#### **Bureau International des Poids et Mesures**

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

Report of the 12th meeting (6– 7 April 2006) to the International Committee for Weights and Measures



#### Comité international des poids et mesures

Bureau international des poids et mesures Organization intergouvernementale de la Convention du Mètre Note:

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

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

A.J. Wallard, Director BIPM

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

as of 6 April 2006

#### **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], Queretaro.

CSIR – National Measurement Laboratory [CSIR-NML], Pretoria.

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

Danish Institute of Fundamental Metrology [DFM], Lyngby.

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 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.

State Laboratory [SL], Kildare.

Swedish National Testing and Research Institute [SP], Borås.

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

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

#### **Observers**

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

Centro Español de Metrología [CEM], Madrid.

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 Office of Measures/Országos Mérésügyi Hivatal [OMH], Budapest.

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

Standards, Productivity and Innovation Board [SPRING], Singapore.

#### 1 OPENING OF THE MEETING; APPROVAL OF THE AGENDA

The Consultative Committee for Amount of Substance: metrology in chemistry (CCQM)\* held its twelfth meeting at the International Bureau of Weights of Measures (BIPM), at Sèvres on 6-7 April 2006.

The following were present: L. Besley (NMIA), A. Botha (CSIR-NML), R. Cavanagh (NIST), P. Charlet (LNE), K. Chiba (NMIJ/AIST), E.W.B. de Leer (NMi VSL), R. Dybkaer (IFCC), H. Emons (IRMM), H. Ent (NMi VSL), A. Fajgelj (IAEA/IUPAC), J.-C. Forest (IFCC), J.A. Guardado Pérez (CENAM), B. Güttler (PTB), H.-P. Haerri (METAS), W. Hässelbarth (BAM), J. S. Kim (KRISS), H.D. Jensen (DFM), R. Kaarls (President of the CCQM), K. Kato (NMIJ/AIST), Y. Kustikov (VNIIM), W. Louw (CSIR-NML), L. Mackay (NMIA), B. Magnusson (SP), M. Máriássy (SMU), W.E. May (NIST), J. McLaren (NRC), M.J.T. Milton (NPL), Y. Mitani (CENAM), D.W. Moon (KRISS), U. Panne (BAM), H. Parkes (LGC), M. Pérez-Urquiza (CENAM), M. Sargent (LGC), L. Siekmann (RfB), T. Steiger (BAM), R. Sturgeon (NRC), A. van der Veen (ISO REMCO), A.J. Wallard (Director of the BIPM), S. Windsor (NPL), S.A. Wise (NIST), Yadong Yu (NIM).

Observers: H. Brandi (INMETRO), M. Gallorini (INRIM), P.K. Gupta (NPLI), W. Kozlowski (GUM), B. Martin (CEM), E. Rizzio (INRIM).

Invited: A. Bristow (NIBSC), C. Cherdchu (NIMT), P. De Bièvre (International Coordination Committee of the Avogadro Project, IAC), E. Gray (NIBSC), V. Ivanova (WADA), D. Ivanova (NCM-SAMTS), G. Massif (Fundacion Chile), I.M. Mills (CCU, Univ. Reading), V.M.L. Ponçano (IPT, CITAC), D.W.M. Sin (Government Laboratory, Hong Kong), A. Squirrell (ILAC), M. Walsh (AOAC International).

Also present: P. Giacomo, T.J. Quinn (Emeritus Directors of the BIPM); M. Esler, R. Josephs, P. Moussay, C. Thomas, J. Viallon, S. Westwood (BIPM), R. Wielgosz (Executive Secretary of the CCQM, BIPM).

Excused: B. Brady (SL), V. de Souza (INMETRO), E. Deák (OMH), I. Kuselman (INPL), F. Sogut (UME), S. Tan (SPRING Singapore), P. Taylor (IRMM), T.L. Ting (Government Laboratory, Hong Kong).

Dr Kaarls welcomed members, observers and guests to the twelfth meeting of the CCQM. He noted that a number of guests were representing international organizations that were not yet members or observers of the CCQM, but had considerable interest in metrology in chemistry. These organizations included ILAC, AOAC International, WADA, NIBSC and CITAC. Professor Wallard added his welcome to everyone to the BIPM. Dr Kaarls noted that this would be the last CCQM meeting to be attended by Dr Hässelbarth who was retiring from the BAM and thanked him for his continuous and useful contributions over many years.

<sup>\*</sup> For the list of acronyms, <u>click here</u>.

#### 2 APPOINTMENT OF A RAPPORTEUR

The President said that Dr Milton had again expressed his willingness to act as the rapporteur for the meeting. Dr Wielgosz would assist him.

#### 3 APPROVAL OF THE AGENDA

The agenda was approved.

#### 4 REPORT OF THE ELEVENTH MEETING

The report of the eleventh meeting was approved.

#### 5 POSSIBLE REDEFINITION OF THE MOLE

The President said that the possible redefinition of the mole was an issue of great interest to the CCQM and that he had invited Prof. Mills, the President of the Consultative Committee for Units (CCU) to discuss the topic.

Prof. Mills said he was delighted to speak to the CCQM, and he had chosen a sub-title for his talk of "the SI units for the 21st century". He explained the definitions of the seven base units of the SI and how these led to certain "fixed" quantities such as the speed of light and the molar mass of carbon-12. He said that the present definitions could be re-worded in a simpler way, although that was not the subject of his proposal. The second and the metre are both defined in terms of fundamental properties of nature, but the kilogram, the ampere, the kelvin and the mole were not. Of these, the ampere and the mole depended on the kilogram.

The desirable properties of a definition of a base unit were that it should be: an invariant property of nature, simple, accurate, and freely available to anyone, anywhere, at any time. He then introduced proposals to redefine:

- the kilogram, in terms of the value of the Planck constant (h),
- the ampere, in terms of the value of the elementary charge (e),
- the kelvin, in terms of the value of the Boltzmann constant (k), and
- the mole, in terms of the value of the Avogadro constant  $(N_A)$ , which would be independent of the kilogram.

He explained that two alternative approaches to redefining the kilogram had been considered. These involved either fixing the Planck constant, or fixing the Avogadro constant. A definition in terms of the Avogadro constant would be easier to understand, but the alternative had a stronger link to fundamental physics; and if in addition to fixing the Planck constant, the ampere were defined by fixing the elementary charge then Josephson voltage measurements and quantum Hall resistance measurements would be traceable to the SI.

He presented a proposal for revised definitions of four of the base units as follows:

"The kilogram, unit of mass, is such that the value of the Planck constant is exactly  $6.626\,069\,3\times10^{-34}$  joule second."

"The ampere, unit of electric current, is such that the elementary charge (the charge on a proton) is exactly  $1.602\ 176\ 53\times 10^{-19}$  coulomb."

"The kelvin, unit of thermodynamic temperature, is such that the Boltzmann constant is exactly  $1.380\ 650\ 5\times 10^{-23}$  joule per kelvin."

"The mole, unit of amount of substance of a specified elementary entity, which may be an atom, molecule, ion, electron, an other particle, or a specified group of such particles, is such that the Avogadro constant ( $N_A$ ) is exactly 6.022 141 5 × 10<sup>23</sup> per mole."

Alternatively, the latter could be expressed in the form:

"The mole is the amount of substance of a system that contains exactly  $6.022\,141\,5\times10^{23}$  elementary entities, which may be atoms, molecules, ions, electrons, other particles, or a specified group of such particles."

The proposals would have several consequences, including the fact that the mass of the prototype of the kilogram would no longer be exact, but would have an uncertainty of 0.17 parts per million. There would be similar consequences for the molar mass of carbon-12, which would need to be determined by experiment, but its value would be consistent with 0.012 kg mol<sup>-1</sup> within an uncertainty of less than two parts in 10<sup>9</sup>. The triple point of water would also no longer be exact, but would need to be determined by experiment, but its value would be consistent with 273.16 K, within an uncertainty of about 0.25 mK. He expressed some optimism that work on the determination of the Avogadro constant by the x-ray crystal density (XRCD) method would reduce the present discrepancy in the realization of the kilogram between the method which relied on Planck's constant and that which required the Avogadro constant.

He concluded by saying that the advantages of the proposed changes included a simpler definition for the mole. He expected that the proposals would be presented to the CGPM in 2011.

The President introduced Prof. De Bièvre who is involved in the XRCD experiment for the determination of the Avogadro constant and was representing the International Coordination Committee (IAC) of the Avogadro project at the CCQM meeting. Prof. De Bièvre explained the basic measurement model for the method, which depends on the concept of measuring the ratio of the atomic volume of silicon to its molar volume. The experiment involved collaboration between many metrology laboratories around the world. The greatest challenge facing the collaboration was the measurement of the relative molecular mass of the silicon used in the artefact.

A silicon crystal would now be grown from silicon that had been enriched in the isotope silicon-28 from the natural level of 92 % to 99.995 %. It was expected that this would lead to a redetermination of the Avogadro constant with a relative uncertainty of a few parts in 10<sup>8</sup>. He concluded by discussing the importance of the measurement of numbers of entities to chemistry, as exemplified by the XRCD experiment.

Prof. Emons welcomed the proposals made by Prof. Mills, and asked for greater clarity about the types of entity that could be used within the definition of the mole. Prof. Mills said he would pay attention to any suggestions to improve the proposed definition of the mole. Dr Milton asked whether the availability of an improved determination of the Avogadro constant would change his proposals? Prof. Mills said it would not; it would actually accelerate the change.

Dr Besley suggested that the redefinition of the mole could be a timely opportunity to develop a new term for "amount of substance". Prof. Mills said he would welcome proposals, but knew of none that would meet with widespread acceptance.

The President thanked participants for their contributions and concluded that there was no objection from the CCQM to the proposals made by the CCU.

Dr Thomas asked members of the CCQM to assist her with the revision of the text relating to the mole in the SI Brochure. The President, together with Drs de Leer, De Bièvre, Hässelbarth and Milton agreed to do this.

#### 6 REPORTS OF THE CCQM WORKING GROUPS

#### 6.1 Organic analysis

Dr May presented his report of progress made by the CCQM Working Group on Organic Analysis (OAWG). The group had met at the IRMM and at the BIPM since the last meeting of the CCQM. He reviewed the terms of reference of the group which emphasised the "critical evaluation and benchmarking of "higher-order" measurement procedures for well-defined organic molecular

entities". He explained how the group had carried out key comparisons and pilot studies required by the health, food and commodity sectors.

During the year, the results of four key comparisons had been reported and discussed and agreement reached on the assignment of KCRVs: <a href="CCQM-K38">CCQM-K38</a> (PAHs in organic solution), <a href="CCQM-K39">CCQM-K39</a> (chlorinated pesticides in solution), <a href="CCQM-K11.1">CCQM-K11.1</a> (glucose in serum) and CCQM-K11.2 (creatinine in serum).

Four pilot studies had also been completed. The first two had been run in parallel to key comparisons: CCQM-P31.a.1, PAHs in organic solution, run in parallel to CCQM-K38; and CCQM-P31.c.1, chlorinated pesticides in solution, run in parallel to CCQM-K39. The third was CCQM-P68 (19-norandrosterone) for which the WADA had granted special permission for the use of some of their samples, and Mrs V. Ivanova had attended the meeting on behalf of the WADA. Statistics for 2004 from the WADA indicated that nandrolone was the second most abused of the anabolic steroids. Participants measured free and glucuronide forms of the metabolite. Four NMIs had participated in the pilot study, each of which had used a different method. The results were all comparable with the reference value within their estimated uncertainties. Dr May explained that there were no plans to proceed to a key comparison on this analyte, but the results would be published in a peer-reviewed journal. The President asked if there were any objections to such a publication being prepared. There were none.

The fourth pilot study completed during the year was CCQM-P69 (PAHs in soils and sediment). Samples had been sent to 11 laboratories, 9 of which had submitted results. The results had indicated some problems with the homogeneity of the samples, and this would be addressed prior to continuing with <a href="CCQM-K50">CCQM-K50</a> and the pilot study CCQM-P69.1, which would be run in parallel to the key comparison.

Dr May reported on two comparisons that were in progress at the time of the meeting:

- CCQM-K47 (VOCs in solution) was being carried out with a parallel pilot study CCQM-P61.1. Whilst results were good for most analytes, they were poor for m-xylene and the coordinating laboratory was investigating the accuracy of the reference value derived from gravimetric preparation, and how the uncertainty of a reference value might be calculated. Dr Wielgosz observed that similar problems had been encountered with earlier calibration solution comparisons, and asked whether the cause of this had been elucidated. Dr May replied that this was being looked into.
- Dr May reported on further work analysing the results of a suite of comparisons (<u>CCQM-K40</u>, CCQM-P31.b.1, -P57 and -P67) aimed at evaluating the uncertainty of measurements of PCBs in mussel tissue. Solutions of five target PCB congenors had been used to determine the influence of extraction on the uncertainty of the analysis. A new statistical approach had been developed to evaluate the consistency of results by different laboratories, from which it had been concluded that chromatographic separation and signal quantification were larger sources of uncertainty than extraction in these systems.

Dr May presented future plans for the development of a series of key comparisons to underpin NMI measurement capabilities for organic high purity materials. He reported that Dr Westwood at the BIPM had proposed a model that used information about the molecular weight and the polarity of

specific compounds to develop a strategy for future key comparisons on purity assessment. The working group had agreed that this was a valid approach, and that  $17\beta$ -estradiol would be an appropriate material for the first key comparison (CCQM-K55.a).

He reported that four pilot studies were being planned:

- CCQM-P77.a and .b (progesterone and cortisol in serum),
- CCQM-P20.e and .f (theophylline and digoxin),
- CCQM-P78 (nutrients in infant formula),
- CCQM-P88 (malachite green in fish).

In addition, the OAWG were discussing the possibility of organizing pilot studies involving:

- residues of veterinary drugs (nitrofurans and/or chloramphenicol),
- apple juice spiked with pyrethroids,
- acrylamide in potato chips, and
- organic components in alcoholic drink.

They were also considering work on method-defined analytes such as moisture in grain. Prof. Yadong drew the attention of the meeting to the interests of the OIML in the measurement of moisture.

In concluding, Dr May said that there was an ever-increasing requirement for studies in the area, which went beyond what could be achieved by the OAWG. He also believed that there should be guidelines developed to help in determining which laboratories might be able to coordinate key comparisons and studies, and that a strategic planning framework for key comparisons should be developed, resulting in a finite number of comparisons.

The President thanked Dr May for the work of his group.

#### 6.2 Inorganic analysis

Dr Sargent presented his report of progress made by the CCQM Working Group on Inorganic Analysis (IAWG), which had met at the BAM and the BIPM since the last meeting of the CCQM. He summarized the current status of key comparisons and pilot studies being undertaken by the working group: reports for six key comparisons and seven pilot studies were completed or being completed in 2005-2006; the reports of a further seven pilot studies were not yet complete, or awaiting comments on the first draft; a further two key comparisons and four pilot studies were in progress; and four new key comparisons and four pilot studies had been proposed.

He presented the results of <a href="CCQM-K44">CCQM-P70</a>), which involved measurements of seven trace metals in a matrix of sewage sludge. The concentration range covered three orders of magnitude and therefore the statement of "how far the light shines" would be broad. He highlighted the results for chromium, where a mixture-model median had been used to calculate the KCRV. Dr Wielgosz asked whether the 5 % relative expanded uncertainty in the KCRV calculated on the basis of this model could be justified? Dr Sargent said it was the result of the calculation, and he believed that the outcome was useful. He went on to report that the BAM had provided a corrected

value after having had access to the results of the study. Dr Wielgosz and the Director said that the acceptance of such a result was not consistent with the way the MRA was implemented across other working groups and by the other consultative committees.

Dr Sargent went on to present the results of <a href="CCQM-K45">CCQM-K45</a> (and CCQM-P72), which involved toxic metals in food at levels close to an EU regulatory limit for tin of 200 mg/kg. There were five participants in the key comparison. The KCRV was calculated as the mean. There were five additional laboratories in the associated pilot study (CCQM-P72), which had also studied other toxic metals (including Cd and Pb) in the same samples. He noted that the results for Sn were typical of the results in previous CCQM studies.

He presented the results of three pilot studies and made proposals for three new key comparisons to follow them:

- trace elements in soyabean powder CCQM-K56/CCQM-P64.1 (following CCQM-P64),
- chemical composition of clay CCQM-K57/CCQM-P65.1 (following CCQM-P65), and
- nitrogen and five trace elements in Si nitride powder CCQM-K58/CCQM-P74.1(following CCQM-P74).

He also proposed a new key comparison with an associated pilot study on nitrite and nitrate in calibration solutions and natural water (CCQM-K59/CCQM-P89). He noted that this would be a new area for the IAWG.

At the previous meeting of the CCQM, he had been requested to respond to a request from the CCT for support from the CCQM in measuring the purity of materials used to realize the fixed points for the international temperature scale. He reported that the request had led to the BAM and the NRC initiating research into glow discharge mass spectroscopy to carry out this type of measurement.

He echoed the comments made previously by Dr May relating to the large number of organizations seeking to participate in the work of the CCQM working groups. The IAWG had held a workshop to discuss these challenges. This resulted in a proposal to carry out a review of existing comparisons which would attempt to identify "generic" descriptions of sample matrices, which would support the development of statements of "how far the light shines" to justify CMCs. They were also planning to develop a formalized timetable of key comparisons that might support a wide range of CMCs.

A proposal to organize a CCQM workshop on neutron activation analysis as a primary method was also being prepared. He concluded that the IAWG were continuing to make good progress with their work.

The President thanked Dr Sargent for his report.

#### 6.3 Gas analysis

Dr de Leer presented his report of progress at the CCQM Working Group on Gas Analysis (GAWG) which had met at the CENAM and at the BIPM since the last meeting of the CCQM. He reviewed the results of four key comparisons:

- <u>CCQM-K23</u> (natural gas). He showed the results of two unknown mixtures each containing 10 compounds. The level of comparability between participants showed an improvement compared with the previous key comparison on natural gas mixtures of this composition (<u>CCQM-K1</u>).
- <u>CCQM-K26.a</u> (NO in nitrogen) which showed good comparability. The final report had been agreed.
- CCQM-K26.b (SO<sub>2</sub> dioxide in air). The KCRV for this key comparison had been defined by reference to a permeation device at the coordinating laboratory (NPL). An outlying result from one participant had subsequently been found to be due to an error in calibrating the flow. A difficulty caused by the import of the travelling standards into Japan had been overcome by a bilateral comparison with a travelling standard from NMIJ being sent to the coordinating laboratory. The coordinating laboratory was investigating possible sources of bias in the data.
- <u>CCQM-K41</u> (H<sub>2</sub>S in nitrogen). There was good comparability between six of the participants and one outlier. This comparison had made use of travelling standards which were calibrated with respect to a stable standard at the coordinating laboratory (NIST). The KCRV had been chosen as a mixture model, although he reported that the group had not been unanimous about this.

Dr de Leer described how he had undertaken a "gap analysis" in order to develop a future programme for comparisons to be carried out by the GAWG. The gaps he had identified included: methane and propane in air, oxygen in nitrogen, nitrous oxide, nitrogen dioxide, carbonyl sulphide, organo-sulphur compounds, formaldehyde, non-methane hydrocarbons, volatile organic compounds at environmental levels and the purity analysis of gases.

His analysis had also identified priorities for repeating key comparisons, these included:

- <u>CCQM-K1.d</u> (SO<sub>2</sub> at emission levels),
- <u>CCQM-K1.c</u> (NO at industrial levels),
- <u>CCQM-K3</u> (automotive emissions),
- <u>CCQM-K4</u> (ethanol in air).

He reported that there had been a strong increase in the amount and quality of work being organized within the RMOs, particularly the APMP and EUROMET.

Dr de Leer also talked about the collaboration of the group with the WMO Global Atmosphere Watch (GAW) project. Two representatives of the GAW had attended the GAWG meeting and had requested that the GAWG take responsibility for maintaining a "scale" for atmospheric VOCs on their behalf. This would require the provision and long-term maintenance of a multi-component standard of various VOC species as well as formaldehyde, DMS, methanol, ethanol and some other species. The GAWG had established a small committee to consider how this request could be met.

The President thanked Dr de Leer for his report.

#### 6.4 Electrochemical analysis

Dr Máriássy gave his report of the work of the CCQM Working Group on Electrochemical Analysis EAWG, which had met at the BAM and the BIPM since the last meeting of the CCQM.

He reported the results of some key comparisons. There had been a bilateral comparison carried out subsequently to <a href="CCQM-K34.1">CCQM-K34.1</a> (assay of KHP). This had shown satisfactory comparability between the BAM and the SMU. He presented the results of <a href="CCQM-K19">CCQM-K19</a> (pH of borate buffer) at three temperatures. The final report was being prepared. He reported that <a href="CCQM-K36">CCQM-K36</a> (electrolytic conductivity) had involved 14 participants each measuring two samples; there was good comparability amongst laboratories at both conductivity levels studied.

He presented the plans of the EAWG for future key comparisons and pilot studies. He observed that the range of work carried out on electrolytic conductivity spanned the range between pure water and seawater. Similarly, the work on pH had covered the range of the pH scale covered by all of the designated "primary standards".

Amongst the six planned key comparisons and studies, he highlighted the intention to carry out a preparative study in which participants would prepare a buffer with a pH close to 7 which would be analysed by the coordinating laboratory (CCQM-P93). He described how the group planned to carry out some work on the salinity of ocean water in order to improve the metrological traceability of these measurements.

The President thanked Dr Máriássy for his report.

#### 6.5 Surface analysis

Dr Unger presented his report of the work of the CCQM Working Group on Surface Analysis (SAWG). He started by re-iterating the view of the previous Chairman that the scope of the group covered not just surface analysis, but also micro- and nano-spatial analysis.

He discussed the results of <a href="CCQM-K32">CCQM-K32</a> and the parallel pilot study CCQM-P84, which evaluated the comparability of measurements of silicon dioxide on silicon and had been presented in full to the previous meeting of the CCQM. Subsequently, there had been some discussion as to why several results were outliers from the KCRV. The problem was being investigated, but had not been fully resolved.

He presented the results of two pilot studies: CCQM-P80 (C in Fe) and CCQM-P81 (N in Fe). These both studied the comparability of EPMA methods which are used widely. The results showed poor comparability, even amongst those laboratories using the same methods. It was believed that the problem might, in part, be due to the data reduction methods. This possibility was being investigated in more detail by the NPL and the BAM.

He described possible future projects for the group involving the quantitative surface analysis of Fe-Ni alloy using CRMs from the KRISS and the determination of F and N in diamond-like carbon films coordinated by the BAM.

Dr Unger said that the SAWG had been involved in the review of CMCs covering BET and surface porosimetry. After some necessary corrections, these had been approved. The SAWG had also invited Mrs Parkes from the BAWG to speak to them. This had led to the identification of some possible areas for research relevant to the life sciences including: the quantification of functional groups at surfaces, the study of surface energy in terms of surface chemistry, the quantification of SIMS images of biological samples and imaging cells at cryogenic temperatures by scanning-electron microscopy.

The President thanked Dr Unger for his report.

#### 6.6 Bioanalysis

Mrs Parkes presented her report of progress by the CCQM Working Group on Bioanalysis (BAWG), which had met at the CSIR-NML and the BIPM since the last meeting of the CCQM. They had reviewed the scope of the group's work and concluded that bioanalysis covered "large" macromolecules, where the target measurand was of biological origin in a biological context. They took the view that bio-measurement included, but was not limited to, the identification and quantification of the active macromolecule in complex matrices and mixtures immediately relevant to its biological function. She went on to describe the progress of four pilot studies that were being organised by the BAWG.

- CCQM-P44.1 was aimed at achieving the quantification of DNA by quantitative use of the polymerase chain reaction (PCR). The results in the second round had shown a significantly reduced variability between laboratories compared with the first round. However, the group still believed that further progress was required before proceeding to a key comparison. Some of the work involved in developing statistics for this study had led to the publication of a joint guidance paper. Mrs Parkes expected to be able to present further results at the next meeting, with the aim of progressing the study to a key comparison.
- CCQM-P58 was studying instrumental and inter-plate effects in assays performed by the ELISA
  method. Results from the first round of measurements indicated a variability of up to 10 % in
  florescence measurements across a plate. This pilot study would proceed to a second phase that
  would involve the measurement of protein samples.
- CCQM-P59 was studying the measurement of proteins by circular dichroism. This had involved
  the distribution of carefully controlled protein samples to participants. The coordinating
  laboratory (NPL) had carried out a comprehensive analysis of the results by principal
  component analysis. The results suggested that the level of comparability between results form
  the participants was poor, particularly for results measured in the far-ultraviolet part of the
  spectrum.
- CCQM-P60 was studying the extraction of DNA. This had made use of real-time-PCR. It had
  led to a conclusion that the percentage of genetically-modified material extracted depended on
  the method and that the major component in the measurement uncertainty was due to a lack of
  method repeatability.

Mrs Parkes noted that two other pilot studies were underway, CCQM-P54.1, which would determine the mass fraction of a defined 20 mer in a sample oligonucleotide, and identify any sequence failure products and their mass fractions, and CCQM-P55 on protein and peptide quantification.

She introduced some proposals for new work including a proposal from the KRISS to study the quantification of DNA methylation (CCQM-P94). There had also been nine proposals submitted by the NIBSC. Three of these would be developed further, and these involved: the assessment of reference materials and methods for cell mediated responses, DNA reference materials and glycoprotein and glycan analysis. She concluded by saying that the BAWG had had a successful year, which had built on its existing collaborations as well as making good progress with the objectives defined on its roadmaps.

Dr Wielgosz asked what range of measurement capabilities the proposed key comparison would underpin. Mrs Parkes said this was being considered.

The meeting of the BAWG was attended by representatives from the US Pharmacopoeia, who concluded that cooperation between the Pharmacopoeia and the CCQM and its working groups would be very useful.

The President thanked Mrs Parkes for her report.

#### 6.7 Key comparisons and CMC quality

The President informed the meeting that Dr McLaren would be retiring from the chairmanship of the CCQM Working Group on Key Comparisons (KCWG) following his appointment to the post of Director-General of the NRC-INMS. He had also joined the CIPM. The President expressed the thanks of the CCQM to Dr McLaren and said that Dr Mackay from the NMIA had indicated her willingness to take on the role of Chair. The meeting agreed with her appointment.

Dr McLaren started his report by reviewing the scope of activity of the KCWG. He explained that its membership included representatives from the CCQM working groups and the RMOs. He thanked Dr Hässelbarth for his involvement in the group and welcomed Dr Steiger also from the BAM who would replace him. He reported that 379 CMCs had been submitted by five RMOs as part of the Cycle VII review process. He observed that the inter-regional review process had worked well for Cycles V, VI, and VII.

He said that there continued to be a problem with the approval of CMCs that were supported by either a key comparison or a pilot study that had been completed shortly before the review. His view was that if the full report from a comparison had not been agreed, then it was only appropriate to use the results with the agreement of the relevant working group.

He said he was very grateful to the staff at the BIPM for supporting the process of CMC review and finished by expressing his confidence that this important activity was being handed over to a very capable new Chairperson.

The President thanked Dr McLaren and asked Dr May to take the thanks of the CCQM to Mrs Parris who had also been strongly involved in establishing the KCWG processes.

Dr Wielgosz asked the President and Director to give some guidance on how a KCRV should be established when a result from a participant had been revised. He observed that a unified approach was not used across the consultative committees, and that this point had already been raised during the presentation of the inorganic analysis working group. The Director confirmed that other committees did not use revised values as part of their calculation of the KCRV. He stated that if a laboratory made a mistake, that mistake should stand.

Dr Thomas said that there was no example of a revised result being used as part of the calculation of the KCRV. Dr May said that although it was preferable to exclude such a result from the calculation of the KCRV, it was not always possible, for example when there were only a small number of participants. The President said he would postpone a detailed discussion of the issue until the CCQM could hold a workshop devoted to issues of this kind. The workshop would allow discussion and harmonization of the different approaches to the uncertainty calculation of the KCRV and discuss how claimed CMC's related to the results of key comparisons.

The President concluded by asking the chairpersons of the CCQM working groups to convey his thanks to all participants in their working groups, for their work which was largely contributing to the success of the CCQM.

#### 7 UPDATE ON THE BIPM KEY COMPARISON DATABASE (KCDB)

Dr Thomas gave a report of progress with the KCDB. At the time of the meeting, it included information relating to 671 comparisons, of which 537 were key comparisons and 134 supplementary comparisons. Approximately 50% of these were organised by the consultative committees and the rest by the RMOs and the BIPM. At the time of the meeting, there were 18 058 CMCs recorded in Appendix C of the KCDB of which 3 521 were in chemistry. She reminded the CCQM that the final approval of the JCRB was needed before CMCs could be entered into the KCDB.

She reported that a new search facility would be incorporated into the KCDB in October 2006.

#### 8 BIPM PROGRAMME ON METROLOGY IN CHEMISTRY

The Director introduced the criteria used in defining the work programme of the BIPM. These were based on the recommendations of the Kaarls Report accepted by the CGPM in 2003. The Director

said that he had asked the section heads at the BIPM to develop ten-year plans in advance of the next meeting of the CGPM which would approve a work programme from 2009 to 2012.

He emphasised that the BIPM was able to make a long-term commitment to maintaining facilities with a core activity of coordinating comparisons and to offer good value for money by taking advantage of the opportunity to network with the NMIs. They should also continue to support a small number of "front line" research programmes, for example the BIPM watt balance. He emphasised that the BIPM occupies a "scientific niche" that does not duplicate the work of the NMIs. He concluded with a summary of the activities involved in planning the 23rd CGPM in 2007.

Dr Wielgosz presented an overview of the programme of metrology in chemistry running in the Chemistry section at the BIPM from 2005 to 2008. The objective of the programme had been discussed at previous meetings of the CCQM (CCQM/01-24, 2001). It incorporated laboratory work on gas metrology and organic analysis (CCQM/04-35, 2004) and also supported the work of the JCTLM.

He went on to describe a summary of the results (CCQM/06-14 and CCQM/06-41) of a chemical metrology and bio-metrology questionnaire (CCQM/06-03) that had been distributed by the BIPM in February 2006 to all NMIs and designated institutes active in the CCQM and its gas, organic and bioanaysis working groups. The questionnaire had also been sent out to directors of NMIs that were not yet active in the CCQM or its working groups. The questionnaire had been drafted to aid the BIPM in the formulation of its 2009-2012 workplan proposals, and would enable the BIPM to monitor the consistency of its proposed programme with current and likely future NMI priorities in the fields of gas, organic and bio-analysis. A total of 31 institutes had provided replies to the questionnaire.

In the field of gas metrology the NMIs had been asked to comment on: their expected priorities for new gas standards and capabilities over the next ten years; their requirements to participate in repeat or new comparisons and resources for their coordination; and the BIPM programmes themes, notably ozone, nitrogen oxides and reactive gases and preparative capability comparisons. The results indicated a very strong interest in gas analysis amongst the NMIs, particularly concerning: preparative comparisons, reactive binary mixtures and multi-component mixtures. The results of the questionnaire implied that there was a deficit between the number of comparisons that NMIs planned to coordinate, around thirty, and the number that they indicated would be required, which was approximately 40 and comparable in number to the 37 carried out during the previous ten-year period. He also talked about the requirements for comparisons of preparative capability, on-going requirements for reactive gas comparisons, and future research themes related to dynamic methods, optical methods and absorption cross section measurements. He concluded that elements of the current programme in gas analysis should be maintained and in some cases extended to meet some of the requirements identified through the questionnaire.

He went on to discuss the responses to the questionnaire relating to organic analysis. Information had been sought on NMI future priorities in the areas of clinical chemistry, food analysis, environmental analysis and forensic and doping control, and notably requirements for matrix, calibration solution, and pure material comparisons and studies. The majority of responses indicated that NMIs would be increasing their work programmes in these areas. Seventy five per cent of respondents projected an increase in their work on food analysis and particularly on residues and

contaminants in food, which would also require programmes related to pure substances and calibrants. He also reported on responses related to the area of environmental analysis, which is generally well established in many NMIs, and for which new persistent organic pollutants would continue to be of interest. In the area of clinical chemistry, nine NMIs had reported established activities and others had expressed interest in starting them. Steroid analysis was an area of common future interest, and additionally (T3/T4) thyroxine and digitoxin were suggested for pure substance studies. He presented the criteria that the BIPM had applied in prioritizing analytes for future comparison needs, and these included: a demonstrated NMI interest; interest driven by strong requirements such as legislative ones; an expansion of the scope of CCQM purity comparisons; a requirement for the substance to be representative of a larger class of materials; relevance of the analyte to several application areas; and the technical feasibility of the study. He concluded by presenting a number of analytes relevant to the clinical, food residue and forensics sectors as possible candidates for future purity/calibration solution studies.

He finished by summarizing the 14 responses received covering the area of bio-analysis. Many of these NMIs were planning to increase their work in the area. He presented the results in terms of the areas of greatest interest for future CRM production and comparisons related to genetically modified organism (GMO) analysis, pathogen quantification, genetic testing, gene expression and protein analysis. He concluded with a recommendation that the BIPM might coordinate a comparison in the area of GMO quantification and support international activity in the area.

Proposals for the 2009-2012 BIPM chemistry programme had been circulated before the meeting (CCQM/06-08). The proposals had been drawn up taking into account the high level drivers and triggers for the BIPM's work, based on an analysis of scientific need, the views of NMIs expressed in the questionnaire responses, and feedback from CCQM working groups and visits to NMIs. They included the continuation of existing activities in six areas as well as eight proposals for new projects. He informed the CCQM that since preparing the paper, the proposal for laboratory work on bio-analysis had been withdrawn, following the recommendation of a CCQM Advisory Group.

The President thanked Dr Wielgosz for his presentation and introduced Dr Sargent who was acting as Rapporteur for an Advisory Group drawn from members of the CCQM, which had reviewed the proposals in a meeting held at the BIPM on 2 April 2006. The Advisory Group had broad representation from across the NMIs and the subject areas within the scope of the CCQM. They had prepared a report (CCQM/06-13 and Appendix Q 2).

Dr Sargent said that the group had taken account of the need for the BIPM's work programme to address the requirements of the global community including the NMIs. The Advisory Group noted that the 2005-2008 chemistry programme comprised scientific activities on gas analysis and purity of organic compounds, together with support for the JCTLM and its database. BIPM had proposed to continue and strengthen these activities in the 2009-2012 programme and, in addition, to introduce a bioanalysis programme, consisting of both laboratory and coordination activities.

The overall gas analysis programme was broadly supported by the group and felt to be a worthwhile extension of the current activities, which are well-regarded and closely linked to the programme of the CCQM Gas Analysis Working Group (GAWG). All projects should demonstrate clearly that they underpin measurement needs for key global issues and be dedicated to climate change and air quality. The Advisory Group noted that it was premature to define the specific tasks in 2009-2012 in

detail, as they may be overtaken by events and that the proposals should, as such, be regarded as indicative. The BIPM should carry out specific tasks with the consultation and cooperation of the GAWG at the appropriate time.

He reported that the group had made a larger number of specific comments about the proposals for the organic analysis programme. It was suggested that BIPM programme should be focussed in one single project entitled 'Primary references for organic analysis'. The overall programme was supported, noting that it placed the main emphasis on comparisons. The Advisory Group accepted that BIPM cannot be too specific at this stage of programme development but requested that more novel science related to method development should be included. It also recommended a focus on primary references to support food, healthcare and forensic applications as indicated by the response to the questionnaire. The Group would welcome development and coordination of a best practice guide on purity determination of organic compounds. This should build on the expertise developed under the BIPM chemistry programme.

The group felt that proposals for a bio-analysis laboratory project were premature and needed wider debate. Activities at BIPM should focus on fundamental metrology and it was not clear that this was proposed or feasible at this stage. The Advisory Group noted that this is a rapidly developing field where most key players are also able to devote substantial resources, far beyond those available to BIPM. It was accepted that staff working on liaison activities benefit from an involvement in relevant science. However, the Advisory Group recommended that BIPM should not develop its own research programme at this time. The BIPM should consider novel alternatives to setting up its own bio-analysis research programme.

The support for international liaisons in the field of bio-analysis was accepted as an important task but BIPM were requested to clarify the proposals. In particular, the Advisory Group saw a need to distinguish between the global role of the BIPM in this respect and the individual representation of member states. The proposed project on bio-analysis liaison with national and international organizations was welcomed but BIPM should provide more information on the impact expected from this activity. In addition, consideration should be given to broadening the liaison beyond GMOs.

In conclusion, the group requested that the BIPM should provide a revised draft of its proposals for the CIPM emphasizing the key top level themes. Notably, the gas work should fall within the area of air quality and climate change, and the organic chemistry programme should address primary references for organic analysis in support of food, healthcare and forensic applications. The BIPM should develop liaison activities in the field of bio-analysis but not a bioanalysis laboratory programme at this time. The Advisory Group welcomed the BIPM's proposals for its 2009-2012 chemistry programme and supported them subject to the implementation of its recommended amendments.

Prof. Emons said that his organization was already very strongly involved in coordinating activities across Europe in several of the areas mentioned, and he was not convinced of the added value of BIPM's proposed coordination activity. The President replied that the BIPM is operating at the global inter-governmental level and not solely within Europe.

Since there were no further questions, the President summarized that the CCQM approved the report of the advisory committee.

#### 9 JOINT COMMITTEE FOR TRACEABILITY IN LABORATORY MEDICINE (JCTLM)

The President introduced the Chairman of the JCTLM, Prof. Jean-Claude Forest.

Prof. Forest said he was pleased to make his first report to the CCQM. He reviewed the history of the JCTLM since its formation in 2002. He described its structure, which included an executive committee and two working groups. The executive committee had been active in promoting the activities of the JCTLM at events around the world. The funding model for the secretariat was being reviewed because of an increase in its workload.

The President thanked Prof. Forest for his report.

#### 9.1 JCTLM Working Group 1

Dr May presented his report of work carried out by Working Group 1 of the JCTLM. He reviewed the terms of reference for the group, and said that the review process it operated was based upon the recommendations of ISO standards 15193 and 15194. The process had been documented and was available from the website. The development of their quality manual had involved summarizing existing procedures as well as improving on them. Reviews of materials and methods were being carried out by twelve review teams each working in a different specialised area.

The lists of higher order reference materials and reference measurement procedures for laboratory medicine and *in vitro* diagnostics developed by Working Group 1 included approximately 100 reference measurements procedures and 190 CRMs. The third call for nominations made at the start of 2006 had resulted in twenty–five new nominations for CRMs and forty new nominations of reference measurement procedures.

He described how the WG had initiated a programme of comparability studies to underpin the review process. He presented an example of a study intended to establish whether there was any bias between the Abell-Kendall and IDMS methods for the measurement of cholesterol in human serum. The studies had concluded that there was not.

Dr Wielgosz said that nominations of scientists who were willing to be active within the JCTLM working groups were welcome. Prof. Yadong asked for clarification as to what was meant by a reference method in this context. Dr Wielgosz said that this information was included within ISO 17511.

The President thanked Dr May for his report.

#### 9.2 JCTLM Working Group 2

Prof. Siekmann gave a report of the work of Working Group 2 of the JCTLM which was concerned with the technical competence of reference measurement laboratories. They had prepared a manual that described their processes and procedures. An open call for laboratories to nominate their reference measurement services had been published through the JCTLM pages on the BIPM website. A total of 210 nominations of reference measurement services from twenty six laboratories had been received in 2006. He reviewed the hierarchy of measurements used in laboratory medicine, which involved the NMIs, reference (or calibration) laboratories and routine (or testing) laboratories. He described the processes that Working Group 2 would use to incorporate reference measurement services of laboratories into their lists and, if necessary, to remove them.

He described the approach that Working Group 2 had developed to structuring a series of interlaboratory comparisons. This involved defining a single "key measurand" representing each class of analyte. In each cycle the "key measurand" would be different. He showed some of the results from the most recently completed comparisons organised by the DGKL on behalf of the IFCC. These included results for cortisol from reference laboratories and he compared these to results reported from routine laboratories from a related comparison. He proposed that the IFCC EQAS scheme for reference measurement laboratories be used more widely by the CCQM as part of the key comparison system. Dr May said it would be difficult for NMIs to participate frequently in these ring trials until they had incorporated them into their long-term plans. Mr Squirrell commented that it was important to maintain effective dissemination of traceability to routine laboratories working in the area of laboratory medicine.

The President thanked Prof. Siekmann for his report.

### 10 COOPERATION WITH INTERNATIONAL AND INTERGOVERNMENTAL ORGANIZATIONS

#### 10.1 World Anti-doping Agency (WADA)

The President introduced Mrs Ivanova, a Science Project Manager of the WADA. She gave a brief history of anti-doping in sport. In 1967 the International Olympic Committee (IOC) established its medical commission and developed its first list of prohibited substances ahead of the Olympics in 1968. Subsequently steroids were banned in 1976 and blood doping was banned in 1986. Ben Johnson's case at 1988 Olympics and the 1998 Tour de France doping scandal caused major international outrage of a growing problem in sport. Following rising concern about danger of doping in sport, the IOC and government representatives gathered for the First World Conference on Doping in Sport in Lausanne in February 1999. The WADA was established in November 1999. The World Anti-Doping Code is the fundamental document of the World Anti-Doping Programme. All

WADA international standards (including standards for testing and for laboratories) are based on the Code provisions and have the same regulatory power as the Code. The Copenhagen Declaration had been signed by 184 countries to indicate their support for the Code.

Mrs Ivanova described the process that WADA uses for the accreditation of laboratories. At the time of the meeting, there were 33 WADA accredited laboratories in 30 countries, each of which maintained accreditation to ISO 17025, participated in the WADA inter-laboratory proficiency-testing scheme, and complied with the WADA international standard for laboratories and related technical documents. The WADA PT scheme includes threshold substances for which quantification and an estimation of measurement uncertainty need to be provided by laboratories, in addition to non-threshold substances on the WADA prohibited list which need to be correctly identified by laboratories. The drafting of a document on measurement uncertainty was a current priority for the WADA Laboratory Committee.

Dr de Leer asked whether WADA set performance criteria for its laboratories? Mrs Ivanova confirmed that they did.

The President thanked Mrs Ivanova for her interesting presentation and concluded that the results of the 19-norandrosterone had been very good and would be published, and hoped for continued collaboration between the WADA and CCQM.

#### 10.2 International Atomic Energy Agency (IAEA)

Dr Fajgelj gave a short report on behalf of the IAEA. He pointed out the Agency's independent international role, which is very much based on the high quality analytical results provided by its own laboratories. The metrological link of the IAEA with the BIPM, CCQM, CCIR and other bodies is therefore of outmost importance.

The IAEA was submitting a phospho-gypsum material to be used for pilot studies by both the CCIR and the CCQM. He described the development of a new V-SMOW isotopic reference material. There would be sufficient quantities of the new material to last for at least 30 years.

Dr Fajgelj was also acting as the IUPAC representative to the CCQM, and noted that the presentation on IUPAC activities was available as working document CCQM/06-36.

The President thanked Dr Fajgelj for his reports.

#### 10.3 International Union of Pure and Applied Chemistry (IUPAC)

Dr Fajgelj was also representing IUPAC and he thanked CCQM and BIPM for sponsoring the International workshop on combining and reporting analytical results — The role of (metrological) traceability and (measurement) uncertainty for comparing analytical results, held in Rome between 6 and 8 March 2006. Proceedings of the workshop will be available by the end of 2006.

The presentation on IUPAC activities was available as working document CCQM/06-36.

The President thanked Dr Fajgelj for his report.

## 10.4 International Organization for Standardization, Committee on Reference Materials (ISO REMCO)

Dr van der Veen, the chairman of the ISO REMCO, described its work, which includes responsibility for the development and publication of ISO Guides 30 to 35. They were preparing Technical Reports on recommendations for transport and a classification scheme for reference materials. He said that several of the guides had been subject to a systematic review.

He explained that new definitions had been developed for the terms "reference material" (RM) and "certified reference material" (CRM). These encompassed qualitative as well as quantitative measurements and recognised that the term CRM referred to a subset of the term RM. He explained that ISO REMCO took the view that calibrants, calibrators and CRMs were all types of RM. He said that there was some dialogue with representatives of the JCGM over the incorporations of these definitions into the new VIM, but that no agreement had yet been reached.

The President said it was essential for the ISO REMCO and the JCGM to come to agreement over definitions for RM and CRM that would be suitable for use within ISO REMCO and the JCGM. Dr Wielgosz said it was becoming a matter of urgency to prevent a proliferation of differing definitions. Mrs Ivanova said that it was also a priority for the WADA. The President summarised the views of the CCQM by saying that the CCQM strongly encourages the ISO REMCO and the JCGM to resolve the problem.

Dr de Leer asked whether the ISO REMCO would work with the ISO CASCO on the revision of Guide 34. Dr van der Veen said that they had good cooperation with the ISO CASCO, and that collaboration would continue as needed through the ISO CASCO representative at ISO REMCO.

The President thanked Dr van der Veen for his report.

#### 10.5 International Laboratory Accreditation Cooperation (ILAC)

Mr Squirrell gave a report on behalf of the ILAC. He said that they were strengthening their technical links with the BIPM. He drew the attention of the CCQM to the joint CIPM-ILAC memorandum and also the BIPM-ILAC-OIML statement on the use of the three Mutual Recognition Arrangement (MRAs). Both of these were available on the BIPM website.

An issue of concern to the ILAC was the effective use of the CMCs defined under the CIPM MRA, and how they related to the BMCs used by accreditation bodies.

Dr de Leer asked whether there were any plans to apply the concept of a "flexible scope" to calibration as well as testing laboratories. Mr Squirrel said this was being discussed within ILAC committees. Prof. Emons said it was a source of frustration that the approaches being used by different national accreditation bodies were not harmonised.

The President thanked Mr Squirrell for his report.

#### 10.6 Forensics

The President reported that he had been invited to give a presentation to the global meeting of the International Association of Forensic Sciences (IAFS), which was held every three years. The forensics community expressed a clear interest in the work of the CCQM, which could support the further improvement of the quality and reliability of forensic measurements and the establishment of comparability of forensic analyses amongst the world's forensic laboratories, which is important in fighting crime and establishing security. Soon after the CCQM the President had arranged a meeting with the chairman of the European Network of Forensic Science Institutes (ENFSI). He hoped to invite a representative from the IAFS and/or the ENFSI to the CCQM in the future.

#### 10.7 Codex Alimentarius and Inter-agency meetings

Dr Wielgosz stated that no reports from the inter-agency meeting or the Codex Committee on Methods of Analysis and Sampling were available, as these meetings would be held after the CCQM this year.

#### 11 REPORTS FROM REGIONAL METROLOGY ORGANIZATIONS

#### 11.1 Asia/Pacific Metrology Programme (APMP)

Dr Sin gave a report on the work of the TCQM of the APMP on behalf of Prof. Yadong its Chairman. They had been considering the requirements of ISO Guide 34 and were developing a strategic work plan for the work of the TCQM. A number of pilot studies were underway together with a number of bilateral and trilateral comparisons needed to underpin CMCs submitted as part of the Cycle VI review.

## 11.2 Southern African Development Community Cooperation in Measurement Traceability (SADCMET)

Dr Louw reviewed the work of SADCMET on metrology in chemistry. He reported that a total of 92 CMCs were being withheld pending accreditation to ISO 17025. A regional proficiency testing scheme had been completed for laboratories carrying out the analysis of water, including microbiology. Pilot studies were being planned for ethanol in water, veterinary residues and certain analytes in food. He reported that there was a plan being developed to bring SADCMET together with other African metrology organisations to form a single pan-African RMO called AFRIMETS.

#### 11.3 European Collaboration in Measurement Standards (EUROMET)

Dr Charlet presented a report on behalf of the EUROMET MetChem technical committee. He reported that it has four sub-committees and that the most recent meeting had involved representatives from 24 countries. The committee has 11 active projects and was initiating a number of new proposals including a cooperation on ocean salinity, measurements to support the European Union Water Framework Directive and a workshop on nanoparticle measurements.

He described the iMERA project, which involved those countries within EUROMET that had significant research activities. It was concerned with the development of a proposal for funding under Article 169 of the European Treaty. It would ultimately require the registration of EUROMET as a legal entity.

#### 11.4 Cooperation in Metrology among the Central European Countries (COOMET)

Dr Kustikov gave a report on behalf of Prof. Konopelko, the chairman of the Physical Chemistry Committee of COOMET. He described the scope of the committee. The strongest area amongst the programme of the participating NMIs is gas analysis. This is reflected in the largest number of CMCs being in this area. In addition to 189 CMCs from the VNIIM, there are seven from the VNIIFTRI and eight from the UNIIM. Three pilot studies and one key comparison were underway within the group.

#### 11.5 Inter-American Metrology System (SIM)

Dr May gave a report on the work of SIM on chemical metrology. There were 15 NMIs participating regularly in the SIM Chemical Metrology Working Group meetings. He described the programmes of a series of workshops being held in developing countries, typically involving 50 or 60 attendees at each. These workshops involved training in chemical metrology as well as a technical discussion of the results of comparisons.

#### 12 CCQM WORKSHOPS

The President said he was proposing to hold a workshop during the same week as the next meeting of the CCQM. Possible topics included strategies for organizing key comparisons and processes for establishing KCRVs. Other proposals for CCQM workshops included neutron activation analysis, primary methods, measurement uncertainty, and a workshop with stakeholders involved in the 'omics' (genomics, proteomics etc.).

#### 13 CCQM RECOMMENDATIONS

Recommendation Q 1 (2006) on metrology in chemistry and biotechnology was circulated to CCQM delegates. The President requested comments on the text. Following discussion on the appropriateness of the term 'biotechnology', the President stated that the text was adopted, but should take into account any further comments received from delegates in the month following the meeting.

#### 14 ANY OTHER BUSINESS AND DATE OF THE NEXT MEETING

Dr May said he wanted to give recognition to the role of the President of the CCQM in managing it so effectively. Dr May's acknowledgement was unanimously supported by the members of the CCQM.

The President announced that the next meeting would be held at the BIPM during 19 and 20 April 2007.

M.J.T. Milton, rapporteur 14 June 2006

WG	Reference No.	Description	Coordinating laboratory	Start date	Status	Comments
CCQM Bioanal	ysis Working Grou	p key comparisons and pilot studies				
BAWG	CCQM-P44	DNA quantification	NIST/LGC	2002	Complete: repeat study	
BAWG	CCQM-P44.1	Q-PCR (repeat)	NIST/LGC	2004	Report in progress	
BAWG	CCQM-P53	DNA profiling	NARL	2003	Complete	
BAWG	CCQM-P54	DNA primary quantification	LGC	2004	Complete: repeat study	
BAWG	CCQM-P54.1	DNA quantification	LGC	2006	Planned	
BAWG	CCQM-P55	Peptide/protein quantification	LGC	2004	Planned	
BAWG	CCQM-P58	Fluorescence in ELISA	NPL/NIST	2004/2005	In progress	
BAWG	CCQM-P59	Protein structural measurements by CD	NPL/NIST		Report in progress	
BAWG	CCQM-P60	DNA extraction – reference method	IRMM	2004/2005	Report in progress	
BAWG	CCQM-P94	Quantification of DNA methylation	KRISS		Planned	
_	•	Working Group key comparisons and pilot		1999	Approved for equivalence	1
EAWG	CCQM-K9	pH 7.0 (phosphate)	PTB	1999	Approved for equivalence	
EAWG	•			1999 2002	Approved for equivalence Approved for equivalence	
EAWG EAWG	CCQM-K9	pH 7.0 (phosphate)	PTB		* * *	
EAWG EAWG	CCQM-K9 CCQM-K9 subsequent	pH 7.0 (phosphate) pH 7.0 (phosphate) PTB-SMU bilateral	PTB PTB	2002	Approved for equivalence	Run in parallel to CCQM-P52
EAWG EAWG EAWG	CCQM-K9 CCQM-K9 subsequent CCQM-K17	pH 7.0 (phosphate) pH 7.0 (phosphate) PTB-SMU bilateral pH 4.1 (phthalate)	PTB PTB PTB	2002	Approved for equivalence  Approved for equivalence	
_	CCQM-K9 CCQM-K9 subsequent CCQM-K17 CCQM-K18	pH 7.0 (phosphate) pH 7.0 (phosphate) PTB-SMU bilateral pH 4.1 (phthalate) pH 10.1 (carbonate)	PTB PTB SMU	2002 2001 2003	Approved for equivalence  Approved for equivalence  Protocol complete	
EAWG EAWG EAWG EAWG	CCQM-K9 CCQM-K9 subsequent CCQM-K17 CCQM-K18	pH 7.0 (phosphate) pH 7.0 (phosphate) PTB-SMU bilateral pH 4.1 (phthalate) pH 10.1 (carbonate)  pH 9.2 (borate)	PTB PTB SMU	2002 2001 2003	Approved for equivalence  Approved for equivalence  Protocol complete  Report in progress draft A	
EAWG EAWG EAWG EAWG EAWG EAWG EAWG	CCQM-K9 CCQM-K9 subsequent CCQM-K17 CCQM-K18 CCQM-K19 CCQM-K20	pH 7.0 (phosphate) pH 7.0 (phosphate) PTB-SMU bilateral pH 4.1 (phthalate) pH 10.1 (carbonate)  pH 9.2 (borate) pH 1.7 (tetroxalate)  Assay of potassium hydrogen phthalate	PTB PTB PTB SMU PTB	2002 2001 2003 2004	Approved for equivalence  Approved for equivalence  Protocol complete  Report in progress draft A  Planned	
EAWG EAWG EAWG EAWG EAWG EAWG EAWG EAWG/IAWG	CCQM-K9 subsequent CCQM-K17 CCQM-K18 CCQM-K19 CCQM-K20 CCQM-K34	pH 7.0 (phosphate) pH 7.0 (phosphate) PTB-SMU bilateral pH 4.1 (phthalate) pH 10.1 (carbonate) pH 9.2 (borate) pH 1.7 (tetroxalate) Assay of potassium hydrogen phthalate (KHP) Assay of potassium hydrogen phthalate	PTB PTB SMU PTB SMU	2002 2001 2003 2004	Approved for equivalence  Approved for equivalence Protocol complete  Report in progress draft A Planned Approved for equivalence	CCQM-P52  BAM/SMU bilateral
EAWG EAWG EAWG EAWG EAWG EAWG EAWG/IAWG EAWG/IAWG	CCQM-K9 Subsequent CCQM-K17 CCQM-K18 CCQM-K19 CCQM-K20 CCQM-K34 CCQM-K34	pH 7.0 (phosphate) pH 7.0 (phosphate) PTB-SMU bilateral  pH 4.1 (phthalate) pH 10.1 (carbonate)  pH 9.2 (borate) pH 1.7 (tetroxalate)  Assay of potassium hydrogen phthalate (KHP)  Assay of potassium hydrogen phthalate (KHP)	PTB PTB SMU PTB SMU	2002 2001 2003 2004	Approved for equivalence  Approved for equivalence Protocol complete  Report in progress draft A Planned Approved for equivalence  Approved for equivalence	CCQM-P52  BAM/SMU bilateral
EAWG EAWG EAWG EAWG EAWG	CCQM-K9 Subsequent CCQM-K17 CCQM-K18 CCQM-K19 CCQM-K20 CCQM-K34 CCQM-K34.1	pH 7.0 (phosphate) pH 7.0 (phosphate) PTB-SMU bilateral  pH 4.1 (phthalate) pH 10.1 (carbonate)  pH 9.2 (borate) pH 1.7 (tetroxalate)  Assay of potassium hydrogen phthalate (KHP)  Assay of potassium hydrogen phthalate (KHP)	PTB PTB SMU PTB SMU	2002 2001 2003 2004	Approved for equivalence  Approved for equivalence Protocol complete  Report in progress draft A Planned Approved for equivalence  Approved for equivalence  Report in progress draft A	CCQM-P52  BAM/SMU bilateral

EAWG/IAWG	CCQM-P19.1	Purity of HCl	NIST	2002	Complete	
EAWG	CCQM-P22	Electrolytic conductivity	DFM	2001	Completed	
EAWG/IAWG	CCQM-P36	Assay of potassium hydrogen phthalate (KHP)	SMU/NIST	2002	Complete; progression to key comparison proposed	
EAWG	CCQM-P37	Fundamental studies of pH standards	SMU	2002	Completed	
EAWG	CCQM-P47	Electrolytic conductivity (low level)	NMi	2003	Planned	
EAWG	CCQM-P52	pH 10.1 (carbonate)	SMU	2003	Protocol complete	Run in parallel to CCQM-K18
EAWG	CCQM-P82	pH 9.2 (borate)	PTB	2005	Report in progress	Run in parallel to CCQM-K19
EAWG	CCQM-P83	Electrolytic conductivity (0.5 mS/m)	DFM	2005	Planned	
EAWG	CCQM-P93	pH 7 preparation study		2008	Planned	
CCQM Gas And	llysis Working Grou	p key comparisons and pilot studies				
GAWG	BIPM.QM-K1	Ozone at ambient level	BIPM	2006	Planned	
GAWG	CCQM-K1.a	CO in N <sub>2</sub>	NMi	1998	Approved for equivalence	
GAWG	CCQM-K1.b	CO <sub>2</sub> in N <sub>2</sub>	NMi	1998	Approved for equivalence	
GAWG	CCQM-K1.c	NO in N <sub>2</sub>	NMi	1998	Approved for equivalence	
GAWG	CCQM-K1.d	SO <sub>2</sub> in N <sub>2</sub>	NMi	1998	Approved for equivalence	
GAWG	CCQM-K1.e,f,g	Natural gases (Types 1, 2, 3)	NMi	1998	Approved for equivalence	
GAWG	CCQM-K3	CO, CO <sub>2</sub> , propane in N <sub>2</sub>	NMi	1998	Approved for equivalence	
GAWG	CCQM-K4	Ethanol in air	NPL	1999	Approved for equivalence	
GAWG	CCQM-K7	Benzene/toluene/xylene (BTX) in N <sub>2</sub> /air	NIST	1999	Approved for equivalence	
GAWG	CCQM-K10	BTX in N <sub>2</sub> (low concentration $10 \times 10^{-9}$ to $30 \times 10^{-9}$ )	NIST/NPL	2001	Approved for equivalence	
GAWG	CCQM-K15	SF <sub>6</sub> , CFCs – emission levels	KRISS	2003	Approved for equivalence	Run in parallel to CCQM-P51
GAWG	CCQM-K16.a	Natural gas (Types IV)	BAM/NMi	2001	Approved for equivalence	Run in parallel to CCQM-P49.a
GAWG	CCQM-K16.b	Natural gas (TypesV)	BAM/NMi	2001	Approved for equivalence	Run in parallel to CCQM-P49.b
GAWG	CCQM-K22	VOCs in air	NMIJ	2003	Report in progress draft B	Run in parallel to CCQM-P71
GAWG	CCQM-K23	Natural gas (repeat)/LPG	NMi	2004	Report in progress draft B	

GAWG	CCQM-K26.a	Reactive gases-ambient levels – NO in N <sub>2</sub>	NPL	2003	Approved for equivalence	Run in parallel to CCQM-P50.a
GAWG	CCQM-K26.b	Reactive gases-ambient levels – SO <sub>2</sub> in air	NPL	2003	Report in progress draft B	Run in parallel to CCQM-P50.b
GAWG	CCQM-K41	H <sub>2</sub> S in nitrogen	NIST		Report in progress draft B	
GAWG	CCQM-K46	Ammonia in nitrogen	NMi	2005	Planned	
GAWG	CCQM-K51	CO in nitrogen (5 µmol/mol)	NMI/CSIR-NML	2006	Planned	
GAWG	CCQM-K52	$CO_2$ in air $(360 \times 10^{-6} - 400 \times 10^{-6})$	NMi/CSIR-NML	2006	Planned	
GAWG	CCQM-K53	O <sub>2</sub> in nitrogen – preparative capabilities	KRISS	2006	Planned	
GAWG	CCQM-K54	n-hexane in methane – preparative capabilities	NMi VSL	2006	Planned	
GAWG	CCQM-P23	CO in nitrogen $(50\ 000 \times 10^{-6},\ 1000 \times 10^{-6},\ 10 \times 10^{-6},\ 10 \times 10^{-6})$ – Gravimetry	NMi	2000	Complete	
GAWG	CCQM-P24	Dynamic mixing methods	LNE	2002	Complete	
GAWG	CCQM-P28	Ozone – ambient levels	BIPM	2003	Complete	
GAWG	CCQM-P41	Greenhouse gases CO <sub>2</sub> , CH <sub>4</sub> – ambient levels	NMi	2002	Complete	
GAWG	CCQM-P45	Purity analysis of parent gases incl. H <sub>2</sub> O	LNE	2002	Planned	EUROMET workshop
GAWG	CCQM-P49.a	Natural gas (Types IV)	BAM/NMi	2001	Completed	Run in parallel to CCQM-K16.a
GAWG	CCQM-P49.b	Natural gas (Types V)	BAM/NMi	2001	Completed	Run in parallel to CCQM-K16.b
GAWG	CCQM-P50.a	Reactive gases – ambient levels – NO in N <sub>2</sub>	NPL	2003	Complete	Run in parallel to CCQM-K26.a
GAWG	CCQM-P50.b	Reactive gases – ambient levels – SO <sub>2</sub> in air	NPL	2003	Report in progress	Run in parallel to CCQM-K26.b
GAWG	CCQM-P51	SF <sub>6</sub> , CFCs – emission levels	KRISS	2003	Complete	Run in parallel to CCQM-K15
GAWG	CCQM-P71	VOCs in air	NMIJ	2003	Report in progress	Run in parallel to CCQM-K22
GAWG	CCQM-P73	Nitrogen monoxide in nitrogen	BIPM	2006	Planned	
GAWG	CCQM-P87	Multicomponent preparative capability study	NPL		Planned	

IAWG	CCQM-K2	Cd and Pb in natural water	IRMM	1998	Completed	
IAWG	CCQM-K8	Elemental solution standards (Al, Cu, Fe, Mg)	EMPA/LNE	1999	Approved for equivalence	
IAWG	CCQM-K13	Pb/Cd in sediments	IRMM	2000	Approved for equivalence	
IAWG	CCQM-K13.1	Pb/Cd in sediments	NIST	2000	Approved for equivalence	
IAWG	CCQM-K14	Ca in serum	IRMM	2003	Approved for equivalence	
IAWG	CCQM-K24	Cd in rice	IRMM	2001	Approved for equivalence	Run in parallel to CCQM-P29
IAWG	CCQM-K28	TriButylTin in sediment	LGC/NRC	2003	Approved for equivalence	
IAWG	CCQM-K29	Anions in calibration solutions	EMPA	2003	Approved for equivalence	
IAWG	CCQM-K29.1	Anions in calibration solutions	SMU		Approved for equivalence	SMU/CENAM bilateral
IAWG	CCQM-K30	Pb in wine	LGC (with CMQ-F assistance)	2003	Protocol complete	Run in parallel to CCQM-P12.1
IAWG	CCQM-K31	As in fish or shellfish	NIST	2002	Approved for equivalence	
IAWG	CCQM-K33	Minor elements in steel	NMIJ/NIST/BAM	2003	Approved for equivalence	Run in parallel to CCQM-P56
IAWG/EAWG	CCQM-K34	Assay of potassium hydrogen phthalate (KHP)	SMU	2003	Approved for equivalence	
IAWG/EAWG	CCQM-K34.1	Assay of potassium hydrogen phthalate (KHP)	SMU	2003	Report in progress draft B	BAM/SMU bilateral
IAWG	CCQM-K35	Sulfur in fuels (lower levels)	NIST	2003	Report in progress draft B	Run in parrallel to CCQM-P26.1
IAWG	CCQM-K42	Constituents of an aluminium alloy	BAM	Oct. 2004	Report in progress draft B	Run in parallel to CCQM-P34.1
IAWG	CCQM-K43	Methyl-mercury in salmon fish	IRMM	Nov. 2004	Approved for equivalence	Run in parallel to CCQM-P39.1
IAWG	CCQM-K44	Trace metals in sewage sludge	IRMM	Dec. 2004	Report in progress draft B	Run in parallel to CCQM-P70, EUROMET 784 and IMEP
IAWG	CCQM-K45	Toxic metals in food (tin in tomato paste)	LGC	2005	Report in progress draft B	Run in parallel to CCQM-P72
IAWG/EAWG	CCQM-K48	Assay of KCl			Planned	
IAWG	CCQM-K49	Toxic and essential elements in bovine liver	NIST	2006	Planned	Run in parallel to CCQM-P85

IAWG	CCQM-K56	Trace elements in soybean powder	NIM	2006	Planned	Run in parallel to CCQM-P64.1
IAWG	CCQM-K57	Chemical composition of clay	CENAM	2006	Planned	Run in parallel to CCQM-P65.1
IAWG	CCQM-K58	Nitrogen and trace elements in silicon nitride powder	NMIJ/BAM	2006	Planned	Run in parallel to CCQM-P74.1
IAWG	CCQM-K59	Determination of nitrite and nitrate in calibration solutions and natural water	SMU/NRC	2006-2007	Planned	Run in parallel to CCQM-P89
	CCQM-P1	Trace elements in water Pb	NIST	1997	Completed 1998	
	CCQM-P7	KCl, NaCl, K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub>	NIST			
IAWG	CCQM-P11	As in shellfish	NIST	2001	Completed; progression to k	ey comparison proposed
IAWG	CCQM-P12	Pb in wine	IRMM	2000	Completed	
IAWG	CCQM-P12.1	Elements (e.g., Cu, Cd, Zn) in wine	LGC (with CMQ-F assistance)	2003	Protocol complete	Run in parallel to CCQM-K30
IAWG	CCQM-P13	Metals in synthetic food digest	LGC	2001	Completed	
IAWG	CCQM-P14	Trace elements (Pb, Se) in serum	NIST/LGC	1999	Abandoned (see next)	
IAWG	CCQM-P14	Ca in serum	IRMM/SP	2001	Completed; progression to key comparison proposed	
IAWG	CCQM-P15	Pb/Cd in sediments	IRMM	1999	Completed; progression to key comparison proposed	
IAWG	CCQM-P16	Elements in synthetic digest solutions	NMi	1999	Abandoned	
IAWG/OAWG	CCQM-P18	TriButylTin in sediment	LGC/NRC	2001	Completed; progression to key comparison proposed	
IAWG/EAWG	CCQM-P19	Hydrochloric acid	NIST	1999	Completed 2001	
IAWG/EAWG	CCQM-P19.1	Purity of HCl	NIST	2002	Complete	
IAWG/OAWG	CCQM-P20.a	TriButylTin in chloride	NARL	2001	Completed	
IAWG	CCQM-P25	Minor elements in steel	NMIJ/NIST/BAM	2002	Complete; progression to ke	y comparison proposed
IAWG	CCQM-P26	Sulfur in fuels	IRMM/NIST	2001	Completed	
IAWG	CCQM-P26.1	Sulfur in fuels (lower levels)	NIST	2003	Report in progress draft B	Run in parrallel to CCQM-K35
IAWG	CCQM-P29	Cd, Zn in rice	IRMM/NMIJ	2001	Completed	Run in parallel to CCQM-K24
IAWG	CCQM-P30	Elemental solution standards (Al, Cu, Fe, Mg)	EMPA/LNE	1999	Completed 2000	
IAWG	CCQM-P32	Anions in calibration solutions	EMPA	2001	Completed; progression to k	ey comparison proposed
IAWG	CCQM-P33	Boron in Si	PTB	2003	Complete	
IAWG	CCQM-P34	Constituents in Al alloy	BAM	2001	Complete	

IAWG	CCQM-P34.1	Consitutents of an aluminium alloy	BAM	Oct. 2004	Report in progress	Run in parallel to CCQM-K42
IAWG/EAWG	CCQM-P36	Assay of potassium hydrogen phthalate (KHP)	SMU/NIST	2002	Complete; progression to key comparison propose	
IAWG	CCQM-P39	As, Se, Hg, Pb, methyl-Hg in tuna fish	IRMM	2003	Complete	
IAWG	CCQM-P39.1	Methyl-mercury in salmon fish	IRMM	Nov. 2004	Complete	Run in parallel to CCQM-K43
IAWG	CCQM-P43	DiButylTin in sediment	LGC/NRC	2003	Completed	
IAWG	CCQM-P46	Preparation of inorgic calibration solutions	NIST	2003	Report in progress	
IAWG	CCQM-P48	Uranium isotope ratio in synthetic saline matrix	IRMM	2003	Report in progress	
IAWG	CCQM-P56	Minor elements in steel	NMIJ/NIST/BAM	2003	Complete	Run in parallel to CCQM-K33
IAWG	CCQM-P62	Trace analysis of high purity nickel	BAM	June 2004	Report in progress	
IAWG	CCQM-P63	Platinum group elements in an automotive catalyst	LGC	Aug. 2004	Report in progress	
IAWG	CCQM-P64	Trace elements in soyabean powder	NRCCRM	Sept. 2004	Complete	
IAWG	CCQM-P64.1	Trace elements in soybean powder	NIM	2006	Planned	Run in parallel to CCQM-K56
IAWG	CCQM-P65	Chemical composition of clay	CENAM	Oct. 2004	Report in progress	
IAWG	CCQM-P65.1	Chemical composition of clay	CENAM	2006	Planned	Run in parallel to CCQM-K57
IAWG	CCQM-P66	Determination of metals in fertilizer	NIST	Oct. 2004	Planned	
IAWG	CCQM-P70	Trace metals in sewage sludge	IRMM	Dec. 2004	Report in progress	Run in parallel to CCQM-K44, EUROMET 784 and IMEP
IAWG	CCQM-P72	Toxic metals in food (tin, lead and cadmium in tomato paste)	LGC	2005	Report in progress	Run in parallel to CCQM-K45
IAWG	CCQM-P74	Composition of fine ceramics	NMIJ	July 2005	Report in progress	
IAWG	CCQM-P74.1	Nitrogen and trace elements in silicon nitride powder	NMIJ/BAM	2006	Planned	Run in parallel to CCQM-P58
IAWG	CCQM-P75	Stable isotope delta values in methionine	IRMM/IAEA	Jan. 2006	In progress	
IAWG	CCQM-P76	Major and minor elements in copper alloy	BAM	Oct. 2005	In progress	
IAWG	CCQM-P85	Toxic and essential elements in bovine liver	NIST	2006	Planned	Run in parallel to CCQM-K49
IAWG	CCQM-P86	Analysis of total Se and Se methionine in pharmaceutical supplements	LGC/NRC	2006	In progress	

IAWG	CCQM-P89	Determination of nitrite and nitrate in calibration solutions and natural water	SMU/NRC	2006-2007	Planned	Run in parallel to CCQM-K59
CCQM Orga	nic Analysis Working	Group key comparisons and pilot studies				
OAWG	CCQM-K5	p,p'-DDE in fish oil	LGC	1999	Approved for equivalence	
OAWG	CCQM-K6	Cholesterol in serum	NIST	1999	Approved for equivalence	
OAWG	CCQM-K6 subsec	quent	NIST	2001	Approved for equivalence	
OAWG	CCQM-K11	Glucose in serum	NIST	2001	Approved for equivalence	
OAWG	CCQM-K11.1	Glucose in serum (subsequent comparison)	KRISS	2005	Report in progress draft B	
OAWG	CCQM-K12	Creatinine in serum	NIST	2001	Approved for equivalence	
OAWG	CCQM-K12.1	Creatinine in serum (subsequent comparison)	KRISS	2005	Report in progress draft B	
OAWG	CCQM-K21	p,p'-DDT in fish oil	LGC	2000	Approved for equivalence	
OAWG	CCQM-K25	PCBs in sediments (PCBs 28, 101, 153, 170)	NIST/NRC	2001	Approved for equivalence	
OAWG	CCQM-K27.2	Ethanol in water (subsequent)	NIST	2006	Planned	Subsequent comparison
OAWG	CCQM-K27.a	Ethanol in aqueous matrix (forensic level $1 \times 10^{-6}$ )	LGC/BAM	2002	Approved for equivalence	
OAWG	CCQM-K27.a subsequent	Ethanol in aqueous matrix (forensic level $1 \times 10^{-6}$ )	NIST	2003	Approved for equivalence	Run in parallel with SIM pilot study
OAWG	CCQM-K27.b	Ethanol in aqueous matrix (commodity level $100 \times 10^{-6}$ )	LGC/BAM	2002	Approved for equivalence	
OAWG	CCQM-K37	VOCs in organic solvents	KRISS/NIST	2003	Planned	
OAWG	CCQM-K38	PAHs in solution	NIST	Nov. 2004	Approved for equivalence	
OAWG	CCQM-K39	Chorinated pesticides in solution	NIST	Nov. 2004	Approved for equivalence	
OAWG	CCQM-K40	PCB congeners in solution	NIST	2004	Approved for equivalence	Run in parallel to CCQM-P31.b.1
OAWG	CCQM-K47	VOCs in solution	CENAM/NIST	2006	Planned	Run in parallel to CCQM-P61.1
OAWG	CCQM-K50	PAHs in soils/sediments	CENAM/BAM	2006	Planned	Run in parallel to CCQM-P69.1
OAWG	CCQM-K55.a	Purity assessment of high purity organic materials	BIPM	2007-2008	Planned	

OAWG	CCQM-P2	p,p'-DDE in isooctane	LGC	1997	Completed	
OAWG	CCQM-P3	NMR study	BAM	1998	Completed 1999	
OAWG	CCQM-P3.2	NMR study	BAM	1999	Completed 2000	
OAWG	CCQM-P4	p,p'-DDE in corn oil	LGC	1998	Completed; progression to ke	ey comparison proposed
OAWG	CCQM-P5	Acetanilide, benzoic acid, and naphthalene	NIST	1998	Completed 1999	
OAWG	CCQM-P6	Cholesterol in serum	NIST	1998	Completed; progression to ke	ey comparison proposed
OAWG	CCQM-P8	Glucose in serum	NIST	1999	Completed; progression to ke	ey comparison proposed
OAWG	CCQM-P9	Creatinine in serum	NIST	1999	Completed; progression to ke	ey comparison proposed
OAWG	CCQM-P10	Gamma-HCH in fish oil	LGC	1999	Repeated (see next)	
OAWG	CCQM-P10.2	Gamma-HCH in fish oil 74 ng/g, 240 ng/g	LGC	2000	Completed	
OAWG	CCQM-P17	PCBs in sediments	NRC/NIST	2000	Completed; progression to ke	ey comparison proposed
OAWG/IAWG	CCQM-P18	TriButylTin in sediment	LGC/NRC	2001	Completed; progression to ke	ey comparison proposed
OAWG/IAWG	CCQM-P20.a	TriButylTin chloride	NARL	2001	Completed	
OAWG	CCQM-P20.b	o-xylene	NIST	2002	Completed	
OAWG	CCQM-P20.c	Atrazine	NARL	2004	Report in progress	
OAWG	CCQM-P20.d	Chlorpyrifos	NARL	2004	Report in progress	
OAWG	CCQM-P20.e.1, -P20.e.2	Purity series: theophylline (2 samples)	BIPM/LGC	2006	Planned	
OAWG	CCQM-P20.f	Digoxin (purity assessment series)	BIPM/LGC	2006/2007	Planned	
OAWG	CCQM-P21	p,p'-DDT in fish oil	LGC	1999	Completed; progression to ke	ey comparison proposed
OAWG	CCQM-P27	LSD in urine	LGC	2001	Completed	
OAWG	CCQM-P27.1	Drugs of abuse in urine	NARL	2004	Planned	
OAWG	CCQM-P31.a	Organic calibration solutions (PAHs)	NIST	2003	Completed	
OAWG	CCQM-P31.a.1	Organic calibration solutions (PAHs)	NIST	2004	Completed	Run in parallel to CCQM-K38
OAWG	CCQM-P31.b	Organic calibration solutions (PCBs)	NIST	2003	Completed	
OAWG	CCQM-P31.b.1	PCB congeners in solution	NIST	2004	Completed	Run in parallel to CCQM-K40
OAWG	CCQM-P31.c	Organic calibration solutions (chlorinated pesticides)	NIST	2003	Completed	

OAWG	CCQM-P31.c.1	Organic calibration solutions (chlorinated pesticides)	NIST	2004	Completed	Run in parallel to CCQM-K39
OAWG	CCQM-P35	Ethanol in aqueous matrix (forensic and commodity levels)	BAM/LGC	2001	Completed; progression to key comparison proposed	
OAWG	CCQM-P40	Organic contaminants in mussel tissue	NIST	2003	Planned	
OAWG	CCQM-P57	PCB congeners in tissue extract	NIST	2004	Report in progress	
OAWG	CCQM-P61	Volatile organic compounds (VOCs) in solution	CENAM/ NIST	2004/2005	Complete	
OAWG	CCQM-P61.1	VOCs in solution	CENAM/NIST	2006	Planned	Run in parallel to CCQM-K47
OAWG	CCQM-P67	PCBs congeners in tissue	NIST	2004	Report in progress	
OAWG	CCQM-P68	Anabolic steroids in urine	NARL	2004/2005	Report in progress	
OAWG	CCQM-P69	PAHs in soils/sediments	CENAM/BAM	2004/2005	Report in progress	
OAWG	CCQM-P69.1	PAHs in soils/sediments	CENAM/BAM	2006	Planned	Run in parallel to CCQM-K50
OAWG	CCQM-P77.a	Progesterone in serum	NIST	2006	Planned	
OAWG	CCQM-P77.b	Cortisol in serum	NIST	2006	Planned	
OAWG	CCQM-P78	Nutrients in infant/adult formula	NIST	2006	Planned	
OAWG	CCQM-P88	Malachite green in fish	LGC	2006	Planned	
OAWG	CCQM-P90	Chloramphenicol in food	PTB	2007	Planned	
OAWG	CCQM-P91	Pyrethroids in apple juice	NIM			
OAWG	CCQM-P92	Moisture in grain (method dependent)	NIST/NMIA			
CCQM Surfa	ce Analysis Working G	Group key comparisons and pilot studies				
SAWG	CCQM-K32	SiO <sub>2</sub> on Si film thickness	NPL		Report in progress draft B	
SAWG	CCQM-P38	SiO <sub>2</sub> on Si film thickness	NPL	2002	Completed; progression to	key comparison proposed
SAWG	CCQM-P80	Carbon in precipitates in Fe	NPL	2005	Report in progress	
SAWG	CCQM-P81	N in surface layers of Fe	NPL	2005	Report in progress	
SAWG	CCQM-P84	SiO <sub>2</sub> on Si, surface analysis	NPL		Report in progress	Run in parallel to CCQM-K32
SAWG		Quantitative analysis of Fe-Ni alloy	KRISS	2006	Planned	
SAWG		Determination of Fe and N in doped DLC films	BAM	2006	Planned	
SAWG	CCQM-P95	Standard-free quantification in EPMA			Planned	

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

# RECOMMANDATION Q 1 (2006) : Métrologie en chimie et en biotechnologie

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

#### considérant

- l'importance de la fiabilité, de la comparabilité et de la traçabilité des mesures en chimie pour étayer le commerce international, pour le contrôle de l'environnement, y compris du changement climatique et de la qualité de l'air, de la qualité et de la sécurité des aliments, de la médecine de laboratoire et de la médecine légale;
- l'importance croissante de la biotechnologie pour la santé humaine, la production alimentaire, la médecine légale et la protection de l'environnement ;
- la fiabilité sans cesse meilleure des produits pharmaceutiques et des règlements sur l'équivalence internationale des résultats de mesure ;
- la mobilité internationale croissante de la population ;

**prenant acte** des travaux du Bureau international des poids et mesures (BIPM) pour coordonner et promouvoir les activités liées à la métrologie, en collaboration avec les autres organisations intergouvernementales et internationales concernées ;

#### recommande que

- les laboratoires nationaux de métrologie continuent à promouvoir et coordonner les activités nationales dans les domaines de la métrologie en chimie et en biotechnologie, en collaboration étroite avec les autorités concernées ;
- les laboratoires nationaux de métrologie définissent, en collaboration avec le Comité international des poids et mesures, les domaines prioritaires et les comparaisons internationales fondamentales pour assurer la comparabilité des résultats de mesure dans les domaines de la chimie et de la biotechnologie, aux niveaux mondial et régional;
- le BIPM continue à œuvrer au niveau international pour faciliter et étayer ces activités.

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

RECOMMENDATION Q 1 (2006):
On metrology in chemistry and biotechnology

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

#### considering

- the importance of reliable, comparable and traceable chemical measurement results in support of world trade, monitoring the state of the environment including climate change and air quality, food quality and safety, laboratory medicine, and forensics;
- the growing importance of biotechnology in human health, food production, forensic medicine and the protection of the environment,
- the increasing reliance of pharmaceutical products and regulations on internationally equivalent measurement results;
- the increasing international mobility of the world population;

**noting** the BIPM's activities in coordinating and promoting the development of metrology in cooperation with other relevant intergovernmental and international organizations;

#### recommends that

- National Metrology Institutes continue to initiate and coordinate national activities in the field of metrology in chemistry and biotechnology, in close cooperation with other relevant bodies;
- National Metrology Institutes in collaboration with the CIPM, work to define the areas of
  priority and essential international comparisons which are key to ensuring the comparability
  of measurement results in chemistry and biotechnology, both worldwide and within regions;
- the BIPM continues to work at the international level in order to facilitate and support these activities.

# APPENDIX Q 1. Working documents submitted to the CCQM at its 12th meeting $\,$

Working documents submitted to the CCQM at its 12th meeting are on restricted access.

#### **APPENDIX Q 2.**

BIPM metrology in chemistry programme: project proposals (2009-2012) Opinion of the CCQM Advisory Group

#### **Background**

The Advisory Group members were invited to comment on draft 6 of the BIPM proposals, dated 15 March 2006. Their comments were discussed at a meeting held at BIPM on 2 April 2006, following presentations by BIPM of the CIPM criteria used to prioritize programmes and of the responses to a questionnaire sent to NMIs and designated institutes. BIPM also gave an overview of progress with delivery of the 2005-2008 chemistry programme.

The Advisory Group regarded the following points as key in applying the CIPM criteria to the proposals for the 2009-2012 programme:

The BIPM needs to be a scientific institute in order to deliver its mission.

The BIPM science programme should focus on specific needs of international metrology and demonstrate clear added value to its customers (being the worldwide measurement community).

Activities of the BIPM should occupy a niche which addresses high level metrology issues and reflects BIPM's global status.

The 2005-2008 chemistry programme comprises scientific activities on gas analysis and purity of organic compounds, together with support for the JCTLM and its database. BIPM has proposed to continue and strengthen these activities in the 2009-2012 programme and, in addition, to introduce a bioanalysis programme. The current level of resource is 5.5 scientists, 2 technicians and 0.75 manyears from NMI scientists on secondment. If accepted in full, the 2009-2012 proposals would require staff numbers to be increased by the addition of 2 scientists, 4 technicians, 1 Post-doctoral research assistant, and 4 man-years from NMI scientists on secondment. Of these, the laboratory-based bioanalysis programme would require 2 scientists, 2 technicians, and 1 man-year from NMI scientists on secondment.

#### Gas analysis programme

The overall programme was broadly supported and felt to be a worthwhile extension of the current activities, which are well-regarded and closely linked to the programme of the CCQM Gas Analysis Working Group (GAWG). All projects should demonstrate clearly that they underpin measurement needs for key global issues and be dedicated to climate change and air quality. The Advisory Group noted that specific tasks identified for 2009-2012 may be overtaken by events and that the proposals should, as such, be regarded as indicative. The BIPM should carry out specific tasks with the consultation and co-operation of the GAWG at the appropriate time. Activities at BIPM should focus in particular on fundamental metrology.

#### Organic purity programme

It was suggested that BIPM programme should be focussed in one single project entitled 'Primary references for organic analysis'. The overall programme was supported, noting that it placed the

main emphasis on comparisons. The Advisory Group accepted that BIPM cannot be too specific at this stage of programme development but requested that more novel science related to method development should be included. It also recommended a focus on primary references to support food, healthcare and forensic applications as indicated by the response to the questionnaire. The Group would welcome development and coordination of a best practice guide on purity determination of organic compounds. This should build on the expertise developed under the BIPM chemistry programme.

#### Bioanalysis programme

It was felt that proposals for a bioanalysis laboratory project are premature and need wider debate. Activities at BIPM should focus on fundamental metrology and it was not clear that this is proposed or is feasible. The Advisory Group noted that this is a rapidly developing field where most key players are also able to devote substantial resources, far beyond those available to BIPM. It was accepted that staff working on liaison activities benefit from an involvement in relevant science. However, the Advisory Group recommended that BIPM should not develop its own research programme at this time. BIPM should consider novel alternatives to setting up its own bioanalysis research programme.

#### International coordination and liaison programme

The support for international liaisons was accepted as an important task but BIPM were requested to clarify the proposals. In particular, the Advisory Group saw a need to distinguish between the global role of the BIPM in this respect and the individual representation of Member States. The proposed project on bioanalysis liaison with national and international organizations was welcomed but BIPM should provide more information on the impact expected from this activity. In addition, consideration should be given to broadening the liaison beyond GMOs.

#### Recommendation

BIPM is requested to review the proposals with regard to the specific comments of the Advisory Group and provide a revised programme for the CIPM emphasizing the key top level themes. Notably, the gas work should fall within the area of air quality and climate change, and the organic chemistry programme should address primary references for organic analysis in support of food, healthcare and forensic applications. The BIPM should develop liaison activities but not a bioanalysis laboratory programme at this time. The Advisory Group welcomes the BIPM's proposals for its 2009-2012 chemistry programme and supports them subject to the implementation of its recommended amendments.

Mike Sargent Rapporteur, CCQM Advisory Group 5 April 2006