

Bureau International des Poids et Mesures

**Consultative Committee
for Amount of Substance
(CCQM)**

7th Meeting (April 2001)

Note on the use of the English text

To make its work more widely accessible the International Committee for Weights and Measures publishes an English version of its reports.

Readers should note that the official record is always that of the French text. This must be used when an authoritative reference is required or when there is doubt about the interpretation of the text.

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**MEMBER STATES OF THE METRE CONVENTION AND
ASSOCIATES OF THE GENERAL CONFERENCE**

as of 4 April 2001

Member States of the Metre Convention

Argentina	Japan
Australia	Korea (Dem. People's Rep. of)
Austria	Korea (Rep. of)
Belgium	Mexico
Brazil	Netherlands
Bulgaria	New Zealand
Cameroon	Norway
Canada	Pakistan
Chile	Poland
China	Portugal
Czech Republic	Romania
Denmark	Russian Federation
Dominican Republic	Singapore
Egypt	Slovakia
Finland	South Africa
France	Spain
Germany	Sweden
Greece	Switzerland
Hungary	Thailand
India	Turkey
Indonesia	United Kingdom
Iran (Islamic Rep. of)	United States
Ireland	Uruguay
Israel	Venezuela
Italy	

Associates of the General Conference

Cuba	Latvia
Ecuador	Lithuania
Hong Kong, China	

THE BIPM AND THE METRE CONVENTION

The International Bureau of Weights and Measures (BIPM) was set up by the Metre Convention signed in Paris on 20 May 1875 by seventeen States during the final session of the diplomatic Conference of the Metre. This Convention was amended in 1921.

The BIPM has its headquarters near Paris, in the grounds (43 520 m²) of the Pavillon de Breteuil (Parc de Saint-Cloud) placed at its disposal by the French Government; its upkeep is financed jointly by the Member States of the Metre Convention.

The task of the BIPM is to ensure worldwide unification of physical measurements; its function is thus to:

- establish fundamental standards and scales for the measurement of the principal physical quantities and maintain the international prototypes;
- carry out comparisons of national and international standards;
- ensure the coordination of corresponding measurement techniques;
- carry out and coordinate measurements of the fundamental physical constants relevant to these activities.

The BIPM operates under the exclusive supervision of the International Committee for Weights and Measures (CIPM) which itself comes under the authority of the General Conference of Weights and Measures (CGPM) and reports to it on the work accomplished by the BIPM.

Delegates from all Member States of the Metre Convention attend the General Conference which, at present, meets every four years. The function of these meetings is to:

- discuss and initiate the arrangements required to ensure the propagation and improvement of the International System of Units (SI), which is the modern form of the metric system;
- confirm the results of new fundamental metrological determinations and various scientific resolutions of international scope;
- take all major decisions concerning the finance, organization and development of the BIPM.

The CIPM has eighteen members each from a different State: at present, it meets every year. The officers of this committee present an annual report on the administrative and financial position of the BIPM to the Governments of

the Member States of the Metre Convention. The principal task of the CIPM is to ensure worldwide uniformity in units of measurement. It does this by direct action or by submitting proposals to the CGPM.

The activities of the BIPM, which in the beginning were limited to measurements of length and mass, and to metrological studies in relation to these quantities, have been extended to standards of measurement of electricity (1927), photometry and radiometry (1937), ionizing radiation (1960), time scales (1988) and to chemistry (2000). To this end the original laboratories, built in 1876-1878, were enlarged in 1929; new buildings were constructed in 1963-1964 for the ionizing radiation laboratories, in 1984 for the laser work, and in 1988 for a library and offices. In 2001 a new building for the workshop, offices and meeting rooms was opened.

Some forty-five physicists and technicians work in the BIPM laboratories. They mainly conduct metrological research, international comparisons of realizations of units and calibrations of standards. An annual report, the *Director's Report on the Activity and Management of the International Bureau of Weights and Measures*, gives details of the work in progress.

Following the extension of the work entrusted to the BIPM in 1927, the CIPM has set up bodies, known as Consultative Committees, whose function is to provide it with information on matters that it refers to them for study and advice. These Consultative Committees, which may form temporary or permanent working groups to study special topics, are responsible for coordinating the international work carried out in their respective fields and for proposing recommendations to the CIPM concerning units.

The Consultative Committees have common regulations (*BIPM Proc.-Verb. Com. Int. Poids et Mesures*, 1963, **31**, 97). They meet at irregular intervals. The chairman of each Consultative Committee is designated by the CIPM and is normally a member of the CIPM. The members of the Consultative Committees are metrology laboratories and specialized institutes, agreed by the CIPM, which send delegates of their choice. In addition, there are individual members appointed by the CIPM, and a representative of the BIPM (Criteria for membership of Consultative Committees, *BIPM Proc.-Verb. Com. Int. Poids et Mesures*, 1996, **64**, 124). At present, there are ten such committees:

- 1 the Consultative Committee for Electricity and Magnetism (CCEM), new name given in 1997 to the Consultative Committee for Electricity (CCE) set up in 1927;

- 2 the Consultative Committee for Photometry and Radiometry (CCPR), new name given in 1971 to the Consultative Committee for Photometry (CCP) set up in 1933 (between 1930 and 1933 the CCE dealt with matters concerning photometry);
- 3 the Consultative Committee for Thermometry (CCT), set up in 1937;
- 4 the Consultative Committee for Length (CCL), new name given in 1997 to the Consultative Committee for the Definition of the Metre (CCDM), set up in 1952;
- 5 the Consultative Committee for Time and Frequency (CCTF), new name given in 1997 to the Consultative Committee for the Definition of the Second (CCDS) set up in 1956;
- 6 the Consultative Committee for Ionizing Radiation (CCRI), new name given in 1997 to the Consultative Committee for Standards of Ionizing Radiation (CCEMRI) set up in 1958 (in 1969 this committee established four sections: Section I (X- and γ -rays, electrons), Section II (Measurement of radionuclides), Section III (Neutron measurements), Section IV (α -energy standards); in 1975 this last section was dissolved and Section II was made responsible for its field of activity);
- 7 the Consultative Committee for Units (CCU), set up in 1964 (this committee replaced the “Commission for the System of Units” set up by the CIPM in 1954);
- 8 the Consultative Committee for Mass and Related Quantities (CCM), set up in 1980;
- 9 the Consultative Committee for Amount of Substance (CCQM), set up in 1993;
- 10 the Consultative Committee for Acoustics, Ultrasound and Vibration (CCAUV), set up in 1998.

The proceedings of the General Conference, the CIPM and the Consultative Committees are published by the BIPM in the following series:

- *Reports of the meetings of the General Conference on Weights and Measures;*
- *Reports of the meetings of the International Committee for Weights and Measures;*
- *Reports of the meetings of Consultative Committees.*

The BIPM also publishes monographs on special metrological subjects and, under the title *The International System of Units (SI)*, a brochure, periodically updated, in which are collected all the decisions and recommendations concerning units.

The collection of the *Travaux et Mémoires du Bureau International des Poids et Mesures* (22 volumes published between 1881 and 1966) and the *Recueil de Travaux du Bureau International des Poids et Mesures* (11 volumes published between 1966 and 1988) ceased by a decision of the CIPM.

The scientific work of the BIPM is published in the open scientific literature and an annual list of publications appears in the *Director's Report on the Activity and Management of the International Bureau of Weights and Measures*.

Since 1965 *Metrologia*, an international journal published under the auspices of the CIPM, has printed articles dealing with scientific metrology, improvements in methods of measurement, work on standards and units, as well as reports concerning the activities, decisions and recommendations of the various bodies created under the Metre Convention.

LIST OF MEMBERS OF THE CONSULTATIVE COMMITTEE FOR AMOUNT OF SUBSTANCE

as of 4 April 2001

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

Bureau National de Métrologie, Laboratoire National d'Essais [BNM-LNE],
Paris.

D.I. Mendeleyev Institute for Metrology, Gosstandart 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], Taejon.

National Institute of Metrology [NIM]/National Research Centre for Certified
Reference Materials [NRCCRM], Beijing.

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

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 [NRC], Ottawa.

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

Office Fédéral de Métrologie et d'Accréditation [METAS], Wabern/ Swiss Federal Laboratories for Materials Testing and Research [EMPA], St Gall.

Physikalisch-Technische Bundesanstalt [PTB]/Bundesanstalt für Materialforschung und -prüfung [BAM], Braunschweig and Berlin.

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

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

Observers

Central Office of Measures/Główny Urząd Miar [GUM], Warsaw.

Centro Nacional de Metrología [CENAM], Mexico.

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

National Measurement Laboratory CSIRO [CSIRO-NML], Lindfield.

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.

Singapore Productivity and Standards Board [PSB], Singapore.

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

**Consultative Committee
for Amount of Substance**

Report of the 7th Meeting

(4 - 6 April 2001)

Agenda

- 1 Opening of the meeting; agenda; appointment of a rapporteur.
- 2 Report of the sixth meeting.
- 3 The Mutual Recognition Arrangement.
- 4 The development of the Appendix C database for CMC claims:
 - 4.1 CMC file formats for chemistry;
 - 4.2 A search engine for chemistry CMC claims;
 - 4.3 CMC claims for chemistry;
 - 4.4 Value assignment for proficiency-testing samples in CMC claims;
 - 4.5 Criteria for the acceptance of CRMs into Appendix C of the MRA.
- 5 Key comparisons and pilot studies:
 - 5.1 Coordination of activities;
 - 5.2 Key comparison reference values;
 - 5.3 Guidelines for the publication of the results of key comparisons;
 - 5.4 Cooperation between regional metrology organizations, and between regional metrology organizations and the CCQM.
- 6 Reports of working groups:
 - 6.1 Surface analysis;
 - 6.2 Metrology in biotechnology;
 - 6.3 Organic analysis;
 - 6.4 Inorganic analysis;
 - 6.5 Gas analysis;
 - 6.6 Electrochemical analysis;
 - 6.7 Key comparisons.
- 7 The importance of pure materials, the need for a world centre of primary pure reference materials.
- 8 Natural isotopic composition.
- 9 A symbol for amount-of-substance content.
- 10 CCQM symposium.
- 11 Traceability in clinical chemistry.

- 12 Traceability in global atmospheric measurements.
- 13 The BIPM programme of metrology in chemistry.
- 14 CCQM recommendations.
- 15 The composition of the CCQM and the CCQM working groups.
- 16 Other business.
- 17 Date of next meeting.

1 OPENING OF THE MEETING; AGENDA; APPOINTMENT OF A RAPPORTEUR

The Consultative Committee for Amount of Substance (CCQM) held its seventh meeting at the International Bureau of Weights and Measures (BIPM), at Sèvres, on 4 to 6 April 2001.

The following were present: K. Carneiro (DFM), T. Catterick (LGC), P. De Bièvre (IRMM/ISO-REMCO), E.W.B. de Leer (NMI VSL), R. Dybkaer (IFCC), G.L. Gilliland (NIST), M. Grasserbauer (IRMM), H.-P. Haerri (METAS), W. Hässelbarth (BAM), R. Kaarls (President), M. Kurahashi (NIMC), Y. Kustikov (VNIIM), J. McLaren (NRC), A. Marschal (BNM-LNE), W.E. May (NIST), B. Milman (VNIIM), M.J.T. Milton (NPL), A. Nomura (NIMC), K. Okamoto (NIMC), U. Örnemark (SP), H. Parkes (LGC), T.J. Quinn (Director of the BIPM), W. Richter (PTB), M. Sargent (LGC), M. Seah (NPL), H.G. Semerjian (NIST), Hun-Young So (KRISS), P. Taylor (IRMM/IUPAC), A.M.H. van der Veen (NMI VSL), M. Weber (EMPA), P. Woods (NPL), Yadong Yu (NRCCRM), A. Zschunke (BAM).

Observers: L. Besley (CSIRO-NML), E. Deák (OMH), M. del Rocio Arvizu-Torres (CENAM), S. Hart (NARL-AGAL), W. Kozłowski (GUM), W. Louw (CSIR-NML), M. Máriássy (SMU), Y. Mitani (CENAM), F. Söqût (UME).

Invited: R.R. Greenberg (NIST), B. King (NARL-AGAL), M.T. López Esteban (CEM), L. Mackay (NARL-AGAL), I. Papadakis (IRMM), M. Plassa (IMGC-CNR), A. Squirrell (CITAC), R. Sturgeon (NRC), M.C. Walsh (The State Laboratory, Ireland).

Also present: P. Giacomo (Emeritus Director of the BIPM); C. Thomas, R. Wielgosz (BIPM).

Excused: IAEA.

Absent: NIM.

Dr Quinn welcomed the CCQM to the Pavillon du Mail. This was the first meeting to be held in the building.

The President replied that the CCQM was very proud to be the first Consultative Committee to meet in the new building. He was sure that the

good spirit amongst participants in the CCQM would ensure that progress was made in the challenging field of metrology in chemistry.

The agenda was approved without modification.

The President expressed his thanks to Dr McLaren for preparing the reports of the CCQM meetings for the past five years. He proposed Dr Milton as rapporteur for the meeting, to be assisted by Dr Wielgosz. This proposition was accepted.

2 REPORT OF THE SIXTH MEETING

The report of the sixth meeting was approved.

3 THE MUTUAL RECOGNITION ARRANGEMENT

Dr Quinn reviewed recent progress with the implementation of the CIPM's Mutual Recognition Arrangement (MRA). He stated that all parties involved were gathering experience in carrying out key comparisons and compiling Calibration and Measurement Capabilities (CMCs) for the database of Appendix C of the MRA. The Joint Committee of the Regional Metrology Organizations and the BIPM (JCRB) was meeting twice yearly and was fulfilling its role in steering international activities under the MRA. These activities also relied on the activities of the Regional Metrology Organizations (RMOs) and, in particular, their nominated contact persons.

A number of lessons have been learnt. For example, entering data into the Appendix B database was proving to be difficult and sometimes small mistakes were made. Identifying such mistakes was difficult, and would become more problematic as the volume of data increased. A project was being planned in collaboration with Dr Cox at the NPL to develop a software package that would prepare data for entry into the Appendix B database.

[Note: after careful consideration it has been decided that this project will not be feasible, owing to the large variation between entries.]

A substantial number of CMCs from other areas had been entered onto the Appendix C database following the procedure agreed by the JCRB. It was important to start to publicise the availability of the BIPM key comparison database (KCDB) and to let potential users know what they are and what they do.

Dr Marschal asked if it was known who the users might be. Dr Quinn answered that the number of users of metrology worldwide was increasing, and that new users needed a mechanism to find the metrology services they required. The President added that the MRA had been developed in response to specific requests from international bodies concerned with accreditation and trade.

4 THE DEVELOPMENT OF THE APPENDIX C DATABASE FOR CMC CLAIMS

4.1 CMC file formats for chemistry

The President opened the discussion by reminding the meeting that the format for entering CMCs for chemistry had been agreed at the sixth meeting of the CCQM and that it should only be changed if there was a very strong reason to do so. Dr Thomas presented the structure of the chemistry database that would follow from the format of the chemistry files currently in use at the time of the meeting.

Dr Dybkaer proposed that the headings of the columns in the database be amended. The term “matrix” should be used to refer to all non-measurands and not to the entire “system” or “material”. In addition, the term “measurand” should also refer to the quantity measured (e.g. amount-of-substance fraction) and not solely to the species measured (e.g. glucose). A number of members agreed with Dr Dybkaer’s proposal to clarify the quantity being measured, but few agreed with the use of the term “matrix” in its strictest sense. The President insisted that the criterion to be used should be whether a user would understand the database and should not be based solely on matters of metrological purity.

At the conclusion of the first part of discussion it was decided that an additional column entitled “quantity”, which would be used to specify the quantity of concern, would be added to the chemistry CMC files.

[Note: The database containing the first CMCs in chemistry was opened to the public on 15 June 2001. Following the CCQM recommendations on this matter, the species measured is now referred to as “analyte or component”, the measured quantity is given, and the term “matrix” has been replaced by the expression “matrix or material”.]

4.2 A search engine for chemistry CMC claims

Dr Thomas explained that 500 lines of CMC claims for gases had been reviewed at the BIPM prior to proposing a method for the operation of a search engine for CMC claims in chemistry. The proposed search engine would allow searching for a specified analyte and matrix. Since more than 170 analytes had been used for gases alone, it was proposed that a method for simplifying the analyte search would be required. In particular, it was illustrated that the system of the “CAS numbers” would be used to overcome problems with the nomenclature of chemical species, and hence, it would be necessary to include this information within the chemistry CMC files.

Dr Thomas also illustrated how the search engine would present results on the BIPM website. The headings used in the CMC files would not appear *verbatim*, and the data would be presented in a more condensed form. She reminded the delegates that only the information contained within the CMC files would be accessible to the search engine. To allow for efficient searching of the database it would be necessary to eliminate inconsistencies in the presentation of data.

Members of the CCQM made a number of useful comments about the proposed format. The headings for the sets of columns listing the capabilities were discussed at length and it was agreed that the wording of the headings used in the CMCs themselves should appear in the results of the search engine.

The President summarized by approving the necessary changes to the CMC columns, which included the addition of new columns and a modification of column headings. A small group including representatives from the BIPM, the CCQM and the RMOs will meet on 9 and 10 April 2001 at the BIPM to discuss the search engine.

[Note: The conclusion of the meeting was to use free text search as the basis of the search engine.]

4.3 CMC claims for chemistry

The President explained that it was the responsibility of the JCRB, not the CCQM to approve CMC claims. Nevertheless, there was a need for the CCQM to facilitate agreement between the RMOs as to the sequence in which the CMC claims for chemistry might be presented.

Representatives from the EUROMET (Dr Deák), APMP (Dr Mackay), SIM (Dr May), SADC MET (Dr Louw) and COOMET (Dr Kustikov) summarized progress in their organizations.

Dr Quinn pointed out that there had been a shortage of information about progress with the inter-regional reviews after the data had been distributed to the regions by the BIPM. The BIPM proposed to provide current information on progress through the JCRB website. Dr Woods commented that as the inter-regional review of CMCs only covered approximately 10 % of all claims submitted, it was therefore essential that each RMO carried out a thorough review of all its own claims. Professor Grasserbauer added that the critical judgement of the national metrology institutes (NMIs) was essential in reviewing the CMCs since it was not evident “how far the light might shine” from many of the key comparisons. Dr May distributed a proposal for the involvement of some NMIs outside the SIM in the review of their CMCs.

Finally the President remarked that in the case where a country had more than one designated NMI, there should be no duplication of CMCs for a value of a particular quantity. Only one NMI per country can claim a CMC for any one value of a particular quantity.

4.4 Value assignment for proficiency-testing samples in CMC claims

The President drew the attention of the committee to a proposal from the NIST to list amongst its CMC entries the value assignment of a proficiency-testing sample as a “means for disseminating their measurement capability”. Dr May explained that in one case the NIST had provided a calibration service to the College of American Pathologists, which was an example of how their capability had been disseminated.

Dr McLaren explained that the entries in Appendix C to the MRA required demonstrable mechanisms for their dissemination. For example, the capability to carry out measurements at the NRC by glow-discharge mass spectroscopy would not appear because there was no means available to disseminate this measurement capability. Several members of the CCQM gave examples of why some caution was necessary when entering capabilities associated with proficiency testing in Appendix C. Mr Squirrel

reminded the CCQM that the results of a proficiency test only provided a “snapshot” of the relative performance of participants, and did not provide any form of traceability. Dr Marschal said that it was important to distinguish carefully between the capabilities themselves and the customers for them.

Summarizing, the President said that in principle the names of customers should not be listed, but in the case of the example discussed by Dr May, the name was a reference to a formal programme that had made use of the capability.

4.5 Criteria for the acceptance of CRMs into Appendix C of the MRA

The President introduced his paper (CCQM/01-8) which proposed criteria for the acceptance of Certified Reference Materials (CRMs) into the Appendix C database. He highlighted a number of difficulties he had attempted to resolve. For example, there were cases where a CRM was developed through a collaboration between the NMI that was responsible for it and other laboratories. Even when such collaboration was completely under the control of the NMI, there was some doubt as to whether such CRMs could support a claim for the competence of that NMI. He reminded the committee that the MRA was in a transition phase, and that many issues, such as this, might not be resolved before it was implemented in full.

Professor Grasserbauer drew the attention of the meeting to document CCQM/01-6 which summarized the operation of the certification process used by the Community Bureau of Reference (BCR). Many members of the CCQM had first-hand experience of this process. Although many BCR CRMs would not be in Appendix C, because they did not support a laboratory’s own capabilities, they did fulfil the appropriate criteria for quality. Since all available CRMs were required for the operation of a structured system to support measurements in chemistry, he suggested that it might even be necessary to develop a further database to list CRMs more widely.

The President repeated the intention of the JCRB that Appendix C should not be a catalogue of CRMs, but that it should show how the NMIs disseminate their measurement capabilities. Nevertheless, the CIPM was concerned that a single system should be established that covered all global needs for information about the services of the NMIs. Professor Zschunke observed that the MRA operated on a national basis, and that reference materials were often used as national standards. Professor Grasserbauer

commented that 70 % of legislation in Europe was “supra-national” and that, by analogy, the IRMM would have a “supra-national” responsibility for reference materials.

Dr Marschal asked whether there was a need for a list of available CRMs when sources of such information were already available? The President confirmed that Appendix C was not intended to duplicate the COMAR, or any other, database of CRMs. Dr Semerjian added that the provision of the CRMs was not the major motivation for the MRA. They were included in order to document measurement services provided by an NMI.

Dr Richter noted that document CCQM/01-8 specifically mentioned NIST Traceable Reference Materials (NTRMs) as being acceptable within Appendix C. He asked whether gas mixtures produced by laboratories accredited under ISO 17025 would also be acceptable, as they played a similar role in disseminating traceable measurements. The difficulty was explained by Dr Woods who pointed out that, in the case of gas mixtures, NIST SRMs were value assigned individually, but that NTRMs were value assigned based on measurements of some samples from the batch. Nevertheless, both types of reference material were issued with a NIST certificate.

In reply, Dr Semerjian, said that traceable reference materials from an accredited laboratory do not provide support for the claimed capabilities of an NMI.

Dr Quinn said that it was clear that the MRA referred to the “calibration and measurements capabilities” of signatories. Document CCQM/01-8 was an attempt to interpret this for CRMs. If this approach excluded a large number of CRMs that were important for trade and regulation, then the MRA might need to be implemented differently. The basic principles, as formulated within the document, were approved by the CCQM. In particular, there was general agreement that the following sentence in the document “The NMI claiming capabilities of dissemination of traceability via deliverable CRMs must have its own measurement capabilities in the field concerned and must actually be involved in the measurement and characterization of the claimed CRM”, defines the level of involvement of NMI measurement capabilities required for acceptable CMC claims. The wording in other parts of the document would be adapted correspondingly. Several members of the CCQM proposed textual changes to the paper. The President undertook to assimilate these corrections and to improve the document, prior to submitting it to the JCRB.

5 KEY COMPARISONS AND PILOT STUDIES

5.1 Coordination of activities

The Chairs and rapporteurs of the RMO working groups on amount of substance were asked to organize a meeting at short notice in order to coordinate and prioritize the activities between the RMOs with respect to their CMCs which are to be presented to the JCRB and need to be underpinned by key comparisons. An additional outcome of such a meeting will be to further the discussion on “How far does the light shine”?

5.2 Key comparison reference values

Dr Thomas showed how the results of an RMO key comparison, which by definition would be linked to a CIPM key comparison, would be presented on the KCDB (CCQM/01-28). This included a fictitious example to illustrate how a bilateral comparison subsequent to a key comparison might be linked to a key comparison. In this case, if the laboratory had also participated in the key comparison itself, it would now have a new degree of equivalence. If an RMO key comparison were held subsequently, it would be necessary for a number of laboratories to participate in both key comparisons. The example had been discussed with the CCQM Gas Analysis Working Group, where the problem of calculating the reference values was less difficult because of the availability of gravimetric data.

Dr May said that this was easy to understand in the case of a laboratory that did not participate in a key comparison and subsequently took part in a bilateral comparison. But in the case where it participated in two comparisons giving rise to two different degrees of equivalence, which would be recommended for use? Dr Quinn said that both would appear in the database, but the more recent would normally be recommended.

Dr de Leer showed how CCQM-K7 (BTX in air) included a satisfactory example of the incorporation of a bilateral comparison into a key comparison. Degrees of equivalence had been calculated for all laboratories involved in both parts of the comparison. Dr Semerjian agreed that this example was readily understood, but that there were more complicated examples where there was no independent reference value. Dr Quinn replied that a more complex example had been handled for key comparisons in the field of electricity and magnetism.

The President suggested that the Working Group on Key Comparisons should develop a response to this issue.

5.3 Guidelines for the publication of the results of key comparisons

The MRA requires that a full report of each key comparison be published in the Appendix B database. Dr Quinn introduced CCQM/01-1 which highlighted important points that the full report should cover, including a one- or two-page summary suitable for publication in the Key and International Comparisons section of *Metrologia*. Consideration had been given as to which names should be included in the author list of such publications. It was concluded that the list of authors is to include all participants of the key comparison. Authors from the pilot laboratory will be listed first, with an explanatory note to this effect.

A number of members of the committee questioned whether papers with extended lists of authors received great scientific credibility. Dr Taylor drew attention to the way the IUPAC distinguished between the authors of its publications. Dr Carneiro asked if the document applied to the key comparison reports from all Consultative Committees (CCs). Dr Quinn said it was expected to be of use to other CCs and RMOs.

Dr Milton asked whether a summary report was required in *Metrologia* in addition to an extended paper in the same journal? Dr Quinn confirmed that a summary report would always be required since it would often be published more rapidly.

5.4 Cooperation between regional metrology organizations, and between regional metrology organizations and the CCQM

The President said he was concerned to improve communication between the RMOs and the CCQM. In future, he would invite rapporteurs from the RMOs to meetings of the CCQM working group chairs. This would help to coordinate key comparisons held by the CCQM and the RMOs. Dr Sargent said that there had been some difficulty achieving such coordination within the Inorganic Working Group, where the CCQM had proposed to perform a pilot study while an RMO proposed to carry out a supplementary comparison on the same sample. Dr Quinn said that the confusion in this case largely arose because the same sample would be used for both comparisons.

Dr Semerjian expressed the view that problems occurred when a proposal that had been agreed at RMO (or CCQM) level was changed, perhaps because of developments in another RMO. He stressed that it was important for all participants to be clear as to the status agreed for each exercise in which they were planning to participate.

A number of members referred to the benefits gained by participants from outside a region participating in an RMO supplementary comparison. This was particularly the case when the majority of world capability existed in just one region. Dr Deák confirmed that EUROMET was prepared to involve laboratories from outside the region.

The President said he did not believe there was a requirement for further rules, but that it was important to ensure that all of those involved kept in good contact.

6 REPORTS OF WORKING GROUPS

6.1 Surface analysis

The President welcomed Dr Seah from the NPL who had acted as the Chair of the *Ad hoc* Working Group on Surface Analysis.

Dr Seah explained some of the challenges facing surface analysis, which unlike much else in chemistry is concerned with properties that are not homogeneous within the sample. This inhomogeneity, particularly with depth at the level of individual atomic layers, is utilized in many manufactured products for their advantage. He outlined the drive to develop surface analysis methods for the measurement of structures as the thickness of films used in different applications reduced. In the next few years, for microelectronics, the required gate oxide equivalent thickness for silicon dioxide on silicon will be less than 2 nm.

He outlined the properties of the SiO₂/Si system proposed for a pilot study (CCQM/01-16). This system has all of the appropriate attributes for an effective study.

Traceability of measurements of layers less than 10 nm thick is generally very poor. In many cases, the manufacturers of instruments quote the precision of such measurements, but not their accuracy. He outlined a series

of measurement methods that the *ad hoc* group considered to be suitable for use in the proposed study. In general, X-ray Photoelectron Spectroscopy (XPS) is the preferred method for this type of measurement. Other methods exist which are extremely slow but have excellent traceability or which are fast but are affected by contamination. Contamination at surfaces is very significant for measurements below 10 nm. Some of the spectroscopic techniques used are not sensitive to this contamination but this is not the case for some of the more popular methods.

The proposed study will use two different substrates, (100) Si and (111) Si, with several oxide thicknesses in the range 2 nm to 8 nm. Each laboratory will be sent up to eight samples. The protocol will include repeat measurements on each sample. Interest in the study, so far, involves seven NMIs (BAM, CSIR-NML, CSIRO-NML, KRISS, NIST, NMIJ and NPL) as well as some well-qualified expert laboratories. Trials at the NPL of samples with native SiO₂ of less than 1 nm thickness on a (100) Si substrate show that these very thin oxides remain stable for at least one year. They also show that spectroscopic techniques, such as XPS, are insensitive to contamination on the surface. Analysis by XPS correctly shows that between the native oxide and the silicon substrate there exist sub-monolayer levels of Si₂O₃, Si₂O and SiO.

Since contamination of the surface during the handling of the samples can give rise to important uncertainties for some methods, the NPL has tested many different cleaning procedures. The *ad hoc* group had approved one of these.

A protocol for the study had been circulated to the *ad hoc* group and endorsed by the participants. Laboratories will use approved cleaning procedures and carry out the measurements within three months. Appropriate batches of sample are still to be obtained but negotiations are currently in hand for premier quality material. Tests will then need to be made of the batch homogeneities.

Professor De Bièvre congratulated Dr Seah on his presentation which involved leading-edge technologies. He suggested that the capabilities described would also be of interest to those who are carrying out high-accuracy measurements on silicon artefacts as part of the CCM Working Group on the Determination of the Avogadro Constant.

Drs Carneiro and Marschal asked whether this work should be considered to be length metrology. In reply, Dr Seah said that the results of modern analytical science were expressed in many ways in addition to amount of

substance. Professor Zschunke suggested that these methods could assume a more fundamental role if they were expressed in terms of the number of atoms in a layer. Dr Máriássy answered that the results were simply amount of substance re-expressed in terms of the thickness of a chemically uniform layer.

Dr Semerjian said that the measurement was based on a spectroscopic technique that was chemical specific. He suggested that the *ad hoc* group should consider other techniques, such as SIMS, that were used for surface analysis.

The President mentioned that it had already been decided that this work would be within the scope of the CCQM, and that the proposed comparison would be a pilot study, designated CCQM-P38. He asked Dr Seah to develop a proposal for the wider work of the group beyond this study. When the CCQM receives such a proposal, the *ad hoc* group could be ratified as a full working group.

6.2 Metrology in biotechnology

The President welcomed Dr Gilliland from the NIST and Dr Parkes from the LGC, the coordinator of the *Ad hoc* Working Group on Metrology in Biotechnology. The *ad hoc* group was established following a decision by the sixth CCQM and first met in November 2000. Further meetings were held early in 2001 to identify the areas of greatest interest for the group's activities. They presented a report from the group (CCQM/01-9) and a short review of the requirements for metrology in the biotechnology sector.

They defined biotechnology as “a set of powerful tools that employ living organisms (or parts of organisms) to make or modify products, improve plants or animals, or develop micro-organisms for specific uses”. Work in the area progresses very rapidly, with entirely new approaches sometimes being invented, commercialized and exploited within the space of five years. Particular examples were the techniques of protein engineering which have led to the analysis of the proteome and might lead to a better understanding of some diseases and the targeted design of appropriate drugs. Developments in tissue engineering have enabled the production of “artificial skin”. Genetically modified crops required the application of less fertilizer and pathogens in water might be detected with greater sensitivity. The economic impact of all of these capabilities will be enormous. For example, the market for highly specific DNA diagnostic tests was expected to be 1.8 billion US dollars by 2005.

They described the role that the NMIs might have in developing a metrology infrastructure for biotechnology. Particular challenges for metrology included the measurement of nucleic acid, where the sequence of base pairs in the acid has to be measured as well as the amount of material present. In the field of protein measurements, molecules had to be identified, the amount measured and their size characterized, and in some cases, the biological activity measured.

Dr Parkes asked whether SI traceability could be established in practice and whether there was any potential for primary direct methods.

Dr Catterick asked how the group planned to contribute to a field that was developing so rapidly. In reply, Dr Gilliland said that the work would have to emphasize fundamental aspects that remained common throughout the development of these techniques.

It was proposed to hold a workshop at the IRMM in the autumn of 2001. The *ad hoc* group was planning a pilot study to determine the capability of laboratories to measure the quantity of DNA in a sample. This type of measurement is required to quantify the amount of genetically modified material in food and animal feedstuffs.

Professor Grasserbauer was of the opinion that biotechnology was a fascinating field of science and that it was imperative that the CCQM was active. He drew the committee's attention to a number of developments within Europe to establish networks of expert reference laboratories. Dr Parkes agreed that numerous inter-laboratory studies were under way, but that the CCQM should seek to identify a comparison that might underpin some of these.

Dr Hart described some work carried out by the AGAL to distinguish erythropoietin from natural and artificial sources. Such measurements of organic analytes are not part of "classical" analytical chemistry, but might fit well into the work programme of the *ad hoc* group. Dr de Leer told the meeting that a survey was in progress to establish a Dutch inventory of needs for metrological support in biology and biotechnology (CCQM/01-26).

Dr Dybkaer said that the term "biometrology" might be taken to refer to all biological measurement, which would be a massive undertaking. It is right to set appropriate priorities and that the IFCC would be able to nominate a suitable representative to the working group. The President proposed that the group become a working group under the chairpersonship of Dr Gilliland and Dr Parkes. The KRISS, the NMi and the NPL indicated an interest in participation.

Dr Milton said that the NPL would like to involve the U.K.'s National Institute for Biological Standards and Control (NIBSC) which is the World Health Organization's (WHO) reference laboratory for biological standards. The President said that he planned to visit the NIBSC with Dr Quinn. Dr Kustikov, Prof. Okamoto and Dr Walsh undertook to discover whether there were suitable representatives from their countries. The President confirmed the position of the group as a formal working group of the CCQM. The first meeting of the newly established working group will be at the NIST in June or July 2001.

6.3 Organic analysis

Dr May presented his report of progress by the Organic Analysis Working Group (CCQM/01-10). Three key comparisons had been completed:

- CCQM-K5 (pp'-DDE in fish oil), for which a draft B report was submitted to the meeting (CCQM/01-11). The KCRV had been calculated as the mean of the results (excluding two laboratories, the NARL and the VNIIM). The key comparison was approved for equivalence by the CCQM.
- CCQM-K6 (cholesterol in human serum) (CCQM/01-12), for which the sample was based on samples from a number of donors working at the NIST. CCQM-K6 was approved for equivalence by the CCQM.
- CCQM-K21 (DDT in fish oil), which followed a successful pilot study on the same system. Samples at levels of 70 ng/g and 170 ng/g were used. The relative standard deviation (RSD) of the results was 3.5 % for the lower-level sample, and 0.99 % for the upper-level sample. If one outlying result were removed (KRIS), the RSD for the lower-level sample would also be less than 1 %. The results of the key comparison were in a draft A report at the time of the meeting (CCQM/01-23).

Four pilot studies had been completed:

- CCQM-P8 (glucose in serum): The samples for the study were a NIST SRM that had been certified in 1996. Dr Dybkaer questioned the use of mg/g as the unit for the results of this study when many practical laboratories would use $\mu\text{mol/l}$. Dr May said that the density for these samples had also been measured at the NIST. The CCQM agreed that the results (CCQM/01-15) were adequate to proceed with a key comparison (CCQM-K11), based on samples used for the IMEP-17.

- CCQM-P9 (creatinine in serum): This study compared measurements made by IDGC/MS and IDLC/MS (CCQM/01-14). The CCQM agreed that the performance of the five participants was adequate to proceed to a key comparison (CCQM-K12), also based on samples from the IMEP-17. Dr Woods asked what traceability was involved if all participants used the same NIST SRM. Dr May said this was not the major source of uncertainty. He suggested that the suite of key comparisons involving glucose, creatinine and cholesterol in serum provided evidence that laboratories had the capability to measure “small molecule health status markers” in general.
- CCQM-P10.2 (gamma-HCH in fish oil): This study was a repeat of a previous one that had very poor results because of the low level of analyte in the samples. This second study had been successful (CCQM/01-13). It was not proposed to carry out a key comparison of this analyte because it would not provide additional information about laboratories’ capabilities.
- CCQM-P17 (PCBs in sediment): Nine sets of results were received for the two samples distributed by the NIST (CCQM/01-17). It is proposed to prepare the results of the study for publication in a journal. The results indicated a much better level of comparability between participants than the NIST had recorded for commercial laboratories in the United States. The CCQM agreed to proceed with a key comparison (CCQM-K25).

Three pilot studies were in progress at the time of the meeting: CCQM-P18 (tributyl tin in sediment) for which the samples would be distributed in April 2001; CCQM-P20 (organic purity assessment of glucose, pp'-DDE, xylene and tributyl tin); and CCQM-P27 (LSD in human serum).

Dr May proposed a series of new comparisons:

- Ethanol in an aqueous matrix (proposed by the LGC and the BAM) in two ranges, one suitable for evidential breath alcohol standards and one in a range suitable for measurements of ethanol in wine. The CCQM agreed that a pilot study could proceed (CCQM-P35).
- Three laboratories (KRISS, NARL and VNIIM) had requested bilateral key comparisons for cholesterol in serum in order to provide linkage to CCQM-K6. The CCQM agreed that these could go ahead.

The President responded to a query from Dr May about whether a pilot laboratory could impose a charge on any laboratory requesting a bilateral comparison. In general this is not done, but if a laboratory has to ask for a

second comparison due to errors made in the first, this may be a point to discuss. The issue was not resolved at the meeting.

6.4 Inorganic analysis

Dr Sargent presented his report of progress made by the Inorganic Analysis Working Group. The group had met in Teddington in the autumn of 2000 and at the BIPM prior to the CCQM meeting. Two key comparisons had been completed.

CCQM-K8 (elemental solution standards) was complete and Dr Weber presented the results of the measurements in solution of Al, Cu, Fe and Mg (CCQM/01-7). The thirteen participants had used a variety of different methods. One participant had submitted results that deviated from the KCRV by 20 %. Dr de Leer and Dr Marschal commented that commercial standards of this type claimed uncertainties in the range 0.2 % to 0.5 %, which was broadly the level of comparability demonstrated by this exercise. Mr Squirrel observed that very few of the results for Mg were comparable with the KCRV. The CCQM agreed to approve the results of the comparison for equivalence following the approval of the revised report by the working group and the working group Chairmen.

Dr Papadakis presented the results of CCQM-K13 (elements in sediment). This key comparison followed a successful study on the same system. A draft A report had been approved by the working Group (CCQM/01-21). Because the uncertainties submitted by the participants had not been calculated on a consistent basis (particularly for the digestion stage), the KCRV for both species had been based on a median, with two outlying results. The relative uncertainty of the KCRV was 3 % for Cd and 0.9 % for Pb. A draft B report was in preparation. Dr de Leer observed that this key comparison was of particular interest because it was the first time that such a complex sample preparation had been required in a CCQM comparison. Perhaps in cases where the preparation uncertainty was very large a primary method (such as IDMS) was not necessarily appropriate. Dr Taylor confirmed that this was an issue of some importance that was shared with the Organic Analysis Working Group.

Dr Papadakis also presented the results of CCQM-P12 (Pb in wine) (CCQM/01-22). Results had been received from fourteen participants. The samples had also been used as part of the IMEP-16 exercise. Dr Taylor said that the results of this study had been presented to an international trade body in the area. Dr Sargent proposed that the same samples be used for a

pilot study (CCQM-P12.1) of measurements of Cu. Additionally, it was hoped that a new sample could be developed by INTEC in Chile for a key comparison of measurements of Pb in wine.

Dr Sargent reported that the Inorganic Analysis Working Group was also continuing with eight pilot studies that had been decided at the sixth meeting of the CCQM. In addition, he proposed that CCQM-P19 (purity of HCl) be repeated. He recommended that a key comparison (CCQM-K24) be carried out for Cd in rice. The same samples would also be offered for measurements of Cd and Zn as a pilot study (CCQM-P29). The IRMM and the NMIJ would act as the pilot laboratories. A new pilot study was proposed by Dr Kurahashi for measurements of Cr, Mn, Ni and Mo in steel (CCQM-P25).

Moreover, the working group was considering a pilot study of boron in silicon (CCQM-P33). This proposal from the PTB would support measurements made by the semiconductor industry. Dr Woods remarked that the area was of significant interest and that there was a considerable amount of work going on in the area, including some based on SIMS measurements that would be part of the programme of the surface analysis *ad hoc* group. Dr Greenberg commented that the NIST had certified an SRM in this area, and that this work should be considered before proceeding with this study. The President referred the matter to Drs Greenberg, Richter, Sargent and Seah to develop a coherent proposal.

Dr Sargent proposed a pilot study of minor constituents in Al alloy to be led by the BAM. Dr Marschal and the President questioned whether work on this system was required at NMI level. Dr Sargent intimated that members of the working group were interested. The CCQM agreed that the pilot study could go ahead (CCQM-P34). Dr Sargent described a proposal from the SMU to carry out a pilot study of measurements of the purity of potassium hydrogen phthalate (CCQM-P36). This was also approved by the CCQM.

Dr Sargent asked if the BIPM could place the reports from its pilot studies on its website. Dr Wielgosz said that it was planned to do this. A series of papers which discussed aspects of neutron activation analysis (CCQM/01-18 to 20) had been received by the meeting. The working group proposed to meet in October 2001 at the IRMM.

6.5 Gas analysis

Dr de Leer presented a report of progress in the Gas Analysis Working Group. The group now has representatives from nineteen countries. He noted

that the report for CCQM-K3 (vehicle exhaust emission gases) had been approved at the sixth meeting of the CCQM and the report of CCQM-K4 (ethanol in air) was presented to the CCQM (CCQM/01-4). The CCQM agreed that the results could be published in the Appendix B database.

He showed the results of CCQM-K7 (BTX in air) and presented a draft B report to the CCQM (CCQM/01-3). This key comparison involved seven participants. Since the sample sent to one participant had been delayed by customs formalities, they had been offered a linked bilateral comparison, which was presented with the results of the key comparison. The results from one participant had been revised prior to the publication of the draft B report. The pilot laboratory was providing a detailed explanation of the sequence of events to the working group prior to their giving approval. A draft B report was being finalized.

A pilot study (CCQM-P23) was being carried out in which a number of laboratories had submitted standards of carbon monoxide in nitrogen at three concentrations to a single laboratory (NMI) for analysis using the same technique. In correcting the results for the influence of atmospheric pressure a problem had arisen which will be overcome following suggestions from members of the working group.

CCQM-K16 (natural gas) will extend the scope of CCQM-K1.e, f and g by incorporating components with larger molecular weights. The standards had been prepared by the BAM and the NMI and were ready for distribution.

CCQM-K10 will compare measurements of benzene, toluene and xylene (BTX) in air at concentrations in the range 3 nmol/mol to 5 nmol/mol, which are levels typically found in the ambient atmosphere. The NIST is the pilot laboratory, and planned to distribute the standards for the comparison later in 2001. Additionally, the Gas Analysis Working Group was developing a proposal for a Pilot Study (CCQM-P24) on dynamic mixing methods.

The results of four key comparisons carried out by the RMOs had also been reviewed by the working group. The draft B report for EUROMET.QM-K3 which concerned automotive emission gases had been approved by the working group. Dr de Leer presented the results (CCQM/01-2), which were approved for equivalence by the CCQM. He also presented the results of APMP.QM-K4 concerning ethanol in air, which had been submitted (in draft A form) to the working group. Dr Milton observed that linking such an RMO key comparison to the relevant CCQM key comparison would require recognition of the fact that although the KCRVs were based on the results

from the gravimetric preparation of the standards, the pilot laboratories in the two comparisons were not the same.

Dr de Leer reported that a detailed presentation of the work programme of the BIPM in the field of gas metrology had been presented to the working group by Dr Wielgosz. He expressed his and the working group's support for this programme.

Dr de Leer presented the plans of the Gas Analysis Working Group for future comparisons. Discussions with the WMO Global Atmospheric Watch (GAW) programme would lead to a detailed specification for CCQM-K14 (ambient carbon dioxide and methane in air) and CCQM-K15 (SF₆ and CFC's at emission levels). A pilot study on the comparability of ozone photometers (CCQM-P28) awaits the outcome of a EUROMET comparison being carried out by the NPL and the PTB, and will take into account the programme on ozone measurements at the BIPM. A comparison of volatile organic compounds is being planned by the NMIJ (CCQM-K22).

He also reported that EUROMET sought approval for two comparisons: EUROMET.QM-K1.c would compare measurements of NO in nitrogen, and would establish a link to CCQM-K1.c. EUROMET also proposed a supplementary comparison of NO and SO₂ at concentrations required to underpin the EU Air Quality Directive. This would be open to participants from other regions. The President suggested that this be made into a CCQM key comparison if there was sufficient interest from around the world.

Dr May asked if the programme of bilateral comparisons between the NIST and the NMi would be listed in the Appendix B database. He stated that these comparisons had taken place over a long period of time and used a strict protocol. Dr Woods replied that the EUROMET Gas Analysis Working Group would prefer these laboratories to take part in CCQM key comparisons rather than bilateral comparisons. Dr Carneiro said that bilateral comparisons were inefficient and should be reconsidered now that the MRA was in operation. The President agreed that in the long term, activity at the CCQM should replace such bilateral arrangements. Similar examples had arisen in the work of other Consultative Committees and guidance was being developed as to how they should be handled. Certainly in a transitional phase the results of bilateral comparisons will be listed in Appendix B. This provision will also extend to the results of future bilateral comparisons.

Finally, Dr de Leer explained that some errors had arisen in the degrees of equivalence for CCQM-K1.g when the pilot laboratory carried out the

calculation. The CCQM approved the necessary corrections to the Appendix B database.

6.6 Electrochemical analysis

Dr Richter presented his report of the work of the Electrochemical Analysis Working Group. He presented the draft B report for CCQM-K9 (phosphate buffers) for which the PTB had been the pilot laboratory supported by the SMU and the NIST. The key comparison had used a standard buffer formulation and a buffer with a formulation unknown to the participants. The KCRV was calculated from the weighted mean of results. Since the standard deviation of the results was twice as large as the uncertainty estimated for the weighted mean, the working group chose to estimate the uncertainty of the KCRV according to the so-called external consistency concept. The CCQM agreed that the final report could be approved for equivalence subject to the approval of the chairs of the working groups.

He then described the working group's plans to carry out comparisons based on the phthalate, carbonate, borate and tetraoxalate buffers (CCQM-K17 to K20). The working group was conducting a pilot study of electrolytic conductivity at the level of 0.15 S m^{-1} (CCQM-P22) with the DFM acting as pilot laboratory. He also proposed a pilot study to investigate the sources of uncertainty giving rise to the dispersion in the results of CCQM-K9.

6.7 Key comparisons

Dr Semerjian presented a table summarizing the status of all of the CCQM key comparisons and pilot studies, which was updated during the CCQM meeting (CCQM/01-27). He reminded the CCQM that this table was presented by sector and requirement. At some time, it would be important for the committee to review the distribution of key comparisons between sectors. He suggested that the CCQM solicit input from external organizations as to the highest priority requirements that were not being addressed. Particular areas that merited further investigation were measurements to support the health care and food sectors.

He said that there were issues arising from the need to link RMO comparisons with CCQM key comparisons, which should be discussed by the JCRB. A paper had been submitted discussing this issue (CCQM/01-5). He also informed the CCQM that the NIST would be holding a conference on metrology and its impact on global trade in the autumn of 2002. The

President added that the CIPM had commissioned a report that would discuss this and other issues.

Dr Walsh commented that measurements of moisture in food, and those related to drink and animal feedstuffs were of great importance. There is very little consensus on methods for these measurements and they might be a suitable area for CCQM activity in the future. Dr Plassa suggested that the CCQM pay greater attention to choosing consistent titles for key comparisons and pilot studies.

7 THE IMPORTANCE OF PURE MATERIALS, THE NEED FOR A WORLD CENTRE OF PRIMARY PURE REFERENCE MATERIALS

The President reviewed discussions that had taken place at previous meetings of the CCQM about a possible role for the BIPM in providing pure reference materials.

Dr Marschal was of the opinion that it would be difficult for the BIPM to provide a service with the resources they had available, but perhaps it could become the basis for a “virtual institute” that brought together information from organizations around the world. Professor Zschunke supported this proposal. He said it would be useful to coordinate the work of laboratories around the world. Dr King said that there were a large number of pure materials available already and that the CCQM could usefully concentrate on identifying important gaps in their availability.

In Prof. Okamoto’s view there were important requirements, but the work involved was time-consuming and expensive. He suggested that a questionnaire be circulated to identify the most important needs. Dr So said that information about such materials was readily available from a search of the internet. Closing the discussion, the President said that he believed it would not be possible for the BIPM to meet such a requirement itself. He suggested that the working groups consider what might be done in each area.

8 NATURAL ISOTOPIC COMPOSITION

Dr Plassa gave a presentation to the meeting about the requirement for accurate values of the natural isotopic composition of pure elements in order to improve the definition of some of the fixed points of the International Temperature Scale of 1990 (ITS-90). In particular, accepted values for the ratio D/H in water spanned the range 89 $\mu\text{mol/mol}$ to 156 $\mu\text{mol/mol}$. Such uncertainties in the determination of isotopic composition lead to an uncertainty up to 0.2 mK in the value of the triple point of water which is required with an accuracy of the order of 0.1 mK. A similar problem existed with the triple point of hydrogen.

Dr Taylor said that he had considered this issue, but that since most users do not use SI-traceable values it was difficult to make useful progress. Professor De Bièvre said that work at the IRMM was attempting to certify several gas standards with SI-traceable values for their isotope ratio. Dr Quinn said that the influence of these effects on the realization of ITS-90 was very small.

9 A SYMBOL FOR AMOUNT-OF-SUBSTANCE CONTENT

Dr Sargent relayed a proposal from Dr Pratt (NIST) to use ν_B as a symbol for amount of substance content. Dr Dybkaer said that the IUPAC and the IFCC had already agreed to this in 1995.

Dr Dybkaer then initiated a wider discussion on terminology. He explained that many practitioners considered the term “amount-of-substance concentration” to be cumbersome. In recent years, different groups had proposed that this be abbreviated to amount concentration or substance concentration. He had proposed the term “chemon” to a previous meeting of the CCQM.

Professor De Bièvre said that the proposal had been made, but that it had not been adopted widely. Dr May reminded the CCQM of the importance of making recommendations that would be agreed upon by practitioners in the field. Dr Máriássy said that amount-of-substance content was being used more widely since it was now possible to measure the mass of a system more

accurately than its volume. The President said that the CCQM should keep the matter under consideration.

10 CCQM SYMPOSIUM

The President reported that he had discussed the possibility of holding another symposium with a number of members of the CCQM. It had been suggested that “traceability” would be an interesting topic for such a symposium that would usefully extend previous discussions on “primary methods of measurement”. Dr Ornemark observed that some useful meetings on the topic had recently been organized by EURACHEM. A further opportunity to review progress in establishing traceability in the field was welcomed by Dr King.

A number of members of the committee suggested that it might be useful to focus on the dissemination of traceable measurements. The President suggested that the chairs of the working groups develop a detailed proposal for a symposium.

11 TRACEABILITY IN CLINICAL CHEMISTRY

The President informed the CCQM that there had been a meeting to discuss traceability in clinical chemistry between the CCQM, the IFCC and the BIPM, following the “Workshop on Measurement Traceability for Clinical Laboratory Testing and In Vitro Diagnostic Test Systems”, held at the NIST in the autumn of 2000. There will be another meeting in May 2001. The President was also planning to visit the WHO’s Reference Laboratory for biological standards, the National Institute for Biological Standards and Control in the United Kingdom.

12 TRACEABILITY IN GLOBAL ATMOSPHERIC MEASUREMENTS

A number of representatives from the CCQM had attended a meeting of the WMO's Global Atmospheric Watch (GAW) programme (CCQM/01-25) immediately prior to the meeting of the CCQM. This programme was founded in 1989 with the objective to "coordinate monitoring and research into the changing atmosphere". It operates twenty-two global observatories with three hundred associated regional stations that monitor a number of gas species of interest to the CCQM, including CO₂, CH₄, N₂O, ozone and several important photochemically-reactive gases. They have established quality assurance centres at four locations and a network of world calibration centres. Dr Wielgosz reported that the meeting held at the WMO had passed a resolution to explore the benefit of a future role for the BIPM and the NMIs in the WMO-GAW Science Advisory Group for Reactive Gases.

13 THE BIPM PROGRAMME IN METROLOGY IN CHEMISTRY

Dr Wielgosz described the programme of work for the Chemistry section at the BIPM (CCQM/01-24), which was formulated in consultation with a group of experts from the CCQM Gas Analysis Working Group and presented to the CIPM in October 2000. He described progress in the redevelopment of laboratories to carry out the BIPM's programme of experimental work.

The experimental programme of work will include a project on ozone photometry to be carried out in conjunction with the NIST, in addition to projects to develop facilities for the comparison of gas standards. He described the requirements for improved ozone photometry to support measurements of ozone in the atmosphere. Research would be required to underpin the ozone photometry by gas-phase titration of NO and NO₂ and through the measurement of absolute cross-sections of ozone in the ultraviolet. He showed how a programme of comparisons based on the infrastructure of the key comparisons and centred at the BIPM would

facilitate the international comparability of ozone measurements. The President thanked Dr Wielgosz for his clear presentation.

14 CCQM RECOMMENDATIONS

There were no recommendations.

15 THE COMPOSITION OF THE CCQM AND THE CCQM WORKING GROUPS

As of the sixth CCQM, the METAS and the EMPA have been admitted as members of the CCQM, while the CENAM, the PSB and the UME have been granted observer status.

16 OTHER BUSINESS

Dr de Leer proposed that the CCQM change its name to incorporate a direct reference to the phrase “metrology in chemistry”. The President explained that the acronym CCQM was recognized widely, and that it would be confusing to change it at this time. However, the phrase “metrology in chemistry” could be added as a subtitle to the acronym CCQM., and this would be discussed during the forthcoming meeting of the CIPM. [Note: The CIPM decided during its October 2001 meeting that the name of CCQM will be “Consultative Committee for Amount of Substance: metrology in chemistry”, but that the acronym CCQM would remain.]

The President commented that a considerable number of working documents were received and distributed to the CCQM for this meeting. He reminded

participants that it was important for documents to be submitted to the CCQM Executive Secretary by the set deadline, to allow delegates sufficient time to read the documents and prepare for the meeting. Dr Quinn added that with the increase in the number of working documents being submitted to meetings of Consultative Committees, it was no longer efficient to distribute these in paper form during or after the meeting. In the future documents would be distributed via a document download page available on the BIPM website. He confirmed that this web facility would be available shortly for the retrieval of working documents related to this meeting.

The President thanked the staff of the BIPM for their efforts in preparing the Pavillon du Mail for the meeting. He thanked all participants and declared the meeting closed.

17 DATE OF NEXT MEETING

The next meeting of the CCQM is scheduled for the 18 and 19 April 2002.

M.J.T. Milton, Rapporteur

May 2001

revised November 2001

APPENDIX Q 1.**Working documents submitted to the CCQM at its 7th meeting**

(see the list of documents on page 49)

LIST OF ACRONYMS USED IN THE PRESENT VOLUME

1 Acronyms for laboratories, committees and conferences

AGAL	Australian Government Analytical Laboratories
APMP	Asia/Pacific Metrology Programme
BAM	Bundesanstalt für Materialforschung und -prüfung, Berlin (Germany)
BCR	Community Bureau of Reference of the Commission of the European Communities
BIPM	International Bureau of Weights and Measures/Bureau International des Poids et Mesures
BNM	Bureau National de Métrologie, Paris (France)
BNM-LNE	Bureau National de Métrologie, Laboratoire National d'Essais, Paris (France)
CC	Consultative Committee of the CIPM
CCM	Consultative Committee for Mass and Related Quantities/ Comité Consultatif pour la Masse et les grandeurs apparentées
CCQM	Consultative Committee for Amount of Substance/Comité Consultatif pour la Quantité de Matière
CEM	Centro Español de Metrologia, Madrid (Spain)
CENAM	Centro Nacional de Metrologia, Mexico (Mexico)
CIAE	Chinese Institute of Atomic Energy, Beijing (China)
CIPM	International Committee for Weights and Measures/ Comité International des Poids et Mesures
CITAC	Cooperation on International Traceability in Analytical Chemistry
COOMET	Cooperation in Metrology among the Central European Countries
CSIR-NML	Council for Scientific and Industrial Research, National Metrology Laboratory, Pretoria (South Africa)
CSIRO-NML	Commonwealth Scientific and Industrial Research Organization, National Measurement Laboratory, Lindfield (Australia)
DFM	Danish Institute of Fundamental Metrology, Lyngby (Denmark)

EMPA	Swiss Federal Laboratories for Materials Testing and Research, St Gall (Switzerland)
EU	European Union
EUROMET	European Collaboration in Measurement Standards
GAW	see WMO-GAW
GUM	Główny Urząd Miar/ Central Office of Measures, Warsaw (Poland)
IAEA	International Atomic Energy Agency
IFCC	International Federation of Clinical Chemistry and Laboratory Medicine
IMEP	International Measurement Evaluation Programme
IMGC-CNR	Istituto di Metrologia G. Colonnetti, Consiglio Nazionale delle Ricerche, Turin (Italy)
INTEC	Corporación de Investigación Tecnológico de Chile, Santiago (Chile)
IRMM	Institute for Reference Materials and Measurements, European Commission
ISO	International Organization for Standardization
ISO-REMCO	International Organization for Standardization, Committee on Reference Materials
IUPAC	International Union of Pure and Applied Chemistry
JCRB	Joint Committee of the Regional Metrology Organizations and the BIPM
KRISS	Korea Research Institute of Standards and Science, Taejeon (Rep. of Korea)
LGC	Laboratory of the Government Chemist, Teddington (United Kingdom)
LNE	Laboratoire national d'essais, Paris (France), see BNM
METAS	(formerly the OFMET) Office Fédéral de Métrologie et d'Accréditation, Wabern (Switzerland)
MRA	Mutual Recognition Arrangement
NARL	National Analytical Reference Laboratory, Canberra and Pymble (Australia)
NIBSC	U.K. National Institute for Biological Standards and Control, Herts (United Kingdom)
NIM	National Institute of Metrology, Beijing (China)
NIMC*	National Institute of Material and Chemical Research, Tsukuba (Japan), see NMIJ/AIST

* Organizations marked with an asterisk either no longer exist or operate under a different acronym.

NIST	National Institute of Standards and Technology, Gaithersburg (United States)
NMi VSL	NMi Van Swinden Laboratorium, Nederlands Meetinstituut, Delft (The Netherlands)
NMI	National Metrology Institute
NMIJ/AIST	National Metrology Institute of Japan, National Institute of Advanced Industrial Science and Technology, Tsukuba (Japan)
NPL	National Physical Laboratory, Teddington (United Kingdom)
NPLI	National Physical Laboratory of India, New Delhi (India)
NRC-CNRC	National Research Council of Canada, Ottawa (Canada)
NRCCRM	National Research Centre for Certified Reference Materials, Beijing (China)
NRLM*	National Research Laboratory of Metrology, Tsukuba (Japan), see NMIJ
OFMET*	Office Fédéral de Métrologie, Wabern (Switzerland), see METAS
OMH	Országos Mérésügyi Hivatal/National Office of Measures, Budapest (Hungary)
PSB	Singapore Productivity and Standards Board (Singapore)
PTB	Physikalisch-Technische Bundesanstalt, Braunschweig and Berlin (Germany)
RMO	Regional Metrology Organization
SADCMET	SADC Cooperation in Measurement Traceability
SIM	Sistema Interamericano de Metrologia
SMU	Slovenský Metrologický Ústav/Slovak Institute of Metrology, Bratislava (Slovakia)
SP	Sveriges Provnings- och Forskningsinstitut/Swedish National Testing and Research Institute, Borås (Sweden)
UME	Ulusal Metroloji Enstitüsü/National Metrology Institute, Marmara Research Centre, Gebze-Kocaeli (Turkey)
VNIIM	D.I. Mendeleyev Institute for Metrology of Gosstandart of Russia, St Petersburg (Russian Fed.)
VSL	Van Swinden Laboratorium, Delft (The Netherlands), see NMi
WHO	World Health Organization

WMO	World Meteorological Organization
WMO-GAW	World Meteorological Organization, Global Atmospheric Watch Programme

2 Acronyms for scientific terms

BTX	Benzene, toluene, xylene
CFC	Chlorofluorocarbon
CMC	Calibration and Measurement Capabilities
COMAR	Database on certified reference materials (COde of Reference MAterials)
CRM	Certified Reference Materials
DNA	Deoxyribonucleic Acid
IDGC/MS	Isotope Dilution Gas Chromatography/Mass Spectrometry
IDLC/MS	Isotope Dilution Liquid Chromatography/Mass Spectrometry
IDMS	Isotope Dilution Mass Spectrometry
INAA	Instrumental Neutron Activation Analysis
ITS-90	International Temperaturre Scale of 1990
KCDB	BIPM Key Comparison Database
KCRV	Key Comparison Reference Value
NTRM	NIST Traceable Reference Material
PCB	Polychlorinated Biphenyl
RSD	Relative standard deviation
SI	International System of Units
SIMS	Secondary Ion Mass Spectrometry
SRM	Standard Reference Material
XPS	X-ray Photoelectron Spectroscopy