Bureau International des Poids et Mesures

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

Report of the 14th meeting
(3–4 April 2008)
to the International Committee for Weights and Measures

Comité international des poids et mesures

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Note:

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

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

A.J. Wallard,
Director BIPM

LIST OF MEMBERS
CONSULTATIVE COMMITTEE FOR AMOUNT OF SUBSTANCE: METROLOGY IN CHEMISTRY
as of 3 April 2008

President

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

Executive Secretary

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

Members

Centro Nacional de Metrología [CENAM], Querétaro.

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

Danish Fundamental Metrology Ltd [DFM], Lyngby.

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

Institute for Reference Materials and Measurements [IRMM].

International Atomic Energy Agency [IAEA].

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

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

International Union of Pure and Applied Chemistry [IUPAC].

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

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

National Institute of Metrology [NIM], Beijing.

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

National Measurement Institute, Australia [NMIA], Lindfield.

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

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

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

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

NMi Van Swinden Laboratorium, Nederlands Meetinstituut [NMi VSL], Delft.

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.

SP Technical Research Institute of Sweden [SP], Borås.

State Laboratory [SL], Co. Kildare.

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 [BIM], Sofia.

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

Centro Español de Metrología [CEM], Madrid.

Hungarian Trade Licensing Office [MKEH], Budapest.

Istituto Nazionale di Ricerca Metrologica [INRIM], Turin.

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

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

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

1 OPENING OF THE MEETING

The Consultative Committee for Amount of Substance: metrology in chemistry (CCQM)* held its fourteenth meeting at the International Bureau of Weights and Measures (BIPM), at Sèvres, on 3-4 April 2008.

The following were present: H. Andres (METAS), L. Besley (NMIA), A. Brewin (NPL), R. Brown (NPL), G. Carroll (SL), P. Charlet (LNE), K. Chiba (NMIJ/AIST), P. De Bièvre (IUPAC/CIAAW), R. Dybkaer (IFCC), O. Efremova (VNIIM), H. Emons (IRMM), H. Ent (NMi VSL), A. Fajgelj (IAEA/IUPAC), B. Güttler (PTB), H.D. Jensen (DFM), R. Kaarls (President of the CCQM), J.S. Kim (KRISS), K. Kato (NMIJ/AIST), Y. Kustikov (VNIIM), H. Li (NIM), W. Louw (NMISA), L. Mackay (NMIA), B. Magnusson (SP), M. Máriássy (SMU), W.E. May (NIST), M.J.T. Milton (NPL), Y. Mitani (CENAM), U. Panne (BAM), S.-R. Park (KRISS), H. Parkes (LGC), S. Prins (NMISA), J.A. Salas Téllez (CENAM), M. Sargent (LGC), L. Siekmann (RfB), R. Sturgeon (NRC), W. Unger (BAM), A. van der Veen (NMi VSL, ISO REMCO), A.J. Wallard (Director of the BIPM), S. Wise (NIST), Y. Yu (NIM).

Observers: O. Cankur (UME), V.S. Da Cunha (INMETRO), J. da Jornada (INMETRO), R. Daroda (INMETRO), P.K. Gupta (NPLI), W. Kozlowski (GUM), M.P. Sassi (INRIM), M. Sega (INRIM), Z.N. Szilágyi (MKEH), A. Zoń (GUM).

Invited: E.J. Amis (NIST), M. Amos (NIST), C. Bertler (SKL), C. Cherdchu (NIMT), S. Sik-Man Choi (GL), P. Chui (HSA), S. Doyran (FAO), S. Ellison (LGC), E. Gray (NIBSC), C.M. Jackson, L.T. Kooi (HSA), I. Kuselman (INPL), C.K. Li (GL), A. Squirrell (ILAC, NATA), P. Totarong (NIMT).

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

Sent regrets: A. Bryden (ISO), T. Fernández Vicente (CEM), V. Ivanova (WADA), J.W. McLaren (NRC), T. Steiger (BAM), P. Taylor (IRMM), L. Yang (NRC), M. Walsh (INAB).

Dr Kaarls, the President, welcomed participants and observers to the 14th meeting of the CCQM. Professor Wallard, the Director of the BIPM, also expressed a warm welcome to all, further adding that there were more participants than ever before.

Dr Kaarls brought to the attention of all the sudden passing of Dr de Leer and proceeded to present a brief eulogy in honour of his significant contributions and experience brought to the CCQM. His warm personality, noting that he was a man of songs, laugh and dance in addition to his scientific activities, was fondly recalled; he will be greatly missed by his many colleagues and friends throughout the world. A respectful moment of silence was observed in memory of Dr de Leer.

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* For the list of acronyms, click here.

The President then commented on presentations made in the days preceding the CCQM, particularly noting the workshop on 2 April devoted to considerations of the Key Comparison Reference Value Working Group (KCRVWG) and the Efficient and Effective Testing of CMC Claims Working Group (EETWG) and to the 15 years of achievements and challenges of the CCQM, as aptly summarized in the "progress" report delivered by Dr H-Y. So, embracing the belief that the CCQM has been "doing the right thing". Dr Kaarls added that although significant progress had been made, there was much more work yet to be done to enhance the efficiency and transparency of many processes. He expressed hopes that the newly established *Ad hoc* working groups on the KCRV and EET would be able to finish their tasks by the end of the year. Dr Kaarls then thanked the BIPM, its staff and Prof. Wallard for the infrastructure support needed to organize the meetings.

2 APPOINTMENT OF A RAPPORTEUR

Dr Kaarls thanked Dr Milton for his seven years of excellent service to the CCQM in his role as rapporteur, but noted that because he was now tasked with chairing the CCQM Working Group on Gas Analysis, that he should be relieved of this duty. Dr Kaarls proposed that Dr Sturgeon act as rapporteur for the meeting. Dr Sturgeon agreed. Dr Wielgosz would assist him.

The introduction of all delegates, observers and international representatives present was then undertaken, following which Dr Kaarls offered his public congratulations to Dr Besley on his appointment as Chief Executive for NMIA. He further noted that because of this, Dr Besley would likely be unable to attend future CCQM meetings.

3 APPROVAL OF AGENDA

Dr Kaarls stated that short reports from both the Codex Alimentarius and CITAC were to be added to the existing agenda. The amended agenda was approved.

4 REPORT ON THE THIRTEENTH MEETING OF THE CCQM

Dr Fajgelj (IAEA/IUPAC) raised the issue of having the technique of instrumental neutron activation analysis (NAA) formally recognized as a primary method of analysis. Although this was discussed at a meeting of the inorganic analysis working group in 2007 and recorded in the report of the last CCQM meeting, he expressed a strong desire to have it more prominently recorded in the minutes. The President agreed that, although the CCQM no longer listed 'primary methods', it was recognised that NAA had claims to a similar status to that of the five methods listed originally by the CCQM and he would return to a reconsideration of this list of techniques in the future. The report of the 13th meeting was subsequently approved and Dr Milton thanked for his efforts.

5 REPORTS OF THE CCQM WORKING GROUPS

5.1 Organic analysis

Dr May presented his report of progress made by the CCQM Working Group on Organic Analysis (OAWG), which had met twice since the last meeting of the CCQM; during October 2007 at the PTB with 40 participants from 21 institutes and earlier in the week at the BIPM, with 45 participants representing 28 institutes. He briefly outlined the terms of reference of the group and emphasised the importance of benchmarking the capabilities of the national metrology institutes (NMIs). Dr May then proceeded to report on four key and four pilot comparisons that had been completed, as well as those in progress at the time of the meeting.

- CCQM-K50.a,b [in parallel with CCQM-P69.1] (PAHs in soil//Particulate matter). Data for five PAHs submitted by most participating laboratories agreed to within ±5 % of the mean for the analysis of the soil sample and within ±10 % for the particulate matter. Discussions within the OAWG relating to sample homogeneity and the effects of temperature on extraction efficiency of the measurands were to be completed with the issue of a Draft B report and proposal of the mean of eligible results as the KCRV and the standard deviation of the mean of the eligible results taken as the standard uncertainty of the KCRV. An "operational" definition of the measurement, commensurate with current state-of-the-art practice, would specify the measurands as "amount of specific PAH in soil/sediment as extracted under exhaustive extraction conditions".
- CCQM-K62 [in parallel with CCQM P78.1] (Nutrients in infant/Adult formula: Vitamins). The test sample was commercially purchased and packaged, and the analytes under study were folic acid, niacin and vitamin A (as retinol). Although only 2 to 3 participants were registered for these comparisons, Dr May reminded all present that the OAWG had adopted a working policy only those laboratories participating in the pilot study organized prior to a key comparison were

eligible to have their data for the respective key comparison considered for the establishment of the KCRV. A Draft B report was to be assembled after the investigation of the possibility of a folic acid calibration bias had been completed.

- CCQM-K63.a, CCQM-K63.b (Non-peptide hormones in serum: Cortisol and progesterone). This study followed a successful pilot study and was designed to underpin work on clinical diagnostics. The arithmetic mean of data from five of six laboratories contributed to the KCRV for cortisol and from three of eight laboratories for progesterone in human female serum. The data not included in the KCRV calculation originated from NMIs that had either not participated in the pilot study or had not made independent measurements on their primary calibrator (unless their primary calibrator was a Certified Reference Material purchased from another NMI).
- CCQM-K47 (VOCs in solution). Dr May noted that this study had begun some four years earlier and there was still no comprehensive explanation to account for the spread of the results for benzene, p-xylene, m-xylene and o-xylene. He suggested that the issue may be re-examined in the future with input from the gas analysis working group, but for certain analytes a KCRV based on the gravimetric preparation of the test samples combined with a standard uncertainty derived from the dispersion of the participants data would be adopted for the present.

Results from a number of pilot studies were then presented:

- CCQM-P20.f (Organic purity assessment series: Digoxin). Determination of the mass fraction of digoxin in a high purity digoxin material attracted thirteen participants. The approach typically involved assessment of purity based on the summation of all detected impurities, including inorganic, volatile and organic impurities. The coordinating laboratory, the BIPM, will expand their report to compare reported mass fractions of individual impurities and groups of related impurities, in order to clarify the reasons for differences in reported results. Dr Wielgosz commented that he would present the CCQM-P20.f results in greater detail during his presentation on the BIPM programme.
- CCQM-P109 (Determination of acrylamide in cooked high-carbohydrate food). The spread of the thirteen participant's results was such that the coordinating laboratory (KRISS) agreed to undertake an additional meta-analysis of the data to be included in a report to be circulated in time for the next meeting of the OAWG. A decision would be taken at that time as to whether another pilot study was warranted or a key comparison could be recommended. No consensus value for the study result was tabled.
- CCQM-P88 (Antifungals in food: Malachite green in fish). Seven participants took part in the measurement of malachite green (MG) and LeucoMG in a salmon tissue. An initial 15 % heterogeneity of the test sample was acknowledged at the start of the study, but it was likely that the resulting 48 % content value of the data for MG and 8 % for LeucoMG arose because of the difficulty some participants had with the chemical instability of MG and with its extraction from the high fat matrix. It was recommended that the first draft summary report by LGC be distributed to the participants and the working group (WG) so as to review the details of the procedures used. A decision would be made regarding repeating the study if a more homogeneous sample became available.

- CCQM-P90 (Chloramphenicol (CAP) in milk). Participant's presentations were made and discussed during the October meeting of the OAWG. A first draft summary report was distributed to the OAWG in March 2008. The IRMM is now preparing a CRM for CAP in a pork muscle tissue and will report on the assessed homogeneity of this material at the next meeting for use as a possible test material. As it was agreed that as the analytical challenge of making measurements of CAP in muscle tissue was greater than that of making the measurements in milk, CMCs in milk would be covered.

Dr May proceeded to summarize proposed pilot and key studies, which are to include:

- CCQM-K55.a: Approach to purity assessment of high purity organic materials: beta-estradiol;
- CCQM-K69: Anabolic steroid in urine – testosterone glucuronide and epitestosterone glucuronide in freeze dried human urine;
- CCQM-P91: Pesticides in foods – pyrethroids in apple juice;
- CCQM-P114: Flame retardants in plastics – selected PBDEs and PPBs.

He then noted that several proposals for additional studies were not adopted, including quinolones in pork, tetracyclines in poultry and acetylcholine in microdialysate, these being deferred because the exponential increase in interest and needs expressed by the CCQM Working Group on Organic Analysis (OAWG) members will not permit the needs of the CIPM MRA to be met either strategically nor practically if activities continue using the same approach and same level of effort. Dr May outlined a proposed way forward, based on a strategic planning framework for key comparisons within the OAWG. He envisaged that future comparisons would be divided into three types: key comparisons that test the core competencies of NMIs in providing primary calibration reference services or accuracy control reference services; key comparisons that assess the equivalence of measurement services provided by NMIs (such as delivery of CRMs and value assignments to PT samples); and finally selected pilot studies that address emerging areas of interest or those that are of strategic importance. This approach would result in a limited number of key comparisons in which all NMIs with CMCs would be expected to participate and at least five NMIs would need to agree to participate in any pilot study to enable it to go forward. He further elaborated this model by elucidating the need to first identify and agree to a list of core competencies required for the delivery of higher order measurement services with the need to design and conduct WG studies to support assessment of these competencies.

Dr May finished by announcing an international conference entitled, "Accelerating innovation in 21st century biosciences: identifying measurement, standards and technological challenges" hosted by the NIST on 20-22 October 2008. Experts from five focus areas, notably medicine, energy, manufacturing, agriculture and environment and workshops would identify and prioritize challenges impeding innovation in these areas. The anticipated outcome would be a list of needs to guide research at NIST as well as measurement and standards communities worldwide. An open invitation to attend was offered to all.

Dr Louw raised discussion regarding the proposed way forward with key comparisons, chiefly the need to clarify how extensive participation must be in pilot comparisons in order to support CMCs, given all of the current pressures on NMIs. Dr May reiterated that if core competencies are being tested, then all NMIs having relevant CMCs must participate. Studies targeting emerging areas, however, may only be of interest to selected NMIs. Dr Máriássy questioned whether the KCRV

should be the best estimate of the truth or simply a comparative reference value; this was addressed by Dr Kaarls, who reminded the CCQM that the answer to this issue was the purview of the KCRV working group.

5.2 Inorganic analysis

Dr Sargent presented his review of the work of the CCQM Working Group on Inorganic Analysis (IAWG). The group had met twice (jointly with the EAWG) since the last CCQM; at the NIST Hollings Marine Laboratory in Charleston (October 2007) during which a workshop on metrological applications of multi-collector ICP-MS techniques was arranged, and the previous week at the BIPM (Novotel, Sèvres) wherein a workshop on uncertainties with isotope ratio determinations was conducted. Significant discussions relating to the establishment of a core competencies matrix were also undertaken. A Gantt chart of all current pilot and key comparisons was presented, permitting him to conclude that a healthy portfolio of activities was underway.

Dr Sargent subsequently reported on four key comparisons (several of which had been conducted with parallel pilot studies) completed since the last reporting period:

- CCQM-K43.1 (As, Hg, Se and methylmercury content in marine fish). A concurrent pilot study (CCQM-P96, As and arsenobetaine content in marine fish) was also conducted in parallel with an APMP study. Relatively few issues arose during the performance of these comparisons, apart from the small number of participants and the presence of "outliers". It was emphasized that technical outliers were typically the result of participation of less experienced laboratories or arose from those that do not provide sufficient detail as to their procedures when questioned. In all cases, the decision to include data in the calculation of the KCRV was based on scientific merit and not on purely statistical tests. Although CCQM-K43.1 was derived from CCQM-K43, it was argued that the KCRV should not be anchored to that of CCQM-K43 because there were insufficient results for a statistical evaluation of the KCRV, and that the test samples constituted different matrices with rather different analyte concentrations. It was proposed that the median be used as the best estimate of the KCRV.
- CCQM-K56 (Trace elements in whole fat soybean powder) was run parallel with CCQM-P64.1. Eleven participants submitted results to the key comparison while only seven of 10 registered laboratories provided results for the pilot. As both the mean and median of the key comparison results were in agreement, the simpler approach of using the mean as the measure of the KCRV was adopted, with the uncertainty of the KCRV being given as the standard deviation of the mean.
- CCQM-K57 (Chemical composition of clay) was run in parallel with CCQM-P65.1. Six NMIs participated in the key comparison with seven laboratories in the pilot study. A variety of techniques was used to assess mass fractions of Si, Al, Fe, Ca and Mg with all participants' data contributing to the KCRV, established as the median of the results. The coordinating laboratory (CENAM) calculated the uncertainty of the KCRV from the median absolute deviation (MAD), adopting a coverage factor of 2.57 (Student's *t* for five effective degrees of freedom).
- CCQM-K58 (Determination of nitrogen and trace elements in silicon nitride powder) was conducted in parallel with CCQM-P74.1. Mass fractions of Al, Fe, Ca, Ti and N were assessed.

Because of the relatively small number of NMIs in this key comparison, the best choice of KCRV for each element was the arithmetic mean of results from all participants. It was noted that there was relatively good agreement amongst the results submitted by NMIs, whereas the laboratories contributing data to the pilot study suffered from more divergent results. Degrees of equivalence were presented.

Dr Sargent made brief mention of several other pilot studies reported in the 2007-2008 period, including:

- CCQM-P96: As and arsenobetaine content in marine fish (NMIJ);
- CCQM-P97: Cd and Pb in herb (*Herba Demodii Styracifolii*) (Government Laboratory Hong Kong);
- CCQM-P100.1: Hg in pure water (PTB);
- CCQM-P100.2: Hg in natural water (PTB);
- CCQM-P107: Purity analysis of zinc (BAM).

Additional key comparisons and pilot studies approved by the IAWG for activity in 2007-2008 included:

- CCQM-K34.2: Assay of potassium hydrogen phthalate (SMU);
- CCQM-K64: Analysis of copper alloy (BAM);
- CCQM-P19.2: Assay of HCl (NIST);
- CCQM-P111: Salinity and seawater composition (PTB);
- CCQM-P112: Assay of EDTA (BAM).

He reported that the IAWG was planning several new comparisons:

- CCQM-P100.3 (PTB): Mercury in natural water along with a corresponding key comparison to be conducted in parallel with a EURAMET project for reference laboratories;
- CCQM-P96.1 (NMIJ): Arsenobetaine along with a parallel key comparison once anomalies with respect to available arsenobetaine calibration standards were resolved;
- Proposed pilot study (INMETRO) for trace elements in biofuels;
- Proposed key and/or pilot studies for trace elements in frozen urine, coal or fly ash.

Future activities included a scheduled joint meeting of the IAWG and EAWG hosted by the IAEA in Vienna (October 2008); continuing work on the refinement of a core competencies matrix; an update of the review of future CMC plans by all NMIs, and the establishment of a system to prioritize KC proposals, likely based on the implementation of recommendations of the *ad hoc* KCRVWG, to fill in the gaps and maintain testing of core competencies. He noted that a key comparison and additional pilot studies would be organized to address stable isotope ratio measurements, as only one such study had been conducted to date.

In conclusion, Dr Sargent summarized by stating that good progress had been made with comparisons, noting that the technical information received from participants had improved but that there were still delays in completing some reports. A strategy to link key comparisons with CMCs is now nearing completion and that priorities need to be set which will “illuminate” the core competencies matrix. Pilot studies continue to play a useful role in assisting newer NMIs with

training and obtaining experience while providing a vehicle for the more experienced NMIs to address problems and new areas of interest. Two meetings per year were considered cost effective as they ensure that the work progresses steadily while permitting sufficient time for a wide range of technical presentations and discussion to be accomplished.

Dr Sargent completed his presentation by also announcing an upcoming meeting, the BERM 12 Conference (Biological and Environmental Reference Materials), scheduled for 8-10 July 2008 in Oxford. This meeting is organized to evaluate the status and asses the future needs of users, producers, accreditation bodies and regulators for CRMs.

Dr Fajgelj commented that the area of isotope ratio measurements is quite broad and that interest from both IUPAC and the IAEA has been found to be sufficiently high that they will be organizing a major workshop in the first half of 2009 (most likely to be in Berlin), to which the CCQM will be invited to participate.

With respect to debate over the uncertainties of results delivered by laboratories participating in comparisons, Prof. De Bièvre noted that the GUM has "arrived" at a very timely period in that it provides the analyst with tools to distinguish Type A and B components of measurement uncertainty and reestablishes the authority of the analyst over the final result, an accomplishment that cannot be achieved by use of statistics alone.

5.3 Gas analysis

Dr Milton prefaced his report of progress made by the CCQM Working Group on Gas Analysis (GAWG) with a brief eulogy to Dr de Leer (chair of the GAWG since 2000), noting how much he was appreciated and would be missed. He then proceeded to note that the WG had met twice since the last meeting of the CCQM; at the NMIA in October 2007 during which a workshop was held to consider general strategies for future activities of the GAWG and gas metrology to support measurements of the atmosphere and ambient air quality, and a second, at the BIPM earlier in the week, with a workshop on ambient VOCs involving the WMO-Global Atmosphere Watch and the development of optical spectroscopic methods for gas analysis. He noted that there had been excellent participation in all comparison activities of the GAWG, covering 28 countries and 33 institutes. He then proceeded to outline a flowchart for the calculation of the KCRV, citing three scenarios in use: calculation based on an independent method (i.e., gravimetric) which was "tested" against the participant's data; calculation of the weighted mean (after rejection of any data based only on technical considerations) and, in those cases where data and uncertainties were not consistent, the KCRV was derived by other means (this has happened only once in the history of the GAWG activities).

Dr Milton summarized progress with four key comparisons:

- CCQM-K46 (Ammonia in nitrogen): seven NMIs using a variety of independent measurement techniques returned a suite of data characterizing ammonia at a nominal mole fraction of 30 µmol/mol with significant dispersion in the results (-6 % to +1.5 % relative deviation), thereby prompting a need for additional technical investigation, which is currently underway.

- CCQM-K52 (Carbon dioxide in synthetic air): a successful intercomparison involving eighteen laboratories.
- CCQM-K53 (Oxygen in nitrogen): this had been a preparative exercise coordinated by the KRISS which had developed a very high accuracy comparison method based on GC-TCD. Some discordance of the results amongst the twelve participants prompted discussion of the possibility that excess Ar may have been present in four of the cylinders, but this argument was rejected and the data replotted to exclude those NMIs not having sufficient sensitivity for the measurement of trace oxygen in their nitrogen balance gas.
- CCQM-K51 (CO in nitrogen at 5 µmol/mol) and CCQM-K65 (Methyl- and ethyl-mercaptan at 20-30 µmol/mol in methane) are currently on-going with some 25 and 3 participants, respectively. This work was required to underpin CMCs relating to the odourisation of natural gas.

Several proposed key comparisons were further outlined:

- CCQM-K66 (Purity of methane): this was the first time an exercise was undertaken on a purity analysis wherein a single vessel of the test gas was decanted into individual 3L cylinders that were distributed to a maximum of 10 participants. Cylinders have been distributed and data are expected by June 2008 with work on a draft report commencing in October 2008.
- CCQM-K68 (Nitrous oxide): this comparison will demonstrate the comparability of measurements at the level of 320 nmol/mol and will also involve two expert global reference laboratories – NOAA and GAW/WCC. The target level is typical of current ambient N_2O levels arising from sources in the oceans, soils and anthropogenic emissions.
- CCQM-K71 (Multi-component stack gas emissions): NMi VSL has proposed work on the first multi-component mixture relevant to monitoring of typical industrial stack-emission gases. Measurands targeted include µmol/mol levels of NO, SO_2, CO, CO_2 and C_3H_8, with nitrogen as the balance.
- New activities for 2008 include CCQM-P110 (NO_2 in synthetic air) with test samples being value assigned at the BIPM using their NO_2 dynamic preparation facility. The registration deadline is set for November 2008, to allow time for the BIPM laboratory to complete validation and stability studies of their analytical method and the gas standards. Participant analysis of mixtures is scheduled for October-December 2009.

Dr Milton continued with a commentary on harmonization of CMC claims, whereby he used an example of current claims for ozone measurements from Appendix C to present a model which would link the uncertainties listed in the CMC to the performance of laboratories in key comparisons. The performance achieved in a key comparison would be linked in some quantitative fashion with the CMC claims in accordance with the thirteen principles elucidated by the *Ad hoc* Working Group on the KCRV chaired by Dr Cox, i.e., the uncertainty permitted in the CMC claims would be related to both the laboratory's degree of equivalence and it's uncertainty in results reported in the relevant key comparison.

Dr Milton continued with the presentation of a strategy proposed for the GAWG to ultimately limit the "proliferation" of key comparisons by classifying them into three groups in accordance with the measurands and their tested ranges, notably: core comparisons utilizing generic techniques for the analysis of stable species; those constituting an analytical challenge for which species may not be

stable in cylinders and preparation of standards may be complex; and finally those for natural gas, which may comprise features of the latter two groups. He proceeded to provide examples of this by selecting a group of species and concentrations for which performance is expected to be linked and plotting data for the relative degrees of equivalence arising from all corresponding GAWG key comparisons (long-term performance) against the amount fraction of the measurands, illustrating that such an approach would be capable of linking broad core competency performance capabilities to corresponding CMCs.

Future challenges raised by the GAWG include nano-particles and trace water vapour detection. Current European particle measurement programmes provide emission results for diesel vehicles using two different methodologies (condensation particle counter and a mobility device) which do not overlap in their stated uncertainties. The NMIJ is addressing this issue by developing an aerosol electrometer as a primary standard for this purpose since a number of regulatory and health study parameters are in need of definitive particle mass concentration, number concentration, size distribution, surface area and other characterization measurements. In the area of trace water vapour measurements, it was noted that some standards are available at the trace level but only NPL has CMCs listed ((10-100) nmol/mol range) and the only comparability study undertaken to date has involved the NIST, NMIJ and PTB in a EURAMET study. Activities in the area of humidity were starting in the CCT community and in APMP, but these were at much higher concentrations.

He concluded by noting that collaborations are underway with the WMO and he would have more to say on this with his next report (below).

Professor Wallard asked if contacts had been made with any regulators in Europe regarding the nano-particle issue to which Dr Milton replied that the European Particle Measurement Programme had been consulted and that the METAS and NPL were engaged in this issue but a fundamental question of importance remained as to how to make such measurements traceable, and thereby comparable. Dr Emons posed more general remarks relating to nano-particles, stating that within the EU links have been established between an ISO committee and the OECD to foster health related studies and that since January 2008 the IRMM has been coordinating such measurement issue discussions and their activities should be linked with those of EURAMET and the BIPM. The President agreed that it was important to ensure that the appropriate agencies had been contacted such that we were seen to be “doing the right thing”.

5.4 Electrochemical analysis

Dr Máriássy presented his report of the work of the CCQM Working Group on Electrochemical Analysis (EAWG), which had met twice since the last meeting of the CCQM (both joint meetings with the IAWG; in Charleston in October 2007 and earlier during the present week). He presented the results of five key comparisons:

- CCQM-K36.1 (Electrolytic conductivity). This comparison was linked to the earlier CCQM-K36 through participation of the DFM, PTB and SMU. A weighted mean was used for calculation of the KCRV. Two NMIs showed evidence of problems that may have been related to cell surface effects.

- CCQM-K18.1 (pH of carbonate buffer). This comparison assessed the measurement capabilities for alkaline buffers and was linked to the earlier CCQM-K18 through participation of SMU. Improved performance of most institutes was evident over that achieved in CCQM-K18. A weighted mean was used for calculation of the KCRV.
- CCQM-K20 (pH of oxalate buffer). This comparison assessed the measurement capabilities for acidic buffers and engaged twelve participants. Most laboratories agreed well and the KCRV was calculated from the weighted mean of the results for all participants save one that submitted no uncertainty budget.
- CCQM-K48 (Assay of KCl): the ability to determine the amount content of chloride in KCl was assessed for seven participants in an exercise that was jointly conducted with the IAWG. With the exception of one laboratory undertaking gravimetry, all others used coulometry. A variety of additional analytical techniques was used (IC, XRF, ICP-MS) for detection of impurities and a KCRV based on the median of the results was calculated. The corresponding uncertainty was very small because of the large number of results that were in close agreement.
- CCQM-K59/P89 (Nitrate and nitrite in calibration solutions and natural water). This key comparison was motivated by the importance of providing traceability for these analytes in food chemistry, water analysis and agriculture. The spread of results was larger than in previous comparisons and although the KCRV was established as that arising from the gravimetric preparation of the test solutions, it was in good agreement with the weighted mean of the participants' results. A significant spread in the submitted data for these measurands in a seawater matrix resulted in values not in agreement even within specified uncertainties. The complexity of the matrix and the 1000-fold lower concentration of NO_2^- and NO_3^- were suggested as reasons for this.

It was noted that CCQM-P111 (Seawater) was in progress and had attracted 24 participants (11 NMIs) for measurement of conductivity. This comparison is important as it will provide traceability of salinity measurements to the SI. Salinity is a significant parameter used by oceanographers and climatologists due to its impact on global temperatures. Results are expected next year as samples were to be shipped in April 2007.

Dr Máriássy summarised the plans made by the EAWG for future comparisons, concluding that key comparisons targeting pH now span the range 1.5 to 10 and are considered complete and will only be readdressed at least three years hence. Similarly, the suite of key and pilot studies undertaken for electrolyte conductivity spans nearly 5 orders of magnitude with seawater on the one extreme but pure water not yet being accessed and lying 100-fold below the lowest conductivity comparisons undertaken to date by the EAWG, i.e., CCQM-P83.

Plans for the future were outlined and include CCQM-P83 (conductivity, 0.5 mS/m), CCQM-P37.1 (pH) and CCQM-P19.2 (HCl), all of which are scheduled for 2008, and CCQM-P93 (pH 7 preparation study) slated for 2009.

Dr Máriássy proceeded to discuss several technical presentations hosted by the ECWG, including: headspace effects in the preparation of carbonate buffers which cause problems with pH due to equilibrium shifts in the bicarbonate system, this problem increasing in importance with the relative headspace volume; concerns with the slope of the acidity function plots and the effect of minor changes in temperature and chloride content; and the issue of pHe measurement in anhydrous

ethanol. The latter measurement problem is deemed particularly important in light of the global interest in ethanol and other biofuels for transportation. The standard methodology [D6423-99(2004)] for this test produces pHe values that are electrode and instrument dependent, a problem compounded by the fact that specified electrode designs adopted by the ASTM and EU are different. There is a need for harmonization since, for example, Brazil, a major biofuels producer, uses equipment from both sources and obtains discordant results. The issue is most likely due to differences in the liquid junction potentials for aqueous buffer calibration standards and the ethanolic test matrix. The standardization organizations need to be contacted.

Dr Brewin raised the issue of a need for non-aqueous calibration standards and a more rigorous methodology for measurement of pHe along with a desire to ensure that the standardization bodies were consulted. Although the EAWG initiated contacts with the ASTM, Dr May cautioned that such actions need to be undertaken at a much higher level in order to coordinate international activities. The President agreed that duplication of effort should be avoided but that, in any case, such information should be made available to the CCQM.

5.5 Surface analysis

Dr Unger presented his report of progress of the CCQM Working Group on Surface Analysis (SAWG), which comprises seventeen institutes and a total of thirty-five contributing "analysts". He noted that the SAWG has applications spanning life sciences, electronics, chemistry, physics and materials science with a portfolio of techniques that span the 10 nm to 10 µm dimension. He immediately proceeded to discuss the status of active key comparisons and pilot studies.

- CCQM-K32/P84 (Gate oxide SiO_2 on Si). The first of two key comparisons, CCQM-K32 addressed the need for traceable determination of the thickness of gate oxides in the semiconductor industry. A suite of nine samples of nominal thickness in the range 1.5 nm to 8 nm on (100) and (111) Si substrates were examined. Nine NMIs participated in CCQM-K32 with an additional 4 in the parallel CCQM-P84 study. A discussion of the Draft B report was completed in November 2007 and a KCRV agreed upon at the 2008 meeting of the SAWG prior to the CCQM meeting. The KCRV was calculated using a weighted mean of the results with an inverse variance weighted uncertainty. As noted in earlier reports, the existence of an effect in some results due to the presence of hydrocarbons, dehydrogenated carbon and physisorbed/chemisorbed water on the samples precipitated a lack of agreement in results. In particular, because the layer was not detected by x-ray reflectometry, results contributed by NMIJ were not included in the calculation of the KCRV. It was noted that all methods used were traceable to the metre either directly or via calibrations against another technique and that traceable thickness determination for gate oxides needs a multi-method approach. It is expected that CMC claims will be advanced for the next cycle but they will be harmonized before submissions are made.
- CCQM-P95 (Amount of low z elements in diamond-like carbon (DLC) films). DLC films are widely applied in recent technologies and low z elements in thin surface layers, such as N, need to be determined by micro-analytical and in-depth analysis methods. This study began in October 2006 with distribution of samples; results were received in February 2007 and a draft

of the final report circulated in March 2008. Six NMIs, a Designated Institute (DI) and a private laboratory contributed results for three test specimens. Data for nitrogen were scattered; uncertainties were mostly underestimated and there is a lack of CRMs. As such, it was concluded that the SAWG is not ready to go forward with a key comparison based on use of electron probe microanalysis (EPMA), and that the measurement of low z elements is very challenging and that the issue will be returned to in the future.

- CCQM-K67/P108 (Amount of Fe and Ni in (200 nm) Fe-Ni alloy film on Si). Thin film alloy compositions are frequently characterized by micro-analytical techniques and a set of traceable Fe-Ni alloys prepared and certified by the KRISS (using ICP-MS) were used for this study. Five NMIs and 4 expert laboratories have settled upon a (primarily XPS) measurement protocol as of earlier this week, and samples are yet to be distributed. A brief review of the performance of laboratories in CCQM-P98 showing excellent agreement amongst results obtained using EPMA, AES and XPS techniques for the determination of Fe in a $Fe_{51}Ni_{49}$ CRM provided the basis for a call for CCQM-K67.

Dr Unger continued his presentation with a consideration of future activities planned by the SAWG. Foremost amongst these was a need to address calibration issues for EPMA. EPMA is extremely important for industrial applications and quantitative measurements are accomplished using a standards/matrix correction methodology whereby the unknown is measured under identical conditions relative to a suite of standards. Earlier pilot studies (CCQM-P80, CCQM-P81 and CCQM-P95) demonstrated that use of this method resulted in differences in measurements larger than the expanded uncertainties, highlighting the need for better standards and CRMs in this field. Modern instrumentation uses solid state detectors whose efficiency needs to be calibrated so as to permit standardless analysis to be undertaken in which only the unknown is measured. This approach will be attempted using the BESSY facilities at BAM to calibrate absolute spectrometer efficiency *via* a transfer (multi-element) calibration standard which can be used to normalize response from future participants' instrumentation. Such a situation will then permit future pilot comparisons on bulk materials as well as implementation of a standardization project under the auspices of ISO TC 202 and subsequently lay the foundations for a key comparison which permits a return to the issue of measurement of low z elements in a thin film sample or a Ni alloy (for the aerospace industry).

Other potential comparisons being considered by the SAWG include a sojourn into the life sciences wherein measurements of: organic functional group surface density; the amount of OH-groups at hydrophilic surfaces; the amount of NH_2-groups at animated surfaces; the amount of DNA on surfaces; and the amount of a drug on a surface and at interfaces could be undertaken. A number of NMIs expressed interest in these directions and noted that productive collaborations/interactions with the BAWG would result.

Dr Unger concluded his presentation with the announcement of the 13th European Conference on Applications of Surface and Interface Analysis to be convened in Antalya, Turkey from 18-23 October 2009, noting that this would be a good venue for promotion of the activities of the SAWG.

Dr Milton raised a question regarding the proposed future CMCs likely to arise from successful completion of CCQM-K32 and asked for more information on how the process of harmonization

will work to integrate the RMOs and EURAMET. Dr Kaarls responded that this would be accomplished through the current Quality System reviews but that the JCRB should further discuss this matter. Dr Louw commented that there were currently no CMCs submitted but that the first such ones will be assessed by the SAWG members in an effort to carefully format/harmonize them prior to submission. Dr Wielgosz asked for clarification regarding what criteria were being used for participation of private companies in pilot studies. It was noted that this is currently in the hands of the coordinating laboratory and that the majority of them came from the semiconductor industry in Korea and that they provided high quality data and were, in fact, usually more experienced than the NMIs. The President interjected that whereas there may be scientific justification for them to participate in pilot studies, care must be exercised in the selection process in order to avoid conflict of commercial interests. Dr Emons concurred with these comments and suggested that, even for pilot studies, some selection criteria should be developed. Dr May added that such decisions should not be left to the coordinating laboratory but that the entire WG be involved because it would be naive to assume that participating commercial laboratories would not use their involvement in these exercises to some financial end.

5.6 Bioanalysis

Mrs Parkes presented her report of the progress made by the CCQM Working Group on Bioanalysis (BAWG) which had met twice since the last CCQM. The twelfth meeting occurred in October 2007 at the NMIA and attracted 27 participants from twelve organizations and included a workshop on "Biotechnology in Australia"; the thirteenth meeting concluded at the BIPM earlier in the week with forty-three participants from twenty-one organizations. She reviewed highlights of the past year:

- CCQM-K61 (Quantitative PCR calibration). This study underpins many applications of the polymerase chain-reaction (PCR) method (e.g., GMO quantification, viral infection quantification, genetic diagnostic assays and prenatal diagnosis measurements). Nine participants used quantitative real-time PCR to characterize two samples using a variety of platforms and reagents. The uncertainty in the results was increased to take into account the non-homogeneity of the distributed samples, and this resulted in satisfactory agreement with a KCRV that had been established independently using ICP-OES for quantitation of phosphorus. The spread in reported uncertainties (1.5 % to 15 % for sample 1 and 2 to 31 % for sample 2) suggested that the issue of threshold selection in the raw data might be a factor in establishing the correct baseline for subsequent interpretation of the PCR amplification curve. This will be examined further.
- CCQM-P55 (Peptide and protein quantification). The question of peptide purity is hampering establishment of traceability and this pilot study involved the determination of peptides in solution. Results for proline and angiotensin I were in good agreement for seven of eight participants. Availability of pure amino acid CRMs would improve traceability and reduce the spread of results but harmonization of uncertainty evaluation also needs to be achieved. Rather than proceed to a key comparison at this time, a decision was made to undertake a subsequent pilot study (CCQM-P55.1) using the same amino acids but a new peptide in an effort to reduce the reported uncertainties. The quantification of peptides, leading up to that of proteins, would eventually underpin the work of the JCTLM.

- CCQM-P94 (Quantitative analysis of DNA methylation). These measurements have implications for the identification of cancerous tissue as methylcytosines are popular targets in cancer epigenetics. Good agreement amongst four of the five laboratories using different methods was achieved. The cause for the single outlier was discussed and assigned to use of too little enzyme for hydrolysis. A subsequent study, CCQM-P94.1, utilized 50 % and 90 % methylated DNA samples, revealing that insufficient sensitivity was achieved to currently permit real world determinations at the 5 % level of methylation.
- CCQM-P101 (Protein glycosylation). This study is deemed to be of high bio-pharmaceutical importance as glycosylation plays a direct role in the modulation of biological activity and such an analysis constitutes an important aspect of the quality control of glycoprotein product. The study, designed to identify and determine relative quantities of glycan species in a digested glycoprotein mixture, was coordinated by the NIBSC and the US Pharmacopoeia and attracted 52 participants from industry, academia, regulatory agencies and NMIs. The large participation from industrial laboratories afforded the opportunity for NMIs to assess what methods are being used and to judge the quality of the results. It was concluded that few (<5) NMIs have the resources to undertake such studies in this important area and questions were raised as to whether results meaningful to the CCQM could be obtained. It was noted that the CCQM and USP will host a workshop in December 2008 at the BIPM on “Measurement Traceability for Pharma and Bio-pharma Measurements”, which will address comparability of analytical and bio-analytical measurements.

Mrs Parkes proceeded to briefly summarize progress in several on-going studies:

- CCQM-P58.1 (Comparability of fluorescence in ELISA). This study evaluated various instrumental and sample handling effects (i.e., sources of measurement uncertainty) in the ELISA assay, an immunological method to determine the quantity of a specific antigen. CCQM-P58.1 will investigate the utility of ELISA to transfer SI-traceable measurements of a protein reference material from a high concentration stock to one at the nanogram per litre (therapeutic) level. Significant independent work is underway in other laboratories examining different techniques, such as mass spectrometry, that will enable the direct quantitation of such marker species. The study is linked to an IFCC initiative for standardization and traceability of measurement of a human myocardial infarction marker (cTnI). These studies provide the basis for any future considerations of CMCs in the area of immunoassays.
- CCQM-P59.1 (Protein structural measurements by circular dichroism). Results of CCQM-P59 had been received and a further pilot study (CCQM P59.1) was in progress involving seven laboratories to improve aspects of sample handling. There had been some discussion about the range of calibration and measurement capabilities for CD measurements that a key comparison based on this study might underpin. One possibility was through the dissemination of a “molar” CD spectrum.
- CCQM-P102 (Quantification of cells with specific phenotypic characteristics). This study was intended to measure the fraction of cells of a given phenotype by flow cytometry. No details of progress were reported.
- CCQM-P103 (Measurement of a multiplexed panel of RNA transcripts). This exercise would build on experience gained from CCQM-K61 with DNA and demonstrate capabilities for RNA.

The LGC and the NIST were coordinating the study involving eight laboratories. No details of progress were reported.

- CCQM-P113 (Relative quantification of genomic DNA fragments). This study had attracted fourteen laboratories and was piloted by the IRMM. No details of progress were reported.

New study proposals included:

- Measurement of α-amylase activity, which is an important issue for food science but provides no added value to the clinical community. Although coordinated by NIM, liaison with Codex Alimentarius will be undertaken and perhaps the study proposal will be redefined to better align it with industry requirements and BAWG strategy by studying enzyme activity.
- Measurement of acetylcholine in a microdialysate, proposed by the NIM and referred to the OAWG.
- Atomic force microscopy measurement of DNA. Although DNA length would not be of direct interest, protein-DNA binding interactions may be and this consideration will be left for discussions at INMETRO in 2009.

Mrs Parkes then proceeded to discuss BAWG activities which overlapped with other international measurement and standardization interests, citing relevant reports from the USP, JCTLM, IFCC and WHO. A number of issues were raised, including representation of BAWG on the KCWG, collaborations with the OAWG (to leverage analytical expertise in mass spectrometry) and the SAWG (for interest in chip arrays and biomarker imaging) and the need to engage in strategic planning to ensure a tightly focussed agenda for the BAWG.

She concluded by reviewing the roadmaps developed in previous years and the manner in which the present set of pilot studies and key comparisons addressed issues. The next meeting for the BAWG was to be hosted by the NMIT in November 2008.

Dr Milton returned to the apparently unresolved issue raised in key comparison CCQM-K61 regarding potential uncertainties introduced by the need to establish threshold and baseline values for the PCR amplification curve. The problem was acknowledged as being recognized but the key comparison results had been accepted as reported. However, the knowledge gained will be useful for subsequent CCQM-P103 and -P113 studies.

Dr Emons cautioned the use of a highly publicised USP/BIPM workshop as being inappropriate since it appear to be focussing on only the USP, stating that other pharmacopoeias should be involved since the issues at stake were global. It was remarked that the real agenda involves USP only as an organizer and sponsor but that a balanced representation had been achieved from international participants. Dr May agreed with Dr Emons that it was important not to have this perceived focus. He then went on to raise the subject of the summary roadmap and asked when CMCs will be proposed since no timeline was given and there was a need to benchmark services now being delivered by several NMIs. Mrs Parkes replied that CMCs were to be discussed at BAWG strategy sessions since there was a need to know what studies were needed to underpin such CMCs but, in the absence of claims, generic studies were being done such that the types of claims that could be derived from them could be subsequently delineated. It was recognized that this was a difficult “chicken and egg” situation as several NMIs deliver services with well defined claims.

5.7 Key comparisons and CMC quality

Dr Mackay presented her report on the work of CCQM Key Comparison and CMC Quality Working Group (KCWG). This WG, comprising 20 members drawn from all of the RMOs, had an opportunity to meet during the preceding days. She presented the timetable for the CMC Cycle VIII review used during 2007, highlighting that 4007 chemistry CMCs were currently in the Appendix C (as of 12 March 2007). Some 486 submissions were made during the Cycle IX process, constituting 384 new claims and 102 revisions in 13 measurement service categories. The timetable for the CMC Cycle IX review was presented and it was noted that the number of NMIs entering claims had increased by two in each of the last two years and new measurement service areas were being added to accommodate surface (NIM for silicon dioxide surface thickness) and bio-analysis (NIST for protein purity) measurement capabilities. Each WG chair has agreed to update the list of all comparisons present on the CCQM website twice per year (June and December), posting current status and availability of reports to support the work of the KCWG.

The KCWG has committed to preparation of a guidance document for the submission and review of chemistry CMCs which will cover all aspects of the process, including a hierarchy of criteria used to assess CMCs, their expected links to KCs, criteria for NMIs having CRMs as a service delivery mechanism (to define uncertainty in measurement capability *vis-à-vis* that associated with the CRM) as well as some specifics for those having calibration services as their service delivery mechanism. It was recognized that activities in the new ad hoc WGs for KCRV and EETWG will impact the work of the KCWG as there will likely no longer be a one to one relationship between claims and KCs and there will be a need to find more effective ways of using more generic comparison results from KCs designed to demonstrate core competencies.

Inconsistencies in the CMC review process at the RMO level were highlighted and for this reason every single claim submitted was re-examined rather than simply resorting to a random check of a small number of claims. For this reason, an attempt to harmonize the RMO process will be made based on the use of a template for the reports that each RMO must submit commensurate with their CMCs. The Inorganic Working Group within MetChem in EURAMET may serve as an example to be followed.

Within the following year the KCWG will commence a review of all existing CMCs, since the first were submitted 10 years ago. This task shall proceed by measurement service category, starting with the gas area, as most claims lie here. As there are approximately 1000 CMCs in this area, a subcommittee of gas experts will be convened to aid with this process. Furthermore, all claims older than 5 years will be reviewed in the future so as to ensure harmonization of CMCs and that supporting evidence is relevant.

The President commended the WG for their hard work the preceding week and opened the floor for discussions. Dr Milton also praised the KCWG for their hard work in having to review all of the Cycle IX CMCs but felt that their agenda was being hijacked each year by having to devote such an enormous amount of time to the validation of CMC claims and that there was a need to break out of this work cycle and begin to focus on issues that were more relevant to the mandate of the KCWG. The President responded by reiterating that the new template and guidance documents being prepared by the KCWG should alleviate this situation somewhat.

A lively debate then ensued. Dr May praised the KCWG members for their excellent work and commented that more meetings of the KCWG per year was not an approach to be undertaken but that more of the burden of work should be taken up by the RMOs. He noted for the record that it was timely to announce and congratulate former CCQM colleague and delegate Dr Hratch Smerjian (NIST) who had been selected for the "wall of fame" as one of NIST's Gallery of Distinguished Alumni. Dr May then went on to state that he opposed any attempts to forge a formal link between CMCs and CRMs because there was no significant justification for it nor had relevant discussions been undertaken with CRM producers. He went on to state that KCs yield the best estimate of the truth but CRMs reflect the results of potentially limitless activity undertaken by an NMI to drive down the uncertainty associated with the result for a measurand by taking advantage of an increased number of results. Dr Mackay agreed that there would likely be differences in the magnitudes of the uncertainties associated with CMC claims for CRM producers and their associated KC data and stated that a request would be made of the producer to submit evidence of the procedures to support lower uncertainty claims. The President argued that the entire process is not transparent to the user of Appendix C. Dr May replied that there were no inconsistencies because the process of value assignment to a CRM is different from a measurement service activity. Dr Emons also disagreed with the President's comment, stating criteria such as that in ISO Guides 34 and 35 serve to make the process transparent. He rationalized that a CRM is not only a "claim", but also a product and that no one else can share any liability or judge it. The President reiterated that he believed the process was still not transparent to the user of this information. Dr van der Veen stated that ISO REMCO was ready to participate in any discussions on this topic. Dr Quinn agreed with Dr May's position stating that the MRA was initiated to provide confidence in the capabilities of NMIs with expectation of their being a one-to-one relationship established between CRMs and CMCs. Dr Chiba expressed concern about the nature of the measurand and noted that many CMCs are method dependent and asked how a measurement could be expressed in a CMC claim, giving examples of those discussed in the SAWG and the BAWG. Dr Mackay noted that this issue will require careful consideration and cited the case of CCQM-K32 which utilized a variety of measurement techniques. The President agreed that it is likely that the CCQM will be faced with an increasing number of method dependent CMCs.

5.8 *Ad-hoc* KCRV Working Group

Dr Milton kindly presented the results of this WG (established by the President in April 2007) on behalf of its chairman, Dr Cox. The terms of reference for the KCRV WG were to examine the various approaches currently in use for the determination of the KCRV and its uncertainty, and to produce a set of guidelines that would be adopted by the CCQM for the calculation of these values. It was clear that harmonization of approaches was necessary but it was indeterminate to what extent this was feasible or desirable. At the January meeting of the WG it became apparent that this task was too extensive to achieve within one year. He noted that document CCQM/08-08 (on restricted access) posted on the CCQM website summarized a set of thirteen basic principles proposed for moving forward but emphasized that further comments were appreciated and that they should be directed to Dr Cox by e-mail. He stressed that there was still a need to understand the link of CMCs to results of KCs, but otherwise the group was moving on to fulfil the remainder of their mandate.

The President expressed hope that the work of this *ad hoc* group could be completed within the next year.

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

The President made a brief report on the activities of the EETWG on behalf of Dr Turk. This *ad hoc* group was established by the President in April 2007 and mandated to provide recommendations to the CCQM on how best to deal with the ever increasing problem of the need for more and more key comparisons arising from the current *modus operandi* by developing policies to minimize their number needed to underpin the CIPM-MRA without compromising their effectiveness. The WG proposed a policy of implementing KCs with a view to testing their core competencies. It was proposed that NMIs are to inform analysis WGs of plans to submit CMCs in the next 3-5 years. It was also suggested that the format for proposal of a KC by a co-ordinating laboratory will change so as to include discussion of the relationship of the proposed KC to completed KCs, as well as information on which CMCs will be supported and what analytical skills or challenges will be tested. It was recommended that the KCWG report to the analysis WGs on the adequacy of the existing KCs in supporting CMCs. Tasks remaining to be completed include a summary of core capabilities by each analysis WG, harmonization of the criteria for these approaches amongst the WGs and an investigation into how to deal with uncertainties when multiple KCs, each demonstrating different capabilities, are used to underpin a CMC.

Professor Wallard opened discussions by airing his concerns about the number of outliers having small uncertainties that appear in several KCs and suggested that, in planning future KCs, the WGs pay more attention to the uncertainties that are expected to be returned before the KC is started, giving consideration to what is to be in such budgets and how they will be assessed. Dr May agreed that this process is not done well but when participating in a KC, “you get what you get” if it is not mandatory to first participate in a pilot study comparison. Professor Wallard felt that there should be more science devoted to these aspects of a KC.

6 UPDATE ON THE BIPM KCDB

Dr Thomas expressed concern in her report that the KCDB Excel file available on the BIPM website is not being properly updated, citing examples of six reports apparently approved in October 2007 which have yet to be posted, as well as some KCs being listed as still in the planning stages but which are in fact completed. She reminded the group that if a KC is not in the BIPM KCDB then the search engine locating an NMI comparison will not be found. The CCQM is the only Consultative Committee which is consistently tardy in this manner. Dr Wielgosz agreed that this was a very serious point that appears to be raised every year and since within the CCs there are WGs that meet twice per year and the Excel file that has been developed is there as a tool to help enter data into the

KCDB as well as keeping up to date records of pilot studies, and there are few excuses for this situation. He noted that the KCWG Excel file will now be updated twice annually and this may prove to be the most reliable vehicle for maintaining the KCs in an up to date state. The Excel file was posted on the BIPM website and available at http://www.bipm.org/utils/common/xls/CCQM_KCs_PSs_140308.xls . This was confirmed by Dr Mackay in that once a report has been approved by the WG it is sent to the other WGs and subsequently also sent to Dr Thomas. Dr Wielgosz suggested that this last step appears to be lacking and thus Dr Thomas reminded the group to please ensure that this was done.

A second point raised by Dr Thomas was that up to nine different "robots" are interrogating the KCDB (apparently one robot is searching every 10 s under "chemistry, Canada"!) at a rate of up to 100 000 hits per month, apparently in an effort to mine URLs. After correction for these anomalous hits, it is estimated that *bona fide* interrogations amount to some 18 000 visits to Appendix C and 14 000 visits to Appendix B per month.

Dr Thomas concluded with two extraneous items – noting that the BIPM Summer School activities, planned for June to 11 July 2008 will be hosting some 92 students and that the "14th International Congress on Metrology" was planned and would be held in Paris at the Palais des Congrès from 22-25 June 2009 with an expectation of 800 participants. One plenary session will be devoted to a celebration commemorating the 10th anniversary of the CIPM MRA and an open invitation to attend was extended to all.

7 BIPM PROGRAMME ON METROLOGY IN CHEMISTRY

Dr Wielgosz presented his report on the work carried out during the year by the Chemistry section at the BIPM. He thanked his colleagues for their contribution to the work and then proceeded to summarise the activities undertaken in the fields of organic analysis and gas metrology being coordinated by the BIPM.

- CCQM-P20.e (Purity of theophylline). Two comparison samples had been prepared – one (CCQM-P20.e.1) by the LGC, which was characterised by the BIPM, and one (CCQM-P20.e.2) of a spiked material that was both prepared and characterised by the BIPM. Excellent agreement was achieved between the mean as well as weighted mean of participants' data that had not been excluded based on technical grounds and the gravimetric reference value for CCQM-P20.e.2. The uncertainty due to preparation and non-homogeneity had been combined to calculate the uncertainty of the reference value.
- CCQM-20.f (Digoxin). Digoxin, used for the treatment of cardiac conditions, is administered over a narrow therapeutic range and is thus of interest for proficiency testing in the clinical laboratory. This was a larger and more challenging measurand, ideally requiring the use of LC techniques. The material was characterized (by LC-UV and LC-MS/MS) for some nine

identified and unknown impurities, as well as assessed for homogeneity and intermediate-term stability, thereby yielding uncertainty components for these factors. Volatile impurities were quantified by GC-MS, and the water content by Karl-Fisher titration. A number of techniques were used to quantify non-volatile residues. The BIPM result agreed well with two other laboratories, but the majority of laboratories were biased with respect to the BIPM value as they had not accounted for volatile organic compounds (some 4 mg/g) in the material. Some further discussion was needed on how to use asymmetric uncertainties to deal with impurities which were below the limit of detection, and would be covered in the report of the comparison.

The BIPM will coordinate CCQM-K55, the first key comparison for purity assessment of organic primary calibrators, which is intended to demonstrate a laboratory's performance in determining the mass fraction of the main component in a high purity organic material. The OAWG had earlier adopted a model based on "molecular weight versus polarity space'', developed by the BIPM, as a way of classifying measurement capabilities for the characterization of pure organic materials. The BIPM has designed CCQM-K55 to consist of a group of three measurands selected to span these capabilities. The first stage, CCQM-K55.a (17 beta-estradiol), is a challenge due to medium structural complexity, intermediate polarity and one wherein related-structure impurities could be quantified by capillary GC or HPLC. The second, CCQM-K55.b, for which oxytetracycline or chloramphenicol are the proposed measurands, will present a medium to high structural complexity measurand of high polarity wherein related structure impurities could be quantified by HPLC but not GC. For the third stage of the comparison, CCQM-K55.c, the measurand will be of low to medium structural complexity and low polarity for which impurities are best quantified by GC. These three comparisons will provide for a broad probing of the "molecular weight-polarity space". Characterisation of the CCQM-K55.a study material, as well as planning for the latter stages of the comparison, was underway at the BIPM. The future comparison programme at the BIPM would also be extended to cover calibration solutions.

Dr Wielgosz then turned attention to the gas metrology programme, beginning with a discussion of how to link a CMC claim with performance achieved in BIPM.QM-K1 (Ozone), noting that in addition to the obvious need for agreement of a participant's KC result with the KCRV, that the claimed uncertainty in the KCDB be at least equal to the uncertainty provided for the KC. However, this latter issue was compounded by the fact that the uncertainty that can be achieved varies with the amount content of the measurand. He then proceeded to present a suggested template for such a CMC claim based on BIPM.QM-K1 that would take this into account.

Attention was then turned to activities planed for the period 2009-2012:

- BIPM.QM-K1 (Ozone): is the first "on-going" key comparison to be organised by the BIPM. The protocols have been agreed and published and Draft B reports circulated. Results of this key comparison would be updated regularly following bilateral comparisons at the BIPM.
- CCQM-P110 (NO_2 in nitrogen): this study presents a difficult analytical challenge due to the reactive chemistry involved in sample handling as well as tests the capability of spectroscopic methods to measure gases. A workshop on FTIR measurement will be undertaken to consider the traceability and uncertainty achieved with optical methods of analysis. It is anticipated that cylinder preparation would start at the end of 2008 with their distribution to participants in

October, 2009. Analysis of mixtures is due to be completed by December 2009 and a Draft A report issued in April 2010 commensurate with the workshop.

Dr Wielgosz then briefly summarised the 2009-2012 BIPM gas metrology programme by highlighting the activities and capabilities of the ozone reference standard comparison facility as well as international comparisons in support of green house gas monitoring and, air quality measurements, including work on CH_4, O_3, NO_2 and CH_2O gas standards.

Coordination activities with a number of agencies were then summarized, including those with the JCTLM, Codex Alimentarius commission, USP, ENFSI, WMO and ISO REMCO.

A successful meeting on activities and challenges for traceability in laboratory medicine was held in Beijing in 2007; a report was made to the Codex Committee on Methods of Analysis and Sampling (CCMAS) in March 2008 which addressed position papers on method selection criteria, harmonization of analytical terminology (VIM3 (JCGM 200:2008) and ISO 3534), uncertainty in sampling and measurement uncertainty in relation to the Horwitz equation. A conference on Measurement Uncertainty was supported in Budapest in March 2008 wherein a workshop on method performance and analytical uncertainty was organized, treating statistics from collaborative trials amongst other topics. The BIPM also participated in an electronic working group with CCMAS, focusing on guidance on measurement uncertainty.

Dr Wielgosz concluded his presentation with a brief announcement of the availability of visiting research scientist opportunities existent at the BIPM for 2008/2009 in both the gas metrology and organic analysis laboratories.

Dr Brown (NPL) commented that with respect to the VIM3 being such an important document, would it be made freely available on the BIPM website. Professor Wallard replied that there was a commitment from ISO to provide the BIPM with the final version for posting on the BIPM website. Dr Kaarls expressed hope that this would indeed transpire.

Mrs Parkes asked whether ISO/TC 34 was active in the Codex Alimentarius in relation to work on sampling and detection of GM foods. Dr. Wielgosz replied that the secretary of ISO/TC 34, Mr J.-B. Finidori was the ISO representative to the CCMAS.

Dr May commended Dr Wielgosz and his team for their work on the organic purity project.

Dr Besley commented that, with respect to the organic purity programme, the “light shines” on perhaps three selected compounds, but how far can it be extrapolated – does the laboratory performance extend to other areas? Dr Wielgosz replied that this still needs to be examined in more detail as there is a learning curve as we move from easy to more difficult systems, but that we will be in a better position to address that question following completing of the three comparisons. Dr May commented that the programme has just recently been designed to probe the weight-polarity space and an answer to this question will have to wait until 2011 when the three studies have been completed.

8 REPORTS ON RMO ACTIVITIES

8.1 APMP

Dr Kato, chair of the Technical Committee on Metrology in Chemistry (TCQM), presented a report on activities of the APMP. At the end of 2007, the APMP comprised 32 Members from 21 Economies. A total of 676 CMC claims are accorded to APMP in Appendix C and 219 new claims were submitted for Cycle IX. Dr Kato proceeded to summarize TCQM key comparisons and pilot studies underway in 2007-08. In addition to eight pilot studies earlier completed in 2007, three key comparisons, notably APMP.QM-K1.c (NO in N_2), APMP.QM-K1.d (SO_2 in N_2) and APMP.QM-K4.1 (ethanol in N_2) were in Draft B or final report stages and three pilot studies, APMP.QM-S1 (N_2 in He), APMP-QM-P10 (Cd and Pb in Herb) and APMP-QM-P11 (Total As and organo-As in swordfish) were in Draft B or running, with APMP-QM-S2 (O_2 in N_2) planned and three others proposed.

Dr Kato summarized a number of APMP meetings held during 2007, including the 23rd General Assembly in Sydney in October, the 5th gas CRM workshop in Xi'an in May, the International MiC Symposium in Xi'an in June, an International Symposium on Chemical Metrology in Hong Kong in June and several meetings (March and November) of the Asian Collaboration on CRMs.

8.2 COOMET

Dr Kustikov presented a report on behalf of Prof. Konopelko, the Chairman of TC1.8, the Physical Chemistry Committee of the COOMET. He noted that two new members had joined in 2007: Armenia and Azerbaijan, and described the main objectives of COOMET activities. With the exception of surface analysis, the five NMIs within COOMET have been active in key comparisons co-ordinated by the OAWG, GAWG, BAWG, IAWG and EAWG. Currently, there are 250 CMC claims for chemistry in Appendix C with more than 90 % contributed by the VNIIM.

Dr Kustikov then detailed several COOMET projects, including No. 379/RU/06 (Moisture content in cereals and cereal products) involving the determination of moisture in wheat grain with results submitted in November 2007. The KCRV was based on the median of results from five participants. A subsequent comparison had been proposed based on the success of the pilot study. Project No. 378/RU/06 (Particle size measurements) was undertaken using an aqueous suspension of latex spheres and involved VNIIM and VNIIFTRI using optical microscopy techniques.

COOMET.QM-K1.a (Gas mixtures: CO in nitrogen), co-ordinated by VNIIM is linked to CCQM-K1.a and involved four laboratories. Samples were gravimetrically prepared and distributed in November 2007 with results returned in January 2008. A Draft A report is expected imminently.

A second key comparison, COOMET.QM-K23.b (Synthetic natural gas), also co-ordinated by VNIIM is linked to CCQM-K23.b and will involve 6 participants with distribution of the samples in April 2008, results by June 2008 and a Draft A report expected in October 2008.

Dr Kustikov finished his presentation with a summary of planned meetings for 2008 which included the 18th COOMET Committee meeting in Minsk in April, the annual meeting of TC 1.8 (Physical Chemistry) in May in St. Petersburg and the 3rd international conference on "Metrological Assurance of Physico-chemical Measurements" in Kiev in November 2008.

Dr Milton enquired about the current status of review of the Quality System of COOMET laboratories and Dr Kustikov replied that this had now been achieved using an on-site peer review process.

8.3 EURAMET

Dr Güttler summarised the work of the MetChem committee of EURAMET, noting that a transition had occurred this past year as a consequence of the 2006 20th EUROMET General Assembly in Vienna recommending the creation of EURAMET e.V. as a legally incorporated society under German law. Its inaugural meeting was held on 11 January 2007 in Berlin. The TC for Metrology in Chemistry comprises Dr Güttler as chair and sub-committee representatives R. Wessel, F. Ulberth, C. Quetel and P. Spitzer for gas, organic, inorganic and electrochemistry, respectively. EURAMET comprises 21 European countries and has a commitment of 250 million euro over seven years.

He also provided an update on the iMERA (implementing Metrology in the European Research Area) project being carried out by EURAMET, which had involved the development of detailed 'road-maps' for their work and a call for expressions of interest in joint research projects amongst the NMIs. The results of these proposals were announced in January 2008 and funding was received for technical programmes in health (T2.J02, T2.J10 and T2.J11) as well as in SI and fundamentals (T1.J1.2, the Avogadro Project). The first EURAMET TC chemistry meeting was held in Istanbul in March, 2008 in which a workshop was convened of iMERA and Cycle IX CMCs were discussed.

8.4 SADCMET

Mrs Prins presented a review on behalf of Dr Louw, the chair of the SADCMET Working Group on Quantity of Matter. SADCMET involved 14 countries and was collaborating with other African metrology organisations under AFRIMETS.

NMISA is still the only NMI within SADCMET to have participated in key comparisons and to have submitted CMCs covered by the CCQM. The NMISA submitted eleven new CMCs and three extensions of scope for Cycle IX and it is expected that Kenya and Egypt will soon be submitting CMCs. She also provided a summary of the current metrology facilities in various countries within Africa. A total of fourteen international comparisons were undertaken in 2007 along with two proficiency testing schemes covering water and aqueous ethanol. Reference materials for aqueous ethanol solutions, sodium fluoride and several primary gas mixtures have been produced. SADCMET activities planed for 2008/2009 include regional proficiency testing (PT) exercises for water quality, aqueous ethanol solutions and CO in stack gases. Future conferences of note included MSSA in Botswana in July 2008, a chemical metrology workshop in Kenya in August 2008 and NanoAfrica in South Africa in February 2009. She concluded by informing the meeting that, in

future, all other sub-regional metrology organizations (i.e., MAGMET, SOAMET, NAFRIMET, CEMACMET, EAMET and SADCMET) will be members of AFRIMETS.

Dr Fajgelj (IAEA/IUPAC) noted that because of the large size of Africa, significantly more effort should be expended in ensuring that closer cooperation with other agencies was achieved.

8.5 SIM

Dr May presented his report on the chemical metrology working group of the SIM. He noted that, as with SADCMET, SIM comprises 20 countries covering a large geographical region constituting five sub-regions. There are some fifteen NMIs and DIs which regularly participate in SIM intercomparison studies, seven countries that actively participate in CCQM meetings, four NMIs that have CMCs, two countries with CMCs in the current Cycle IX review and one country that will contribute CMCs in the next few years. The SIM programme in chemical metrology is implemented through a cooperative arrangement between the OAS/SIM and the German Government. The principal activities of the group involve outreach and awareness, proficiency assessment activities and training in CMC preparation and review.

Dr May noted communication activities over the past year during which several conferences hosted workshops and technical seminars, notably the SIM Chemical Metrology Working Group meetings in Ecuador in May 2007, the General Assembly in Ottawa in September 2007 which celebrated SIM's Chemical Metrology Working Group's tenth anniversary.

Dr May elaborated on plans for a change in the operational structure within SIM as it is recognized that the needs and metrology in chemistry programmes vary widely among SIM countries. As a consequence, these needs were to be better met through WG sub-groups populated according to the level of development and needs within each country which would stimulate and facilitate cooperation among countries of a similar level of development in a more direct manner than can be achieved with all, in one large group. Thus, sub-group I will comprise NMIs or DIs participating in the CIPM-MRA and having CMCs. Sub-group II will comprise NMIs with no CMCs and sub-group III will comprise those countries having no NMI for chemical metrology activities. The primary needs of sub-group I are key and supplementary comparisons to support their CMCs; those of sub-group II are awareness seminars, assistance with the preparation of CMCs and proficiency assessment studies while those of sub-group III are awareness seminars, assistance in framing arguments for obtaining sustainable government support and for conducting needs assessments with relevant customers.

Dr May concluded by summarizing 2008-2009 programme plans for CARIMET, CAMET and SURAMET.

Mr Squirrel framed a general question of relevance to all reports from the RMOs in that he encouraged closer cooperation and communications between the RMOs and the corresponding regional accreditation agencies because of the limited resources.

Dr May qualified that there was nothing unique about this proposed sub-structure arrangement and argued that every RMO in the world comprised a mix of NMIs having varying experiences. He

offered an invitation to other RMOs that may be interested in the programme that is going to be developed within SIM to examine the process.

Dr Güttler (PTB) noted that CMC claims from SIM make reference to their pilot studies that are difficult to examine and accept for substantiation of CMC claims. He asked if it would be feasible to run both such pilot studies and supplementary comparisons in the future. Dr May replied that it was a question of engaging laboratories with no experience in such exercises and this could be done by giving assurances of public anonymity to avoid embarrassment if results were poor. The newly proposed sub-group structure would eliminate this problem because key comparisons would not be involved and parallel pilots could be available to substantiate performance claims but in such cases the laboratories would have to agree to release of the results publicly. Key comparisons would thus continue to be available to support CMC claims.

9 METROLOGY FOR BIOFUELS

The President extended a warm welcome to Dr Jornada, President of INMETRO, who proceeded to introduce the topic of metrology with respect to biofuels. He drew attention to a briefing document distributed to all Delegates, a White Paper prepared by a tripartite Task Force comprising Brazil, the US and the EU on “Internationally Compatible Biofuel Standards”. Dr da Jornada noted that this was a challenging new opportunity for metrology and proceeded to outline general problems, developments and co-operations in this field. Biofuels are central to major global concerns and will have a significant impact on energy and the environment with respect to sustainability, security of fuel supply and competitiveness. Many world governments are responding to these concerns: the EU will require 10 % of all fuels to be derived from biomass by 2020; the US Energy Independence and Security Act calls for development of ethanol and biodiesel production. World production of biofuels has reached some 60 billion litres in 2007 and specifications as to energy content/purity/origin are now of interest. This fast rising global commodity will bring with it new international/national regulations and the large economic consequences will demand uniformity of quality for measurements to support its use and trade. Some of the anticipated metrology needs include net energy, harmful impurities, other physico-chemical characteristics (e.g., jellation temperature) and provenance. Practical needs will include CRM development, PT organization, instrument development, basic scientific research and measurements relating to impact (environment, engine corrosion, health). Possible roles of the CIPM were presented, including encouragement of CMCs to support this area, development of recommendations to guide regulators, accreditors and standards bodies, development of measurement methodologies and the fostering of exchange of information amongst NMIs.

Dr da Jornada concluded by describing INMETRO’s activities for biofuels, noting the release of a CRM for ethanol in water, participation in CCQM-K27 (Ethanol in aqueous matrix) as well as a programme to identify provenance based on ESI-MS techniques. He briefly elaborated on a

collaboration between NIST and INMETRO to develop CRMs for these purposes, a bioethanol by INMETRO and a biodiesel by NIST, and an invitation to IRMM to join these activities. A Tripartite Agreement for Biofuels was established in June 2007 amongst Brazil, EU and the US will allow the biofuel issue to move forward by creating a task force to prepare a report on metrology and biofuels with recommendations to the CIPM/BIPM and NMIs and begin sharing such findings with other standards bodies, such as ISO.

The President summarized by noting that many points raised were for the CCQM regarding establishment of comparability and traceability of measurements in this field but there was a need to have a clearer understanding of what was to be measured, including ranges and target uncertainties. Both the IAWG and the OAWG should be engaged in these activities as it is not seen to be a complex problem. As Secretary to the CIPM, he will ensure that this topic is discussed at its next meeting.

Dr Emons agreed that biofuels was commanding significant attention in the EU and that there was a need to understand both the metrology issues and the politics surrounding this matter. Notwithstanding the need for reliable measurements, discussions between the CIPM/BIPM and relevant standards organizations was called for in an effort to ensure harmonization with, for example ASTM and ISO.

Dr Sommer (PTB) viewed the whole field as not being particularly challenging but suggested that physical and chemical measurements need to be combined so as to include both solid/liquid and gaseous biofuels. He stressed that the area needs international coordination efforts and noted that there would be a workshop in November in Strasbourg organized by PTB and LNE for stakeholders to determine the future needs in biofuels. He further suggested that a task force or *ad hoc* group to coordinate activities should be established. The President concurred, stating that this was feasible.

Dr Siekmann remarked that the German Ministry of the Environment needs to withdraw the 10 % ethanol content in fuels because of an excessive risk of engine damage in vehicles. The question is whether this is the result of the ethanol itself or contaminants.

Dr Sargent indicated that some of the parameters already identified can be tackled and it will be possible to co-ordinate a joint session of the OAWG and IAWG during the fall of 2009 when the meeting is hosted by INMETRO.

The President stressed that it was necessary to know what parameters required measurement, what CRMs were needed to support this and once this was known, other stakeholders could be rationally identified. The international conference on “Accelerating Innovation in 21st Century Biosciences” scheduled for October 2008 at the NIST would be useful for this purpose as energy is a target for discussions.

Dr Emons emphasized the need to have organized communication channels, suggesting that, in this regard, the CIPM would be an exceptionally useful portal to other organizations since the importance of biofuels will only continue to expand.

The President suggested that a workshop could be organized in much the same manner as had been undertaken in the past for foods but there was a necessity to avoid duplication of work underway that

could have another route forward. Dr da Jornada indicated that Brazil could organize a workshop in 2009.

Dr Charlet (LNE) stated that further information could be made available to those interested in the PTB/LNE workshop in Strasbourg and the President asked that this be co-ordinated through the CCQM Executive Secretary, Dr Wielgosz.

10 REDEFINITION OF THE MOLE

Dr Milton presented a progress report on behalf of the *ad hoc* working group focussing on the redefinition of the mole. He briefly summarized the background of this issue, noting that the CCQM had passed a Recommendation in 2005 relating to the re-definition of the kilogram which emphasised the importance of resolving any outstanding discrepancies between available values of Planck's constant as derived from measurements made with moving-coil watt balances and that derived from the "Avogadro experiment" involving X-ray crystal diffraction on a silicon artefact. In 2006, the CCQM had received a comprehensive presentation from Prof. Ian Mills, the President of the CCU, in which he had elucidated many of the options for wording possible re-definitions of the kilogram, the kelvin, the ampere and the mole. The CCQM and the other CCs had been asked to give their views on these proposals and, as a consequence, the President asked Dr Milton to assemble and chair an *ad hoc* WG in April 2007, which ultimately consisted of Drs Milton, Kaarls, Wielgosz, Besley, Prof. De Bièvre and Dr Salit. Consultations had been held with Prof. Mills and a paper submitted to the CCQM after a meeting of the group in Sydney during the fall of 2007. Resolution 12 of the 23rd CGPM meeting in 2007 recommended that NMIs and the BIPM, amongst other things, initiate awareness campaigns to alert user communities to the possibility of such redefinitions and their implications.

Dr Milton reiterated his view from the 13th CCQM meeting that there was no fundamental objection to moving to a definition of the mole based on a fixed value for the Avogadro constant since it is, in fact, an amount of substance. He proceeded to outline the relationship between atomic mass and the fundamental constants, noting that the new definition will result in moving from a fixed molar mass of carbon with no associated uncertainty at present to a system wherein the uncertainty of the atomic mass unit is also picked up by the molar mass unit and that of the Avogadro constant then assigned zero uncertainty. Dr Martin and Prof. Mills are intending to publish a paper on this and issue a wording of the revised definition of the mole, noting that other stakeholders, such as the ACS and IUPAC, would be fully engaged in the process and, as a consequence, refrained from presenting such a definition at this time.

Dr Quinn congratulated Dr Milton on his presentation and noted that while these matters were in one sense very simple, they were also very subtle, and the more widely it is discussed the more people will think about it and the clearer it will become. He continued on to note one additional important

point not directly mentioned was that by redefining the mole based on fixing the value of the Avogadro constant we are deliberately breaking the link with mass, decoupling it from the kilogram, and this is important and helpful because, in his view as a physicist, there has always been slight confusion between the amount of substance and mass when units were defined on the basis of the fixed mass of carbon.

Professor De Bièvre admitted that, during the considerable time he has devoted to giving consideration to this matter over the past few years, he could not agree more with Dr Quinn's remark on the concept of breaking the link between amount of substance and mass (and defending that against physicists) since, in principle, the amount of substance is a base quantity. Therefore, the new definition will result in a more simplified approach overall as all academics are currently confused over the current definition of the mole in any case.

11 A EUROPEAN NETWORK OF FORENSIC SCIENCE INSTITUTES (ENFSI)

The President welcomed Dr Bertler, chairperson of the Quality and Competence Standing Committee, ENFSI, and representative of the European Network of Forensic Science Institutes, who presented an overview of activities in the forensic science community. The ENFSI was founded in the mid-90's as a forum for information exchange and currently fifty-four members from 31 countries strive to ensure the quality of development and delivery of forensic science throughout Europe. She outlined the structure of the network and the aims of the various expert working groups which foster harmonization of methods, promotion of quality assurance, engage in training seminars, author best practices manuals and exchange information to promote the science. In addition, two standing committees, i.e., the European Academy of Forensic Science and Quality and Competence Committee constitute the organization. The latter develops policies and provides advice to the expert working groups and ENFSI members. All member laboratories have achieved (24 to date), or are taking steps towards achieving accreditation to ISO/IEC 17025 or ISO/IEC 17020 with ILAC-G19:2002 (Guideline for Forensic Laboratories) serving as a guide. Dr Bertler noted that appropriate CRMs are rarely available and thus in-house reference materials and standards are frequently utilized. Proficiency testing/collaborative exercises are typically arranged by the expert groups. Harmonization of techniques is required for collection and comparison of information as there is an increasing need for compatible databases with the rise in "internationalism" of crime. Future activities will focus on accreditation of more forensic laboratories, education of stakeholders, tackling the issue of compatible databases, sampling techniques, reference materials, proficiency tests and uncertainty of both quantitative and qualitative measurements.

The presentation generated significant discussion amongst the Delegates. The President noted that there was an obvious need for traceability in international forensic results and in response to his question about linkages with the global community it was noted that a Memorandum of Understanding (MoU) has been signed for the regional members and that this will eventually be

extended to an international level. Dr Güttler commented that there are clearly long- and short-term goals to be achieved but that the availability of reference materials is long term in nature and wondered if there was any priority list of needs available. Dr Bertler responded that this issue will normally be undertaken by the expert working groups but, as a whole, common ones such as those relating to terrorism (e.g., fingerprints) are required but there was no overall priority list exists within ENFSI. The President remarked that such a situation makes it difficult to obtain support from the CCQM and it would be better to establish what is needed. Mrs Parkes remarked that, in an effort to share information, the BAWG could invite ENFSI to working group meetings when DNA is topical. The President noted that such invitations should be encouraged for the other WGs of the CCQM. Dr Bertler invited the BIPM to attend the next board meeting of the ENSFI. Dr Güttler suggested that there was a need to expand the communications between ENFSI and the CCQM WG members, noting that this could be accomplished through their website at www.enfsi.eu. Dr Besley questioned the extent to which the uncertainty in forensic measurements is considered by the court systems. Dr Siekmann noted a parallel situation between the activities of the ENSFI and the global network of anti-doping laboratories in that both desire traceability; the PT schemes currently undertaken by WADA may be a valuable resource for the ENFSI in that the prepared samples might be shared. In regard to the issue of uncertainty, Dr Emons asked how this is handled in the many forensic situations for which a simple presence/absence testing is conducted. Dr Bertler replied that proficiency testing is still needed to support such testing and the issue is in the early stages of consideration, whereupon Dr Emons noted that several other organizations are also working on this issue (a WG in ISO REMCO for example) and suggested that joint activity would be beneficial. Professor Wallard expressed a desire for such partnerships. Mr Squirrell noted that defence lawyers are more frequently making reference to traceability of results. Dr Dybkaer added that a large report on quantitative analysis and uncertainty, some 2-3 years old, is available from the EC.

12 CODEX ALIMENTARIUS COMMISSION REPORT

The President welcomed Dr Doyran from the Codex Alimentarius Commission to the meeting. She outlined the background to the Codex Alimentarius Commission and explained that it was a joint organisation involving the Food and Agriculture Organisation (FAO) and the WHO. She then presented her report on the work of the Committee on Methods of Analysis and Sampling which hosted its 29th meeting in Budapest, March 2008. Several of the highlights included:

- guidelines for evaluating acceptable methods, which is expected to be finalized within four years;
- guidelines to governments for settling disputes over analytical results (to require QA in the laboratories, report of uncertainties, use of methods recommended by CODEX or otherwise validated methods, and possible arbitration by a third party laboratory using reserve samples);

- guidelines for measurement uncertainty, including an appropriate approach for sampling uncertainty which would encompass the work of other organizations (EURACHEM, NordTest);
- draft guidelines on analytical terminology, which were revised in 2003 and are undergoing re-revision to take into account terminology in the VIM3; this is expected to be completed in 2009;
- criteria for the methods for detection and identification of foods derived from biotechnology, an emerging area distinct from the considerations of labelling, to provide a document addressing definitions, method development to formal validation, method acceptance criteria, collaborative trial requirements and validation of PCR and protein based methods, units of measurement, uncertainty, reference materials and sampling;
- the conversion of methods for trace elements into criteria: new criteria were finalized for adoption by the Commission concerning applicability, limits of detection and quantitation, precision, recovery and trueness and the examination of current methods to ensure that they meet the new guideline criteria.

The President remarked that he was pleased with the cooperation between the BIPM and the Codex Commission. He asked that the Codex Commission inform the BIPM of what its priorities were. Dr Fajgelj commented that it was pleasing to see that CCMAS was adopting terms from the VIM3 and harmonizing terminology.

13 THE JOINT COMMITTEE ON TRACEABILITY IN LABORATORY MEDICINE (JCTLM)

13.1 JCTLM WG1

Dr Wielgosz presented an update on the work of the JCTLM and its WG1 on Reference Materials and Measurement Methods. Its mandate is to review nominated higher-order reference materials and methods for publication in the JCTLM database. A number of technical review teams, and another dealing with quality systems carry out the work of WG1. The list of materials is divided into two parts – one for which SI traceability is available, and one for which it is not. A searchable database on the JCTLM website contains some 202 CRMs (50 % from North America), 139 approved traceable and validated reference measurement procedures (RMPs) and 98 laboratory reference measurement services (RMS, 90 % from Europe). Approximately 725 visits per month were registered for the database, primarily from the IVD industry and hospitals.

Dr Wielgosz then proceeded to summarize the activities of the JCTLM for 2007, which included reviews of nominations for CRMs, methods and reference measurement services, Executive Committee and WG meetings as well as the symposium on “Activities and Challenges for Traceability in Laboratory Medicine” held in Beijing in October, the formation of a task group on Laboratory Network Accreditation and participation in the review of ISO TC 212 WG2 Standards. It was noted that ISO standards for higher order RMs and RMPs include ISO 17511, 18153, 15193,

15194 and 15195. A tutorial on the use of the JCTLM database illustrated which higher order RMs can be used by an IVD manufacturer for calibration along with its traceability and listed reference methods with potential laboratories capable of delivering the measurement service.

After highlighting a liaison with the EC Enterprise and Industry Directorate-General, noting that their draft comment on the compliance of RMs needed to be modified to cover all manadated written standards, he then turned to a consideration of activities for 2008, mentioning the development of national activities on traceability in laboratory medicine, accreditation of laboratory networks, and discussions aimed at ensuring consistency of the BIPM KCDB and the JCTLM database for CMCs and RMSs.

Dr Wielgosz concluded with an outline of requests to the CCQM for experts from NMIs to participate on JCTLM teams addressing drugs, vitamins and non-peptide hormones and a request for any NMIs offering CMCs or reference measurement services to please submit them for review and publication in the JCTLM database; currently only PTB is registered. He noted that both the WG1 Cycle V call for CRMs and RMs and WG2 Cycle III call for RMS nominations close as of 30 May 2008 and that meetings of both WGs are scheduled for Washington in July 2008 with an Executive and Stakeholders meeting at the BIPM in December 2008.

13.2 JCTLM WG2

Professor Siekmann described the work of WG2 of the JCTLM, which is concerned with reference measurement laboratory services. He reported that of the 197 services from 21 laboratories covering 74 measurands which had been nominated in 2007, 104 services, 14 laboratories and 32 measurands had been recommended for approval by the review teams for listing on the JCTLM database. The primary reasons for rejection were that either no metrologically acceptable reference procedure was used or there was no evidence of (annual) participation in PT schemes or a quality assessment of the laboratory in accordance with ISO/IEC 17025 and 15195 (or an NMI being listed in Appendix C). He then outlined the procedure for listing or delisting a laboratory and the information that reference laboratories were asked to submit to the working group. In 2003 the IFCC created a proficiency testing system for use by such reference laboratories (EQAS) and the number of measurands has risen from 94 to 168 by 2008. He noted that reference laboratories have acceptance limits that are set at one-quarter those limits achieved by commercial laboratories operating in Germany.

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

Dr Milton remarked that the Youden plots were interesting but wanted to know why there appeared to be a bias between field laboratories and reference laboratories. Professor Siekmann replied that some rather large deviations occurred because of the commercial test kits that were used and in trying to address this problem it became evident that some 2-3 years were needed because it was necessary to inform the EU authorities and the FDA and then wait for manufacturers to correct the problem.

In response to Dr Dybkaer's question of what happens if a competent laboratory exists but can provide no evidence of participation in PT schemes, Prof. Siekmann stated that such a laboratory would be requested to submit a nomination for its procedure and any information on new measurands. He acknowledged that it was also possible that only one laboratory may be available to make a particular measurement and, as such cases do exist, this being all the more reason why NMIs should participate in such studies to provide a second set of data for comparison.

The President remarked that this was similar to the case of the CCQM undertaking a bilateral comparison when a new NMI wishes to exhibit capability and thanked Prof. Siekmann for his presentation.

14 WORLD HEALTH ORGANIZATION – NIBSC

Dr Gray presented a report on 2007/2008 standardization activities of the National Institute for Biological Standards and Control, the major WHO laboratory. She prefaced her remarks by briefly outlining the objectives of the WHO (attainment by all people of the highest possible level of health) and noted that it was mandated to develop, establish and promote international standards for biological products. In this respect, WHO produces both norms and physical standards for biological products, noting that NIBSC produces and holds >95 % of the WHO standards. In 2007, thirteen new international standards were established in the fields of antigens, blood products, cytokines, growth factors, endocrinological substances and diagnostic reagents; these have been released and are currently being evaluated. She briefly summarized the preparation/activity and status of each of them. New projects proposed for 2008 in the areas of virology, bacteriology, immuno/ endocrinology, haematology, genetic reference materials and haemostasis were then presented.

The development of the first international standard for parathyroid hormone 1-84 (PTH) was singled out for significance as it exemplifies the pressing requirement for an international standard that is calibrated in mass or molar units, reflecting the current trend for peptide measurements away from activity units. This has become a reality due to developments in instrumentation and concepts of traceability which make such links to the SI possible. This need is recognized by WHO and for well-characterized biologicals this will be successful as the measurand can be specified; limitations will continue in those situations where the ability to define the biological reagent remains unclear.

Dr Wielgosz questioned the general move to mass basis for PTH and asked how reproducible the identity of PTH was in that if a second batch of PTH was to be prepared would it possess the same mass to activity ratio as the current one, to which Dr Gray stated that, although it was not her area of expertise, this remained to be determined.

Mr Squirrell asked how new standards, released each year in October, were correlated to the activity of the old batch, noting that this is likely to change, thus altering calibration and response characteristics. Dr Gray replied that this was a recurrent question that there was no clear answer for it other than to state that the new batch of calibrant defines a new International Unit but the new batch is calibrated against the old one in an effort to maintain continuity. However, she did admit that there may be circumstances in which some drift in the unit may occur and in such case the end user is informed. This effectively means that the activity as defined by the old standard will no longer be used.

Dr Mitani asked how to implement suggestions and recommendations from WHO so as to ensure that standards can be made traceable to NMIs, to which Dr Gray replied that project leaders for NIBSC studies are always looking for collaborative laboratories such that the range of measurement methods used is as wide as possible, suggesting that NMIs contact NIBSC to get onto their circulation list for future studies. The President agreed that this would certainly be a good recommendation.

15 WORLD METEOROLOGICAL ORGANIZATION – GAWG

Dr Milton presented a short report describing progress in the collaboration between the GAWG and the WMO-GAW. It had been proposed several years earlier that certain NMIs active in the GAWG would provide the central calibration laboratory for the GAW with calibration standards for volatile organic compounds (VOCs). The collaboration thus principally concerns the provision of standards for VOCs at the parts-per-billion level, which are of interest to the GAW because of their role in the generation of ozone in the troposphere. Measurements of such VOCs were required to be stable to better than 1 % per year; 10 such compounds were selected for study (ethane, propane, iso-butane, acetylene, 2-methyl butane, n-pentane, isoprene, benzene and toluene) out of some 30. Results for the first technical international comparison, EUROMET.QM-S2, involving NMi VSL, NIST, KRISS and NPL (co-ordinating laboratory), showed no significant bias with those from the GAW being equivalent to the results from the NMIs and no drift over a 12-month period from the gravimetric values within the 95 % CI.

Dr Milton then outlined the next steps to the collaboration which would address additional VOCs (terpenes, dimethylsulfide, formaldehyde, methanol, acetonitrile, ethanol and acetone) and engage in discussions with GAW on how delivered SI-traceable values could be understood by their users in terms of their current “scale” so as to normalize results to historical values. A draft Memorandum of

Cooperation initiated by the NIST between those NMIs active in the provision of low concentration VOC gas standards (KRISS, NIST, NMi VSL and NPL) and the WMO-GAW remains under preparation as a result of the need to clarify objectives and deliverables. A workshop on VOCs is scheduled for July 2009 at EMPA. A BIPM/WMO workshop on 'Measurement Challenges for Global Observation Systems and Climate Change Monitoring' is being organized and will be held in Geneva with the intention of bringing together a number of relevant CC groups and WMO experts.

16 ISO REMCO

Dr van der Veen, chairman of ISO REMCO, presented a short update of activities by stakeholders (standardisation bodies, NMIs and international and regional liaison partners) and clients (ISO committees, reference material users and producers and accreditation bodies). He briefly outlined the structure of REMCO, summarized developments in the revision and drafting of new ISO Guides, as well as activities relating to development of new reference materials to support qualitative measurements (chemical weapons, colour, fraud detection and drug testing). Qualitative measurements account for some 95 % of the testing activity in industry for which compliance/non-compliance decisions are taken and although some CRMs do exist for this purpose, guidance on good practice is lacking. ISO Guide 30 (Terminology) has been reviewed with terminology based on the VIM. A new guide on quality-control reference materials (ISO Guide 80) is close to completion and will address "in-house" preparation of RMs and distinguishes between certified RMs and non-certified RMs. ISO Guide 33 is under revision and will provide a wider scope in covering applications of RMs as well as addressing calibration (formerly covered in Guide 32). ISO Guide 34 was subjected to limited revision to improve the text and provide more explanation based on assessors experience and feedback and will undergo a final vote this fall. ISO Guide 79 provides for an introductory guide to RMs/CRMs and a discussion on its exact contents is on-going but an opportunity will be taken to develop a rule book for CRM users, legislators, regulators and standards developers. A report on the 30th meeting of ISO REMCO is available in *Accred. Qual. Assur.*, 2008, **13**, 53-55.

Dr Fajgelj commented on the Committee structure, noting that with most members of the steering and working groups present at the current CCQM meeting, it is difficult to understand why issues relating to harmonization of definitions such as RM and CRM amongst the two bodies persist, generating an unhealthy state of affairs. Furthermore, stakeholders include the members of the BIPM and as ISO REMCO documents are circulated to various standardization bodies this should be done more comprehensively in the future by involving the BIPM directly. The President agreed with the regrettable nature of this situation and expressed hope that this would soon be resolved.

Dr Wielgosz asked a more general question of how the ISO system deals with different vocabularies and the harmonization of different definitions, to which Dr van der Veen replied that ISO does not deal with harmonization and noted that there are several terms with different meanings freely being

used by each Technical Committee and as there is no hierarchy of vocabularies, they all have the same status. However, it is recognized that there is a need to accomplish this in connection with the VIM to eliminate these differences. Dr Emons noted that the same questions arose at a recent workshop on nanotechnology where there are three to five different definitions for the same term and that the problem exists within and among organizations, highlighting the need for closer ties to the BIPM to effect such harmonization. The President commented that within the ISO organization there is clearly room for better infrastructure. Mr Squirrell said that such issues have been continually raised but there appears to be no progress in these matters. He noted that a new group, the Technical Interface Group (TIG), had now been set up by ISO to specifically address this shortcoming of communications between groups and is meeting next week. He strongly recommended that someone from metrology be represented on the TIG. ILAC is extremely concerned about resolving this situation and feels that the TIG may be able to finally provide a vehicle to address this and that it is a positive move forward by ISO, especially if the BIPM obtains formal membership on the TIG.

17 INTERNATIONAL ATOMIC ENERGY AGENCY

Dr Fajgelj reported that the IAEA would be hosting the joint meeting of the IAWG and the EAWG in October and that a workshop on uncertainty of measurement of stable and light element isotope ratios would be organized with IUPAC.

18 INTERNATIONAL UNION OF PURE AND APPLIED CHEMISTRY

Dr Fajgelj presented his (and Prof. De Bièvre's) report on activities, announcing that the 41st IUPAC World Chemistry Congress and 43rd IUPAC General Assembly had convened in August 2007 in Turin. He noted that all copies of *Pure and Applied Chemistry* since 1964 were now freely available electronically, that the 3rd edition of "Quantities, Units and Symbols in Physical Chemistry" had been published in 2007 and that the on-line version of the "Gold Book" addressing general terms had been updated but would no longer be available in hard copy. IUPAC is proposing 2011 to be the International Year of Chemistry. Required approval by UNESCO and the UN is pending. In the case that this approval is not granted, IUPAC still plans to go ahead and organize a Year of Chemistry. IUPAC is looking for support by other organizations having interest in chemistry, including the BIPM and the CCQM.

Professor De Bièvre then proceeded to provide some information about the work of the Commission on Isotope Abundance and Atomic Weights (CIAAW), summarizing a number of recently approved documents that present the most recently published and evaluated data. The CIAAW convenes every two years (2007 in Pisa, 2009 in Glasgow). Data for atomic weights have been reviewed and approved in 2005 and appeared in *Pure and Applied Chemistry* in 2006. The latest data for isotopic compositions of the elements appears in the *Journal of Physical and Chemical Reference Data* in 2005. Professor De Bièvre emphasized, through reference to several selected Commission approved documents, that the atomic weights should not be considered as "fundamental" constants of nature. He concluded by announcing that the IUPAC project "Metrological Traceability of Measurement Results in Chemistry" is nearing completion. Its aim is to demonstrate the scientific basis of metrological traceability and provide practical examples of traceability chains in chemical measurements. A final draft of the document was presented to the IUPAC General Assembly in Turin and sent for comments to 14 international organizations, including the CCQM. Although it was posted on the IUPAC website from October 2007 to February 2008, Prof. De Bièvre emphasized that comments were still welcomed.

Dr Milton asked for clarification of what the next process would be for this traceability document after all comments had been received, to which Prof. De Bièvre replied that work is continuing but that by the end of the summer it would be completed and ready for publication in *Pure and Applied Chemistry*, as no second draft was anticipated. Dr Milton asked if there would not be a second chance for comments on the "final" draft. Dr Fajgelj stated that based on these discussions and the concerns noted that they will reconsider what will be done with a possible second draft but did not commit to the process. The President noted that while the position of IUPAC was clear it would be in their best interests to consider this. Dr Wielgosz noted that the BIPM was one of the organizations that did send comments, noting that the original draft did contain some fundamental errors and felt that there should be an opportunity for a second round of comments on a revised version. Dr Fajgelj reiterated that IUPAC may reconsider their policy regarding the next step of the process, to which Prof. De Bièvre agreed that it was important enough to ensure that all expert comments were taken into account.

19 INTERNATIONAL LABORATORY ACCREDITATION COMMITTEE

Mr Squirrell provided some information on behalf of ILAC, noting that 128 members in 18 countries comprised 98 accrediting bodies of which 59 are signatories to its multi-lateral agreement. Some 30 000 laboratories are now accredited (mostly to ISO 17025) with approximately 10 000 of these undertaking chemical measurements. That being said, there remains a much larger suite of unaccredited laboratories. Approximately 50-60 % of NMIs are accredited for their routine calibration services and the ILAC is attempting to develop an international MRA for reference materials producers to give them a sense of elevated recognition that they deserve.

Mr Squirrell then presented a general overview of the 2001 MoU, outlining collaborations between the BIPM and ILAC, noting that activities between the Regional Cooperation of Accreditation Bodies (RCAB) and RMOs need to be strengthened. He remarked that ILAC was able to participate in the APMP meeting last year in Sydney and had been invited to the CGPM. Specific task groups were working on issues relating to CMCs, ISO REMCO and several ISO TCs. He then turned to a consideration of the CMC issue.

He was pleased that BMCs and CMCs were now considered to be the same but cautioned that there was still a need for more understanding on this matter and of what exactly goes into the CMC database, which needs to be used more frequently by assessors. Situations must be avoided wherein the CMC for an NMI has an uncertainty greater than that of an accredited laboratory. There are also cases where the CMC and the results of a key comparison are not in agreement and this needs closer examination. He noted that some NMIs go through an accreditation process and some do not, opting for an on-site peer review as well as review of KC and PT performance. ILAC is thus working on guidance documents addressing harmonized assessment processes for those NMIs that choose accreditation that will take account of RMO processes so as to avoid any duplication of efforts. Improved communications between the RMOs and the RABs would achieve a better understanding of the issues. He concluded by remarking that the close cooperation between ILAC and the BIPM has produced several positive results of benefit to both parties and that ILAC is most appreciative of the opportunities for discussions on issues of interest to both organizations.

Dr Fajgelj commented on the output of peer reviews of NMIs and specifically the IAEA and would like to see a more formal document to recognise successful peer review. Professor Wallard welcomed the very positive report and noted that the greatest change has been the move from very high level agreements, such as those relating to the issue of BMC and CMC, to move further downstream wherein within the next few years the real benefits will arise. The recent third meeting between the RMOs and RABs was very positive and showed a commitment and agreement to making policy documents and he expressed hope that the next few years will show continued progress to demonstrate competencies in traceability systems in laboratories.

20 CITAC

The President welcomed Dr Kuselman, the current chair of CITAC, who proceeded to provide a brief overview of their structure and board, noting that it was created in 1993 and currently comprises some 34 members with 4 representatives from 25 countries. The primary mission of CITAC is to globally improve traceability and comparability of the results of chemical measurements by dissemination of information accumulated by NMIs. A multi-faceted strategy was delineated to achieve this goal, including close cooperation with many organizations, societies and institutions, publication of scientific papers addressing metrological principles, organization of seminars, symposia and workshops, assisting analytical laboratories with needed tools, clarifying

concepts and disseminating them globally. Three new CITAC Guides were under development, including: EURACHEM/CITAC Guide “Assessing performance in qualitative analysis. “The expression of uncertainties in qualitative analysis and testing”; EURACHEM/CITAC Guide “The fitness for purpose of analytical methods – a laboratory guide to method validation and related topics”; and IUPAC/CITAC Guide “Selection and use of proficiency testing schemes for a limited number of participants”. He concluded by announcing the CITAC Award to highlight authors of remarkable papers and its nomination procedure and highlighted the new CITAC logo.

21 CCQM WORKSHOPS

The President summarized a number of announcements and discussions relating to workshops that had been aired during the past two days of the meeting, including the special workshop with the pharmacopoeia community 4-5 December 2008; the biofuels related meetings in Strasbourg organized by PTB/LNE for November and the NIST meeting in October. He welcomed the final reports from the KCWG and EETWG within a year from now and indicated that these would be supported by appropriate workshops. The President then welcomed further suggestions; none were raised.

22 CCQM RECOMMENDATIONS

The President summarized by stating that there were no special major Recommendations or Draft Resolutions that needed to be conveyed to the CIPM or CGPM.

23 ANY OTHER BUSINESS

Dr Fajgelj returned to the issue of neutron activation analysis to note that in the minutes of last years’ meeting it was agreed that this technique would be added to the list of potential primary methods of analysis but that he felt this decision should be formulated in a more prominent manner.

The President agreed that this was in the minutes of the last meeting and that they had been accepted and further reminded all Delegates that a year ago he had promised to return to this list of potential primary methods and that in consideration of their metrological importance he would indeed do this.

Dr Besley expressed concern that the last few CCQM meetings devoted too much time to the BIPM key comparison database and associated comparisons and not enough time on strategic issues. However, he did find evidence in this meeting which may suggest a move back to more scientific issues being discussed and he encouraged this trend. He pointed out that CIPM-MRA issues must not be permitted to take over our primary tasks by overemphasizing their importance.

Dr Sargent suggested that the activities of the CCQM need to be more widely publicized and suggested the idea of a newsletter to highlight them.

Professor De Bièvre praised the IAWG and OAWG for the high quality of their work being done, suggesting that it should be more openly available to the public and urged that publication should be encouraged. He added that he would be pleased to help facilitate this by offering the services of *Accred. Qual. Assur*.

Dr Mitani was of the opinion that there was a need for more discussions on the issue of CMCs and the preparation of guidelines with respect to use of key comparisons.

Professor Wallard mentioned IUPAC's interesting proposal to have the International Year of Chemistry in 2011 and noted the similarity to the re-launch of World Metrology Day on 20 May for which UNESCO had to again be consulted. World Metrology Day is now in its fourth year and has surpassed all the Director's expectations. This year's 20 May poster will appear in some 90 languages with the theme of Olympic Sport.

24 DATE OF NEXT MEETING

The next meeting of the CCQM Plenary was fixed for 23-24 April 2009 at the BIPM with the prior days reserved for meetings of the CCQM WGs and a workshop.

24.1 Coordination of CCQM working groups meetings

The President was grateful to INMETRO for extending an invitation to host a meeting of the CCQM WGs during the week of 26-30 October 2009 in Rio de Janeiro (Brazil). This will be the first time that the CCQM will meet in South America. Hopefully, there would be no conflict with APMP or SIM General Assemblies and details would be developed and provided in the coming year.

24.2 Closure

The President closed the meeting by thanking all participants for their contributions to a successful plenary and prior week of meetings and expressed best wishes for safe travel to all participants.

R.E. Sturgeon, rapporteur

22 April 2008, revised July 2008

APPENDIX Q 1.

Working documents submitted to the CCQM at its 14th meeting

Working documents submitted to the CCQM at its 14th meeting are on restricted access.