Bureau International des Poids et Mesures

Consultative Committee for Amount of Substance (CCQM)

5th Meeting (February 1999)

Note on the use of the English text

To make its work more widely accessible the Comité International des Poids et Mesures 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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THE BIPM AND THE METRE CONVENTION

The Bureau International des Poids et Mesures (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 m^2) 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 Comité International des Poids et Mesures (CIPM) which itself comes under the authority of the Conférence Générale des Poids et Mesures (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) and to time scales (1988). 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 and in 1984 for the laser work. In 1988 a new building for a library and offices 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, published in the *Procès-Verbaux des Séances du Comité International des Poids et Mesures*, 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:

- *Comptes Rendus des Séances de la Conférence Générale des Poids et Mesures;*
- *Procès-Verbaux des Séances du Comité International des Poids et Mesures;*
- *Reports of Meetings of Consultative Committees.*

The BIPM also publishes monographs on special metrological subjects and, under the title *Le Système International d'Unités (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 *Procès-Verbaux* of the CIPM.

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 10 February 1999

President

Dr R. Kaarls, member of the Comité International des Poids et Mesures.

Executive secretary

Dr R.S. Davis, Bureau International des Poids et Mesures [BIPM], Sèvres.

Members

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

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

Danish Institute of Fundamental Metrology [DFM], Lyngby.

Institute for Reference Materials and Measurements [IRMM], European Commission.

International Organization for Standardization: Committee for 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 Physical Laboratory [NPL]/Laboratory of the Government Chemist [LGC], Teddington.

National Research Council of Canada [NRC], Ottawa.

National Research Laboratory of Metrology [NRLM]/National Institute of Material and Chemical Research [NIMC], Tsukuba.

Nederlands Meetinstituut [NMi], Delft.

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 Bureau International des Poids et Mesures [BIPM], Sèvres.

Observers

Central Office of Measures [GUM], Warsaw.

CSIRO, National Measurement Laboratory [CSIRO], Lindfield.

National Office of Measures [OMH], Budapest.

Office Fédéral de Métrologie [OFMET], Wabern.

Slovenský Metrologický Ústav [SMU], Bratislava.

Consultative Committee for Amount of Substance

Report of the 5th meeting

(10-12 February 1999)

Agenda

1 Opening of the meeting; agenda; appointment of a rapporteur.
2 Minutes of the 4th meeting.
3 Nomenclature for key comparisons and other comparisons or studies.
4 Discussion of Appendix C of the Mutual Recognition Agreement.
5 Membership in the CCQM and CCQM working groups.
6 Reports of working groups:
  6.1 Working group on key comparisons;
  6.2 Organic analysis;
  6.3 Inorganic analysis;
  6.4 Gas analysis;
  6.5 pH.
7 The role of certified reference materials in metrology.
8 The katal and the SI.
9 Draft CGPM resolutions on metrology in chemistry and metrology in biotechnology.
10 Primary methods.
11 BIPM programme of metrology in chemistry.
12 Other business.
13 Date of next meeting.

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

The Consultative Committee for Amount of Substance (CCQM) held its 5th meeting at the Bureau International des Poids et Mesures (BIPM), at Sèvres. Five sessions took place on 10, 11 and 12 February 1999.

The following were present: K. Carneiro (DFM), T. Catterick (LGC), P. De Bièvre (IRMM, ISO/REMCO), E.W.B. de Leer (NMi-VSL), M. Grasserbauer (IRMM), W. Hässelbarth (BAM), Euijin Hwang (KRISS), F. Ingman (IUPAC), H. Jancke (BAM), R. Kaarls (President), Yu. Koustikov (VNIIM), M. Kubota (NIMC), M. Kurahashi (NIMC), B. Lundgren (SP), J. McLaren (NRC), A. Marschal (BNM-LNE), W.E. May (NIST), B. Milman (VNIIM), M.J.T. Milton (NPL), U. Örnemark (SP), Xiurong Pan (NRCCRM), T.J. Quinn (Director of the BIPM), W. Richter (PTB), M. Sargent (LGC), H.G. Semerjian (NIST), Hun-Young So (KRISS), C. Takahashi (NRLM), P. Taylor (IRMM), Min Zhao (NRCCRM).

Observers: E. Deák (OMH), H. Felber (OFMET/EMPA), S. Hart (CSIRO/NARL), B. Inglis (CSIRO), B. King (CSIRO/NARL), W. Kozlowski (GUM), M. Máriássy (SMU), D.W. Zickert (OFMET).

Invited: R. Dybkaer (IFCC), F. Hengstberger (CSIR-NML), I. Papadakis (IRMM), M. Plassa (IMGC-CNR), V.M.L. Ponçano A. Silva (IPT), A. Squirrell (CITAC).

Also present: Prof. P. Giacomo (Director emeritus of the BIPM); R.S. Davis, C. Thomas (BIPM).

Apologies for absence were received from Mr A. Alink (NMi-VSL) and Dr H.B. Kristensen (DFM).

Absent: NIM.

The President opened the meeting and welcomed the participants. He noted that the attendance (nearly fifty members, observers and invited guests) is the largest in the history of the CCQM. He introduced Dr René Dybkaer, representing the International Federation of Clinical Chemistry and Laboratory Medicine (IFCC). He noted that both the increasing attendance at this meeting and at earlier meetings of the various working groups signal continuing progress of, and growing interest in, the activities of the CCQM.

The Director of the BIPM, Dr Quinn, added his own words of welcome. He also drew the attention of the meeting to the availability of the 1999 Directory of Consultative Committees and of the 7th edition of the SI Brochure.

Dr McLaren was appointed Rapporteur, to be assisted by Dr Davis.

The agenda was adopted without modification.

2 MINUTES OF THE 4TH MEETING

The final version of the minutes of the 4th meeting was not made available to the members until just prior to this meeting. Members were asked to review these minutes before the end of the meeting. Subsequently, the minutes were accepted, after a few remaining errors had been noted.

3 NOMENCLATURE FOR KEY COMPARISONS AND OTHER COMPARISONS OR STUDIES

Dr Quinn drew the attention of the participans to document CCQM99-17, in which a new system of nomenclature for key comparisons (KCs), supplementary comparisons and other comparisons or studies identified by the Consultative Committees is described. Key comparisons identified by the CCQM will have the syntax QM-K1, 2, ...*n*. Supplementary comparisons will have the syntax QM-S1, 2, ...*n*. Other studies, such as pilot studies which might lead up to a key comparison, will have the syntax QM-P1, 2, ...*n*.

At the time that the comparison or study is executed, the prefix “CC”, the acronym of a regional metrology organization, or “BIPM” is added to the identifier to indicate responsibility for the execution. Thus, for example, key comparisons carried out by the CCQM will have the syntax CCQM-K1, 2, ...*n*, while supplementary comparisons carried out by EUROMET would be identified as EUROMET.QM-S1, 2, ...*n*.

Dr Kaarls noted that the existing key comparisons of the CCQM will be renumbered to be consistent with the new system.

4 DISCUSSION OF APPENDIX C OF THE MUTUAL RECOGNITION AGREEMENT

To initiate discussion of this agenda item, Dr Quinn provided a brief review of the reasons for which mutual recognition of national measurement standards and of calibration and measurement certificates issued by national metrology institutes (NMIs) is important. He reminded the participants that a draft mutual recognition agreement (MRA) had been initialled by NMI Directors in 1998, and that, during the 21st meeting of the CGPM in October 1999, the Directors will sign the MRA, thereby accepting the process and outcome. The importance of key comparisons to this process was explained. The results of the key comparisons will be maintained in a database which will underpin degrees of equivalence documented in Appendix B of the MRA (i.e. list of results of the comparisons). Information in Appendix B will in turn be used by regional metrology organizations (RMOs) and by the Joint Committee of the Regional metrology organizations and the BIPM (JCRB) in the approval process for the listing of calibration and measurement capabilities of individual NMIs in Appendix C of the MRA (i.e. list of quantities for which calibration and measurement certificates are recognized). The JCRB will have responsibility for the coordination and final approval of data provided by the RMOs, and for examining the validity of claimed measurement capabilities in Appendix C with respect to the corresponding evidence of measurement capability in Appendix B. (The process is described in CCQM99-20).

This introduction was followed by a discussion as to what should be the content of Appendix C in the case of amount of substance measurements. Dr Semerjian encouraged the CCQM to develop a position on this topic. It was agreed that the interpretation of the term “national measurement standards” in the case of chemical measurements was not obvious, but that it probably should include both measurement capabilities of an NMI (including uncertainty claims) and mechanisms for transfer of these capabilities (e.g. by means of certified reference materials) to other laboratories in its country.

Dr Milton expressed the view that a national measurement system should include measurement standards, a measurement capability and a mechanism for their dissemination (see CCQM99-04). In the document CCQM99-11, Dr Hässelbarth expressed the view that both measuring systems and primary reference materials could constitute national standards. Dr King suggested that, in the case of measurement capabilities, it may be possible to learn from the experience of accreditation programs, for which considerable specificity (e.g. analytes, concentration ranges, matrices) is required.

5 MEMBERSHIP IN THE CCQM AND CCQM WORKING GROUPS

Dr Kaarls, noting that a number of institutes have enquired about membership in the CCQM, reviewed the CIPM criteria for membership in Consultative Committees. First the institute must represent a member country of the Metre Convention. Secondly, the institute must have a demonstrated ability to contribute to the technical activities of the Consultative Committee in more than one area. This broad capability is most often found in the NMI. In the case where the expertise required for a specific Consultative Committee activity is not available in the NMI, the NMI may delegate national responsibility for this area to another institute in the same country, provided that the institute also has a viable mechanism for transfer of this capability to secondary laboratories. Laboratories not ready for full membership may apply to become observers.

Participation in key comparisons is open to laboratories having the highest technical competence and experience. Normally this will include members and observers of the CCQM or laboratories delegated by the NMI to have responsibility for a specific measurement standard or capability. Participation in the activities of the various CCQM working groups is, however, open to non-members. Broader participation in CCQM activities is also possible through the regional metrology organizations.

6 REPORTS OF WORKING GROUPS

6.1 Working group on key comparisons

Dr Semerjian made a presentation on the role of key comparisons in ensuring global measurement traceability and comparability, based in part on CCQM99-20, in which he delineated the roles of the Consultative Committees, the regional metrology organizations and the JCRB. He listed the roles of the Consultative Committees as follows: to identify appropriate key comparisons; to carry out some or all of these; to review and approve results for inclusion in Appendix B of the MRA; to select methodology for the determination of the stated reference values; and to establish the range of applicability for each set of key comparisons. He noted that the appropriate Consultative Committee working group, rather than the key comparison pilot laboratory, should select the participating laboratories for a key comparison, and that this selection should take into account the need for sufficient regional representation to support the goal of global comparability. He reiterated that participation in key comparisons is restricted to Consultative Committee members or delegated laboratories. The role of the regional metrology organizations was identified as follows: to influence the choice of key comparisons in order to meet regional needs; to carry out some of these (e.g. to facilitate linkage with regional comparisons); to review the results of key comparisons conducted by the regional metrology organizations; to carry out supplementary comparisons (e.g. to include measurement techniques other than those used in related key comparisons); to conduct other activities (e.g. training) intended to support mutual confidence of NMIs in the region; and, to review the claimed capabilities of regional NMIs, submitted for inclusion in Appendix C of the MRA, to ensure consistency between these claims and demonstrated performance as documented in Appendix B and the associated key comparison database. The role of the JCRB was identified as one of coordination of the examination, validation and final approval of the data provided by the regional metrology organizations.

Following this summary of roles and responsibilities, Dr Semerjian indicated a number of necessary conditions for the successful functioning of the MRA. First, the laboratories participating in the key comparisons must represent the pinnacle of the national measurement system. Secondly, these laboratories must actively participate in calibration activities, reference materials development and other elements of the infrastructure for the dissemination of

traceable standards. Thirdly, the methods used in the key comparisons must cover and test the principal techniques used in the field. Finally, key comparisons must be selected while keeping in mind all the areas of application and the most commonly used measurement methods. (In subsequent discussion, Dr Marschal suggested that the third condition be modified to indicate that methods used for key comparisons need to be connected to those employed in dissemination).

Dr Semerjian then presented a framework for selection of CCQM key comparisons, developed at a joint meeting of working group chairmen held at the LGC in the autumn of 1998. He noted that the proposed key comparison areas (e.g. health, food, environment, etc.), listed with possible examples in the document CCQM99-20, had been reviewed and discussed during the working group meetings held immediately prior to this meeting of the CCQM, and that several modifications had been made as a result of these discussions. He proposed that the CCQM develop a relatively long list of possible key comparisons for transmission to CCQM members in order to canvass interest and establish priorities. This list was prepared and revised by Dr Semerjian, based on the reports of the working group chairmen heard later in the meeting. It was reviewed in the final session of the meeting, and is attached to this report as Table 1 (p. 86). This table contains information about studies and key comparisons which have already been completed or are in progress, in addition to proposed future activities. It is intended to provide an historical record of CCQM activities as well as a framework for the planning of future activities. The nomenclature, past and present, given to these activities is shown in Table 2 (p. 90). The following actions related to Table 1 were approved.

1 It will be sent to working group chairmen and the CCQM executive secretary for checking and validation.

2 It will be sent to all CCQM members and observers to identify the following: areas that are of interest for their nation (region); key comparisons in which they would like to participate; pilot studies in which they would like to participate; areas of interest that are not included in the list, and indicate the driving force(s) for the measurement needs; and areas that should be deleted from the list, and provide justification.

3 It will be sent to interested organizations (regulatory agencies, trade organizations, standards organizations, etc.) for review and suggestions.

Dr Semerjian's presentation stimulated considerable discussion, both of the presentation material itself and of a number of policy issues related to key comparisons. Dr Grasserbauer asked what would distinguish many of the proposed key comparisons from comparisons carried out by other groups of expert laboratories to demonstrate comparability. He also asked how traceability to the SI would be addressed. To the first question, Dr Taylor replied that it would be necessary to use methods which yield results with the smallest combined uncertainty. To the second question, Dr Quinn replied that the primary methods used in key comparisons provide traceability to the SI by virtue of having a complete uncertainty budget.

A number of policy issues related to key comparisons were also discussed. It was agreed that there must be a clear *a priori* identification of a particular exercise as a key comparison (as opposed to a study or pilot project) prior to commencement of the exercise, and that in the future it will not be permissible, *a posteriori*, to declare a study a key comparison. The issue of withdrawal of unsatisfactory results from a key comparison was also discussed. Dr Quinn reviewed the policy on this matter, with reference to the BIPM guidelines for key comparisons, noting that a laboratory may withdraw results communicated only to the participants to the key comparison, but that this withdrawal of data then has to be reported in the final (Draft B) report of the key comparison. Dr Squirrell supported a firm policy on this matter, noting that the guidelines were consistent with the philosophy of ISO Guide 25 and ISO Guide 43 regarding withdrawal of results. This point led to an extended discussion. There was a strong body of opinion that a hard line should be taken so that results, once communicated, could only be changed with the agreement of all participants. [*Note*: This question was subsequently taken up by the JCRB and this policy was confirmed. The *Guidelines* for the key comparisons were therefore modified (see BIPM web site)].

There was also considerable discussion of mechanisms for arriving at key comparison reference values (KCRVs) for amount of substance key comparisons, and of the interpretation of the term "degree of equivalence". Dr Quinn noted that it was common for key comparisons in physical metrology to speak both of the degree of equivalence of a laboratory with the KCRV, and of degree of equivalence of two laboratories A and B which had participated in a key comparison. Dr Milton drew attention to the discussion paper CCQM99-15 on the interpretation of the MRA with respect to KCRVs for amount of substance measurements, prepared by Dr Davis, Dr Kaarls and himself. The distinction was made between key comparisons in which a KCRV is established *a priori*, for example by gravimetric comparison (as was

done, for example, in the case of the CCQM-1 study on the determination of Pb in water by IDMS), and those in which this cannot be done (as, for example, in the case of CCQM-9). Commenting on this document, Dr Máriássy cautioned against the use of the term "gravimetry" when referring to gravimetric preparation of test samples. There was general agreement that there will be many instances of key comparisons in which an *a priori* reference value determined by gravimetric preparation of the test sample will not be available. Dr Hässelbarth suggested that in this case, the use of a primary method to determine the KCRV should be mandatory, but that a check should be made for consistency between the KCRV value and the results of the participants. Dr De Bièvre drew attention to his discussion paper CCQM99-03, in which it was proposed that equivalence of laboratories participating in amount of substance key comparisons might better be based on a reference range rather than a KCRV. This view was opposed by Dr Kaarls and Dr Quinn, and it seemed to some others that even if a "reference range" rather than KCRV were used in statements about degrees of equivalence, there would nevertheless be a tendency to think of the mean value of this range as a KCRV. Dr De Bièvre also cautioned against the use of the phrase "horizontal traceability" in discussion of comparisons (including key comparisons) with reference to his submission CCQM99-08.

Dr Hässelbarth was invited by Dr Kaarls to comment briefly on his discussion paper CCQM99-10, concerning the importance of correlation terms in the calculation of uncertainty budgets for many types of chemical analysis. He noted that consideration of correlation terms does not always result in increased overall uncertainty, and can indeed sometimes result in a reduction of the overall uncertainty.

Dr Kaarls also asked Dr Milman to comment briefly on his paper CCQM99-01, concerning the inclusion of "substance identification uncertainty" in combined uncertainty budgets. Dr de Leer noted that this was a subject which was also of interest to CITAC. Dr Hässelbarth noted that this topic has also been discussed by the EURACHEM working group on uncertainty, and invited further contributions from other interested parties. Dr May indicated that Dr Milman's paper had been discussed earlier in the week at the meeting of the working group on organic analysis and might form the basis of some future activity of that group.

6.2 Organic analysis

Dr May presented a summary of the results of the 1998 activities of the working group on organic analysis and a proposed plan of future activities prepared at a meeting of the group earlier in the week. (Copies of his presentation slides were provided to committee members as CCQM99-24).

The first part of Dr May's presentation was a summary of the results of four activities carried out by the group in 1998, as follows: CCQM-4, NMR spectroscopy of mixtures (H. Jancke, BAM); CCQM-5, determination of pp'-DDE in corn oil (K. Webb, LGC); CCQM-6, characterization of pure organic substances (R. Parris, NIST); and CCQM-7, determination of cholesterol in human serum (M. Welch, NIST). This part of the presentation drew on results presented in four reports provided to members of the working group, as follows: CCQM-4, WGORG99-01; CCQM-5, WGORG99-02; CCQM-6, WGORG99-03; and CCQM-7, WGORG99-04.

Study CCQM-4 was an investigation of the performance of NMR spectroscopy as a candidate primary ratio method for measuring the concentrations of organic compounds in liquid mixtures. The pilot laboratory was BAM. The test sample was a gravimetrically prepared mixture of five organic compounds in $CDCl_3$, as follows: 1,2,4,5-tetramethylbenzene, 81.502; ethyl 4-toluene sulfonate, 13.253; cyclododecane, 2.701; octamethylcyclotetrasiloxane, 2.226; 1,3-dimethoxybenzene, 0.319, where all numbers are given as mole fraction in percent. Laboratories (not all of them NMIs) from ten countries participated in the study. The results indicated an encouraging level of agreement on the concentration of the major component (1,2,4,5-tetramethylbenzene), but poorer agreement on some of the other constituents, either for technical reasons related to details of the data acquisition, or, in the case of one constituent, an unexpected decomposition. It was agreed that the results of this first study were sufficiently encouraging to warrant a second study which would mimic a purity determination by means of a sample with one major constituent and several minor ones.

Study CCQM-5 was a comparison on the determination of pp′-DDE in a corn oil matrix by isotope dilution mass spectrometry. This exercise was more challenging than a previous comparison of the determination of pp′-DDE in solvent (CCQM-3) in that a sample clean-up step was required prior to GC/MS analysis. Samples containing the analyte at two mass fractions (0.072 µg/g and 4.74 µg/g) were prepared by the pilot laboratory (LGC) by gravimetric addition of appropriate amounts of pp′-DDE in 2,2,4-trimethylpentane. Eight sets of results were received from NMIs of seven

countries (both BAM and PTB participated). At the higher of the two mass fraction levels, all agreed to within about 1 % with the gravimetrically determined reference value. At the lower mass fraction, however, only about half of the laboratories achieved a result within about 1 % of the reference value. The majority of participants made use of a suggested uncertainty calculation, distributed with the samples, as the basis for calculating their uncertainties for this comparison.

Study CCQM-6 was piloted by the NIST, on the characterization of pure organic substances by a variety of purity assessment techniques (e.g. DSC/melting point depression, HPLC, GC/MS). Samples (two each) of three substances (benzoic acid, acetanilide and naphthalene) were analysed by seven NMIs using methods of their choice. As expected, the results were useful mostly as a means of identifying the issues which need to be resolved in future studies of this type. One such issue is whether an approach using a single technique (DSC/melting point depression), which may underestimate the total impurities, is satisfactory for purity assessment. Many issues surrounding the calculation of uncertainty budgets for purity assessments also remain unresolved.

Study CCQM-7 was a comparison of measurements of cholesterol in human serum, also piloted by the NIST. Two natural unspiked human serum materials were distributed to participating laboratories; material A was NIST SRM 965 (glucose in frozen human serum), while material B was NIST SRM 1951a (lipids in fresh frozen human serum). Material A is not certified for cholesterol, but the cholesterol level falls within the healthy range. A certified value for cholesterol (2.704 mg/g) is available for material B. Although it was left to the individual participating laboratories to choose their method(s), all laboratories which had submitted results by the time of the meeting used isotope dilution GC/MS. Results for material A from six laboratories ranged from 1.663 mg/g to 1.741 mg/g (coefficient of variation = 1.99 %); results for material B ranged from 2.607 mg/g to 2.704 mg/g (coefficient of variation = 1.49 %). In the view of the study organizer, the interlaboratory precision was satisfactory for clinical analyses, but perhaps not for an exercise intended to demonstrate equivalence among NMIs.

Dr May then presented a set of proposed activities for 1999 based on the 1998 studies. It was proposed to conduct a key comparison, piloted by LGC, on the determination of pp′-DDE in a cod liver oil matrix. Two additional analytes, hexachlorocyclohexane and a pesticide to be determined, would be added to the same sample(s), but determination of these analytes would not form part of the key comparison. It was also proposed to conduct a key

comparison, piloted by NIST, on the determination of cholesterol in human serum and to initiate studies for glucose and creatinine. It was suggested that future studies on the characterization of pure substances be focussed on materials that are analytes in key comparisons and studies. These proposals have been incorporated into the framework for key comparisons proposed by Dr Semerjian, as shown in Table 1.

6.3 Inorganic analysis

Dr Sargent presented a summary of the results of the 1998 activities of the working group on inorganic analysis and a proposed plan of future activities prepared at a meeting of the group earlier in the week. (Copies of his presentation slides were provided to committee members as CCQM99-22).

The first part of Dr Sargent's presentation was a summary of the results of two activities carried out by the group in 1998, as follows: CCQM-8, characterization of pure inorganic substances (K. Pratt, NIST); and CCQM-9, determination of Cd and Pb in natural water by IDMS (P. Taylor, IRMM). This part of the presentation drew on results presented in two reports provided to members of the working group, as follows: CCQM-8, WGIN99-01; CCQM-9, CCQM99-06.

Study CCQM-8, piloted by NIST, was intended to compare various approaches to determining the purity of inorganic substances. Samples of NaCl, KCl and $K_2Cr_2O_7$ were sent at the end of September 1998 to thirteen laboratories which had registered for participation. By the end of January 1999, fifteen sets of results had been received. The methods used for analysis were coulometry, gravimetry, potentiometric titration, and summation of impurities determined by instrumental analysis (e.g. ICP-MS). The results indicated that the methods often gave different results, and that the most appropriate method for a particular compound might depend on its intended use. The results for NaCl indicated a particular need for laboratories to agree beforehand on the drying procedure to be followed, as those laboratories which dried the material at 500 °C obtained a different result from those which dried at a temperature around 100 °C, because occluded water was removed at the higher temperature. There was considerable variation with regard to estimation and reporting of uncertainties. The order of inter-laboratory precision, from highest to lowest, was coulometry, summation of impurities, gravimetry and titrimetry. Participants were asked to submit comments, amendments, and uncertainty budgets to Dr Pratt by 10 March 1999. A report summarizing the results, conclusions and recommendations

for possible future work will then be prepared. It was felt by the participants that it would be premature to publish these results.

In discussion of this study, Dr Felber suggested that the precision of the titrimetry results would probably have been better if participants had used the same drying procedure, otherwise the proposed level of precision is not valid. Dr Milton commented upon the difficulties in developing robust uncertainty budgets for purity analyses, noting the many inconsistencies in this study. He expressed the hope that new thinking would emerge at forthcoming workshops on uncertainty. Dr Marschal felt it would be useful to include information about individual impurities (e.g. Br^-) in these materials in the final report. Dr King questioned the wisdom of not publishing the results, but Dr Sargent reiterated the view of the participants that the work is incomplete.

Study CCQM-9 was a comparison involving the determination, by isotope dilution mass spectrometry, of Cd and Pb, at amount contents of approximately 83 $nmol \cdot kg^{-1}$ and 63 $nmol \cdot kg^{-1}$, respectively, in a natural fresh-water sample. The exercise was piloted by the IRMM in conjunction with the IMEP-9 comparison, which involved determination of these elements and others in the same sample. Ten NMIs submitted results for CCQM-9. One laboratory subsequently withdrew its results, which were much higher than those of all other laboratories, because of suspicion that the sample had been contaminated by opening for a customs inspection during transit. The remaining nine laboratories agreed to within 2.6 % of the IMEP-9 certified value for the Cd concentration and to within 2.1 % for the Pb concentration. As the IMEP-9 sample was not prepared gravimetrically, there were no gravimetric reference values available. There were, however, reference values which had also been determined by IDMS for the IMEP-9 comparison. Three of the nine CCQM-9 laboratories (IRMM, KRISS and NRC) also participated as "reference laboratories" in the IMEP-9 comparison, along with two other laboratories which did not take part in CCQM-9. In neither case did the reference values determined for use in IMEP-9 differ significantly from the mean of the CCQM-9 results. Although there appeared to have been some confusion as to whether CCQM-9 had been designated a key comparison at the 4th CCQM meeting, it was agreed by the participants to propose that CCQM-9 be designated a key comparison. A final (Draft B) report will be prepared, and a revised report with a summary of the main approaches to estimating the combined uncertainty will be prepared for publication. Dr Carneiro supported the view that this study should be considered a key comparison. Dr Semerjian recommended a clear statement of policy regarding modifications and withdrawals of data from key comparisons.

Dr Taylor made some additional comments on CCQM-9 in the context of the IMEP-9 comparison. He noted that by combining the two, a successful CCQM comparison having a transparent link to results at other metrological levels had been achieved.

In discussion of this study, Dr Hässelbarth commented that, in his opinion, this was a good example of a means of establishing a key comparison reference value when no target value from another source (e.g. gravimetric preparation of the test sample) was available. Dr King asked whether neutron activation analysis (NAA) might also be a candidate for designation as a primary method for trace analysis. Dr May reported that NIST NAA specialists are in the process of documenting procedures with a view to having the method considered primary. Dr Squirrell commented that the combination of CCQM-9 and IMEP-9 results showed the advantages of using the same sample for a key comparison amongst NMIs and additional comparisons.

Dr Örnemark made a brief presentation on an earlier IMEP comparison concerning the determination of trace elements in human serum (IMEP-7). The report to participants in this exercise was distributed to CCQM members as document CCQM99-05. Copies of Dr Örnemark's transparencies were distributed as CCQM99-13.

Dr Sargent then presented a list of potential key comparisons that had been developed by the working group on inorganic analysis as a starting point for further discussion and comment by correspondence. These have been incorporated into the framework for key comparisons proposed by Dr Semerjian, as shown in Table 1.

Dr Sargent then presented a plan of suggested studies to commence in 1999. These included a key comparison on elemental calibration solutions, to be organized by the EMPA (Dr Felber) in collaboration with the BNM-LNE (Dr Marschal), as well as a key comparison on the determination of trace elements in a sample of drinking water, which will be used for the IMEP-12 comparison. In subsequent discussion of the latter proposal by the CCQM, it was concluded that there was insufficient interest in this key comparison because of its apparent similarity to the recently completed CCQM-9 key comparison. While the advantages of combining a key comparison with a comparison involving a broader population of laboratories had been well illustrated by the CCQM-9/IMEP-9 exercise, it was felt that it would be preferable to conduct a CCQM study on the determination of trace elements in a sediment material, in conjunction with IMEP-14.

6.4 Gas analysis

Dr Alink (NMi) was unable to attend the meeting due to illness. Therefore, Dr Milton presented the summary of the results of the activities to date of the working group on gas analysis. He also proposed a plan of future activities. He drew attention of the participants to two documents circulated before the meeting, CCQM99-09, the minutes of the meeting of the working group held at NPL in December 1998, and CCQM99-12, the draft final report on the first comparison on primary standard gas mixtures (CCQM-2).

Study CCQM-2 was a seven part exercise, begun in 1993, involving analysis of the following five major groups of gas mixtures: A, CO in nitrogen; B, CO_2 in nitrogen; C, NO in nitrogen; D, SO_2 in nitrogen; E, F and G, three types of natural gas. The comparison was piloted by the NMi, and attracted ten participants. In most cases, laboratories agreed to within 1 % of the gravimetrically determined reference values. On behalf of the working group, Dr Milton proposed that CCQM-2 be designated a key comparison, and that the final report be submitted for publication in *Metrologia*.

Comparison CCQM-10, on the determination of CO, CO_2 and C_3H_8 in nitrogen has recently been completed. All results have been received, but full uncertainty statements have yet to be received from some participants. This exercise is also a key comparison.

Study CCQM-11, a comparison on the determination of ethanol in air, was just under way. The next comparison planned will be for benzene, toluene and xylene (at volume fractions less than 50 parts in 10^9) in N_2 or air. The pilot laboratory will be the NIST.

Dr Milton then reviewed several comparisons which are currently under discussion. The first of these concerned "global warming" gases (CO_2 and CH_4 in air at ambient levels, i.e., a few parts in 10^6) and SF_6 and CFCs (at emission levels). A proposed comparison on "air quality" gases (SO_2, NO_2, and ozone) is expected to present a serious technical challenge to NMIs if a target uncertainty of about 1 % at low concentrations is set. These proposals have been incorporated into the framework for key comparisons proposed by Dr Semerjian, as shown in Table 1.

Dr Milton then presented some preliminary results from the CCQM-10 key comparison on "automotive" gases. A total of thirteen laboratories from twelve countries participated in this exercise, piloted by the NMi. Nominal mole fractions of the components (in N_2) were as follows: CO, 2×10^{-2} mol/mol to 4×10^{-2} mol/mol; CO_2, 10×10^{-2} mol/mol to 14×10^{-2} mol/mol; and C_3H_8, 1800×10^{-6} mol/mol to 2200×10^{-6} mol/mol.

The results indicate that agreement to within 1 % of the reference values will be achieved. The working group expects to have the Draft B report of this key comparison ready for approval by the CCQM next year. The next meeting of this group has been scheduled to take place at the NIST in September 1999.

6.5 pH

Dr Richter reported that no comparisons had yet been carried out by the working group on pH, but that a IUPAC working group which was formed in 1997 had met twice in 1998. The primary task of this group was to address concerns about traceability of pH measurements and the possibility of confusion resulting from the existence of two pH scales. The working group has reached a consensus on the most important components of new recommendations to IUPAC, as described by Dr Milton in CCQM99-07.

On behalf of the CCQM working group on pH, Dr Richter proposed a key comparison involving pH determinations in two phosphate buffer mixtures. The first exercise would be the measurement of pH in a Na_2HPO_4/KH_2PO_4 buffer solution at a molality of 0.025 mol/kg. The second exercise would measure a phosphate buffer solution of unknown composition. This exercise is to be piloted by the PTB, with participation by six CCQM member institutes (DFM, KRISS, NIMC, NIST, PTB and VNIIM) as well as four other institutes (CENAM, GUM, OMH and SMU).

7 THE ROLE OF CERTIFIED REFERENCE MATERIALS IN METROLOGY

Dr Kurahashi made a presentation based on document CCQM/98-24, tabled at last year's meeting but not yet discussed, and CCQM99-18. It was proposed that the CCQM initiate an activity for the production of certified reference materials (CRMs) for international use as "national measurement standards", and that emphasis be given to high-purity, primary standard materials rather than to matrix CRMs. He noted that the NIMC has a plan to develop high-purity organic materials.

There was general agreement that, while primary standard (or other) CRMs could be considered as "national measurement standards", they need to be

complemented by measurement capability, as described in Dr Milton's submission CCQM99-04. Dr Semerjian and Dr Grasserbauer commented that ownership, responsibility for maintenance and eventual replacement of CRMs produced by the CCQM would be very problematic.

8 THE KATAL AND THE SI

Dr Kaarls brought the Committee's attention to a proposal, submitted by the IFCC to the Consultative Committee for Units (CCU), which calls for adoption of the derived unit "katal" (abbreviation "kat") to denote mol/s when expressing enzyme catalytic activity in terms of the rate of conversion of a specified indicator reaction. Dr Quinn explained that the CCU does not normally endorse new derived units, but had made an exception in this case. This is partly because a precedent had already been set by the creation of a number of other derived units in the field of health physics. The CIPM had approved the CCU recommendation in principle but, before proceeding further, had asked for the opinion of the CCQM on the matter.

A number of documents on this topic had been circulated to CCQM members prior to the meeting. Document CCU1998-7 is the letter to the CCU from the IFCC regarding the katal. Recommendation U 1 was submitted by the CCU to the CIPM recommending adoption of the katal. An exchange of correspondence between Dr Dube and Dr Richter of the PTB and Dr Dybkaer was circulated as CCQM99-14 and CCQM99-14a. Dr Dybkaer indicated that he fully concurred with the suggestions made by Drs. Dube and Richter. After some further discussion about possible misuse of the unit "katal", the CCQM agreed to support the CCU Recommendation U 1, subject to the addition of the following text to the recommendation.

> *"...**and further recommends** that when the katal is used the measurand must be specified by reference to the measurement procedure, which must identify the indicator reaction."*

[*Note*: The CIPM subsequently approved a draft resolution to be put to the 21st CGPM in October 1999].

9 DRAFT CGPM RESOLUTIONS ON METROLOGY IN CHEMISTRY AND METROLOGY IN BIOTECHNOLOGY

Dr Kaarls drew the attention of the CCQM to two draft resolutions which he will introduce at the next meeting of the CGPM (October 1999): draft Resolution J, concerning metrology in chemistry and draft Resolution K, concerning metrology in biotechnology. Members of the committee had several suggestions for the improvement of the wording of Resolution J, which resulted in the following version:

"**considering**

- the worldwide development of trade agreements under the World Trade Organization,
- the need to eliminate measurement related technical barriers to trade, particularly in the areas of food science, pharmaceuticals and high technology materials,
- that many environmental and public health decisions are based on globally recognized measurements in chemistry,
- that the development of traceability for measurements in chemistry is still far from complete worldwide,

recommends that national metrology institutes

- continue to initiate and coordinate national activities in the field of metrology in chemistry, in close cooperation with other relevant bodies,
- in collaboration with the Comité International, work to define the areas of priority and essential international comparisons which are key to the comparability of measurements in chemistry, both worldwide and within regions."

Dr Kaarls advised the CCQM that, while it was too late to make amendments to the resolution, he would take the members' comments into account in his presentation.

There was also considerable discussion of draft Resolution K. Dr Marschal asked why the types of measurements mentioned in the resolution were not defined in greater detail. Dr Quinn replied that the resolution was intended to

encompass all fields of measurement relevant to biotechnology. Dr King asked why it was felt necessary to single out biotechnology, as opposed to other areas of technology, for a special resolution. Dr Kaarls asked whether the CCQM needs to do more to develop its relationship with the clinical chemistry community. Drs. Marschal, May and So could see no reason for the CCQM to give special emphasis to clinical chemistry rather than assessing measurement problems and needs in many fields. Dr Quinn explained that the resolution is intended to point to a new area of potential interest. He suggested that it is inappropriate in such a resolution to recall existing activities, important as they may be.

Dr Dybkaer pointed out that perhaps such an emphasis could be justified by the fact that the health industry does more chemical measurements than any other sector, and furthermore that many of these are not done well. Dr de Leer reminded the committee of the potential implications for clinical analysis of the forthcoming EU directive on "in vitro diagnostic medical devices". Dr Semerjian suggested that the CCQM should ask the IFCC and other appropriate organizations to review the proposed list of CCQM key comparisons for relevance. Dr Kaarls suggested that the chairman of the working group on key comparisons (Dr Semerjian) coordinate a gathering of information from experts in clinical chemistry. Dr Marschal proposed that this could be done in a 1-2 day meeting of NMI representatives with experts in the clinical chemistry field.

10 PRIMARY METHODS

Dr Kaarls initiated discussion of the CCQM definition of a primary method of amount of substance measurement. Dr Milton briefly reviewed the history of the current definition, with its attached explanatory notes. In his view, the definition should not be changed, but some ambiguity remains in the interpretation of the phrase "of the highest metrological quality". He proposed the addition of a fourth explanatory note to the definition, as follows.

> *"The condition 'highest metrological quality' need not be applied when establishing whether a method provides traceability to the SI. It serves to emphasise the role of primary methods of the 'highest*

metrological quality' in achieving the smallest possible uncertainty as well as traceability to the SI."

Dr Taylor commented that this additional note did not address all of the criticisms of the current definition outlined in his discussion paper CCQM99-02. He suggested that the following revised version of the definition would be more consistent with the current definitions of "measurement" and "method of measurement" found in the *International Vocabulary of Basic and General Terms in Metrology* (VIM). (Proposed changes are indicated in boldface).

"A primary method of measurement ***in the SI*** *is a method having the highest metrological qualities whose* ***model (mathematical equation) and realization*** *are completely described and understood in terms of SI units.*

The use of *a primary direct method results in a value of an unknown quantity without reference to a standard of the same quantity.*

The use of *a primary ratio method results in a value of the ratio of two values of the same quantity without reference to a standard of the same quantity.*

In both cases, the results must be accompanied by a complete uncertainty statement."

Dr Quinn pointed out that the term "highest metrological quality" is wording taken from the VIM definition of "primary standard" but its meaning is not elaborated in that document. No consensus was reached on any modifications to the definition of a primary method. After much discussion, it was agreed that it may be very difficult to arrive at a definition which will be readily understood by the broader analytical chemistry community. In this regard, it was Dr Hässelbarth's opinion that some of the issues which arose in the discussion might be better addressed by the publication of one or more articles on the subject in *Metrologia* rather than further modification of the definition.

Dr Pan reviewed the changes that had been made to the working document on coulometry (CCQM99-16) introduced at an earlier CCQM meeting. Dr Koustikov expressed some concern that the Russian sample of $K_2Cr_2O_7$ for which results were compared in Table 2 of this document was not suitable for this purpose.

Dr Zhao briefly summarized her report (CCQM99-19) on the application of the melting point depression method for the CCQM-6 exercise on the determination of the purity of several organic compounds.

11 BIPM PROGRAMME OF METROLOGY IN CHEMISTRY

Dr Quinn briefly reviewed the history of the development of a plan to establish a programme of metrology in chemistry at the BIPM which had led to a decision in the autumn of 1998 to establish a programme in the area of gas analysis. Dr Davis reviewed the major steps leading up to the planned initiation of scientific work by the end of 2000. A small working party will convene in March 1999 to assist the BIPM with the development of a detailed technical programme. Refurbishment of facilities is to begin in the autumn of 1999. The recruitment of a section head and up to three additional staff will commence in the spring of 2000.

Dr Hässelbarth mentioned the international gas analysis symposium that will be arranged by the ISO/TC 158 in November 1999 in The Netherlands, and invited BIPM staff members to participate. In view of the general interest, Dr Davis agreed to arrange for the distribution of the annoucement to CCQM members.

12 OTHER BUSINESS

Dr Kaarls drew the attention of CCQM members to a meeting of representatives of interested NMIs on viscosimetry on 14-15 September 1999. He also indicated that he would work with the BIPM to organize a meeting at the BIPM which would combine a workshop on uncertainty calculations with CCQM working group meetings.

Dr King made a brief presentation on his paper CCQM99-23, which was a summary of a recent survey of current activities and future requirements for metrology in chemistry in Europe.

Dr De Bièvre reminded the members that revision of the VIM is ongoing. He drew their attention to his paper CCQM99-21, intended for discussion at the next CCQM meeting**,** on the possible need for refinement of the definition of "traceability" in the VIM.

13 DATE OF NEXT MEETING

It was agreed that the next meeting of the CCQM will take place during the week of 10-14 April 2000.

J. McLaren, Rapporteur

March 1999

Table 1. A framework for CCQM comparisons and studies

(classified by field)

Description	Reference number	Pilot lab.	Date beg.	Old number
Health				
Clinical diagnostic markers				
Cholesterol in serum	CCQM-P6	NIST	1998	CCQM-7
	CCQM-K6	NIST	1999	
Glucose in serum	CCQM-P8	NIST	1999	
Creatinine in serum	CCQM-P9	NIST	1999	
Major electrolyte elements (Na, K, Ca) in serum and urine				
Trace elements (Pb, Se) in serum/urine/blood	CCQM-P14	NIST/ LGC	1999	
Anabolic steroids in urine				
Hormones in serum				
Food				
Pesticide residues				
pp´-DDE in isooctane	CCQM-P2	LGC	1997	CCQM-3
pp´-DDE in corn oil	CCQM-P4	LGC	1998	CCQM-5
pp´-DDE in cod liver oil	CCQM-K5	LGC	1999	
Hexachlorocyclohexane and a trade-related pesticide in cod liver oil	CCQM-P10	LGC	1999	
Toxins in food				
As in fish or shellfish	CCQM-P11	NIST	1999	
Pb in wine	CCQM-P12	IRMM	1999	
Cd in rice				
Metals in synthetic food digest	CCQM-P13	LGC	1999	
Antibiotics in meat				
Growth hormones in meat				
Vitamins and minerals				

Drinking water

Organics (EPA list)

Trace elements

Microbiological

Environment

Water

Waste water (EPA list)

Cd and Pb in natural water	CCQM-K2	IRMM	1998	CCQM-9

Air EPA HAPs list and ozone

Global Warming Gases

CO_2, CH_4 - ambient levels

SF_6, CFCs - emission levels

Ozone - ambient levels

Point source emissions CO, CO_2, THC, NO_x, SO_2,…,VOCs

Primary standard gas mixtures

CO in N_2	CCQM-K1a	NMi	1998	CCQM-2
CO_2 in N_2	CCQM-K1b	NMi	1998	CCQM-2
NO in N_2	CCQM-K1c	NMi	1998	CCQM-2
SO_2 in N_2	CCQM-K1d	NMi	1998	CCQM-2
Natural gases	CCQM-K1e,f,g	NMi	1998	CCQM-2
CO, CO_2, propane in N_2	CCQM-K3	NMi	1998	CCQM-10
Benzene/toluene/xylene in N_2/Air	CCQM-K7	NIST	1999	
SO_2, NO_2 air quality gases				

Contaminants in soils/sediments/incinerator ash

Elements in sediments	CCQM-P15	IRMM	1999	
Elements in synthetic digest solutions	CCQM-P16	NMi	1999	
PCBs in sediments	CCQM-P17	NRC	1999	
Organometallics in sediment	CCQM-P18	NRC	1999	

Metals in hard rock mine wastes

Metals in biological tissues

Toxic metals in recycled plastics PET

Advanced Materials

Semiconductors

Ultratrace metals in high-purity semiconductors GaAs

Metal Alloys

Pb in Al alloys

Polymers and Plastics

Leachates
Trace metals

Catalysts

Pt, Rh in vehicle exhaust catalysts

Commodities

Industrial SO_2 in stack emissions	see CCQM-K1d under ***Environment***			
Sulfur and moisture in fossil fuels				
Metals in lubricating oils				
Natural gases	see CCQM-K1e,f,g under ***Environment***			
Sucrose				
Cement Ca, Si, Al, S, Ti, Na, Mg				
Ore composition				
Rare-earth elements				
Precious metals				
Source of origin/adulteration				
Honey				
Alchohol content				
Ethanol in water				

Forensics

Drugs of abuse				
Explosives residues				
Ethanol in air breathalyzers	CCQM-K4	NPL	1999	CCQM-11

DNA profiling

Pharmaceuticals

Biotechnology

DNA profiling
DNA diagnostics

General analytical applications

Purity of materials metals, salts, organics, etc.

KCl, NaCl, $K_2Cr_2O_7$	CCQM-P7	NIST	1998	CCQM-8
Hydrochloric acid	CCQM-P19	NIST	1999	
Acetanilide, benzoic acid and naphthalene	CCQM-P5	NIST	1998	CCQM-6
Cholesterol, creatinine, pp´-DDE, organometallics, xylene	CCQM-P20	NIST/ NARL	1999	
NMR study	CCQM-P3	BAM	1998	CCQM-4

Calibration solutions

Trace elements in water Pb	CCQM-P1	NIST	1997	CCQM-1
Elemental solution standards	CCQM-K8	EMPA/ BNM-LNE	1999	
pH Standards	CCQM-K9	PTB	1999	

Isotopic standards

Table 2. CCQM key comparisons and studies

Old number	New number P	K	S	Description
CCQM-1	CCQM-P1			Trace element (Pb) in water
CCQM-2		CCQM-K1a		CO in N_2
		CCQM-K1b		CO_2 in N_2
		CCQM-K1c		NO in N_2
		CCQM-K1d		SO_2 in N_2
		CCQM-K1e		Natural gas I
		CCQM-K1f		Natural gas II
		CCQM-K1g		Natural gas III
CCQM-3	CCQM-P2			pp'-DDE in isooctane
CCQM-4	CCQM-P3			NMR study
CCQM-5	CCQM-P4			pp'-DDE in corn oil
CCQM-6	CCQM-P5			Purity of acetanilide, benzoic acid, naphthelene
CCQM-7	CCQM-P6			Cholesterol in serum
CCQM-8	CCQM-P7			Purity of KCl, NaCl, $K_2Cr_2O_7$
CCQM-9		CCQM-K2		Cd and Pb in natural water
CCQM-10		CCQM-K3		CO, CO_2, propane in N_2
CCQM-11		CCQM-K4		Ethanol in air
		CCQM-K5		pp'-DDE in cod liver oil
		CCQM-K6		Cholesterol in serum
		CCQM-K7		BTX in N_2, air
		CCQM-K8		Elemental solution standards
		CCQM-K9		pH standards
	CCQM-P8			Glucose in serum
	CCQM-P9			Creatinine in serum
	CCQM-P10			Hexachlorocyclohexane (etc.) in cod liver oil
	CCQM-P11			As in shellfish
	CCQM-P12			Pb in wine
	CCQM-P13			Metals in synthetic food digest
	CCQM-P14			Trace elements (Pb, Se) in serum/urine/blood
	CCQM-P15			Elements in sediments
	CCQM-P16			Elements in synthetic digest solutions

Old number	New number P	K	S	Description
	CCQM-P17			PCBs in sediments
	CCQM-P18			Organo-metallics in sediment
	CCQM-P19			Assay of HCl
	CCQM-P20			Purity of: cholesterol, creatinine, pp'-DDE, organometallics, xylene

Column 1 gives the former designation of the comparison, column 2 gives the new designation of “pilot studies”, column 3 gives the new designation of “key comparisons” and column 4 gives the new designation of “supplementary comparisons” (of which there are currently none). The comparisons are briefly described in column 5.

APPENDIX Q 1.
Working documents submitted to the CCQM at its 5th meeting

(see the list of documents on page 44)

LIST OF ACRONYMS USED IN THE PRESENT VOLUME

1 Acronyms for laboratories, committees and conferences

BAM	Bundesanstalt für Materialforschung und -prüfung, Berlin (Germany)
BIPM	Bureau International des Poids et Mesures
BNM	Bureau National de Métrologie, Paris (France)
BNM-LNE	Bureau National de Métrologie: Laboratoire National d'Essais, Orsay and Paris (France)
CCQM	Consultative Committee for Amount of Substance
CCU	Consultative Committee for Units
CENAM	Centro Nacional de Metrologia, Mexico (Mexico)
CGPM	Conférence Générale des Poids et Mesures
CIPM	Comité International des Poids et Mesures
CITAC	Cooperation on International Traceability in Analytical Chemistry
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, Dübendorf, St Gall and Thun (Switzerland)
EPA	Environmental Protection Agency, Washington DC (United States)
EU	European Union
EUROMET	European Collaboration in Measurement Standards
GUM	Glówny Urzad Miar/ Central Office of Measures, Warsaw (Poland)
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)

IPT	Instituto de Pesquisas Tecnologicas, São Paulo (Brazil)
IRMM	Institute for Reference Materials and Measurements, European Commission
ISO	International Organization for Standardization
ISO/REMCO	International Organization for Standardization: Committee on reference materials
ISO/TC 158	International Organization for Standardization: Technical committee on gas analysis
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, Taejon (Rep. of Korea)
LGC	Laboratory of the Government Chemist, Teddington (United Kingdom)
LNE	Laboratoire National d'Essais, Orsay and Paris (France), see BNM
NARL	National Analytical Reference Laboratory, Canberra and Pymble (Australia)
NIM	National Institute of Metrology, Beijing (China)
NIMC	National Institute of Material and Chemical Research, Tsukuba (Japan)
NIST	National Institute of Standards and Technology, Gaithersburg (United States)
NMI	National Metrology Institute
NMi-VSL	Nederlands Meetinstituut: Van Swinden Laboratorium, Delft (The Netherlands)
NPL	National Physical Laboratory, Teddington (United Kingdom)
NRC	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)
OFMET	Office Fédéral de Métrologie, Wabern (Switzerland)
OMH	Országos Mérésügyi Hivatal/National Office of Measures, Budapest (Hungary)
PTB	Physikalisch-Technische Bundesanstalt, Braunschweig and Berlin (Germany)

RMO	Regional Metrology Organization
SP	Sveriges Provnings- och Forskningsinstitut/Swedish National Testing and Research Institute, Borås (Sweden)
SMU	Slovenský Metrologický Ústav, Bratislava (Slovakia)
VNIIM	D.I. Mendeleyev Institute for Metrology, St Petersburg (Russian Fed.)
VSL	Van Swinden Laboratorium, Delft (The Netherlands), see NMi

2 Acronyms for scientific terms

BTX	Benzene, Toluene, Xylene
CFC	Chlorofluorocarbon
CRM	Certified Reference Materials
DNA	Deoxyribonucleic Acid
DSC	Differential Scanning Calorimetry
GC	Gas Chromatography
HAP	Hazardous Air Pollutant
HPLC	High Performance Liquid Chromatography
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
IDMS	Isotope Dilution Mass Spectrometry
KC	Key Comparison
KCRV	Key Comparison Reference Value
MS	Mass Spectrometry
NAA	Neutron Activation Analysis
NMR	Nuclear Magnetic Resonance
PCB	Polychlorinated Biphenyl
PET	Polyethylene Tetraphtalate
SI	International System of Units
SRM	Standard Reference Material
THC	Total Hydrocarbon
VOC	Volatile Organic Compound