Bureau International des Poids et Mesures

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

Report of the 9th meeting
(10–11 April 2003)
to the International Committee for Weights and Measures

Comité international des poids et mesures

Bureau international des poids et mesures

Organisation intergouvernementale de la Convention du Mètre

Note:

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

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

Working documents for the meetings are listed at the end of each Report and those which the Consultative Committee decides are for public use are available also on the website.

T.J.Quinn,
Director BIPM,
November 2003.

LIST OF MEMBERS OF THE CONSULTATIVE COMMITTEE FOR AMOUNT OF SUBSTANCE: metrology in chemistry

as of 10 April 2003

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

Bundesanstalt für Materialforschung und -prüfung [BAM]/Federal Institute for Materials Research and Testing, Berlin.

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], Daejeon.

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

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

National Measurement Laboratory CSIRO [NML CSIRO], Lindfield/ National Analytical Reference Laboratory – Australian Government Analytical Laboratories [NARL-AGAL], Pymble.

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.

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

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

State Laboratory [SL], Dublin.

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

Swiss Federal Office of Metrology and Accreditation [METAS], Wabern/Swiss Federal Laboratories for Materials Testing and Research [EMPA], St Gall.

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

Observers

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

Centro Español de Metrología [CEM], Madrid.

Centro Nacional de Metrología [CENAM], Mexico.

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

Istituto di Metrologia G. Colonnetti, Consiglio Nazionale delle Ricerche [IMGC-CNR], Turin.

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

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

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

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

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

The Consultative Committee for Amount of Substance: metrology in chemistry (CCQM) held its 9th meeting at the International Bureau of Weights of Measures (BIPM), at Sèvres on 10-11 April 2003.

The following were present: L. Besley (NML CSIRO), P. Charlet (BNM-LNE), K. Chiba (NMIJ/AIST), P. De Bièvre (IRMM/ISO-REMCO), E.W.B. de Leer (NMi VSL), R. Dybkaer (IFCC), H. Emons (IRMM), H. Ent (NMi VSL), A. Fajgelj (IAEA), G.L. Gilliland (NIST), B. Güttler (PTB), H.-P. Haerri (METAS), Qiao HAN (NRCCRM), W. Hässelbarth (BAM), R. Hearn (LGC), H.D. Jensen (DFM), R. Kaarls (President of the CCQM), Y. Kustikov (VNIIM), L. Mackay (NARL-AGAL), B. Magnusson (SP), M. Máriássy (SMU), A. Marschal (BNM-LNE), R. Matschat (BAM), W.E. May (NIST), J. McLaren (NRC), M.J.T. Milton (NPL), K. Okamoto (NMIJ/AIST), H. Parkes (LGC), T.J. Quinn (Director of the BIPM), W. Richter (PTB), M. Sargent (LGC), M. Seah (NPL), Hun-Young So (KRISS), R. Sturgeon (NRC), P. Taylor (IRMM/IUPAC), J. Thijssen (University Medical Centre Utrecht), M.C. Walsh (SL), M. Weber (EMPA), P. Woods (NPL), Yadong YU (NRCCRM).

Observers: I. Akdag (UME), M. del Rocio Arvizu-Torres (CENAM), E. Deák (OMH), M. Gallorini (IMGC-CNR), W. Kozlowski (GUM), K. Lal (NPLI), M.T. López Esteban (CEM), W. Louw (CSIR-NML), Y. Mitani (CENAM), M. Pérez-Urquiza (CENAM).

Invited: V. de Souza (INMETRO), M.A. Getrouw (INMETRO), A. Padilla (WHO), V.M.L. Ponçano (IPT), A. Squirrell (NATA).

Also present: P. Giacomo (Director Emeritus of the BIPM); M. Esler, S. Maniguet, J. Viallon, R. Wielgosz (BIPM).

Excused: T. Catterick (LGC), S. Hart (NARL-AGAL), H.G. Semerjian (NIST).

Absent: NIM.

The President, Dr Kaarls, welcomed participants to the 9th meeting of the CCQM, noting that this was the tenth anniversary of its creation. The Director welcomed the CCQM to the BIPM.

The agenda was approved with a slight re-ordering of the items. The President thanked Dr Milton for preparing the report of the eighth meeting. He proposed Dr Milton as rapporteur for the meeting, to be assisted by Dr Wielgosz. The proposition was accepted.

2 REPORT OF THE EIGHTH MEETING

The report of the eighth meeting was approved. It was noted that the second paragraph on page 68 should have referred to work being carried out on APMP.QM-K3 (vehicle emissions) and not APMP.QM-K4 (ethanol in air); and that the description of CCQM-P40 on page 65 should have referred to the measurement of organic contaminants in mussel tissue and not muscle tissue.

3 REPORTS OF WORKING GROUPS

3.1 Surface analysis

Dr Seah provided the detailed background to his written report (CCQM/03-08) on the progress of pilot study CCQM-P38 (silicon dioxide on silicon). The NPL was acting as the pilot laboratory. There were participants from twelve countries including twenty-six laboratories, and nine different measurement techniques had been used.

He said that the results of the study had led to a reassessment of the performance of the different techniques. It had emerged that XPS could be the pre-eminent method when proper procedures are applied (which required data traceable to other methods), and that TEM had not performed as well as had been expected. Ellipsometry had been used to map the relative thickness of all of the wafers, and highly homogenous regions selected to form the samples. Previous inter-laboratory studies had not used this procedure. Substrates with (100) and (111) orientation effectively behaved the same for all of the methods. The samples distributed for the pilot study had nominal thicknesses of SiO_2 on the Si substrates in the range 2 nm to 8 nm. All samples had a contamination layer of approximately 1 nm consisting of carbonaceous material and water. The results submitted by laboratories were assessed against NPL data, resulting in a least squares fitted straight line, where the gradient is a relative scaling factor and the intercept is the zero-thickness offset.

The XPS results from those laboratories using an NPL supplied reference geometry and reference input values for material constants gave results that deviated least from the NPL data. XPS results for the analysts' original choices showed a spread which was five times greater for the relative scaling factor and four times greater for the zero-thickness offset. Results for ellipsometry showed a very low spread in value for the scaling constant but zero offset values approaching 1 nm due to the contamination layer. A result from the SIMS performed poorly because of its intrinsic non-linearity. Results from TEM have limited accuracy because of the non-analytical contrast of the images at the interface. The RBS, NRA, GIXRR and MEIS all showed offsets of approximately 0.5 nm arising from the oxygen in the contamination layer which they do not distinguish from the oxygen in SiO_2. These offsets had not been quantified previously.

Dr Seah explained that it would be possible to proceed to a key comparison based on measurements from fresh samples from different wafers made from the batches of material prepared for the pilot study. Ten laboratories had indicated their interest in participation. The meeting agreed that the work should proceed to a key comparison (CCQM-K32).

The President thanked Dr Seah for his presentation which was in a very special area of chemical analysis, of great interest to industry. Dr Marschal observed that this study was not a classical comparison of national standards, but an approach that harmonized measurements globally. Dr Seah agreed and said that distributing reference materials had not proven to be a useful approach for harmonizing measurements for these very thin surface layers.

The Director highlighted the possible importance of this work for the attempts to redetermine the Avogadro constant. Dr Güttler confirmed that the PTB were planning to grow a thermal oxide on their silicon artefacts in the future. Dr Seah noted that oxide growth on the artefacts would be dependant on surface orientation, and that the artefact was multifaceted.

Dr Seah also presented his report on progress with the establishment of a working group (CCQM/03-17). He said that surface analysis work was placed variously in materials, chemistry, electronics or physics departments in the national metrology institutes (NMIs). The draft terms of reference discussed at the last meeting of the CCQM had been refined by the working group. Proposals for more than a dozen different studies had been discussed and possible pilot laboratories from five countries identified.

The President commented that the work plan was impressive and of growing interest to the NMIs.

Dr May asked whether the proposed key comparison (KC) would be method specific? Dr Seah confirmed that it would measure the thickness of SiO_2 on Si.

The President agreed that ultimately this KC would underpin NMIs' capabilities to make this type of measurement. He also asked the meeting to approve the recommendation to the CIPM that it ratify the group as a full working group of the CCQM. This was agreed.

3.2 Organic analysis

Dr May presented his report of progress made by the Working Group on Organic Analysis. The group had met twice in the previous year and had also developed interactions with the Working Group on Inorganic Analysis in the area of TBT analysis, with the Working Group on Gas Analysis on the purity of organics and the Working Group on Bio-Analysis on the characteristics of DNA. A report [CCQM/03-26] was available.

He noted that the reports for three key comparisons, which had been approved for equivalence after circulation to working group chairmen, (CCQM-K11, CCQM-K12 and CCQM-K21) had been submitted for publication and proceeded to report in detail on the results of three other key comparisons:

- CCQM-K6-subsequent (cholesterol in serum). This "subsequent" key comparison was organized in order to allow two laboratories that had not participated in CCQM-K6 to develop degrees of equivalence for this measurand. The NIST had participated in both phases of

CCQM-K6 and acted as the reference result to link the two. The comparison was approved for equivalence by the CCQM.

- CCQM-K25 (PCBs in sediment). Five PCB congeners had been selected as representative of the total number of approximately 150 found in typical environmental samples. The key comparison reference value (KCRV) was based on the results from laboratories carrying out their analysis by GC-MS with a ^{13}C labelled internal standard. Agreement was good within the range of claimed uncertainties of 1.6 % to 5 %. The results of this key comparison would be used to assess laboratories' capabilities to measure PCB congeners in samples of sediment. The report of the comparison was in draft B stage at the time of the meeting.
- CCQM-K27.a (ethanol in aqueous matrices). This key comparison used two spiked samples that were representative of typical forensic samples. The working group proposed to use the gravimetric value as the KCRV for these samples, whereas the standard uncertainty of the KCRV would be taken as the standard deviation of the mean of the results. A discussion of this proposal ensued during which the appropriateness of dealing with the determination of the KCRV and its uncertainty in this way was questioned. CCQM-K27.b used a commercial red wine as the material, representative of ethanol in aqueous solution at a level present in a traded commodity. The working group proposed to use a weighted mean of the results as the value of the KCRV for measurements on this material. No major differences were observed in the results from each laboratory between the two types of sample. The reports of both comparisons were in draft B stage at the time of the meeting. A subsequent comparison for CCQM-K27.a is planned, with the NIST as the pilot laboratory, and will be run in parallel to a SIM pilot study.

Dr May displayed the results of these key comparisons using a new type of graph developed by the group to represent the possible distribution of uncertainties of the KCRV. He also described the results of CCQM-P20.b (ortho-xylene purity) which showed good comparability for the purity of a small sample of approximately 98 % purity expressed as a mass fraction. Eleven out of the twelve participants had used GC-FID-based methods, and several laboratories had also used more complex methods (including DSC and NMR).

The group had reviewed a wide range of possible comparison studies and Dr May sought the meeting's approval for the following comparisons and studies:

- CCQM-K37 (volatile organic compounds in organic solvents), to be piloted by the KRISS or NIST;
- CCQM-P20.c (purity of atrazine), piloted by the NARL;
- CCQM-P20.d (purity of chlorpyrifos), piloted by the NARL;
- CCQM-P31 (organic solutions), piloted by the NIST to include:
  - CCQM-P31.a – five target PAHs amongst thirty others in toluene solution,
  - CCQM-P31.b – five target PCBs amongst twelve others in i-octane solution,
  - CCQM-P31.c – four target chlorinated–pesticides amongst ten others in i-octane solution.

The CCQM approved these comparisons and studies.

Additionally, the group was making good progress with an exercise agreed at previous meetings of the CCQM, CCQM-P40 (organic contamination in mussel tissue). He also described how the

Working Group on Organic Analysis could support Working Group 1 of the Joint Committee on Traceability in Laboratory Medicine (JCTLM) in their work of evaluating the comparability of available reference materials nominated as reference materials of higher order for clinical measurements.

The President thanked Dr May for his very comprehensive presentation. In answer to some discussion, the Director confirmed that the practice of calculating the KCRV and unilateral, but not bilateral, degrees of equivalence for a key comparison was acceptable and permitted by the CIPM Mutual Recognition Arrangement (MRA).

3.3 Inorganic analysis

Dr Sargent presented his report of the work of the Working Group on Inorganic Analysis which had met twice since the last CCQM.

He presented the results of three key comparisons, which the CCQM approved for equivalence:

- CCQM-K13-subsequent (Cd and Pb in sediment). This comparison was held subsequently to the first round of CCQM-K13 in order to develop degrees of equivalence for the CENAM (for Cd) and the PTB (for Pb). In both cases, results from the NIST were used to provide a link to the first round. The exercise was based on a sample from Chesapeake Bay which would be certified as NIST SRM 2702.
- CCQM-K14 (Ca in serum, at a clinically-relevant level). The pilot laboratory was the IRMM which had used the same samples as for the global IMEP-17 exercise.
- CCQM-K31 (As in shellfish). The comparison was based on a natural matrix sample which was blended from samples used in CCQM-P11. The NIST had acted as pilot laboratory.

He proposed three new key comparisons, which were agreed by the CCQM:

- CCQM-K33 (Cr, Mo, Mn and Ni in steel). This would follow on from CCQM-P25 which had included ten NMIs as well as fourteen industrial laboratories. There had been no significant difference between the results from the NMIs and the industrial laboratories. The BAM, the NMIJ and the NIST would act as pilot laboratories.
- CCQM-K34 (assay of KHP) which would follow on from CCQM-P36. The pilot laboratory would be the SMU.
- CCQM-K35 (sulphur in fuel) that would follow on from CCQM-P26, which had yielded inconclusive results. This key comparison was required as the basis for the review of proposed calibration and measurement capabilities (CMCs) in this area. It would be organized in parallel with a pilot study (CCQM-P26.1) using the same samples. The NIST was planning to act as the pilot laboratory.

Dr Sargent reported that three key comparisons were in progress (CCQM-K28 (TBT in sediment), CCQM-K29 (anions in solution) and CCQM-K30 (Pb in wine)). The working group had reviewed the results from five pilot studies:

- CCQM-P13 (metals in food digest),
- CCQM-P19.1 (purity of HCl),

- CCQM-P34 (constituents in Al alloy),
- CCQM-P25 (minor elements in steel),
- CCQM-P36 (assay of potassium hydrogen phthalate, KHP).

Three other pilot studies were in progress:

- CCQM-P12.1 (elements in wine (Cu, Cd, Zn)),
- CCQM-P39 (Hg, Pb, organo-Hg in tuna fish),
- CCQM-P43 (DBT in sediment).

Four pilot studies were in preparation:

- CCQM-P26.1 (sulphur in fuel),
- CCQM-P33 (boron in Si),
- CCQM-P46 (preparation of inorganic solutions),
- CCQM-P48 (U isotopic signature in salty matrix).

The working group had also discussed a range of issues, in particular the assignment of a KCRV and its uncertainty. They had also supported the process of reviewing CMCs [CCQM/03-10] where difficulties had arisen and had worked with the JCTLM in its work on clinical chemistry.

Dr Sargent thanked all of the participants for their contribution to the working group and the ten laboratories that had carried out pilot laboratory activities during the year.

The President thanked Dr Sargent for his report.

3.4 Gas analysis

Dr de Leer presented his report on the work of the Working Group on Gas Analysis which had met twice since the last CCQM. The group involved twenty-four laboratories from twenty countries. He described progress with a number of key comparisons:

- CCQM-K15 (CF_4 and SF_6 at emission levels) was being piloted by the KRISS. This key comparison would involve four participants and two other laboratories would participate on the basis of a pilot study numbered CCQM-P51. The samples would be distributed in June 2003.
- CCQM-K16.a (natural gas type IV) and CCQM-K16.b (natural gas type V) had been completed and the draft B report approved by the working group. It had shown good comparability with most results lying within 1 % of the KCRV. There had also been one participant making use of a calorimeter to measure the calorific value of the gas directly. Two laboratories had participated with the same samples on the basis of a pilot study (subsequently numbered CCQM-P49.a and CCQM-P49.b).
- CCQM-K22 (eight hazardous air pollutants – chlorinated VOCs). This key comparison was being piloted by the NMIJ with the CERI. There would be six participants and one laboratory would submit results informally on the basis of a pilot study. After a feasibility study, it had been decided to omit acrylonitrile from the standards to be used for the key comparison.

- CCQM-K26.a and CCQM-K26.b (NO and SO_2 at ambient levels). This key comparison would make use of stable gas standards that do not have a reliable value for their amount fraction based on their gravimetric preparation. The KCRV would be established by labelling them with high-accuracy dynamic methods. It was proposed to carry out a pilot study (CCQM-P50.a and CCQM-P50.b) in parallel based on the same standards.

Dr de Leer also gave an outline of progress with a number of pilot studies:

- CCQM-P23 (study of gravimetry based on CO in N_2). This pilot study was undertaken in order to investigate the uncertainty of the gravimetric values associated with gas standards which have an uncertainty an order of magnitude smaller than is achievable by analysis. The analysis carried out by the pilot laboratory had been biased in certain cases because of the sensitivity of the NDIR analyser they had used to the $^{13}C/^{12}C$ ratio of four of the samples in the study. However, the study demonstrated that commercially available carbon monoxide could be isotopically depleted. He concluded that a more thorough testing of the protocol might have foreseen these difficulties.
- CCQM-P24 (dynamic mixing methods). The pilot study proposed by the BNM-LNE would now take the form of a bilateral comparison between the BNM-LNE and BAM.
- CCQM-P28 (ozone, ambient level). The BIPM was the pilot laboratory for this comparison, for which the protocol had been completed and for which measurements were to start later in the year.

He summarized that CCQM-P41 (greenhouse gases) was underway, whereas the protocol for CCQM-P42 (hydrogen sulphide) was under development. The protocol for CCQM-P45 (purity analysis of gases) would be developed with input from the outcome of a EUROMET workshop to be organized by the BNM-LNE.

The working group was also planning a range of future activities, including: a pilot study to investigate the accuracy with which gas standards are prepared from liquid samples and key comparisons of noble gases, ammonia and non-methane hydrocarbons. The working group had also started to consider how the operation of key comparisons could be improved. Dr de Leer observed that the working group had sometimes experienced difficulty in making a clear statement of "how far the light shines" from each comparison.

Dr de Leer reviewed gas analysis activities in the regional metrology organizations (RMOs). The APMP had completed APMP.QM-K3 (automotive emission gases) and APMP.QM-K4 (ethanol in air). The final reports were in preparation and mechanisms were being developed to link the results to those of the corresponding CCQM key comparisons. The EUROMET had completed EUROMET.QM-K1.c (NO in N_2) and a report was in preparation.

The President thanked Dr de Leer for his report. He agreed with the practice of permitting less experienced laboratories to participate in pilot studies run in parallel with a key comparison. He also confirmed that a clear statement about "how far the light shines" from each comparison was required in each final report. Dr Besley observed that such information could be misunderstood, but Dr May felt that it provided a useful context for the application of the work.

A number of participants explained that they had encountered difficulties with import and export of samples. The President said that this was a perennial problem that might be improved through a coordinated initiative by international organizations.

3.5 Electrochemical analysis

Dr Máriássy presented a report of the work of the Working Group on Electrochemical Analysis, which had met twice since the last CCQM and had also held joint meetings with the Working Group on Inorganic Analysis. He presented the final report (CCQM/03-07) of CCQM-K17 (pH of phthalate buffer) and the CCQM approved it for equivalence.

He described the results and progress of agreed studies and comparisons:

- CCQM-P37 (fundamental studies of pH standards) was aimed at identifying the sources of uncertainty giving rise to the dispersion in the results of CCQM-K9 and CCQM-K17. It had yielded useful insight into the operation of Harned cells. Specific problems had been identified, including: the use of incorrect HCl molality, incomplete saturation of the solution with hydrogen, hydrogen electrode poisoning and concentration changes during filling or due to evaporation or condensation of water. He expected an improvement in future comparisons.
- The pilot study CCQM-P19.1 (purity of HCl) had been organized together with the Working Group on Inorganic Analysis. It had shown much better agreement between participants than the original study (CCQM-P19), which had been compromised by some instability of the samples. Results from acidimetric coulometry showed excellent comparability for the low-concentration sample.
- The pilot study CCQM-P47 (electrolytic conductivity) on lower level conductivity standards (~0.005 S/m and 0.05 S/m) was in its preparation stage and samples would be distributed in September 2003.
- Samples would be distributed for CCQM-K18 (pH of carbonate buffer) in September 2003. As there were some additional measurement problems with this system, it was planned to hold a study in parallel (CCQM-P52) for those laboratories not ready to participate in a key comparison.

He proposed two new key comparisons that were approved by the CCQM:

- CCQM-K34 key comparison (held in cooperation with the Working Group on Inorganic Analysis) to follow CCQM-P36 (assay of potassium hydrogen phthalate). This was expected to start at the end of 2003 and would be piloted by the SMU.
- CCQM-K36.a and CCQM-K36.b key comparisons to assess the measurement capabilities at both higher and lower conductivity levels, that would start after the end of CCQM-P47.

Dr Máriássy informed the meeting about the revision of OIML R56 (standard solutions reproducing the conductivity of electrolytes). It had been decided that there would be no change, but that a revision should be discussed again in five years to accommodate developments in metrology. He reported that a problem had been encountered with the European Pharmacopoeia which included incorrect values for the conductivity of standard solutions. The issue had been raised with the European Pharmacopoeia Commission, but a response had not been received.

He also highlighted an increasing trend with measuring instruments; most models on the market allowed the user to specify the property value of the reference material in use, but some models only used automatic standard recognition. This approach was of concern because it could introduce errors into the calibration step resulting from the inherent batch-to-batch variability of reference materials. The President recommended that the NMIs should enter into contact with the producers of such instrumentation in their own countries.

The President thanked Dr Máriássy for his report.

3.6 Bio-analysis

Dr Gilliland and Mrs Parkes presented their report of the work of the Working Group on Bio-analysis. The group had held a "Thinkshop" at the IRMM in April 2003 with forty-two participants from twenty-one countries including academics, industrialists and representatives of international organizations. It had also involved the NMIs and nominated expert laboratories. The meeting had considered three themes: genes, proteins and cells. Various measurement issues had arisen, particularly concerning the identification of appropriate measurands and the requirement to achieve comparable measurement results between different measurement technologies. An interesting contribution had been made by the NIBSC that compared the requirements of ISO 17025 with the requirements laid down in guidelines from the World Health Organization (WHO). Finally, the meeting had identified several examples where traceability to the SI could be achieved for bio-measurements.

Dr Gilliland reported on progress with CCQM-P44 which attempted to confirm whether the quantitation of a DNA sequence was independent of the method and equipment used. The study was making use of technology developed by the LGC and the NIST to insert an oligonucleide into plasmid DNA that had been expressed in *E.coli.* Procedures were being developed for use in different commercial assay methods. A blank plasmid had been developed to define a background level for the measurements. The optimum concentration of the reagents required for the reaction had been identified. The proposed protocol would use a six-point standard curve based on ten-fold dilutions. Materials would be distributed by August 2003 and the data would be presented to the working group meeting in November 2003.

The chairmen explained that the group proposed to carry out pilot studies in the following areas:

- CCQM-P53 (DNA profiling by AFLP). This proposal had been developed by the NARL and would involve amplified fragment-length polymorphism (AFLP) which was a widely used method for profiling "unknown" DNA.
- CCQM-P54 (DNA primary quantification). This pilot study would develop an IDMS method for determining the amount of DNA. It would be piloted by the LGC and organized in collaboration with the Working Group on Organic Analysis.
- Fluorescence spectral correction. Fluorescence is one of the most important methods for the detection of biological samples and a key comparison was needed in the area. The NIST, the BAM and the NPL had agreed to develop a proposal for this activity which would be very relevant to the important area of micro-arrays.

- Circular-dichroism spectroscopy of protein solutions. This proposal from the NPL addressed an important method used to determine secondary and tertiary structure to meet regulatory requirements. This activity would also be relevant to the development of protein-activity relationships.
- CCQM-P55 (peptide/protein quantification). This proposed study would involve a protein/peptide admixture for calibration of mass spectrometers and would be developed by the LGC, the NIST and the PTB.

The President thanked the chairmen for their report and said it was important to develop a series of pilot studies in the area of bio-analysis.

Dr May said it was important to clarify the differences between the work of the Working Group on Bio-Analysis and other collaborations between the NMIs. Dr Thijssen said that the IFCC had dedicated working groups in many areas of interest to the Working Group on Bio-analysis and there should be greater collaboration. Dr Padilla reported that the WHO had started discussions in 2000 amongst the international professional societies in order to make progress towards the use of SI units.

Dr Gilliland informed the CCQM that he would step down from his role of co-chair of the working group because of changes in his duties at the NIST. The President thanked him for his strong contribution and expressed his thanks to Dr Vincent Vilker, also from the NIST, who would now be vice-chair of the group.

3.7 Key comparisons

The President informed the CCQM that Dr Hratch Semerjian wished to resign from the chairman of the Working Group on Key Comparisons. He thanked him for his very valuable contribution to the work of the CCQM over a period of many years.

A full list of CCQM key comparisons and pilot studies indicating their status as of April 2003 is included as Table 1 at the end of this report.

4 MEMORANDUM OF UNDERSTANDING BETWEEN THE WORLD HEALTH ORGANIZATION AND THE CIPM

Dr Quinn informed the meeting that the CIPM had recently agreed a Memorandum of Understanding with the World Health Organization (CCQM/03-16). He said that it had a similar format to that agreed previously with the World Meteorological Organization (WMO). Dr Padilla from the WHO said that it was an important agreement that confirmed a commitment from the two organizations to work together. She also looked forward to a greater involvement from the

developing countries. Dr Quinn said that the CIPM encourages this. There are now ten associates of the General Conference of Weights and Measures (CGPM) and that at least a hundred States and economies are involved through their participation in the RMOs. The BIPM has approached NMIs in a further fifty-three countries about the possibility of their becoming Members States of the Metre Convention or Associates of the CGPM.

5 REPORT ON THE ACTIVITIES OF THE JOINT COMMITTEE FOR TRACEABILITY IN LABORATORY MEDICINE, JCTLM

Dr Thijssen presented a report (CCQM/03-13) on the activities of the JCTLM. He said that closer collaboration between the fields of metrology and laboratory medicine would follow on from the establishment of the JCTLM. It was hoped that the committee could be established on a more formal basis, but this had proved to be difficult and discussions were continuing. He said the IFCC would do their utmost to make the collaboration successful. He went on to explain that a major challenge in clinical chemistry was the question of how to define the measurand. For example: activity, function and structure were all important properties.

Two working groups had been established. Working Group 1 covered “Reference Materials and Methods” and Working Group 2 covered “Reference Laboratories”.

The European Union’s IVD Directive had been an important trigger for the formation of the JCTLM and for ISO 17511, which describes a system for establishing traceable measurement results for clinical measurements. The mission for the JCTLM was “to support the worldwide comparability, reliability and equivalence of measurement results in laboratory medicine…” [CCQM/03-14].

Dr May described the work of Working Group 1 [CCQM/03-20]. He reported that the IVD Directive requires traceability to “reference materials or reference measurement procedures of a higher order”. WG 1 would identify and recommend a list of reference materials that meet these requirements as defined in several ISO standards. Eight priority areas had been initially identified: electrolytes, enzymes, metabolites, proteins, nucleic acids, drugs, hormones and coagulation factors. A review team had been established for each area, and each of these teams had good global representation.

Drawing on experience from developing the KCDB, a procedure had been developed starting with organizations nominating their own, or others, reference materials and reference measurement procedures that might meet the requirements of being of “higher order”. The nominations had included 79 methods and 501 reference materials. Most of the nominated reference materials do not appear in the KCDB. All of the nominations will be reviewed by a team of experts in each area followed by a joint meeting in order to ensure that a harmonized approach had been used. This required sorting all nominations into one of three lists:

- alpha list – meets requirements,

- beta list – additional information needed to determine compliance,
- gamma list – does not meet requirements.

There will also be a process to identify the most important gaps in what has been reviewed. He suggested that there would be an important role for the working groups of the CCQM in reviewing and analysing some of these candidate reference materials in order to audit the review process.

Working Group 2 of the JCTLM had collected nominations of laboratories that wished to be considered as reference measurement laboratories. Such laboratories would need to be proficient in performing reference measurement procedures. The IFCC have already established international networks of such laboratories in the field of enzymes as well as for HbA1c.

Mr Squirell said that this was a very important initiative which would be supported by the ILAC.

The Director said it was important to meet the requirements being addressed by the JCTLM since they were both metrology-based and existed across the world. The question was how to respond to such requirements; it was not reasonable for the CIPM to decide to ignore such a substantial issue.

Dr Padilla said that, although the WHO was not formally involved in the establishment of the JCTLM, she felt it could contribute to its success and could bring information about the WHO's own activities with biological standards. She also suspected that the work of the JCTLM might be closely related to that of the medicine control agencies in their different jurisdictions. Dr May confirmed that the work was focused on diagnostics and not therapeutics.

6 UPDATE OF THE BIPM KEY COMPARISON DATABASE, KCDB

6.1 Update of CMC claims for chemistry and review of existing claims (report from inter-regional review meetings)

Dr May reported on the work carried out at the inter-regional CMC review meetings (CCQM/03-11). The third such meeting had taken place in April 2003. He reported that there had been four "cycles" in the review process (CCQM/03-09): the first cycle covered 821 claims related to gases; the second covered more than 1000 claims in all other categories; the third and fourth cycles had covered 752 and 372 claims, respectively. He said that he expected the current cycle to be completed by September 2003.

Dr Wielgosz asked how it could be assured that CMC claims, such as those for optical filters, were reviewed by the best qualified group of experts. Dr May said it was important that all experts interested in the process should be involved. The President noted that other Consultative Committees having competence and responsibilities in the fields concerned would also be involved in reviewing those CMCs. The President thanked Dr May and Miss Parris for their extremely valuable work carried out on behalf of the inter-regional review process.

6.2 Linking RMO key comparisons (and subsequent bilateral comparisons) with CCQM key comparisons

The President said that the chairmen of the working groups had given a number of good examples of how RMO and CC key comparisons could be linked.

6.3 Calculation and publication of degree(s) of equivalence

The President drew the attention of the meeting to Dr Quinn's earlier advice that there was no need to compile a table showing every possible bilateral degree of equivalence in the final reports of key comparisons.

Dr Richter asked whether laboratories could be declared to be "equivalent". In reply, Dr Quinn said that this was not the approach that had been developed in the CIPM MRA, where the calculation of degrees of equivalence for particular measurands had been adopted. Dr de Leer pointed out what he considered to be an inconsistency between Appendix B, where a laboratory's own estimate of its measurement uncertainty was accepted, and Appendix C, where a laboratory's estimated uncertainty was only accepted after regional and inter-regional review. In reply to a question from Dr Taylor, the President said that an important task for the future would be to determine how the KCDB is actually used.

6.4 Rules for the publication of reports of key comparisons

The President drew the attention of the meeting to the proposals contained in CCQM/03-03, which he hoped would help key comparisons run more smoothly.

6.5 Revisions to the KCDB

Dr Stéphanie Maniguet, from the BIPM, gave a short presentation of recent changes to the KCDB. These included improvements in both presentation and the underlying web programming.

7 ISO-REMCO

Prof. De Bièvre made a short presentation on ISO-REMCO. Since its establishment in 1975 it had worked as an ISO Council Committee on matters relating to reference materials. It had developed the ISO 30 series of guides relating to the preparation, certification and use of reference materials. He reported that its work was being brought into line with both the *International Vocabulary of*

Basic and General Terms in Metrology (VIM) and the *Guide to the Expression of Uncertainty in Measurement* (GUM). The ISO-REMCO had passed a resolution (CCQM/03-05) at its May 2002 meeting for transmission to the CCQM.

Dr McLaren welcomed the contribution from the ISO-REMCO and said that ISO Guides 34 and 35 were now in widespread use amongst the NMIs. The President confirmed that CCQM would note future contributions from the ISO-REMCO, and that Dr Wielgosz would attend the forthcoming ISO-REMCO meeting in order to present the CCQM position.

8 PROPOSAL FOR THE MODIFICATION OF "CRITERIA FOR THE ACCEPTANCE OF CRMS INTO APPENDIX C OF THE CIPM MRA"

Dr Taylor presented his paper (CCQM/03-19) written jointly with co-workers at the IRMM, the BAM and the LGC. It proposed modifications to the CCQM's agreed policy on the acceptance of CRMs into Appendix C of the CIPM MRA (CCQM/01-08), and pointed to inconsistencies in the document as it stands today, namely the fact that ISO Guides 34 and 35 were not mentioned. He stressed that for CRMs the technical issues addressed in ISO Guides 34 and 35 should receive due consideration in the CMC review process, which needed to go beyond the issue of a review of the quality system. The paper also proposed possible improvements in the presentation of the KCDB to users of CRMs.

The President confirmed that an update of the document is due but that this should be carefully looked at by the newly formed Working Group on Key Comparisons and CMC Quality. Dr McLaren agreed to do this.

9 ESTABLISHMENT OF A CCQM WORKING GROUP ON KEY COMPARISONS AND CMC QUALITY

The President proposed that Dr J. McLaren from the NRC take over the role of chair. The working group's role would be extended to cover CMC quality, and would be responsible for the organization of the inter-regional review meetings. The proposal was agreed.

Dr McLaren presented his proposals for some revised terms of reference of the renamed "Working Group on Key Comparisons and CMC Quality". He proposed to constitute the group by seeking one nomination (other than the chairman) from each of the six working groups of the CCQM – taking

into account the future needs for CMCs in the areas of surface and bio-analysis. Further members would be included to ensure adequate representation from each of the RMOs, which would bring the total number of members to approximately fifteen. In general, it would be desirable, but not essential, for members to have previous experience of the CMC review process.

He emphasized that his proposals would have to be consistent with the recommendations for the establishment of such groups from the JCRB. He thanked Miss Parris from the NIST for her support and was delighted that she would continue to be available to carry out this role.

In response to a question from Dr Marschal, the Director confirmed that the review activities carried out at the inter-regional level should be additional to those carried out within the regions.

The President proposed that Dr McLaren go ahead and complete the terms of reference. The President added his thanks to Dr May and Miss Parris for their vital contribution to the inter-regional review meetings for CMCs.

10 REVIEW OF TEN YEARS SINCE FOUNDATION OF THE CCQM

Dr Quinn gave a summary of the development of activities in chemistry under the Metre Convention. He highlighted the role of a number of people in bringing this about. Dr Alex Williams had been the first to suggest to him that the CIPM consider including work on chemistry. Subsequently, Dr John Lyons had done a great deal to support the proposal at the CIPM. The President of the CCQM, Dr Kaarls, had also been extremely influential in ensuring its success. In addition, the staff at the BIPM had done much to support the CCQM; these included the first Executive Secretary, Dr Davis, the current Executive Secretary, Dr Wielgosz, and Dr Thomas who had done much to adapt the KCDB to the needs of chemistry.

Dr Marschal gave a short personal view on the progress made by the CCQM. He said that the success of the CCQM had demonstrated that analytical chemistry was a part of measurement science, and concluded that the CCQM was “on the right track and going in the right direction”.

New initiatives: The President said that a meeting would be held in November 2003 with key representatives from the food sector to establish collaboration on the comparability of measurements in that area.

11 REPORT OF THE AD HOC WORKING GROUP ON VISCOSITY, AHWGV

The President reported that an ad hoc working group on viscosity had been founded. It was carrying out a key comparison and the results were ready for entry into the KCDB. The group would continue with an "ad hoc" status until the CIPM had decided which Consultative Committee it should report to. He believed that it should probably report to the Consultative Committee for Mass and Related Quantities (CCM); this was agreed by the meeting.

12 NOMINATION AND DESIGNATION OF LABORATORIES

The President drew the attention of the meeting to examples of measurements that required highly specialized facilities, such as nuclear reactors for neutron activation analysis. He suggested that their use should be acceptable under the MRA. The proposal was agreed, subject to the drafting of suitable criteria.

13 BIPM PROGRAMME OF METROLOGY IN CHEMISTRY

Dr Wielgosz presented his report of progress with the BIPM programme of metrology in chemistry. The BIPM team had four staff members, which had been the strength foreseen when it started in 2000.

The BIPM now maintains three NIST standard reference photometers (SRPs). Since the last CCQM, the BIPM had completed the first comparison of a standard reference photometer with the CHMI (*Rapport BIPM*-2003/03). There continues to be an uncertainty in the absorption cross-section of ozone which the BIPM is planning to address through the use of NO/NO_2 gas-phase titration to determine the concentration of ozone by an independent method. The BIPM will now proceed to act as pilot laboratory for CCQM-P28. The comparisons were expected to take place between July 2003 and September 2004, with over twenty laboratories expected to participate.

The BIPM had also stimulated some work (GAWG/02-05) within the Working Group on Gas Analysis on the mole fraction of argon in the atmosphere which is required to make an accurate correction for buoyancy effects in mass metrology. A redetermination of the argon mole fraction in air had been requested by the CCM. Measurements of argon mole fraction had been reported by the

KRISS on ambient air collected at a WMO-GAW site, and the CCQM-P41 study had been extended to argon mole fraction measurements.

Following the last CCQM, the BIPM had continued to develop a programme in the area of pure organic reference materials (CCQM/03-18). Dr Wielgosz had worked with Dr Henrion from the PTB to establish the requirements amongst the NMIs. They had reported to the CIPM in October 2002, and to the CCQM Working Group on Organic Analysis at its last two meetings, the last of which had been held in the two days preceding the meeting of the CCQM. The working group was in agreement with the aims of the programme of work at the BIPM, which would " facilitate the development of robust approaches and methodologies for the determination of purity." This would require the identification of appropriate materials for study; the co-ordination, development and extension of the CCQM-P20 series of comparisons for purity determination; the establishment of a BIPM laboratory to support these activities; and the establishment of international liaison to support and promote the programme. The laboratory facilities would include an appropriate ensemble of techniques (selected from e.g.: GC-FID, GC-MS, HPLC, DSC, HPLC-MS and Karl-Fischer titration). The programme would thus develop the series of international comparisons to provide agreed and documented methodologies for purity determination. The group at the BIPM would eventually include two people working in this area.

In response to a question from Prof. Emons, Dr Wielgosz said that the BIPM did not have the resources to produce reference materials, but only to refine the techniques used in the assessment of their purity. Dr Mackay said that the Working Group on Organic Analysis would prefer the work to emphasize direct assay techniques because the group would appreciate some support in developing them. Dr Wielgosz replied that DSC, which was commonly employed in NMIs as a direct assay technique, would be developed at the BIPM, and would need to be validated by independent methods. Currently, NMR and adiabatic calorimetric direct assay techniques were outside the scope of the programme, but collaboration with NMIs' experts in these techniques would be sought.

The President thanked Dr Wielgosz for his presentation, and looked forward to further news on the implementation of the programme.

14 VOCABULARY

Dr Quinn said that progress was being made towards the important objective of reviewing the VIM.

15 FUTURE CCQM WORKSHOPS

The President said that the CCQM had held successful workshops on traceability, measurement uncertainty and primary methods. He suggested that it might be useful to plan another one and asked for proposals. Various suggestions were put forward including: purity analysis, definition of the correct measurand, metrological approaches to proficiency testing and the interface with users and the dissemination of information.

16 BIPM SUMMER SCHOOL 2003

Dr Quinn informed the meeting about the summer school to be held at the BIPM. It would involve eighty students from twenty-four countries and twenty lecturers including several from the CCQM.

17 CCQM RECOMMENDATIONS

There were none.

18 DATE OF NEXT MEETING

The next meeting of the CCQM will be held during the period 15-23 April 2004. The CCQM Working Groups will hold their meetings during 15 to 21 April 2004 at the BIPM, followed by the plenary meeting of the CCQM on 22 and 23 April 2004 at the BIPM.

The President thanked all members of the CCQM for their participation and asked the chairmen of the CCQM working groups to convey his thanks also to all the members and observers of the CCQM working groups who are doing such an excellent job on behalf of the CCQM.

M.J.T. Milton, Rapporteur

Table 1. A framework for CCQM key comparisons and pilot studies

Description	Reference No.	Pilot laboratory	Start date	Status (as of 04/11/2003)	Comments	Working group
Health						
Clinical diagnostic markers						
Cholesterol in serum	CCQM-P6	NIST	1998	Completed; progression to key comparison proposed		OAWG
Cholesterol in serum	CCQM-K6	NIST	1999	Approved for equivalence		OAWG
	CCQM-K6 Subsequent	NIST	2001	Approved for equivalence		OAWG
Glucose in serum	CCQM-P8	NIST	1999	Completed; progression to key comparison proposed		OAWG
Glucose in serum	CCQM-K11	NIST	2001	Approved for equivalence		OAWG
Creatinine in serum	CCQM-P9	NIST	1999	Completed; progression to key comparison proposed		OAWG
Creatinine in serum	CCQM-K12	NIST	2001	Approved for equivalence		OAWG
Electrolyte elements, steroids and hormones in serum and urine						
Trace elements (Pb, Se) in serum	CCQM-P14	NIST/LGC	1999	Abandoned (see next)		IAWG
Ca in serum	CCQM-P14	IRMM/SP	2001	Completed; progression to key comparison proposed		IAWG
Ca in serum	CCQM-K14	IRMM	2003	Approved for equivalence		IAWG
Anabolic steroids in urine						
Hormones in serum						
Food						
As in shellfish	CCQM-P11	NIST	2001	Completed; progression to key comparison proposed		IAWG
As in fish or shellfish	CCQM-K31	NIST	2002	Approved for equivalence		IAWG
Pb in wine	CCQM-P12	IRMM	2000	Completed		IAWG
Pb in wine	CCQM-K30	IRMM	2003	Protocol complete	Run in parallel to CCQM-P12.1	IAWG
Elements (e.g., Cu, Cd, Zn) in wine	CCQM-P12.1	IRMM	2003	Protocol complete	Run in parallel to CCQM-K30	IAWG
As, Se, Hg, Pb, methyl–Hg in tuna fish	CCQM-P39	IRMM	2003	Protocol complete		IAWG
Cd, Zn in rice	CCQM-P29	IRMM/NMIJ	2001	Completed	Run in parallel to CCQM-K24	IAWG
Cd in rice	CCQM-K24	IRMM	2001	Completed	Run in parallel to CCQM-P29	IAWG
Metals in synthetic food digest	CCQM-P13	LGC	2001	Completed		IAWG

Organic contaminants in mussel tissue	CCQM-P40	NIST	2003	Planned		OAWG
Antibiotics in meat						
Growth hormones in meat						
Vitamins and minerals						
GMO's (DNA, proteins)						
Pesticide residues						
p,p´–DDE in isooctane	CCQM-P2	LGC	1997	Completed		OAWG
p,p´–DDE in corn oil	CCQM-P4	LGC	1998	Completed; progression to key comparison proposed		OAWG
p,p´–DDE in fish oil	CCQM-K5	LGC	1999	Approved for equivalence		OAWG
Gamma–HCH in fish oil	CCQM-P10	LGC	1999	Repeated (see next)		OAWG
Gamma–HCH in fish oil 74 ng/g, 240 ng/g	CCQM-P10.2	LGC	2000	Completed		OAWG
p,p'–DDT in fish oil	CCQM-P21	LGC	1999	Completed; progression to key comparison proposed		OAWG
p,p'–DDT in fish oil	CCQM-K21	LGC	2000	Approved for equivalence		OAWG
Drinking water						
Organics (EPA list)						
Trace elements						
Microbiological						
Environment						
Water						
Waste water (EPA list)						
Cd and Pb in natural water	CCQM-K2	IRMM	1998	Completed		IAWG
Atmospheric pollutants						
Greenhouse gases CO_2, CH_4 – ambient levels	CCQM-P41	NMi	2002	In progress		GAWG
SF_6, CFCs – emission levels	CCQM-K15	KRISS	2003	Protocol complete	Run in parallel to CCQM-P51	GAWG
SF_6, CFCs – emission levels	CCQM-P51	KRISS	2003	Protocol complete	Run in parallel to CCQM-K15	GAWG
Ozone – ambient levels	CCQM-P28	BIPM	2003	Protocol complete		GAWG
Primary standard gas mixtures						GAWG
CO in N_2	CCQM-K1.a	NMi	1998	Approved for equivalence		GAWG
CO_2 in N_2	CCQM-K1.b	NMi	1998	Approved for equivalence		GAWG
NO in N_2	CCQM-K1.c	NMi	1998	Approved for equivalence		GAWG

SO_2 in N_2	CCQM-K1.d	NMi	1998	Approved for equivalence		GAWG
Natural gases (Types 1,2,3)	CCQM-K1.e,f,g	NMi	1998	Approved for equivalence		GAWG
NO in N_2 (EUROMET)	EUROMET-QM-K1.c	NMi	2002	Report in progress draft A		GAWG
Natural gas (Types IV)	CCQM-K16.a	BAM/NMi	2001	Report in progress draft B	Run in parallel to CCQM-P49.a	GAWG
Natural gas (TypesV)	CCQM-K16.b	BAM/NMi	2001	Report in progress draft B	Run in parallel to CCQM-P49.b	GAWG
Natural gas (Types IV)	CCQM-P49.a	BAM/NMi	2001	Report in progress	Run in parallel to CCQM-K16.a	GAWG
Natural gas (Types V)	CCQM-P49.b	BAM/NMi	2001	Report in progress	Run in parallel to CCQM-K16.b	GAWG
Natural gas (repeat)/LPG	CCQM-K23	NMi	2004	Planned		GAWG
CO, CO_2, propane in N_2	CCQM-K3	NMi	1998	Approved for equivalence		GAWG
CO, CO2, propane in N_2 (EUROMET)	EUROMET-QM-K3	NMi	2000	Approved for equivalence		GAWG
CO, CO2, propane in N_2 (APMP)	APMP-QM-K3	KRISS	2000	Report in progress draft B		GAWG
CO in nitrogen ($50\,000 \times 10^{-6}$,1000×10^{-6}, 10×10^{-6}) gravimetry	CCQM-P23	NMi	2000	Report in progress		GAWG
Benzene/toluene/xylene (BTX) in N_2/air	CCQM-K7	NIST	1999	Approved for equivalence		GAWG
BTX in N_2 (low conc 10×10^{-9}–30×10^{-9})	CCQM-K10	NIST/NPL	2001	Approved for equivalence		GAWG
Dynamic mixing methods	CCQM-P24	LNE	2002	In progress		GAWG
NO_2 in air (10×10^{-6})		NIST	2003	Abandoned		GAWG
VOCs in air	CCQM-K22	NMIJ	2003	Planned		GAWG
COS and H_2S in methane	CCQM-P?		2004			GAWG
H_2S in air/nitrogen	CCQM-P42		2003	Planned		GAWG
NH_3 or HCl in air/nitrogen	CCQM-P?		2004			GAWG
NMHC	CCQM-K?		2004			GAWG
Reactive gases-ambient levels – NO in N_2	CCQM-K26.a	NPL	2003	Protocol complete	Run in parallel to CCQM-P50.a	GAWG
Reactive gases-ambient levels – SO_2 in air	CCQM-K26.b	NPL	2003	Protocol complete	Run in parallel to CCQM-P50.b	GAWG
Reactive gases-ambient levels – NO in N_2	CCQM-K50.a	NPL	2003	Protocol complete	Run in parallel to CCQM-K26.a	GAWG
Reactive gases-ambient levels – SO_2 in air	CCQM-K50.b	NPL	2003	Protocol complete	Run in parallel to CCQM-K26.b	GAWG

HAP's						
Contaminants in soils/sediments/incinerator ash						
Pb/Cd in sediments	CCQM-P15	IRMM	1999	Completed; progression to key comparison proposed		IAWG
Pb/Cd in sediments	CCQM-K13	IRMM	2000	Approved for equivalence		IAWG
Pb/Cd in sediments	CCQM-K13 subsequent	NIST	2000	Approved for equivalence		IAWG
Elements in synthetic digest solutions	CCQM-P16	NMi	1999	Abandoned		IAWG
PCBs in sediments	CCQM-P17	NRC/NIST	2000	Completed; progression to key comparison proposed		OAWG
PCBs in sediments (PCBs 28,101,153,170)	CCQM-K25	NIST/NRC	2001	Report in progress draft B		OAWG
TriButylTin in sediment	CCQM-P18	LGC/NRC	2001	Completed; progression to key comparison proposed		IAWG/OAWG
TriButylTin in sediment	CCQM-K28	LGC/NRC	2003	In progress		IAWG
DiButylTin in sediment	CCQM-P43	LGC/NRC	2003	In progress		IAWG
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						
Boron in Si	CCQM-P33	PTB	2003	Planned		IAWG
SiO2 on Si film thickness	CCQM-P38	NPL	2002	Completed; progression to key comparison proposed		SAWG
SiO2 on Si film thickness	CCQM-K32	NPL		Planned		SAWG
Metal alloys						
Minor elements in steel	CCQM-P25	NMIJ/NIST/BAM	2002	Report in progress; progression to key comparison proposed		IAWG
Minor elements in steel	CCQM-K33	NMIJ/NIST/BAM	2003	Planned		IAWG
Constituents in Al alloy	CCQM-P34	BAM	2001	Report in progress		IAWG
Polymers and plastics						
Leachates						
Trace metals						

Catalysts						
Pt, Rh in vehicle exhaust catalysts						
Commodities						
Industrial SO_2 in stack emissions see	CCQM-K1.d					GAWG
Moisture in fossil fuels						
Sulfur in fuels	CCQM-P26	IRMM/NIST	2001	Completed		IAWG
Sulfur in fuels (lower levels)	CCQM-P26.1	NIST	2003	Planned		IAWG
Sulfur in fuels (lower levels)	CCQM-K35	NIST	2003	Planned		IAWG
Metals in lubricating oils						
Natural gases see	CCQM-K1.e,f,g	CCQM-K16.a,b				
Sucrose						
Cement – Ca, Si, Al, S, Ti, Na, Mg						
Ore composition						
Rare-earth elements						
Precious metals						
Source of origin/adulteration						
Alcohol content						
Ethanol in aqueous matrix (forensic and commodity levels)	CCQM-P35	BAM/LGC	2001	Completed; progression to key comparison proposed		OAWG
Ethanol in aqueous matrix (forensic level 1×10^{-6})	CCQM-K27.a	LGC/BAM	2002	Report in progress draft B		OAWG
Ethanol in aqueous matrix (forensic level 1×10^{-6})	CCQM-K27.a Subsequent	NIST	2003	Planned	Run in parallel with SIM pilot study	OAWG
Ethanol in aqueous matrix (commodity level 100×10^{-6})	CCQM-K27.b	LGC/BAM	2002	Report in progress draft B		OAWG
Forensics						
LSD in urine	CCQM-P27	LGC	2001	Completed		OAWG
Drugs of abuse in urine	CCQM-P27.1	NARL	2004	Planned		OAWG
Explosives residues						
Ethanol in air	CCQM-K4	NPL	1999	To Appendix B		GAWG
Ethanol in air (EUROMET)	EUROMET-QM-K4	NPL	2000	Approved for equivalence		GAWG
Ethanol in air (APMP)	APMP-QM-K4	NMIJ	2000	Report in progress draft B		GAWG
Pharmaceuticals						

Biotechnology						
Genomics, proteomics						
DNA quantification	CCQM-P44	NIST/LGC	2002	Protocol complete		BAWG
DNA profiling	CCQM-P53	NARL	2003	Planned		BAWG
DNA primary quantification	CCQM-P54	LGC	2004	Planned		BAWG
Peptide/protein quantification	CCQM-P55	LGC	2004	Planned		BAWG
Fluorescence measurements						
Immunoassays						
General analytical applications						
Purity of materials metals, salts, organics, etc.						
KCl, NaCl, $K_2Cr_2O_7$	CCQM-P7	NIST				
Hydrochloric acid	CCQM-P19	NIST	1999	Completed 2001		EAWG/IAWG
Purity of HCl	CCQM-P19.1	NIST	2002	Report in progress		EAWG/IAWG
Acetanilide, benzoic acid, and naphthalene	CCQM-P5	NIST	1998	Completed 1999		OAWG
TBT chloride	CCQM-P20.a	NARL	2001	Completed		OAWG/IAWG
o-xylene	CCQM-P20.b	NIST	2002	Report in progress		OAWG
Atrazine	CCQM-P20.c	NARL	2004	Planned		OAWG
Chlorpyrifos	CCQM-P20.d	NARL	2004	Planned		OAWG
Purity analysis of parent gases incl. H_2O	CCQM-P45	LNE	2002	Planned	EUROMET workshop	GAWG
NMR study	CCQM-P3	BAM	1998	Completed 1999		OAWG
NMR study	CCQM-P3.2	BAM	1999	Completed 2000		OAWG
Assay of potassium hydrogen phthalate (KHP)	CCQM-P36	SMU/NIST	2002	Report in progress; progression to key comparison proposed		EAWG/IAWG
Assay of potassium hydrogen phthalate (KHP)	CCQM-K37	SMU	2003	Planned		EAWG/IAWG
Calibration solutions						
Trace elements in water Pb	CCQM-P1	NIST	1997	Completed 1998		
Elemental solution standards (Al,Cu,Fe,Mg)	CCQM-P30	EMPA/LNE	1999	Completed 2000		IAWG
Elemental solution standards (Al,Cu,Fe,Mg)	CCQM-K8	EMPA/LNE	1999	Approved for equivalence		IAWG
Anions in calibration solutions	CCQM-P32	EMPA	2001	Completed; progression to key comparison proposed		IAWG
Anions in calibration solutions	CCQM-K29	EMPA	2003	Protocol complete		IAWG
Organic calibration solutions (PAHs)	CCQM-P31.a	NIST	2003	Planned		OAWG
Organic calibration solutions (PCBs)	CCQM-P31.b	NIST	2003	Planned		OAWG
Organic calibration solutions (chlorinated pesticides)	CCQM-P31.c	NIST	2003	Planned		OAWG

Preparation of inorganic calibration solutions	CCQM-P46	NIST	2003	Planned		IAWG
VOCs in organic solvents	CCQM-K37	KRISS/NIST	2003	Planned		OAWG
pH standards						
pH 7.0 (phosphate)	CCQM-K9	PTB	1999	Approved for equivalence		EAWG
pH 7.0 (phosphate) PTB-SMU bilateral comparison	CCQM-K9 subsequent	PTB	2002	Approved for equivalence		EAWG
pH 4.1 (phthalate)	CCQM-K17	PTB	2001	Approved for equivalence		EAWG
pH 10.1(carbonate)	CCQM-K18	SMU	2003	Protocol complete	Run in parallel to CCQM-P52	EAWG
pH 10.1(carbonate)	CCQM-P52	SMU	2003	Protocol complete	Run in parallel to CCQM-K18	EAWG
pH 9.2 (borate)	CCQM-K19	PTB	2004	Planned		EAWG
pH 1.7 (tetroxalate)	CCQM-K20			Planned		EAWG
Fundamental studies of pH standards	CCQM-P37	SMU	2002	Completed		EAWG
Electrolytic conductivity	CCQM-P22	DFM	2001	Completed		EAWG
Electrolytic conductivity (low level)	CCQM-P47	NMi	2003	Planned		EAWG
Electrolytic conductivity (0.5 S/m)	CCQM-K36.a			Planned		EAWG
Electrolytic conductivity (0.005 S/m)	CCQM-K36.b			Planned		EAWG
New proposals						
Uranium isotope ratio in synthetic saline matrix	CCQM-P48	IRMM	2003	Planned		IAWG
Metal in lubricating oil		LGC				
Precious metals		LGC				
Trace metals in plastics		IRMM				
Metals in vehicle exhaust catalysts		IRMM				
Trace metals in plastics						
Total protein in serum						
Growth hormone(s) in meat						
Cortisol in serum 50–230 ng/g						
Moisture in crude oils						
Moisture in food and commodities						
Food toxins						
PCDD and PCDF in tissue and in fly ash						

APPENDIX T 1.
Working documents submitted to the CCQM at its 9th meeting

Working documents submitted to the CCQM at its 9th meeting are on restricted access.