will entail a rolling process of adjustments in response to outcomes of the CIPM *ad hoc* WG as well as planned KCs and strategic plans adopted by the various CCs. Dr R. Wielgosz noted that the final draft of the BIPM 2016-2019 programme of work will need to be ready by October 2013, and will therefore require discussion by the CCQM in April 2013. A timetable of activities, based on the assumption that the development of a CCQM strategy document would follow the same deadlines as those set for the BIPM programme of work formulation, was presented. The timetable started with the distribution of a draft CC body strategic template document in April 2012, drafting a costing of BIPM proposed activities based on the CCQM strategy document by April 2013 and finalization of the proposed activities with cost scenarios and impacts by February 2014. This information will be distributed to Member States in April 2014, ready for the 25th meeting of the CGPM in October 2014. He noted that the proposed BIPM programme of work would need to be submitted by this deadline irrespective of the status of CC Strategy documents.

With regards to BIPM gas metrology activities proposed for 2016-2019, Dr R. Wielgosz itemized a current portfolio of O<sub>3</sub>, HCHO, NO, NO<sub>2</sub> and CH<sub>4</sub>/CO<sub>2</sub>, as delineated by the earlier noted comparisons. In addition to these, one additional measurand could be anticipated (such as ammonia) but, in future, no new capability could be envisioned unless it replaced a currently existing facility (i.e., 1 in 1 out policy). Regarding companion OAWG activities, it was envisioned that a limited number of comparisons relevant to Track A goals would be addressed to cover core capabilities consistent with the timetable presented earlier by Dr L. Mackay (section 11.4). In support of the OAWG/BAWG strategy, an effort would be made to extend capabilities to molecules of higher molecular weight.

Dr R. Wielgosz then described the draft template for the CC and CC WG strategy document, highlighting the requested information. He presented the required input, including the terms of reference, stakeholders, current status of activities, future scan, rationale for activities, required Key Comparisons and pilot studies, resource implications and summary table of comparisons, dates and required resources.

Dr R. Kaarls opened the floor for comments and questions regarding the current and future BIPM programme of work as well as the CC template. Dr Z. Mester queried the impact of the fact that the development of the BIPM programme of work may not proceed in synchronization with that of the CCQM WGs and wondered how proposals could be promulgated and supported. Dr R. Wielgosz hoped that all development would ideally occur in parallel but he reiterated that the programme outline was not starting from scratch and that the BIPM programme of work was always discussed and took CCQM WG future planning and requirements into account so that no radical changes were envisioned and a 'business as usual environment' was assumed. Dr R. Kaarls emphasized that a draft programme of work would have to be tabled for consideration by April 2013 and expressed the hope that input from the WGs would be forthcoming in the months ahead.

Dr B. Güttler expressed his pessimism at attempting to look 10 years into the future for CC planning purposes – stating that this is too difficult to predict and thus strategies should be set but these usually change after two years, making the value of this approach very challenging. Dr R. Kaarls agreed that it was very difficult to predict exactly what key comparisons were needed but the CCQM Working Groups already had strategic plans under discussion including repeat frequencies and core competencies and this could be documented. Prof. M. Kühne acknowledged the difficulty of the process but was of the opinion that Directors of Institutes needed to know how resources would be utilized and one of the weak spots in the current planning process is that only four-year periods were being examined. There was a need to look beyond this for proper planning of required resources and a 10-year horizon on this was not too demanding. Dr M. Sega (INRIM) commented that the issue of resource allocation is faced in every sector at every institute, and appreciated the work carried out by the BIPM in coordinating comparisons for NMIs. There was a need to address how to move forward under such conditions and to find the resources to continue; this pointed to a need for continued cooperation among NMIs. Dr W. May expressed his discomfort with a request for final proposals and was not convinced that all of the information that was being asked for should be presented in a detailed manner until feedback from the CIPM clarified this situation. He believed that, for example, it was not necessary to change the current BIPM programme of work at all and that the request for information was coming from reasonably minded individuals who were aware of the limitations of such a process and would be open to discussion. The Directors are only asking for clarification over what amount of their resources are being requested. Dr H. Emons agreed that significant time resources should not be invested into this planning process unless the CCQM has made the process clearer. Dr R. Kaarls agreed that minute details such as those concerning the exact nature of KCs was not possible but that a general direction of the developments was needed. Dr R. Wielgosz reiterated that the BIPM strategic plan would be revisited and asked for some further input into this process. With regard to the meeting schedule, he asked what level of action could be expected to be achieved before the next WG meetings. Dr R. Kaarls requested that WG strategies be discussed at the next meetings of WGs. Dr W. May stated that the BIPM programme of work was already a result of significant strategic planning. Few changes would be needed since sufficient flexibility exists to alter activities within a 5 %-10 % range. Prof. M. Kühne felt that the BIPM had a fairly precise view of what the programme of activities should be for 2013-2015 and that there was a good idea of what 2016-2019 should comprise. However, this may be different in other areas, citing the field of ionizing radiation wherein a shift in technology is foreseen in the future. Long-term strategic thinking is clearly required. Dr W. May reiterated his assertion that the CCQM was already well-positioned in its strategic thinking. Dr H. Emons suggested that the BIPM programme planning for this programme cycle would need to be

decoupled from the CC strategy documentation process. This position was supported by Dr W. May, noting that each WG could proceed with the idea that all such plans would be drawn together into one coherent plan for the entire CCQM in the future.

## 16. COMMENTS ON WRITTEN REPORTS OF RMO ACTIVITIES FROM INTERNATIONAL ORGANIZATIONS IN LIAISON WITH THE CCQM

Dr R. Kaarls noted that all RMOs have tabled their reports and opened the floor for comments or questions. Mr A. Squirrell noted that there was significant activity in Africa and stressed that discussions on comparisons within the RMOs such as APLAC and APMP needed clearer links to ease the work of the accreditation bodies. Dr R. Kaarls noted that the degree of interaction amongst RMOs was highly variable and to some degree depended on how well developed they were. Nevertheless, he encouraged greater activity.

## 17. JOINT COMMITTEE FOR TRACEABILITY IN LABORATORY MEDICINE (JCTLM)

Dr R. Wielgosz presented a brief summary of the status of the Joint Committee for Traceability in Laboratory Medicine (JCTLM) database since the 10th meeting of the JCTLM Executive Committee in December 2011. A revision of the text of the Declaration of Cooperation between the CIPM, IFCC and ILAC impacted appendices III (JCTLM Framework) and IV (participation of organizations in the JCTLM) including corrected references to updated international standards and revised obligations of JCTLM Member Organizations. A change in the Chair of WG 1 was announced with the stepping-down of Dr W. May and his replacement by Dr K. Phinney (NIST) with co-chair Dr H. Schimmel (IRMM). Chairs for WG 2 were reappointed and a review of the terms of reference was undertaken.

The next JCTLM meeting of WGs will be held at the American Association for Clinical Chemistry (AACC) conference in Los Angeles, USA, in July 2012. Two sessions will take place: the annual business meeting and an invitation-only session on HbA1c reference measurement system listings in the JCTLM database. During the second day, discussions relating to the *in vitro* diagnostic (IVD) industry and updating of the IVD Directive from the EU will be undertaken; the implications of this are as yet unknown.

The 10th meeting of the JCTLM Executive Committee discussed how to better link JCTLM and CCQM activities (possibly through shared samples for CCQM comparisons), liaison with the Global Harmonized Task Force on guidelines for traceability, and liaison with ISO TC 212 (to consider updating of ISO 17511 and a possible revision of ISO 15195).

The JCTLM database is operating well and contains 264 Reference Materials, 158 Reference Methods and 63 Reference Measurement Services declared by 7 reference laboratories accredited against ISO 15195 and 17025. Overall, there was a decrease in the number of listed Reference Measurement

Services as a consequence of accreditation requirements, but this decline is expected to be reversed with the participation of additional laboratories from Asia.

A call for nominations for the 2012 review cycle was launched with a closing date of 20 May 2012. Review Cycle 9 for reference materials and methods (WG 1) requires that reference material (RM) producers initially listing CRMs reviewed against ISO 15194:2002 would need to re-submit their nomination using the ISO 15194:2009 template, otherwise the database will specify that these CRMs (about 70 % of the current list) have been evaluated against the 2002 standard. Review Cycle 7 is under way for Reference Measurement services (WG 2).

A feedback form ([JCTLM Database Customer Survey](#)) for users has been posted on the website since March 2012 and customers are able to use this form to comment on the appropriateness of information in the database as well as to identify specific or potential needs and concerns relevant to materials, methods or laboratory services.

Ms H. Parkes opened discussions by asking how the activities of the JCTLM were communicated to stakeholders. Dr R. Wielgosz replied that this is achieved through the WGs. She commented that the May 2012 deadline for receipt of nominations for the current review cycle was too short a timeframe to allow submissions. Dr R. Wielgosz stated that requests were normally sent out in January with the period from May to December left for the review process. Dr W. May noted that he did not see any recognition of the Quality Systems group in the presentation and asked whether it had been disbanded. Dr R. Wielgosz replied that all documents were regularly updated and the group is still active but that a maintenance stage has essentially been reached. Ms H. Parkes expressed her concerns that emerging areas of relevance may not be adequately addressed by the JCTLM, to which both Dr R. Kaarls and Dr R. Wielgosz replied that it is the purview of the JCTLM Executive to consider how to address new areas of relevance to the industry more effectively.

## **18. COMMENTS ON WRITTEN REPORTS FROM INTERNATIONAL ORGANIZATIONS IN LIAISON WITH THE CCQM**

Dr R. Kaarls noted that reports from ILAC and ISO/REMCO had been received. Dr H. Emons announced that during the annual meeting of ISO/REMCO in June 2012 in Vienna, Austria, there will be a special brainstorming session entitled, “Certified Reference Materials and Metrological Traceability” with opportunities to join the brainstorming session via videoconference (contact Dr A. Fajgelj).

Dr S. Westwood (BIPM) complimented Dr H. Emons on the efficient work and activity undertaken at ISO/REMCO and noted that a technical report on traceability and a review of ISO Guide 35 are under way. Additionally, a revision to ISO Guide 30 is now in the voting stage with a deadline of 10 May 2012. He also noted that there was a difference in the definition of Reference Material from that advocated by the VIM and asked if there could be any input from the CCQM to ISO/REMCO to harmonize this term. Dr R. Kaarls commented that although this is a long-standing issue that does not appear to be resolvable, for the time being it is acceptable. Mr A. Squirrell expressed his appreciation on behalf of ILAC to ISO/REMCO and the numerous meetings that have been held which have strengthened links between the ILAC MRA and the CIPM MRA. As this was his last attendance at a CCQM meeting he took the



opportunity to thank the CCQM and all participants on behalf of ILAC for their support. Dr R. Kaarls personally thanked Mr A. Squirrell for his input and help when attending CCQM discussions as well as to ILAC for the extensive and helpful collaborations with the metrological community and the CCQM in particular. He wished Mr A. Squirrell a productive and long future.

## **19. CCQM WORKSHOPS**

Dr R. Kaarls noted that Dr W. May had earlier suggested a workshop or symposium devoted to “Twenty years of the CCQM: progress made, impacts provided, lessons learned and future challenges” for April 2013. He asked Dr W. May to expand on this suggestion. Dr W. May envisioned that the symposium would be a retrospective reporting on what the CIPM MRA has accomplished for the measurement community as well as providing some strategic thinking for the way forward. If done well, any such ideas could perhaps be incorporated into the reports expected from the WG Chairs.

There were no other suggestions for workshops.

## **20. CCQM RESOLUTIONS**

No resolutions were promulgated during the meeting.

## **21. ANY OTHER BUSINESS**

Dr A. Fajgelj reminded the attendees that 13th International Symposium on Biological and Environmental Reference Materials (BERM 13) will be held in Vienna, Austria, on 25-29 June 2012. He noted that at this time there was another week remaining for submission of posters for inclusion in the programme, and invited everyone to attend.

## 22. CLOSURE AND DATE OF NEXT CCQM MEETING

Dr R. Kaarls proposed that the next meeting of the CCQM will take place on 15-19 April 2013 with the KCWG meeting held on 12-13 April 2013. He also noted that meeting dates for the second half of the 2012 had already been arranged among the WGs.

The President of the CCQM, Dr R. Kaarls closed the meeting at 13:00 and thanked everyone for their contributions, reports, feedback, active participation and suggestions all of which help towards providing more effective support for our customers. As this was Dr R. Kaarls last meeting as President of the CCQM, he was particularly appreciative of all the help he had received. He thanked the staff of the BIPM for their support and expressed best wishes for safe travel to all participants.

R.E. Sturgeon,

rapporteur 1 May 2012

revised : 6 August 2012

## DECISIONS AND ACTIONS FROM THE 18TH MEETING OF THE CCQM

1. Suggestions from CCQM participants on a more extensive list of frequently asked questions related to the redefinition of the mole and the other SI units to be sent Dr M. Milton and Prof. I. Mills. (see agenda point 6)
2. Dr M. Milton to draft an easy to understand paper on the “redefinition and realization of the mole”, which would help CCQM representatives to carry out consultations and communicate the proposed change and impact to user communities. (see agenda point 6)
3. Draft document CCQM/12-14 (Draft *mise en pratique* for the mole) to be posted for open access on the BIPM website. (see agenda point 6)
4. A report on the activity of the *ad hoc* Steering Group on Microbial Measurements to be submitted to the April 2013 meeting of the CCQM. (see agenda point 7)
5. The BIPM to review its policy on the publication of contact details of participants in CC and CC Working Group meetings and the distribution and periodic modifications to passwords of working group and CC restricted webpages. (see agenda point 8)
6. CCQM working groups to include agenda points and discussions on future strategies for comparisons in their fields of activity during their autumn 2012 meetings (see agenda point 10.4) and completed strategy templates to be submitted to the CCQM Presidents by 20 November 2012. (see CCQM/12-39)
7. Dr L. Mackay (NMIA) was appointed Chair of the OAWG. (see agenda point 10.2)
8. Dr D. Sin (GLHK), was provisionally appointed Chair of the KCWG, with formal approval pending the decision of the CIPM to modify their rules and permit NMI representatives from Associates of the General Conference to act as CC working group Chairs. (see agenda point 10.3)
9. Dr D. Sin (GLHK), as chair of the KCWG, and Dr M. Sargent (LGC) as Chair of IAWG to complete forms for proposing exceptions to the CIPM MRA traceability rules and submit these for consideration by the CCQM at its next meeting in April 2013. (see agenda point 11.2)
10. The *ad hoc* working group on the KCRV to complete its current set of tasks and report on these at the next meeting of the CCQM in April 2013 together with any further proposed activities which would be considered by the CCQM. (see agenda point 11.8)
11. A CCQM workshop on “Twenty years of the CCQM: progress made, impacts provided, lessons learned and future challenges” will be organized during the April 2013 CCQM meetings at the BIPM headquarters. (see agenda point 19)
12. The next meeting of the CCQM will be held on 15-19 April 2013 with the KCWG meeting to be held on 12-13 April 2013. (see agenda point 22)

## APPENDIX Q1. WORKING DOCUMENTS SUBMITTED TO THE CCQM AT ITS 18TH MEETING

Working documents submitted to the CCQM at its 18th meeting are on restricted access.

Documents restricted to Committee Members can be accessed at the [restricted website](#).

Document

CCQM/

12-01	Draft agenda of the 18th meeting of the CCQM, 1pp
12-02	Timetable of CCQM meetings 13-20 April 2012, 1pp
12-03	Draft agenda for the CCQM Workshop on the Mole, 18 April 2012, 1pp
12-04	IUPAC Technical Report: Metrological Traceability of Measurement Results in Chemistry, 63pp
12-05	IUPAC-ICTNS 2011 meeting report, 18pp
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- 12-38 Draft '*Mise en pratique*' for the mole (replaces CCQM/12-14), 3pp
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