Bureau International des Poids et Mesures

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

Report of the 20th meeting
(10-11 April 2014)
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. Milton
Director BIPM

LIST OF MEMBERS OF THE CONSULTATIVE COMMITTEE FOR AMOUNT OF SUBSTANCE: METROLOGY IN CHEMISTRY

as of 10 April 2014

President

Dr W.E. May, member of the International Committee for Weights and Measures also National Institute of Standards and Technology, NIST, Gaithersburg.

Executive Secretary

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

Members

Centro Nacional de Metrología [CENAM], Querétaro.

D.I. Mendeleyev Institute for Metrology, Rosstandart [VNIIM], St Petersburg.

Danish Fundamental Metrology Ltd [DFM], Lyngby.

Federal Institute for Materials Research and Testing/Bundesanstalt für Material-forschung und –prüfung [BAM] Berlin.

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

Institute for Reference Materials and Measurements [IRMM], Geel.

Institute Nacional de Matrologia, Qualidade e Technologia [INMETRO], Rio de Janeiro.

International Atomic Energy Agency [IAEA], Vienna.

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

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

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.

Laboratory of the Government Chemist [LGC Ltd], Teddington.

National Institute of Metrology [NIM], Beijing.

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

National Measurement Institute, Australia [NMIA], Lindfield.

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

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

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

National Physical Laboratory [NPL], Teddington.

National Research Council of Canada [NRC], Ottawa, Ontario.
Physikalisch-Technische Bundesanstalt [PTB].
Slovak Institute of Metrology/Slovenský Metrologický Ústav [SMU], Bratislava.
State Laboratory [SL], Co. Kildare.
Technical Research Institute of Sweden [SP], Borås.
VSL [VSL], Delft.
Director, International Bureau of Weights and Measures [BIPM], Sèvres.

Observers

Agency for Science, Technology and Research [A*STAR], Singapore.
Bulgarian Institute of Metrology, General Directorate "National Centre of Metrology" [BIM], Sofia.
Central Office of Measures/Glówny Urzad Miar [GUM], Warsaw.
Centro Español de Metrología [CEM], Madrid.
Cooperation on International Traceability in Analytical Chemistry [CITAC], Trappes.
Health Sciences Authority [HSA], Singapore.
Hong Kong Government Laboratory [GLHK], Kowloon.
Hungarian Trade Licensing Office [MKEH], Budapest.
Instituto Português da Qualidade [IPQ], Caparica.
National Institute of Metrology [NIMT], Pathumthani.
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 twentieth meeting at the International Bureau of Weights of Measures (BIPM), Sèvres, on 10-11 April 2014.

The following were present:, H. Andres (METAS), P. Brewer (NPL), R.J.C. Brown (NPL), G. Carroll (SL), D. Craston (LGC Ltd), B.J. de Vos (NMISA), S. Ellison (LGC Ltd), H. Emons (IRMM, ISO/REMCO), G. Favre (LNE), P. Fisicaro (LNE), T. Fujimoto (NMIJ/AIST), A.C. Gören (UME), B. Güttler (PTB), A. Hioki (NMIJ/AIST), E. Hwang (KRISS), H.D. Jensen (DFM), J.M. Juarez-Garcia (CENAM), J.S. Kim (KRISS), Y. Kustikov (VNIIM), H. Li (NIM), L. Locascio (NIST), L. Mackay (NMIA), B. Magnusson (SP), M. Máriássy (SMU), W.E. May (President of the CCQM), J. Meija (NRC), Z. Mester (NRC), Y. Mitani (CENAM), J. Morrow (NIST), U. Panne (BAM), S.R. Park (KRISS), H. Parkes (LGC Ltd), A. M. Rossi (INRIM), M. Sargent (LGC Ltd), M.P. Sassi (INRIM), M. Sega (INRIM), P. Silva (NIMT), A. Steele (NRC), P. Unger (BAM), A. van der Veen (VSL), S. Vaslin-Reimann (LNE), R.L. Watters (NIST), S. Wise (NIST).

Observers: F. Dias (IPQ), T.F. Vicente (CEM), P.K. Gupta (NPLI), W. Kozlowski (GUM), T.K Lee (HSA), P. Silva (NIMT), D. Wai Mei Sin (GLHK), R. Sinweeruthai (NIMT), Z. N. Szilágyi (MKEH).

Invited: M. Buzoianu (INM), P. De Bièvre, P.A. Gatti (INTI), D. K. Koech (KEBS), M. Khan (DRCiM), R. Pawlowicz (University of Columbia), R. Parris (NIST), O. Zakaria (NML-SIRIM).

Also present: S. Maniguet (BIPM), P. Moussay (BIPM), S. Westwood (BIPM), R. Wielgosz (Executive Secretary of the CCQM, BIPM).

Sent regrets: M. Adeogun (NPL), E. Anklam (IRMM), V.S. Da Cunha (INMETRO), A. Fajgelj (IUPAC also IAEA), I. Kuselman (INPL), M.J.T. Milton (Director of the BIPM), R. Kaarls (CIPM, CCQM Past President).

Dr May, the President of the CCQM, officially opened the 20th meeting of the CCQM on the morning of 10 April 2014.

Dr May initiated a round table self-introduction by all participants and observers. He then proceeded to give a summary of the status of the CCQM, detailing the number of members, attendances at working groups, and the number of comparisons being undertaken.

Dr May highlighted some of the challenges currently facing the CC, namely: the increase in the membership of the CC and working groups which is putting pressure on space and resources for meetings; the growing number of comparisons being undertaken and the number of CMC claims requiring review; and the need to ensure continued effective and efficient working practices, particularly to ensure that CMCs remain relevant to actual services delivered by NMIs and DIs, whilst continuing to increase the science focus within the CC. Dr May further reminded participants that the strategic planning frameworks being put in place by the CCQM should help to address many of these issues. He also took the opportunity to remark that CMCs should underpin services delivered by NMIs and DIs on a regular basis.

* For the list of acronyms, click here.

Dr May then listed the many measures that the CCQM had already taken to improve efficiency and effectiveness. This had included, but was not limited to: review of the terms of reference of the CCQM and of the WGs, the closure of WGs that had completed their tasks, the creation of the strategic planning WG, extension of the core comparisons approach to reduce the number of key comparisons required over the next ten years, acceleration of the discussions on the optimal presentation and management of future CMC claims and reviews, reduction in the size of meetings, development of criteria for participation for non-NMI and non-DI organizations in pilot studies, and a review of the CCQM working group structure to better address the CCQM terms of reference and the metrology space that the CC occupies.

Finally, Dr May took the opportunity commemorate the substantial contributions to world metrology and to the CCQM of Dr Laurie Besley, who had recently passed away. Dr May read excerpts from the tribute to Dr Besley that he had delivered on the occasion of a colloquium in Dr Besley's honour. Dr May summed up by quoting a line from the Sydney Morning Herald's obituary: "Besley was that rarest of people, a serious intellectual totally without pretensions".

2. APPOINTMENT OF A RAPPORTEUR

Dr May proposed Dr Brown as rapporteur for the meeting; Dr Brown agreed.

3. APPROVAL OF THE AGENDA

The agenda was approved with the addition of a presentation from Eurachem after point 8. It was noted that comments on the written report from EURAMET TC-MC would be taken at the end of the first day of the meeting since Dr Sega, the chair of the EURAMET TC-MC, could not attend the second day of the meeting.

4. REPORT ON THE NINETEENTH MEETING OF THE CCQM

Dr May thanked Dr Brown, rapporteur for the nineteenth meeting of the CCQM, for producing the meeting report. Dr Wielgosz reported on progress with the decisions and actions arising from the 19th meeting of the CCQM which were included at the end of the report. Some actions were still in progress, namely:

1. Dr W. May to review the activities, structure and name of the CCQM and report back to the 20th Meeting of the CCQM.
2. Dr W. May and Dr R. Kaarls will draft a set of CCQM guidelines for electing CCQM WG chairs and deputy-chairs by November 2013, with the aim of a final draft to be approved at the 20th Meeting of the CCQM.

3. Dr W. May and Dr R. Kaarls will produce a first draft of a document describing the history of the CCQM by the 20th Meeting of the CCQM.
4. Dr R. Kaarls will establish and chair a new group to look at how the CCQM can carry out its activities to address the goals of the CIPM MRA in the most efficient and effective manner. This might include reassessing the current CMC focus and intent; the process of how CMC generation, formatting, presentation and review could be improved in a CCQM context, etc. and report back to the 20th meeting of the CCQM for further discussions.
5. The CCQM agrees that the first two possible traceability exceptions proposed in CCQM/13-11concerning inorganic analytes are to be considered as 'covered by current working practices and CMCs should not be rejected on these grounds', but that further decision on these be delayed until the text of Note 4 of CIPM/2009-24 is reviewed and clarified.
6. The CCQM agrees with the traceability exception related to *delta* scale isotope ratio measurements, and that a list of certified reference materials that constitute accepted references for traceability statements is agreed and maintained by the IAWG. The text of the exception will be modified accordingly by Dr M Sargent for discussion and action by SPWG.
7. Dr Wielgosz, in consultation with Mr A. Henson, will draft a standard letter and form describing the conditions that guest laboratories participating in CCQM pilot studies are required to agree to in order to allow their participation. Agreement and signature of the forms shall be mandatory for guest laboratories to participate in CCQM pilot studies. (Reminder: Guests are not to be listed as members of a CCQM Working Group).
8. Dr May to invite the President of VAMAS to give a presentation at the 20th meeting of the CCQM (April 2014) and attend the SAWG meeting.

In the absence of Dr Kaarls, Dr Wielgosz reported that the first draft of a document describing the history of the CCQM was still in progress. He also mentioned that the VAMAS presentation had been made to the CIPM bureau and a follow-up workshop is planned for 2015.

Except for the above cited two items, Dr Wielgosz noted that all other outstanding items would be covered during the course of the 20th meeting.

5. LEADERSHIP OF THE CCQM WORKING GROUPS

Dr May outlined to the CCQM the current changes in the method by which CIPM members would be elected. He then described the procedures outlined in the new "Guidelines for Selection of CIPM Consultative Committee Presidents" and "Good Practices for Selection of Consultative Committee Working Group Chairpersons and Working Group Deputy Chairpersons" which provides details of the criteria for selection of working group chairs and deputy chairs and the personal qualities which these individuals were expected to have. Dr May stated that it was expected that similar changes would be introduced for the methods by which CC working groups chairs and deputy chairs were appointed, whereby: Following each election of a new CIPM by the CGPM and subsequent appointment/reappointment of CC presidents, WG chairs would be selected by the CCQM President and subsequently the deputy-chairs would be proposed by the WG chairs, with approval by the CC president who would ensure suitable global diversity where possible. The duration of these

appointments would be until the CCQM meeting following the election of a new CIPM, typically a period of four years.

Dr May described the ways in which he would consider rearranging the structure of the CCQM to better recognize its evolving structure should he be re-elected to the CIPM at the forthcoming CGPM meeting and subsequently reappointed as CCQM President. In short, this comprised (in addition to the name change of the CC to the Consultative Committee for Amount of Substance: metrology in chemistry and biology) the possible merger of the Inorganic Analysis WG and the Electrochemical Analysis WG, inclusion of 'chemical' in the name of the Surface Chemical Analysis WG to emphasize that the measurands being addressed should relate to amount of substance, the formation of a new WG on Microbiology to succeed the current steering group, and the evolution of the Bioanalysis WG to form three new WGs on Nucleic Acids, Proteins and Cells.

Following Dr May's presentation, there were extensive discussions of the issues raised. Dr Steele strongly endorsed the approach stating that the split between biology and chemistry made the meetings more focused. Dr Locascio expressed support for the proposed process for the appointment of WG chairs and deputy-chairs as long as there was sufficient global representation amongst these posts. Dr Ellison enquired as to where a shortlist for such selections might come from and whether there would be a nomination process. Dr May stated he would expect to do this informally as part of the process.

At this stage Dr May presented to the CCQM the proposed selections for the WG deputy-chairs. These were – BAWG: Dr S-R. Park, EAWG: Dr S. Seitz, GAWG: Dr P. Brewer, IAWG: Dr P. Fisicaro, KCWG: Dr A. Botha, OAWG: Dr A. Windust, and SAWG: Dr T. Fujimoto. It was also noted that Dr D. Clarke had been proposed as the deputy-chair of the Microbiology steering group, but that this appointment could not be confirmed at this stage since there had not yet been the opportunity to ask Dr D. Clarke whether he was willing to accept the post. Dr Mackay also stated that the appointment would need to be formally agreed with the NMIA.

There was agreement from the CCQM that the selection process and the names proposed for WG deputy-chairs should be supported. Dr May noted that this specific set of appointments would be for approximately one year as a new CIPM will be elected at the 25th CGPM (2014). The new CIPM will appoint CC presidents for the next term. This newly appointed CCQM President would have the authority to extend the mandate of the current CCQM working group chairs and deputy chairs for another term or replace them. Dr May presumes that the CCQM President would always welcome suggestions for WG chair and WG deputy-chair positions.

Dr Steele remarked that in agreeing these processes for the selection of WG chairs and WG deputy-chairs the CCQM was doing what is done in other CCs and warmly commended the selection process and the succession planning issues addressed by the selection of WG deputy-chairs. He also remarked that in his opinion continuity would be greatly prized by the CGPM and the CIPM. Dr May agreed, reiterating he would select WG chairs and would then work with these WG chairs to select the WG deputy-chairs. He also noted that the appointments must have the support of the parent NMIs and Member States involved.

6. UPDATE ON THE CCQM STRATEGIC PLANNING DOCUMENT

Dr Wielgosz gave a brief update on the status of the CCQM Strategic Planning Document. He stated that positive and constructive written comments had been received from 10 NMIs and DIs (PTB, METAS, DFM, INRIM, JV, NPL, SMD, LNE, CEM, NMIJ) in response to the circulation of the draft document and that these comments had been addressed in the latest version of the strategy. In particular, the case studies had now been moved to the Appendix. Version 1.1 of the strategy document had been published online in January 2014 (http://www.bipm.org/utils/en/pdf/CCQM-strategy-document.pdf).

Dr Wielgosz took the opportunity to highlight how the case studies demonstrated that the CCQM was responding to key measurement challenges in the chemistry sector – particularly noting how the CCQM was responding to requirements to:

i) develop and maintain an effective, efficient and manageable programme of comparisons to underpin the broad range of measurement standards and capabilities at NMIs,

ii) deal with new and emerging fields,

iii) develop and manage CMCs in the fields of chemistry and biology.

Dr Wielgosz also commented that it is expected that the parts of the document that detailed forthcoming comparisons would be updated regularly at each WG meeting. This prompted Dr Brown to ask more generally what the timescale for revision of the strategy would be. Dr Wielgosz replied that a major review was scheduled to be finished in four years' time (by the end of October/November 2018) in time for the CGPM meeting of that year. However, a decision has still to be made about whether this is to be to a one or two year process, requiring a start of the review in either April 2016, or April 2015, respectively. Dr May stated that it would be the prerogative of the newly elected CCQM President to make a decision on this point in 2015.

7. BIPM PROGRAMME ON METROLOGY IN CHEMISTRY

Dr Wielgosz presented progress with the BIPM Chemistry Department's programme. He reiterated the three major themes of the programme as:

i) International equivalence of gas standards for air quality and climate change monitoring,

ii) International equivalence for organic primary calibrators,

iii) Support for CCQM, JCTLM and international liaison activities.

Dr Wielgosz proceeded to describe recent comparisons piloted by the BIPM Chemistry Department and a number of collaborations between the BIPM Chemistry Department and other NMIs; the NMIs in question were thanked for their support. Dr May asked how NMIs provide support to the BIPM Chemistry Department. Dr Wielgosz replied that this support was provided through secondments, provision of resources (e.g. standards and reference materials) and in the form of grants to develop measurement capabilities for comparisons to be coordinated by the BIPM.

A number of secondment opportunities to the BIPM Chemistry Department had recently been advertised and Dr Wielgosz announced the secondees who had been successful (from NIM, NPL, METAS and INMETRO) and the projects that they would be working on (two in the gas metrology area and two in the organic purity area).

A number of highlights from the recent work of the BIPM Chemistry Department were then presented. These included a number of key comparisons being coordinated by BIPM and recent work to resolve the differences between reference methods measuring the ozone adsorption cross section. The ozone cross section work had highlighted a 1.8 % discrepancy with currently accepted values and future activities would have to be developed to ensure global uptake of the new reference values. Dr Wielgosz then described CCQM-K82 on methane at ambient levels, a prime example of how a structured programme of repeat comparisons had resulted in a significant decrease in measurement uncertainties and a vast improvement in equivalence. He stressed that this work will have a significant positive impact on global atmospheric monitoring. It was reported that work was under way for a similarly important ambient level key comparison of CO_2 in 2015, coordinated by the BIPM.

Dr Wielgosz outlined progress in organic purity projects being conducted by the BIPM Chemistry Department. In particular, he highlighted the CCQM-K55 series of comparisons of primary references for organic analysis. Dr Wielgosz discussed the use of qNMR in these purity studies and how this approach differed from the current mass balance approach to purity assessment. He also demonstrated that there was no significant relationship between qNMR accuracy and instrument field strength and that this had positive implications for the cost and resource requirements that NMIs would face in procuring such capabilities. He thanked the NMIJ for its assistance in obtaining the commitment from a manufacturer to donate an NMR instrument to the BIPM for work on qNMR in support of future comparisons to be coordinated by the BIPM. He noted that the BIPM is also working with a number of NMIs on a new IUPAC technical report on 'SI Value Assignment of the Purity of organic Compounds for use as Reference Materials and Calibrators'. Dr Westwood, BIPM, is the project chairman. Mention was also made of important progress with the first CCQM comparison on large molecule purity (human C-peptide) which is being jointly coordinated by the BIPM and NIM. A total of 16 NMIs and DIs have expressed interest in participating; currently homogeneity studies have been completed and stability studies are ongoing.

Dr Wielgosz finished by outlining plans for the BIPM chemistry programme for 2016-2019, that were based on the CCQM strategy document. The plans also included two additional projects for which the BIPM was unlikely to have the resources to fund, even though strong support had been expressed by some NMIs for the comparison on organic purity 500 Da to 1000 Da compounds. Dr Wielgosz hoped that additional funds could be found to carry out this work, which was of interest to the BIPM's stakeholders.

8. REPORTS FROM CCQM WORKING GROUPS

8.1 Key Comparisons and CMC Quality (KCWG)

Dr Sin reported on the work of the KCWG. She opened by reiterating the need for face-to-face meetings of the KCWG to resolve problems and to make progress with the CMC review. She then

highlighted the membership of the KCWG and commented in particular on some changes in the EURAMET delegation in 2014. So far in 2014, about 443 CMCs have been reviewed (290) or re-reviewed (153). The largest number of submitted CMCs this year had come from Category 4 - Gases (125) but there has also been a large number from Category 1 – High Purity Chemicals (103) and Category 11 – Food (70). Re-review of the natural gas CMCs in Category 4 this year had been complicated by a lack of agreement on what constituted 'natural gas'. The timetable for the re-review of existing CMCs was stated (2015: some gases, 2016: pure chemicals and sediment, 2017-2018: organic solutions and biological fluids and materials) and the service categories that had not yet undergone re-review were highlighted. As of December 2013 the number of CMCs in the Chemistry and Biology area stood at 5539.

Dr Sin commented on the different approach taken to core competencies by the different WGs. She expressed her desire that in future CMCs should not be submitted on a one-to-one equivalence basis but instead could move towards a regime where a much more flexible scope could be claimed. She postulated that this would significantly reduce the number of CMCs being reviewed and re-reviewed. Such flexible claims could be supported by the type of 'report card' approach being pioneered by the BIPM and the OAWG and the core competencies approaches being developed in the WGs. Dr Sin pointed in particular to a very progressive claim from the SIM in organic purity which simply referred to the purity determination of low-polarity and low molecular weight high-purity chemicals. Whilst this claim required more discussion, this was highlighted as a starting point to a flexible CMC approach, which could be underpinned in future by the OAWG's 'four track' approach to comparisons.

Dr Sin highlighted to the CCQM the current issues to be addressed in order to further improve the review process and make it more effective and efficient. She stated that fixed deadlines were now in place and that, where these were not met, the claims in question would be transferred from the fast-track into the non-fast-track, as was the case for all claims from one RMO in 2014. Dr Sin proposed the possibility of holding, where possible, short face-to-face informal meetings during the WG meetings in the second half of the year to resolve any outstanding issues. She also mentioned an initiative to be trialled in EURAMET and AFRIMETS to move away from e-mail based circulation of spreadsheets for review in favour of adopting a multi-user web-based document. Dr Sin finished by mentioning the continuing requirement for a clear way forward with respect to CMCs in the biology area.

Dr Sargent remarked that consistent and broad participation of NMIs and DIs over a prolonged period of time was of most importance when judging CMC claims of broader scope. Prof. Emons asked whether there was enough time in the KCWG meeting to review all the CMC claims submitted. Dr Sin replied that most claims are dealt with extremely quickly and it is only the complex or controversial claims that take time. Dr Wielgosz interjected that this is what should happen when the KCWG meets i.e. most of the review should have already been completed at the RMO level and the KCWG position is only to review the sub-set of submissions that could not be agreed upon at the RMO level. Dr Brown stated that fixed deadlines and a clear demonstration of full and effective processes by individual RMOs were also a key part towards achieving these aims. Dr May replied that the regional review process was still evolving and the thoroughness of review can change as participants change.

There was a discussion of the requirement for the re-review of CMCs. Dr Mester asked where the requirement for a re-review period of five years came from. Dr Steele countered that the five year re-review period is actually a requirement for the review of quality systems within RMOs, and the five year review period of CMCs was a working practice that had been adopted by the CCQM.

8.2 Steering Group on Microbial Measurements (MBSG)

Dr Morrow reported on the work of the steering group, starting by outlining the membership and the group's current role. She reminded the CCQM of the measurement focus areas of the group: DNA sequence comparability and comparability of whole cell quantitation – measuring in particular cultures of cells and not individual cells. Dr Morrow commented that whilst the group aims to move towards running key comparisons in the future this is currently some way off.

Dr Morrow then outlined the results of a questionnaire aimed at identifying current and planned metrology developments in the field of microbiology at NMIs and DIs and expressed thanks to the organizations that took the time to respond (CENAM, INTI, IRMM, ISP, NIMC, NIST, NMIA, NPL). The results confirmed the interest in DNA marker quantification and also in the quantification of bacterial colonies. Dr Morrow also noted that Chile was the first country with a DI specifically for microbiology, and noted that in 2014 ISP-Chile would conduct a round robin trial for enumeration of *Staphylococcus aureus* and for the detection of *L. monocytogenes* in milk powder. It was also reported that a pilot study on the quantification of colony forming units on solid media had been proposed by the NMIA with a focus on the measurement uncertainty of the measurement.

Dr Morrow then updated the CCQM on the progress of the microbial identity investigative study to establish laboratory comparability for sequencing the 16S rRNA gene. There were six participants and three different sequencing platforms and sample preparation methods had been used. It was noted that the dataset characteristics were dependent on the sequencing platform and DNA preparation method. The results were described as a valuable step in establishing comparability in microbial identity sequencing data. The work has been written up ready to be submitted to a peer-reviewed journal. A follow up study proposal on the microbial sequence analysis of gDNA *S. enterica* strain LT2 has already been proposed. Dr Morrow then listed a number of proposals for future studies that were under consideration by the group including DNA purity assessment in collaboration with the BAWG.

Dr May commended the rapid and substantive progress made by the group, but cautioned that the desire to formulate possible CMCs was not the job of the group and should not provide the drivers for the studies proposed, instead it was the job of the group to arrange comparisons to support areas where NMIs and DIs have interest in putting CMCs forward. He further noted that this is a general problem in the bio-area where there is less experience of the CMC process. Prof. Emons noted that the majority of services requested in microbiology have to do with identity but he doubted whether there was currently the ability to assess traceability and uncertainty for nominal properties. Prof. Emons went on to note that contrary to this the JCTLM has a sub-group for this area and is already assessing a number of claims. Dr May replied that the JCTLM operated differently to the CCQM by first defining requirements and then establishing materials and services which meet these requirements. The CCQM approaches this from the other direction such that NMIs may provide any service they wish and could submit a CMC to support this and the CCQM must develop comparisons to support these claims. Dr Steele observed that the current CMC service categories are evolving as the related science evolves. He added that the object of the review exercise was to review the claims on the basis of the evidence one receives, which need not be limited to or necessarily have to include a CCQM comparison. Dr Locascio reiterated her approval for the approach taken by the group and asked whether the field would develop to address all identification and quantification issues. Dr Morrow replied that the requirements were being driven by the community and the group was

following these closely adding that currently the food area is being addressed and then there would be plans to address clinical problems.

8.3 Working Group on Surface Analysis (SAWG)

Prof. Unger reported on the activities of the SAWG. The results of CCQM-P140 on $CuInGaSe_2$ alloy composition were presented with generally good equivalence observed. The final report is due to be delivered by June 2014 with the likelihood that KRISS will coordinate a follow-on key comparison starting in the second half of 2014. The results of CCQM-P130 on the electron beam micro-analysis (EMPA) of AuCu alloys were presented. Participants measured *k*-ratios, which are the primary result of the EMPA analysis. Dr Unger then proposed a key comparison to follow on from CCQM-P130 where Institutes would convert their measured *k*-ratios into mass concentrations using their own protocols and compare these against a conversion performed by NIST using a single traceable approach (CalcZAF). At this stage Dr Brown interjected that, as no new measurements were proposed only data manipulation, this constituted a conversion of a pilot study to a key comparison after the fact, and that surely this approach could not be allowed. Dr Wielgosz agreed that whilst the data analysis is an important part of the measurement procedure this approach violated the rules of the key comparison process and could not be allowed. Dr May agreed that to allow this would set a difficult precedent. Dr Güttler mentioned that in the past pilot studies have still been permitted as evidence in support of CMC claims. Dr Sargent agreed that the study could not now be converted into a key comparison to support CMC claims for mass concentration determination. Dr Steele mentioned that in other areas key comparisons sometimes use an independent source for the calculation of coefficients. Dr Brown countered that whilst this may be true the main issue in this case was that this was a proposed key comparison where the results were known in advance. Dr May agreed that the CCQM could not allow a key comparison after the fact and that, whilst the additional data reduction comparison was very useful, the exercise must remain as a pilot study. Dr Wielgosz reiterated that the pilot study could still be used to support CMCs if required, and reminded Prof. Unger that for mature measurement areas it was more efficient to move straight to key comparisons. Dr May agreed that it was probably best to only repeat the study as a new key comparison if a large number of participants were in agreement.

Prof. Unger moved on to talk about proposed new studies for the SAWG. The first topic mentioned was a pilot study on the measurement of BET surface of Alumo gels. Dr May stated that he thought BET measurement was a fairly mature area and that this should probably proceed straight to a key comparison. Dr Magnusson cautioned that there may still be equivalence problems between instruments used for these measurements. Dr Unger mentioned that UNIIM, the proposed pilot laboratory, would be asked to provide a letter of support in order to guarantee that a representative from the laboratory would be able to attend SAWG meetings throughout the lifetime of the comparison. Prof. Unger went on to state that in the absence of such a letter BAM would perform the coordination of the study. The first core competency study in the surface analysis area, a repeat of CCQM-K32 'Amount of silicon oxide as a thickness of SiO_2 on Si', was also being planned and there was also some consideration of HfO_2 on Si measurement as a different, but highly relevant, oxide system.

Prof. Unger concluded by mentioning some pilot studies that were in the initial stages of planning. These included many ideas as outputs of projects funded by the European Metrology Research Programme or from the European Union's Seventh Framework Programme. Dr May warned against

the European research agenda leading the direction of future pilot studies. Ms Parkes asked about what service the proposed 'adsorbed DNA in microfluidic channels of PCR devices' study would support. Dr May replied that such a comparison need not map onto services on a one-to-one basis but instead could be indicative of a more general measurement capability.

8.4 Working Group on Bioanalysis (BAWG)

Ms Parkes reported on a number of highlights from the BAWG's recent work; in particular, the CCQM-P154 study on absolute DNA quantification which was a route to traceability for nucleic acid measurement. Good equivalence was observed across the 10 participants, with Ms Parkes commenting that an in-depth discussion of the results within the WG had helped resolve some issues which had improved the equivalence further. It was noted that this was a big advance in metrology for quantifying DNA and it was hoped that NMIs and DIs would soon be able to confidently claim CMCs and disseminate traceability in this area. However, Ms Parkes remarked that it was still unclear as to how the KCRV would be calculated, what form a CMC in this area would take, what the source of traceability would be, and whether CMCs could be claimed without a key comparison. It was noted that discussion on this topic was ongoing. Prof. Emons pointed out that CMC claims based on the DNA study reported would not need the word 'absolute'. Dr Park replied that this was because no calibrant was used. Prof. Emons commented that this usage was associated with the description of the method being used, but should not be a property of the result.

Ms Parkes went on to explain that the BAWG has discussed a number of issues to do with the protein measurement space; in particular, an examination of current activities, gaps in knowledge, future directions and requirements for studies in this area. Issues associated with reference material (RM) and certified reference material (CRM) production as pure materials and as matrix materials were also highlighted, and Ms Parkes described the current and future studies being undertaken by the group which aim to address these challenges.

Ms Parkes proceeded to elaborate on proposed future studies associated with hGH quantification in human serum and quantification of CD34+ cell counts. It was also noted that the CCQM-K110/P113.2 study on Bt63 in GM rice had been suspended since shipping issues had adversely affected the majority of study participants so that the sample integrity could no longer be relied upon. The BAWG had decided to conclude the work as an 'investigative study' which would not be approved for the support of CMC claims. Ms Parkes also reported that the BAWG had actively participated in discussions in the CCU over a new working party to look in more detail at dimensionless quantities.

Ms Parkes then took the opportunity to raise concerns about the CMC process in biology and what were perceived to be the current difficulties in CMCs from the BAWG being accepted into the KCDB. In particular Ms Parkes expressed support for generic CMCs covering a range of analytes and matrices.

As a result of some concerns over the reorganization of the CCQM WGs expressed by Ms Parkes, on behalf of BAWG participants, towards the end of her presentation, Dr May took the opportunity to widen the discussion of this topic, rather than discussing it under original agenda item 12 'New name and proposed structure of the CCQM'. This was because most of the discussion was associated with the biology area. Dr May reiterated the proposed change of name of the CCQM to 'Consultative Committee for Amount of Substance: Metrology in Chemistry and Biology'. In light of some of the

issues raised in Ms Parkes's presentation Dr May emphasized that there may be aspects of the work which the CC undertakes which are outside the strict understanding of amount of substance measurement, and indeed that some non-SI issues would be addressed.

Dr May suggested that, under the new arrangements for the CC, he proposed that the SPWG, KCWG and the CCQM continue to meet at the BIPM every April, but that the other WGs no longer meet at this time and instead hold their meetings at some other time of year and in a different location. As such WGs may only meet once a year. Ms Parkes expressed concern that the resources and numbers of participants from institutes were limited and unless the four new biology-related WGs met in a series arrangement it would be unlikely that they would get enough participants to be viable. Dr Mackay mentioned that the biology area is still very diverse and that there may be some benefit in a bigger WG continuing and addressing all these sub-areas. Dr May stated that once WG chairs had been appointed they could organize the meetings in any way they saw fit and this certainty did not preclude the series arrangement suggested. Prof. Emons commented that whilst the structure of the chemistry-based WGs was already clear the arrangement of WGs in the biology area was less so and that other groupings could have been selected. Dr May replied that these are based on subject areas where distinct activity at NMIs and DIs already exists. Dr Locascio expressed support for the idea of sequential meetings of the proposed new biology WGs whilst the evolution of the structure takes place. Dr Sassi commented that the proposed new biology WGs must still continue to address fundamental metrology and focus on traceability and the identity of measurands, especially where traceability is to units other than the mole.

Continuing the discussion on the proposed biology WGs, Dr Morrow acknowledged the issues of resource limitation and stated that it is something that is already being taken into account. Dr Mester added that current resources are a closed system and so it is very important for each of these new WGs to be clear in its aims and not to replicate work or get involved in areas that are already mature. Dr Watters commented that initially the chemistry activity in NMIs was small when the CCQM first started but it has now grown into a very large endeavour, and that the same could well happen in the biology area. Dr Steele made the point that structure and logistics were different issues and that whilst it is easier to achieve progress with smaller groups the CCQM must be cautious about expanding too rapidly. He also made the point that it would have been possible to further split the chemistry WGs into two, namely 'physical chemistry' and 'inorganic/organic' groupings, in a similar fashion to the way the CCRI is arranged, although he stated that he was not necessarily in favour of this since the plenary meeting could then become too exclusive.

Dr Mitani asked what effect the proposed increase in the number of working groups in biology would have on the metrology programme at the BIPM. Dr Wielgosz replied that the BIPM programme was formulated based on user needs, the CCQM strategy and advice from the SPWG, rather than organizational changes to the CC.

8.5 Working Group on Electrochemical Analysis (EAWG)

Dr Mariassy reported the results of a number of CCQM and RMO comparisons. In particular the arrangements for the linking study of amount content of oxidants expressed as potassium dichromate in CCQM-K96.1 were presented and the process for the generation of the link between NIM and KRISS was explained in detail. The results from APMP.QM-K19/P25 for the measurement of borate buffer were presented. It was noted that many of the secondary measurements agreed extremely well with the primary measurements made using the Harned Cell. Dr Mariassy also presented the good

equivalence observed during the SIM.QM-K92 study on electrolytic conductivity which linked to CCQM-K92.

It was reported that the deadline for the CCQM-K99 comparison on the pH of physiological phosphate buffer had been extended following delivery problems experienced by 5 of the 19 participants. Prof. Emons asked whether the delays in transport would compromise the stability of the samples. Dr Mariassy replied that the stability was being monitored throughout the lifetime of the study by PTB and so this was under control but was not expected to be a problem as long as the bottles were not opened in transit.

The results were presented for the CCQM-P142 study on the conductivity of seawater. There were 18 participants including oceanographic laboratories. It was noted that the inconsistencies observed decreased with temperature and increased with conductance. These differences were larger than expected. The salinometers operated by the oceanographic laboratories, which are traceable to the practical salinity scale, showed extraordinarily good agreement with the reference value and with much lower uncertainties than produced by the primary conductance measurements at the NMIs.

Dr Mariassy then went on to report on the resolution to the linking of conductivity study COOMET.QM-K36 with CCQM-K36. The linking procedure used was described in document COOMET-R14 and it had been the opinion of the EAWG that the linking uncertainty was too small since it only included repeatability contributions. The matter was referred to the BIPM Director's Advisory Group on Uncertainties where no significant issues with the procedure were found. Dr Mariassy reported that the report had therefore been accepted but with the comment that future implementations of such linking comparisons needed further discussion.

Finally, Dr Mariassy reported on the outcome of CCQM-P37.2 on the international comparison of Ag/AgCl electrodes for pH measurement being piloted by the NPL. The study tested NMIs' ability to prepare Ag/AgCl reference electrodes with the aim of improving the future repeatability and equivalence of primary pH measurements. The results suggested that the differences in preparation of these electrodes at different institutes may not be the major course of variability in Harned Cell measurement. Dr Brown commented that the differential performance of electrodes in different solutions, especially when the electrodes depart significantly from ideality, may still be a contributing factor to lack of equivalence since this was not fully tested by the comparison.

8.6 Working Group on Organic Analysis (OAWG)

Dr Mackay reported on the work of the group reiterating the four track approach to comparison work of the OAWG. First the Track A comparison CCQM-K55.c on the purity of L-Valine was presented showing encouraging equivalence across the majority of the 20 NMIs and DIs taking part. This was followed by a discussion of the CCQM-P150 qNMR pilot study which in particular was focussed on better understanding the sensitivities of results to sample preparation, data acquisition and data processing methods. Again, the results showed good equivalence across the vast majority of participants. Dr May expressed his wish that this study would be written up for publication in a peer-reviewed journal.

Dr Mackay then introduced the Track C comparison on the purity of avermectin B1a. It was noted that the results would need normalization back to dry mass owing to significant uptake of water after the sample bottles had been opened. Dr Mackay also reported that there had been significant discussion about the measurand in the study because of the large number of diastereomers present.

Dr Mackay reminded the CCQM of the wide ranging results from the first Track A 'Matrix' key comparison on pesticides in tea and then introduced a follow-up comparison that was planned on PAHs in tea so that some laboratories would be able to demonstrate better equivalence in the future.

Dr Mackay reported that there was significant discussion on the follow-up studies K6.2, 11.2 and 12.2. Whilst the results from cholesterol had been good, the comparison of glucose and creatinine had once again caused problems. NIST was the linking laboratory to the previous study, however NIST, PTB and KRISS were asked to re-perform the measurements to provide additional linkage information. These laboratories agreed well for creatinine, but there was significant disagreement for glucose. Further investigation had been carried out to investigate the effect of sample equilibration time for the isotopic internal standards and the effect of lipid content, and both of these aspects demonstrated the stability of the samples over time so this had thus been discounted as a possible reason for lack of equivalence. Dr Mackay reminded the CCQM that comparisons with this suite of key analytes had been continuing for 14 years at the CCQM and RMO level and that the links back to laboratories taking part in comparisons previously had assumed that the performance of these laboratories had remained constant. It was now clear, concluded Dr Mackay, that linking back over such long periods was meaningless and that these comparisons would have to be treated as new, separate studies.

Dr Mackay then referred to the excellent work done by the BIPM in producing 'report cards' for NMIs' and DIs' performance in purity key comparisons, making it very easy to gauge competence and experience of the different laboratories. The presentation was concluded with a brief mention of future studies, including CCQM-K55.d on the purity of folic acid, CCQM-K102/P138 on brominated flame retardants in sediment, CCQM-K109/P148 on high-polarity analytes in biological matrix (measurands: urea and uric acid in serum), and a planned track C comparison on pharmaceuticals in surface water.

Opening the discussion on the presentation, Prof. Emons expressed concern that, in relation to the avermectin B1comparsion, dry mass was an operationally defined parameter and asked which protocol would be used for its measurement. Dr Wielgosz interjected that the measurements had already been carried out and this was not a question of choosing a conventional method of drying, but rather the water data would be ignored since the comparison failed on this and could not be used to compare capabilities for measuring water in purity determinations. Dr Watters asked how the KCRV was being determined for the purity comparisons. Dr Westwood stated that for the K55 series this was currently based on the consensus measurement of the four impurity types, although he also remarked that in future qNMR results might be incorporated into the calculation since it was able to measure the purity directly. Returning to the K6.2, 11.2 and 12.2 studies, Dr Watters expressed concern that the linking had failed and this reflected badly on the shelf life of CMCs. Dr May replied that this was still a relatively small dataset and more information was needed to truly understand the shelf life of CMCs. Dr Brown added that the validity of CMCs was less likely to be simply related to elapsed time but would instead be more significantly affected by stage boundary events such as changes in staff or equipment, which could happen on any timescale.

8.7 Working Group on Inorganic Analysis (IAWG)

Dr Sargent gave a brief review of the activities of the IAWG. In particular he presented CCQM-K106/P128.1 on Pb, As and Hg in cosmetics where there had generally been extremely good equivalence. However, Dr Sargent noted that the only participant not registered for the

key comparison had been a Brazilian laboratory which intended to use neutron activation analysis (NAA) but which had not received the sample because it had been blocked by Brazilian customs. As a result the pilot study part of the comparison had been cancelled. The results of CCQM-K107/P146 on elements and Se speciation in human serum were also presented. Again, equivalence was generally very good, but the agreement between laboratories for the Se speciation measurement was not as good as the measurements of the element. The results from CCQM-K98/P134 on isotopic composition of Pb in solution and in bronze also showed extremely good agreement across a large number of the isotope ratios requiring measurement. There was also very good equivalence for the optional measurement of the molar mass of Pb in bronze.

Dr Sargent then introduced a number of new key comparisons and pilot studies which had been agreed during 2013-2014. CCQM-K124 on trace elements and chromium speciation in drinking water was proposed as a benchmarking exercise where sample preparation is simple and all those NMIs and DIs with relevant CMCs were being encouraged to participate. Dr Sargent explained that the original UNIIM proposal for a comparison on nitrogen factors had been reworded to be consistent with CCQM aims, such that it now required the measurement of mass fraction of nitrogen in glycine and milk powder. It was noted that CCQM-P160 on the measurement of isotope ratios and molar mass measurements of Si isotopes in isotopically enriched silicon was related to the Avogadro project but would also be useful for other areas of study. Dr Sargent then presented the IAWG 5-year plan for key comparisons and pilot studies, also highlighting that isotope *delta* values did not fit into any of the current service categories.

Dr Sargent concluded by stating that the number of actively participating institutes in the IAWG was still growing and now included all RMOs. Further, he proffered the opinion that two meetings a year was cost-effective in terms of ensuring that work progresses steadily and so allowing a wide range of technical presentations and discussions. Dr May replied that since the cost of meetings had to be supported by NMI directors it was important to have their support for the frequency of meetings proposed. Dr Steele agreed with Dr May and stated that he thought most NMI directors would also agree, and encouraged much more work to go on between meetings. Dr Steele also expressed the opinion that, whilst international collaboration is at the heart of a successful metrology system, from a budgetary point of view it was not the responsibility of mature laboratories to train newer laboratories. Dr Güttler agreed that it was important for all laboratories to derive the maximum benefit from participation in studies and from the results that are obtained.

Dr Ellison raised a question about whether k=1 or k=2 uncertainties should be plotted on results graphs. Dr Wielgosz replied that the rules on the KCDB were quite clear: results must be plotted as k=1 and degrees of equivalence as k=2. Dr Sargent insisted that users only looked at the result graph and here k=2 was the most useful plot. Dr Ellison added that was also a question of the best style of presentation for meetings. Dr May reiterated that the KCDB rules were clear but that for the sake of presentations he would leave it to WG chairs as how to present the data.

In concluding the discussions on the presentation Dr May highlighted three issues that he wanted the SPWG to consider going forward:

i) appropriate mechanisms and guidelines to deal with guest laboratories that are not NMIs and DIs being invited to, and participating in, WG meetings.

ii) unified nomenclature for the core comparison approaches being undertaken by different WGs to avoid confusion when communicating outside the CCQM.

iii) guidelines for the conduct of linking comparison studies, in particular how closely in time they must follow the original comparison.

8.8 Working Group on Gas Analysis (GAWG)

Dr Kim reported on the work of the GAWG, in particular one final report and six progress reports on comparison studies. Following extensive discussions about the uncertainty of the KCRV, the CCQM-K93 study on ethanol in nitrogen had now been completed with good equivalence shown across participants at the 120 µmol/mol level. Dr Kim then presented the results of CCQM-K82 on ambient level CH_4, coordinated by the BIPM. As mentioned by Dr Wielgosz earlier, in his presentation of the BIPM Chemistry Department activities, the equivalence observed was extremely good and a significant improvement when compared to CCQM-P41 in 2003. Dr May remarked that the value of one of the standards from the World Meteorological Organization (WMO) laboratory taking part, NOAA, disagreed slightly with the results of the other NMIs and DIs and asked what the significance of this was. Dr Kim replied that the NOAA had found the comparison useful and were able to investigate and resolve the source of this bias. He further explained that the WMO scale was based on stability and measurements in their networks that monitored changes from year to year. The WMO could decide to make a change to this scale (as had previously been done) and data in the WMO databank could be altered if required. Dr Brewer added that another advantage of equivalence with the WMO scale was that certified reference materials could also be supplied by NMIs, which would help alleviate the growing supply and demand mismatch for such materials for global monitoring purposes.

Dr Kim then presented preliminary results from CCQM-K101 on oxygen in nitrogen where results were generally good, and an update on CCQM-K90 on formaldehyde in nitrogen where there had been some delays caused by cylinder transportation legislation, which NIST and the BIPM were currently trying to resolve. Dr Kim then expounded on the proposed scheme for the first GAWG core comparison, CCQM-K111 on propane in nitrogen, which would be followed closely by RMO linking studies. This led into a wider presentation of the GAWG strategy for the measurement of core comparison mixtures and also for purity assessment. The documentation to underpin this approach is at the draft stage and will be circulated to the GAWG ahead of the meeting in the second half of 2014.

A number of updates on future studies were then given including: CCQM-K112 on biogas, CCQM-K113 on noble gas mixtures, CCQM-K116 on water in nitrogen, CCQM-K117 on ammonia in nitrogen, CCQM-K118 on natural gas, CCQM-K119 on LPG and CCQM-K120, a repeat study on CO_2 in air at ambient levels. Dr Kim concluded by presenting the outline programme for GAWG comparisons to 2018.

In reference to CCQM-K117, Dr May enquired whether there was any indication that this was in fact a repeat of CCQM-K46. Dr Wielgosz replied that there was no standardized nomenclature or common usage and practice for this in the CCQM at present. Dr Sin agreed that the KCWG did not always know which comparisons were repeat studies. Dr van der Veen mentioned that the natural gas and ammonia comparisons are not strictly repeats since the concentrations being measured are different to those in the original studies. Dr Wielgosz reminded the CCQM of the facility to archive historical comparisons, which no longer support CMCs, into an appendix on the KCDB, but noted that the CCQM had not yet used it. He wondered whether the time had come to start using the facility. Concluding the discussion Dr May expressed the opinion that there ought to be an agreed

system of nomenclature for the numbering of CCQM studies to unambiguously distinguish between follow-on comparisons, repeat comparisons and linking comparisons and also guidance as to what constituted a follow-on or repeat study. He asked the KCWG to look into this issue.

9. EURACHEM PRESENTATION

Dr Magnusson introduced the Eurachem organization and described its aims and operation. In particular, he emphasized that the primary audience for Eurachem's activities were laboratories performing analytical measurement and accreditation bodies and related organizations. In particular, Dr Magnusson highlighted the workshops run by Eurachem and the best practice guidance documents, often produced in cooperation with the Cooperation on International Traceability in Analytical Chemistry (CITAC). Some information was given about the number of times these guides had been downloaded, and also the impact of these guides in terms of the number of ISO documents containing reference to Eurachem guides. Dr Magnusson noted that Eurachem was currently preparing a guide on qualitative analysis.

Dr May asked whether any consideration had been given by Eurachem as to whether to address similar measurement issues in the biological area. Dr Magnusson replied that this was subject to ongoing discussion within Eurachem but that currently the expertise was lacking. He noted that Eurachem would welcome input in this area from new members. Considering the demonstrable impact demonstrated by Eurachem in terms of downloads of guides and references to guides in published standards Dr May asked WG chairs together with the SPWG to propose methods and activities to measure the impact that CCQM is having in the end user community. Dr Wielgosz stated that he expected the performance of routine laboratories to be influenced by the appropriate use of measurement standards, documentary standards and quality systems including accreditation. The challenge would then be to differentiate between the impact of measurement standards and the other influencing factors. However, he added that there were clearly sectors where the impact of CCQM activities could be more easily measured, for example in the area of air quality and clinical chemistry. Prof. Emons agreed that we must all look to improve our impact in order to justify funding.

10. TRACEABILITY IN THE CIPM MRA (AND CCQM LIST OF EXCEPTIONS)

Dr Wielgosz introduced document CIPM/2009-24 'Traceability in the CIPM MRA'. He reiterated that Note 4 was the most relevant to the CCQM and had led to problems in meeting the requirements of document CIPM/2009-24. This had required the drafting of a 'statement on working practices for traceability and elemental calibration solutions' (CCQM/14-06) to avoid these IAWG cases becoming traceability exceptions. Dr Wielgosz reminded the CCQM that there was an outstanding action from the last meeting for the CCQM to propose to the CIPM an alteration to Note 4 to cover these situations, *i.e.* when in-house purity capabilities exist and are used in the preparation of elemental calibration solutions but are not being provided as a service to external customers and hence not meeting one of the conditions for a CMC.

The initial suggestion was to replace "…by applying their own measurement capabilities as described and recognized within published CMCs." with "…by applying their own peer-reviewed measurement capabilities." or "…by applying their own peer-acknowledged measurement capabilities."

A long and complex discussion ensued about the possible changes that could be made to Note 4 to make it acceptable to the CCQM whilst also providing an acceptable level of rigour in terms of quality. Many of the arguments centred on the nuances of the English language, in particular what the term 'peer-reviewed' actually required. It was generally accepted that in this instance it should not mean a visit to an NMI site to assess the measurement capability. Dr Mackay suggested that many NMIs and DIs should be participating in CCQM comparisons on purity, for example, to prove their capability, even if they then did not subsequently claim purity. Dr Steele asked why, if the purity capability existed at an NMI or DI, it was not offered as a service. Dr May responded that the chemical measurement community differed from the physical measurement community in that it often offered its services for matrix materials requested by users, for which the measurement challenge was different to that of highly accurate calibration materials. He further commented that it would be undesirable to fill the KCDB with CMCs which were not disseminated as services. Dr Silva added that a peer-acknowledgement process would be quicker and more efficient than going through the full CMC process. Dr Mester, whilst agreeing with the spirit of the change, was concerned that the introduction of a peer-review component would cause additional work. Ms Li added that customers might find the term 'peer-acknowledged' somewhat confusing. Dr Brown expressed the opinion that proof of the capability actually comes from the resultant CMCs for elemental calibration solutions that are submitted based on performance in comparisons. He added that in a case where the criterion was either 'peer-reviewed' or 'peer-acknowledged' the weaker requirement, in this case 'peer-acknowledged', would be the limiting factor and suggested that only this requirement should be included.

A further discussion subsequently took place over the requirement for the reference to CRMs which occurred earlier in the note. This was because it was currently unclear whether the proposed change to the text at the end of the paragraph referred just to the high purity chemical reference materials or also to other CRMs. Dr Mariassy suggested that a change in the ordering of the clauses would remove this ambiguity. Dr May wondered whether the mention of CRM was needed at all since such a material was most likely to originate from an NMI or DI and be the result of a tested CMC claim on the KCDB. Dr van der Veen agreed that the CRM part of the clause was redundant. Dr Mitani and Dr Gören both felt the changes suggested were necessary otherwise it could prove a barrier to new NMIs and DIs obtaining CMCs in future.

In the absence of full agreement on the text of any proposed change it was decided that the discussion on the exact wording of a proposed change should be continued in the SPWG and it should report back to the CCQM with a draft replacement for Note 4 in due course.

The discussion then proceeded to the proposed traceability exception from the IAWG on isotope ratio delta values. Dr Wielgosz introduced the proposed text (CCQM/14-18). Dr Sargent noted that the recent publication of the IUPAC report on international reference materials for isotope ratio analysis had allowed this document to be referenced as part of the traceability exception. The proposed exception was accepted by the CCQM.

11. REPORT FROM THE *AD HOC* WG LOOKING THE IMPROVEMENT OF CMC GENERATION IN A CCQM CONTEXT

In the absence of Dr Kaarls, Dr Wielgosz presented information on this topic. He opened by providing data on visits to the KCDB that had recently been collected via a pop-in questionnaire on the KCDB. This showed that nearly 25 % of all "visitors" to the data on comparisons, and nearly 35 % of all "visitors" to the data on capabilities are from outside the NMI community. This confirms that the CIPM MRA has growing visibility amongst its wider community of users. Calibration and testing laboratories constituted the largest non-NMI/DI percentage looking at key and supplementary comparisons (7 %), and CMCs (15 %). It was also noted that data was related to visitors to the KCDB as a whole, but that no specific information on visits related to Chemistry/Biology information in the database was available.

Dr Wielgosz then delivered a preliminary analysis of answers to the CCQM questionnaire on the CMC process in the CIPM MRA. Out of a maximum possible number of respondents of about 70 there had been 43 replies, although 10 of these were partial replies or did not include a named contact or NMI identity, and were not analysed further. There was a clear majority of respondents who found CMCs and key comparisons useful and felt that they should continue, albeit with many of those who provided additional comments thinking that changes to operation were required, especially for CMCs. There was also very clear support for a move toward accepting CMCs with a flexible scope, although the comments provided suggested that it was very important that the usefulness of the CMC entries was maintained. There were conflicting responses regarding the information provided on CMCs with many respondents thinking that their customers were the primary target for CMCs whilst at the same time thinking that the database was far too complicated for customers and that few customers referred to it. There was, however, agreement that the CMCs should link to catalogued measurement services. Many respondents felt that the current level of information supplied with CMCs to demonstrate that they were linked to measurement services was sufficient with no clear consensus as to what extra documentation should be offered. There was general agreement that the efficiency of the CMC process would be improved by having fewer CMCs and also making accreditation reports and on-site peer review documentation available for the CMC review process. It was also generally felt that some changes to the CMC template would be useful to accommodate biology and other new areas where CMCs might be claimed. It was felt by respondents that clearer documentation would help those who were new to the CMC process.

Dr Wielgosz emphasized that this analysis was preliminary and that the *ad hoc* WG would have firmer conclusions to present once a more detailed analysis of the data had been carried out. Dr Goren expressed the opinion that customers found the KCDB too complicated. Dr Watters replied that the chemical CMCs where generally easier to find and understand than that for other areas. He added that it was helpful that relative uncertainties are presented. Dr May mentioned that there should also be a strong role for local RMOs in assisting those NMIs and DIs who were new to the CMC process. Dr May mentioned that in other areas of metrology the development of NMIs and DIs takes place mostly within the relevant RMO, and that CCQM had been rather an exception to this general principle. Dr May stated that in the future the CCQM may wish to consider if it needs to undertake less training of newer NMIs and DIs.

Dr Watters, responding to the information from Dr Wielgosz that there would be a further opportunity to gather further information from a 'pop-in' window on the KCDB, asked who would specify these questions. Dr Watters continued by mentioning that, in particular, the pop-in did not

collect information on why the user had visited and whether at the end of their visit they had been successful in getting the information they required. Dr Wielgosz said that the process was being managed by the JCRB but that the CCQM was welcome to make suggestions about what questions should be included. Dr Thomas gave further information on the process and topics that had been discussed in the JCRB. She mentioned that the JCRB had suggested two further enquires along the lines of those suggested by Dr Watters, but it was likely that this would not be for a few months to avoid overly burdening visitors to the KCDB. Dr May supported the proposals stating that, in order to take positive action, the CCQM needs as much information from the chemical measurement community as possible.

12. OUTCOME OF WORKSHOP ON THE REDEFINTION OF THE MOLE AND THE DRAFT MISE-EN-PRATIQUE

Dr Wielgosz thanked those who participated in the workshop on 9 April 2014, the day prior to the start of the twentieth CCQM. There were a number of informative presentations given, expressing the various opinions on this topic. There had also been a lively round table discussion of the issues surrounding redefinition and the implications for metrology in chemistry. Dr Wielgosz stated that a paper would be written up to summarize the outcomes of the workshop and that these outputs would be taken forward by the CCQM *ad hoc* group on the redefinition of the mole and also by the new IUPAC project group established to examine the redefinition. Dr Wielgosz also stated that members of CCQM would be represented on the new IUPAC project group. Dr De Bièvre praised the workshop held by the CCQM as a good opportunity to discuss fundamental issues. He stated, however, that he did not think that the original requirement set by the IUPAC Interdivisional Committee on Terminology, Nomenclature and Symbols prior to redefinition of the mole had yet been met, although he did recognize that whilst ISO handles definitions of quantities the definition of units was the responsibility of the CIPM. Dr De Bièvre also encouraged the CCQM to work closely with the CCU to ensure they were up to date with the current wording of any new definition.

13. DATE(S) FOR THE NEXT MEETINGS OF THE CCQM

Dr May stated that the next meeting of the CCQM and associated working groups would take place during the period from 15-24 April 2015 at the BIPM. The exact format and timings of the meetings would be decided in due course.

13.1 Coordination of CCQM WG meetings to be held during 2nd half of 2014

Dr Fujimoto presented plans for the CCQM WG meetings during the 2nd half of 2014. He stated that these would take place in Tsukuba, Japan, from 13-16 October 2014. In addition to the WG meetings there would be a tour of the NMIJ laboratories and a workshop on the ‘Impact of Chemical Analysis and Reference Materials in Regulation on Food’. Dr Fujimoto stated that more information was

available on the dedicated website that had been set up for the meetings: https://www.nmij.jp/CCQM2014.

Dr May stated that he did not think there needed to be a SPWG meeting during these WG meetings.

14. CC DIRECTORY AND MEMBERSHIP OF CCQM WORKING GROUPS

Dr Wielgosz presented the current membership list of the CCQM. He stated that there was a need to make sure this list was up to date since there were currently some ambiguities. Dr Wielgosz reported that the membership of the CCQM continues to increase and that, whilst this was consistent with the policy of inclusivity upheld by the CCQM, because of the limitations on resources to hold meetings at the BIPM and elsewhere, this increase will eventually cause problems for hosting meetings. In particular Dr Wielgosz requested that the CCQM consider a policy on the attendance of guests and on standing invitations for guest participation in working groups.

In reply, Dr May stated that he thought the CCQM would in the near future need to make the requirements for membership more stringent. He noted that, currently, any NMI or DI with a programme in chemical metrology is welcome to participate, but as the number of NMIs and DIs entering the field around the world increased this could not continue without a limit.

Dr Brown drew parallels with RMO chemical metrology meetings, especially the EURAMET TC-MC meetings recently held at the NPL and LGC in Teddington where the proliferation of DIs in the chemical metrology field has significantly increased attendance and was starting to make it difficult to accommodate everyone who wanted to attend. Dr Watters commented that because of the wide chemical space covered by the CCQM there would always be a lot of participants wishing to attend.

The CCQM was supportive of the policy of inclusivity when operating meetings for the time being, whilst also recognizing that the capacity of the BIPM and other institutes to accommodate increasing numbers of delegates is not unlimited and solutions to this would need to be found in the future.

Dr Wielgosz then introduced the latest draft of form CCQM-F-01 “Participation in CCQM Pilot Studies: Request Form for Guest Laboratories”. This was a new form that had been produced and approved by the CIPM. The form, it was stated, would need to be completed and approved by the CCQM President prior to approval for any guest laboratory to participate in a CCQM pilot study. Dr Wielgosz reminded the CCQM of the background to the need for such a form for pilot studies, and also stated that participation in key comparisons was not open to guest laboratories. Dr Wielgosz explained that once the form is completed it would be forwarded to the CCQM President whose decision on participation would be final with no right of appeal.

Dr Güttler asked whether a distinction would be made between commercial companies and universities, where the motivation to use the result of a comparison for commercial gain would be substantially different. Dr May replied that such matters would be taken into consideration when the president made a decision about participation. Dr May considered that there was also an issue for equitability to be considered: if one company or university was allowed to participate it would be difficult to justify the non-participation of others. Dr Ellison remarked that some NMIs and DIs already operated as commercial companies so using this distinction to deliberate on participation must be treated with caution. He went on to mention that universities may also gain a financial

advantage by participation in CCQM studies because it could enable them to be more successful in winning grant funding subsequently.

In the context of the above discussion, a separate discussion ensued regarding DIs. Dr Güttler mentioned that perhaps the CIPM should think about these issues more widely, i.e. which entities should be allowed to become a DI, for instance whether this should ever be a possibility for commercial companies. Dr May stated that the decision about designation was one for each Member State of the Metre Convention. Dr Steele added that the JCRB has produced a set of guidelines for Member States when considering the designation of laboratories within their country.

Prof. Unger questioned the order of sections on the form, but Dr Wielgosz assured the CCQM that when the form was used in practice the sections were in a logical order. The CCQM approved the form and Dr Wielgosz stated that the final form, with minor editorial changes, would be approved as a CCQM document and published on the publicly available CCQM webpage.

15. COMMENTS ON WRITTEN REPORTS FROM RMOS

Ms Parkes remarked that she welcomed the inclusivity of the EURAMET TC-MC meetings in including issues relevant to bio-metrology even though the EURAMET TC-MC did not have a formal bioanalysis sub-committee. Dr Sargent noted that RMOs were invited to give a presentation at the IAWG summarizing relevant regional activities. Dr Brown added that this was also the case in most other WGs. Dr May stated that in future he would expect TC-MC RMO chairs to say a few words on major developments within in chemical metrology in their RMOs to complement their written reports posted on the CCQM website.

16. COMMENTS ON WRITTEN REPORTS FROM THE JCTLM

Dr Wielgosz reported briefly on the recent work of the JCTLM and in particular the outcome from the recent Stakeholder's meeting, where the successes of the JCTLM Database had been recognized, and the area of non-SI traceable measurands targeted as a focus for the future. He noted that the JCTLM review teams currently had vacancies in the areas of drugs, non-electrolyte metals, non-peptide hormones, proteins and vitamins, and urged those with expertise in these areas to participate in the review teams and nominate themselves by completing the relevant form available from the JCTLM webpages. Dr Wielgosz also mentioned that future changes to the funding of the JCTLM may impact its future structure and the scope of its work. Dr May asked whether, similarly to the KCDB exercise, it would also be possible to get information about who visits the JCTLM database. Dr Wielgosz stated that he would look into this request.

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

No comments were forthcoming.

17.1 Presentation from the Joint SCOR/IAPSO/IAPWS Committee on the Thermophysical Properties of Seawater (JCS)

Prof. Pawlowicz gave an interesting update on the activities of the Joint Committee on Seawater. In particular, Prof. Pawlowicz mentioned the basic measurement requirements of oceanography, namely measurements of temperature, pressure (as a surrogate for depth measurement), salinity (as a bulk measure of the amount of dissolved inorganic matter in seawater), carbonate system parameters including pH, and macronutrients and dissolved oxygen. Prof. Pawlowicz explained the oceanographic community's desire to make all of these measurements SI traceable rather than dependent on a scale. He went on to describe current seawater standards, in particular TEOS-10, and the formation of the Joint Committee on the Properties of Seawater (JCS) whose remit included the maintenance and update of TEOS-10 and related standards. There is already substantial CC involvement in the JCS subgroups on salinity, density, seawater pH and moist air relative humidity. It was noted for conductivity ratio (as an SI traceable measurement comparable to salinity) that the SI traceable measurement uncertainty was an order of magnitude higher that the JCS requirement for this parameter. It was also noted that the definition and measurement of pH in seawater was currently a significant challenge. Prof. Pawlowicz then presented progress with the BIPM-IAPWS draft roadmap for cooperation. Salinity and pH were identified as disciplines where the CCQM was already making a significant contribution. Further, he listed the ways in which NMIs could help ocean scientists. Aside from salinity and pH there was interest in a review of the solubility of gases in seawater and their effects on density and other properties, and also in high accuracy traceable measurement of seawater composition.

Dr Güttler stated that providing traceable conductivity measurements which provide a measurement uncertainty equivalent to current salinity measurements is much more complicated than first thought. He also mentioned that density might be an alternative surrogate measurand which could provide a solution. Prof. Pawlowicz agreed, remarking that density can be measured with such a low uncertainty that the limiting factor then becomes the stability of the standard seawater materials. Prof. Pawlowicz urged the CCQM to continue its work in the seawater arena since it had proved extremely useful so far, especially the collaboration with the EAWG. In response to some of the questions posed, Dr May wondered whether the CCQM should have a reference data function, especially where there were data quality issues to be resolved.

18. FUTURE CCQM WORKSHOPS

Dr May observed that topics for CCQM workshops usually emerged as a result of informal discussions and as such there was probably no need to discuss these in detail at this point. However

Dr May stated that he was always receptive to suggestions for workshops throughout the year. Following the earlier presentation from Prof. Pawlowicz, Dr Locascio wondered whether a workshop on reference data would be of interest.

19. CCQM RESOLUTIONS

The 20th meeting of the CCQM produced no resolutions.

20. ANY OTHER BUSINESS

None.

21. CLOSURE

In the absence of further business, the CCQM President closed the meeting at 15:30 and thanked participants for their contributions, reports and participation in the discussions. Dr May thanked the staff of the BIPM for their support in hosting the meetings and wished all participants a safe journey home.

Dr R.J.C. Brown

Rapporteur, 30 April 2014

DECISIONS AND ACTIONS FROM THE 20TH MEETING OF THE CCQM

1. As rapporteur, Dr R.J.C. Brown to draft "Decisions and Actions" document and "Report of 20th Meeting of the CCQM".
2. CCQM approved the report of the 19th Meeting of the CCQM.
3. Outstanding actions from the 19th Meeting of the CCQM to be progressed (as listed in CCQM/14-32).
4. The President described the procedures outlined in the new "Guidelines for Selection of CIPM Consultative Committee Presidents" and "Good Practices for Selection of Consultative Committee Working Group Chairpersons and Working Group Deputy Chairpersons". The CCQM supports the selection of chairs and deputy-chairs of CCQM WGs according to this process. Following each election of a new CIPM by the CGPM and subsequent appointment/reappointment of CC Presidents, WG chairs would be selected by the CCQM President and subsequently the deputy-chairs would be proposed by the WG chairs, with approval by the CC President who would ensure suitable global diversity where possible. The terms of these appointments would be until the CCQM meeting following the election of a new CIPM, typically a period of four years.
5. The CCQM President stated that if re-elected he would welcome nominations from the CCQM for the posts of WG Chairs for inclusion in the selection process.
6. The WG deputy-chairs proposed for the CCQM WGs were approved by the CCQM subject to local approval at parent NMIs. The initial term of these deputy-chairs is until the 2015 CCQM meeting or soon thereafter in accordance with the end-of-terms of the current CCQM Working Group Chairs and appointment of new WG Chairs by the newly appointed CCQM Chair. Thereafter, these terms would be as described in Item 4.
7. The CCQM decided that the follow-on to CCQM-P130, undertaken in the surface analysis working group does not meet the criteria of a Key Comparison, and must be a pilot study for the conversion of the k-ratio data to mass concentrations.
8. The CCQM recognized that, despite the proposed name change of the CC to the Consultative Committee on Amount of Substance – Metrology in Chemistry and Biology, the work of the CC, especially in the biology area, may include activities not associated with amount of substance determination.
9. The KCWG to consider unified nomenclature for the core comparison approaches being undertaken by different WGs to avoid confusion when communicating outside the CCQM.
10. The KCWG to consider drafting guidelines for the process of linking subsequent comparisons to an original key comparison, in particular how closely in time they should follow the original comparison.
11. The KCWG to consider an agreed system of nomenclature for the numbering of CCQM studies to unambiguously distinguish between subsequent comparisons, repeat comparisons, multiple-analytes, and multi-part comparisons conducted over a period of time.

12. The SPWG to propose methods and activities to measure the impact that CCQM is having in the end user community.
13. The SPWG to develop proposals for changes to the text of Note 4 of document CIPM/2009-24 to accommodate the IAWG statement on working practices for traceability and elemental calibration solutions and other future similar cases which might otherwise be regarded as traceability exceptions. The CCQM agreed to accept the wording of the traceability exception proposed by the IAWG for Isotope Ratio Delta Values (CCQM/14-18), and this will be forwarded to the CIPM for approval.
14. The CCQM agreed to continue its policy of inclusivity when operating meetings for the time being whilst also recognizing that the capacity of the BIPM to accommodate increasing numbers of delegates is not unlimited.
15. The CCQM approved document CCQM/14-09, the request form for guest laboratories to participate in CCQM studies, and the final form with minor editorial changes would be approved as a CCQM document and published on the publicly available CCQM webpage.
16. Guidelines related to guest laboratories that are not NMIs and DIs and are being invited to, and participating in, WG meetings are described in CIPM-D-01. Item 15, addresses CCQM/14-09, which is the required form for guest laboratories to participate in CCQM studies. The President asked the KCWG to investigate if a revised version of this document with less required documentation could be used for non-profit guest laboratories, and guest laboratories that WGs are inviting to participate on a regular basis.
17. The period 15 April to 24 April 2015 was reserved for next year's CCQM meetings at the BIPM. A full timetable will be developed in due course. WG meetings will be hosted at the BIPM in this time period.
18. The CCQM President and Executive Secretary together with WG Chairs will further develop proposals for CCQM meetings in 2016, following up on proposals that CCQM WGs will not meet at the BIPM in April 2016, but at sites/locations agreed to by individual WGs and that the CCQM Plenary, a workshop, KCWG, and SPWG meetings will be held at the BIPM in April 2016.

APPENDIX 1
WORKING DOCUMENTS SUBMITTED TO THE CCQM AT ITS 20TH MEETING

Working documents submitted to the CCQM at its 19th meeting are on restricted access.

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

Document CCQM/	
14-01	Draft agenda for the 20th meeting of the CCQM, 1p
14-02	Timetable of CCQM meetings 3-11 April 2014, 1p
14-03	Visiting Scientist secondment opportunities in the BIPM Chemistry Department 2014-2015, 6pp
14-04	Agenda for the CCQM workshop on the redefinition and realization of the mole, 1pp
14-05	CCQM questionnaire on CMC generation, review and presentation within the CIPM MRA, 5pp
14-06	CCQM IAWG statement on working practices for traceability and elemental calibration solutions, 1pp
14-07	Papers and articles on the redefinition of the mole.
14-08	Report of the 12th meeting of the JCTLM Executive Committee, December 2013, 17pp
14-09	Draft CCQM Pilot Study: Request form for pilot laboratories (1.b) (SPWG/14-05), 5pp
14-10	CCQM and CCQM WG passwords, 1pp
14-11	EURAMET Report to CCQM, 4pp
14-12	COOMET report to CCQM 2014, 6pp
14-13	APMP report to CCQM 2014, 15pp
14-14	AFRIMETS report to CCQM 2014, 4pp
14-15	REMCO report to CCQM 2014, 2pp
14-16	IUPAC Atomic weights of elements 2011, 32pp
14-17	IUPAC report on CRMs for isotope ratio-analysis, 43pp
14-18	IAWG proposed traceability exception on isotopic delta scales, 2pp
14-19	CCQM Bioanalysis WG update, H. Parkes, 31pp
14-20	Towards SI traceability of seawater measurements, Rich Pawlowicz, 13pp
14-21	*Ad Hoc* Steering Group On Microbial Measurements, Jayne B. Morrow, 37pp
14-22	CCQM 2014 in Tsukuba, Japan, 4pp
14-23	Report of the WG on Electrochemical Analysis, Michal Máriássy, 35pp
14-24	CCQM - Gas Analysis Working Group, Jin Seog Kim, 23pp

14-25	Report of the Inorganic Analysis Working Group, Mike Sargent, 14pp
14-26	CCQM Key Comparison and CMC Quality Working Group Update, Della SIN, 24pp
14-27	2014 OAWG Report to CCQM, Lindsey Mackay, 37pp
14-28	Eurachem - a focus for analytical chemistry and quality related issues in Europe, 19pp
14-29	Surface Analysis Working Group (SAWG) - Report for 12th Meeting (2013/14), Wolfgang Unger, 32pp
14-30	Welcome to the 20th Meeting of the CCQM, 44pp
14-31	BIPM Chemistry Department Programme Progress, 28pp
14-32	Updates on decisions and actions from the 19th meeting of the CCQM, 3pp
14-33	Summary of visits to the BIPM KCDB in early 2014, C. Thomas, 6pp
14-34	Preliminary analysis of responses to the CCQM questionnaire on the CMC process, 11pp
14-35	Maintenance and development of the CCQM Strategy document to 2027, 9pp
14-36	Decisions and actions from the 20th meeting of the CCQM, 2pp