TC-MC

Technical Committee - Metrology in Chemistry

- Plenary Meeting -

04 – 05 February 2010

INRIM, Torino (Italy)

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1. Welcome Address (*A. Sacconi*)

INRIM Science director Dr. Attilio Sacconi gives an overview to the Italian metrology system.

2. Introduction and approval of agenda (*B. Güttler*)

The agenda of the 2008 TC MC meeting is approved without any changes.

3. Tour de Table (*all delegates*)

The chairman asks the delegates of the plenary session to introduce themselves.

4. TC Chair's report on EURAMET TC Chair's meeting and EURAMET GA
(*B. Güttler*)

The number of Euramet member NMIs increased from 32 in 2009 to 34 in 2010. IMBiH, Bosnia-Herzegovina, was accepted as the latest member NMI while three additional NMIs apply for membership. In 2010, 68 Designated Institutes (DIs) have the status of an *associate*.

Leslie Pendrill (SP, Sweden) is the new Euramet chairperson following M. Kühne, who has been elected as the new director of the BIPM. The current board of Euramet directors reflects the diversity of the members in respect of geography, level of metrological development and metrological impact in Europe.

A new TC-IM Focus group on "Facilitating National Metrolgy Infrastructure development" has been established. Their objectives are the promotion and development of the metrology infrastructure in the countries of its members by an increased cooperation and the facilitation and acceleration of the integration of its member NMIs into EURAMET activities.

Two guidance documents "Euramet procedures and review criteria for CMCs" and "Euramet and the operation of NIMs" have been released in 2008. New in 2009 are the "guidelines on the determination of uncertainty in gravimetric volume calibration". All guides are available from the Euramet website for free.
Also available from the website is the Euramet Newsletter, published every 6 month.

The next Euramet General Assembly will be held in Lisbon, 25-28 May 2010.

TC Chair's report on status of EURAMET QM CMCs
(*B. Güttler*)

B. Güttler summarizes the April 2009 KCWG meeting. According to a commitment from KCWG, key comparisons that are not on the list of completed key comparison from the WG chairs, must not be used to support CMCs. CMC claims shall be discussed prior to KCWG meeting, the review of existing CMCs shall be discussed in light of new intercomparison results.

86 Euramet TC-MC claims were approved in 2009 (CCQM 186 claims) and no pending

claims exist at the beginning of cycle XI. Most Euramet claims (61%) were from IAWG, 22% from OAWG, 14% from GAWG and 3% from EAWG.

B. Güttler remembered the delegates that deferred claims must be resubmitted in the next cycle.

5. Convenors report on subcommittees activities

Information about TC-MC Convenors Meeting:
Inside Information about the 2010 TC-MC convenors meeting are given by B. Güttler.

M. Milton suggests a later start of the CMC review process. End of Nov/beginning of Dec is too early because the reports that are discussed and approved during the Nov CCQM meetings should enter into the list of completed key comparison that should be used in the forthcoming CMC review process.
B. Güttler suggests to circle around the list of comparisons as soon as it appears in 2010.
B. Güttler announces a change in the annual TC-MC meeting timetabe. The Monday shall be used for discussion of ongoing EMRP projects (JRP workshops).

SC Gas Analysis (*R. Wessel*)

30 attendees from 16 countries met to discuss 14 projects, 5 final reports are due very soon, 3 new projects started in 2009, draft A for ethanol comparison was presented.

4 new proposals have been discussed (2 x NPL, LNE, IPQ)

Re-Review of CMC claims: Cycle XI is dominated by claims based on CCM-K51, -K52, -K65, and BIPM.QM-K1 (ozone).

Discussion arises on the timescale of repetition of KCs underpinning re-review of CMC claims (currently 10 years).

Ozone claims: Formula-type uncertainty for European CMCs are given.

The question arised that labs need ISO guide 34 accreditation. Who will check this, TC-Q or TC-MC?

SC Organic Analysis (*G. O'Connor*)

13 participants from 9 institutes representing 8 countries attended the meeting.

16 CMC cycle XI claims were submitted for consideration by Euramet; 2 claims were accepted outright, 4 claims need further modifications of which 3 bio-related claims involve expert opinion from BAWG. 10 claims are withdrawn and shall be resubmitted when new evidence is available.
It was requested to make available a brief set of clear guidelines to NMIs/DIs who submit CMCs. It was further suggested to ensure consistency of new Biological claims with other RMOs .

In the future, education aspects of the SC shall be maintained. Dissemination of traceability for low level measurands like organic POPs was identified as one of the grand challenges in environmental analysis.

R. Kaarls: Education aspects are of high importance to new members, e. g. in the field of CMC preparation.
M. Sargent: Education and related activities shall be made consistent throughout all the SCs.

SC Electrochemistry (*P. Spitzer*)

The final reporting of Euromet project 898 (electrolytic conductivity of pure water level) was discussed. Further comparisons within the Tracebioactivity JRP10WP4 project are planned to compare PTB, DFM and SP.

Main part of cycle XI CMC business was the re-review of CMCs in **pH and electrolytic conductivity** (cat. 6: 36 CMCs, cat. 7: 26 CMCs).

Re-submission of 62 CMCs (36 pH, 26 EC) to cycle XI started at the Euramet level; CMCs were sent to two SCEC members for comments (BY and CZ are still missing). For pH the situation is less complicated, core- and extended capability buffers are clearly defined.
For EC, the situation is more complicated: There is inconsistency in reporting of uncertainty, declaration of sources and procedures of traceability (units?) to ensure routinely traceability to SI standards and procedures.

Electrochemistry for water analysis was suggested (INRIM) as possible future EMRP work with the focus being on water quality. Possible methods and pollutants are presented

P. Spitzer points to the fact that there are rare conductivity (calibration) measurements that are routinely traceable to primary SI-standards/procedures. Most traceability statements refer to rather old primary measurements. Its seems necessary to perform a key comparison using independent primary measurement procedures.

R. Wielgosz: Re-measurement will probably end up in larger uncertainties; it is not yet clear how to handle this issue.

SC Inorganic Analysis (*C. Quetel*)

An overview on recent and current SCIA meetings and workshops was given.

The projects 563, 724, 763, 784, 785 and 894 have been closed; project 924 is still on-going.

C. Quetel presents a CMC claim statistics for the cycles VI to XI. In total, 268 SCIA CMCs have been submitted and discussted, 203 have been accepted after discussion.

The results of the project 924 (step 3, 117 participants), CCQM-K70 (10 participants) and P100.3 (1 participant) were presented and discussed in detail.

A workshop on the “traceability of the reference values” was held in Helsinki (FI) covering various aspects of sample preparation and measurement during testing and calibration.

During the SCIA meeting, a round table discussion on “Metrological traceability of the reference values we use and/or produce - How do we understand/explain in practice” was held. The discussion (1st round) starts in small groups about “what is traceability” and ends up with statements. The 2nd round was on ”Proposed traceability statement on calibration certificate” and ends up with two traceability statements for publishing CMCs.

53 CMCs have been submitted for cycle XI, 31 were accepted as “fast-track”, 2 will be possibly accepted at a later stage; 20 CMCs were not approved, mainly due to the fact that traceability of the measurement results is unclear because of the use of commercial calibration standards. TUBITAK-UME, NCM-BIM and BRML-INM declared during the meeting that state of practice is now different in each of these organizations: (1) commercial calibration standards are not used anymore and (2) CRMs from fellow NMIs are used instead.

6. Information from President of CCQM (*R. Kaarls*)

Robert Kaarls informs the delegates about recent CCQM activities. Per 1 Feb 2010 CCQM has 54 member states with the latest members being Kazakhstan, Croatia and Kenya.
Till now, 37 countries are accepted as associate members.

The establishment of GULFMET as a new RMO is in preparation; Jordan and Syria are new APMP associates; Albania, FYR Macedonia, Montenegro and EU became Euramet associates.

The CIPM MRA now exists for 10 years and has been very successful in addressing the needs of industry, trade, regulators and society. It is signed by 74 NMIs from 48 member states and covers > 96% of the world trade; further growth is expected.
The MRA is based on results of key-, supplementary- and bilateral comparisons and includes a regional and inter-regional review of claimed calibration and measurement capabilities.
Currently, more than 300 key comparisons are in progress both within and between RMOs.
Published are the Calibration and Measurement Capabilities (CMCs), that are the services of the NMIs and other designated institutes, which are normally delivered to the customers.
The database on www.bipm.org/kcdb now contains some 21 600 CMCs of which 4500 chemical, 3800 ionizing radiation, 13300 physical.

The CIPM MRA documents on http://www.bipm.org/en/cipm-mra/documents/ are mainly related to policy and guidance on CMCs. A couple of new (2009) CIPM/JCRB documents is introduced which have either been released or have been proposed to the CIPM for approval.

Two different routes exist to establish traceability of CMCs to the SI namely (1) via a primary realization or representation of the unit of measurement concerned or (2) via another NMI or DI having relevant CMCs with appropriate uncertainty published in the KCDB or through calibration and measurement services offered by the BIPM. A list of possible exceptions is maintained by the BIPM and is available in the CIPM MRA documents part of the BIPM website. Some additional notes explaining various issues on the application of traceability routes are given.

CCQM work is organized in 7 permanent and 3 ad-hoc (KCRV, EET, redefinition SI) working groups.
Currently the WG on the KCRV drafts a guidance document for calculating the KCRV.
A guidance for determining the relation between the uncertainty in claimed CMCs and the Degree of Equivalence (DoE) determined by the results obtained in a Key Comparison is still needed. Some additional notes on details of KC/PS report preparation are given.

The CCQM proposed future strategy includes key comparisons that test core competencies for providing primary calibration and accuracy control reference services, key comparisons that assess the equivalence of measurement services provided and pilot studies in emerging areas of interest and/or strategic importance.

Various core competencies and sub-capabilities have been defined in different fields of chemical analysis and will be adressed by the working groups.

Several CIPM CCQM recommendations on the possible re-definition of the mole and the kilogram are presented.

R: Kaarls finishes by summarizing some other issues concerning international cooperations, VAMAS, BIOREMA and mentions that a CCQM workshop on microbiology and milk issues is in preparation (2nd half 2010). The contence of BIPM programme of work for 2013-2016 is roughly outlined (ozone, green house gases, bio metrology).

2009-2010 events and meetings are summarized.

B. Magnussen: What about cooperation with OIML?
R. Kaarls: Currently, there is more coordination than cooperation.

7. Workshop EMRP TP Environment

7.1 Report: "Recent developments of the EMRP programme" (*L. Erard, LNE*)

L. Erard summarizes the stages to be passed on the timetable for establishing the new EMRP programmes "Metrology for industry and environment". He outlined the common characteristics of a call and in particular the issues relevant for both 2010 calls "Energy" and "Environment" which might be likewise relevant for future TPs.

The principle objective of "metrology for industry" is to support metrological research (1) to improve the **competitiveness** of European industry, (2) to generate knowledge to ensure industry's transformation from a resource intensive to a **knowledge-intensive** base and (3) to achieve fundamental optimizations within the European industries.

The call "Metrology for environment" shall address geographically localised environmental challenges such as related to (1) pollution of water, air and soil, (2) waste management (including radioactive waste), (3) radiation measurement and protection and (4) global metrological challenges for climate control.

Some of the general goals of a JRP to be addressed in the EMRP are (1) to address metrological challenges without duplication of other programmes, (2) to address research which could/would not be addressed in a single NMI alone or other than with EMRP and (3) to address "big challenges".

The budget/size of JRPs should be in the range between 2.7 M€ and 3.2 M€ and must not exceed 4.5 M€.

Article 169 allows the commission to to cofinance a programme of member states which means that the EMRP is a EURAMET programme, not a Commission programme. The commission expects scientific, management and financial integration.

Organisations which are eligible for project funds are NMIs, designated institutes (DIs) or institutes of the EC. Other organisations may participate as *unfunded partners* (research lab, NMI outside Europe) or *collaborators* (industry, politics).

The JRP-coordinator is responsible for (1) writing the programme of work for the JRP, (2)

preparing a poster detailing the JRP, and presenting this to the Referees at the Review Conference, (3) agreeing with the participants of each JRP and (4) reporting progress to the Commission during the lifetime of the JRP.

Finally, national commitments and voting weights in the EMRP committee are compared.

7.2 Report: "Facilitating the TP Energy – Lessons learnt"
(*K.-D. Sommer, PTB, presented by L. Erard, LNE*)

Several problems concerning the costing process, scientific and metrological excellence, REGs, and facilitation encountered during the recent TP *Energy* are decribed and possible solutions are discussed.

7.3 Preparation for an EMRP Targeted Programme "Environment"

Several presentations were given covering a broad range of environmental metrological challenges:

Challenges in gas analysis and EMRP opportunities in the Environment and Industry call (*Rob Wessel, VSL*)

Metrology requirements for the environment and EMRP opportunities in gas and particle analysis (*Martin Milton, NPL*)

Metrology of particles (*Jürg Schlatter, METAS*)

Graphene-based sensors for environmental monitoring (*Michela Sega, INRIM*)

Underpinning metrology for seawater salinity measurements (*Petra Spitzer, PTB*)

Strengthening of the metrological basis of dissolved oxygen concentration Measurement (*Ivo Leito, Univ. Tartu*)

LNE proposals for challenging metrological research topics in environmental Studies (*Sophie Vaslin-Reimann, LNE*)

Incorporating Chem/Bio metrology into collaborative "Big Picture" PRT's from climate change to industrial process monitoring (*Helen Parkes, LGC)*

Metrology in support of European environmental legislation (*Andrea Held, IRMM*)

Metrology in Chemistry and Biology for Environment (*M. Suchanek*)

Metrology for European water legislations (*Detlef Schiel, PTB & T. Win, BAM)*

Topics for TP Environment discussed in the Sub-Committee Inorganic Analysis *(C. Quetel, IRMM)*

8. Reports on CCQM WG meetings

EAWG activities (*M. Mariassy*)

M. Mariassy starts his overview by stating the *terms of reference* of the group followed by a summary of the main working activities (pH, conductivity, coulometry) of the group members.

The target measurement ranges of recent conductivity and pH sudies are summarized and detailed results are presented for several ongoing or recently finished studies:
P83 (low conductivity): A good comparability was achieved but there is still a factor of 100 down to ultra pure water.
P37.1 (problems in primary pH measurement): Questionaire was sent out, but limited response has been received to date.
P112 (assay of EDTA): The coordinating laboratory will be BAM (supported by SMU and PTB), measurements will be centrally at performed at SMU (reproducibility), some problems are still not solved.
K73 (Assay of H+ in HCl @ ~ 0.1 mol/kg): Results show some discrepancies, possible reasons to be discussed during the April CCQM meeting.
Further studies and comparisons are on the way.

The main part of current CMC activities is the resubmission of all claims in pH and conductivity; harmonization of the format of the CMC Excel sheets shall be achieved.
An EAWG draft CMC document is distributed for discussion, the issues addressed are (1) format, (2) compliance with comparison results, (3) comparisons necessary to support specific claims, (4) HFTLS and (5) future comparison strategy.

IAWG activities (*M. Sargent*)

The main objectives of CCQM IAWG are summarized which are (1) to evaluate and publicly demonstrate the equivalence of inorganic chemical measurements made at different NMIs, (2) to organise a programme of comparisons, (3) to collaborate on new measurement capabilities and (4) to encourage and assist new NMIs and designated institutes.

The IAWG work programme includes organizing laboratory comparisons, the development and improvement of procedures and measurement methods and the organization of regular meetings.

Two meetings (04/09: Sevres, 11/09: Rio) were held, both include EAWG joint sessions.

Overview on current status of key comparison and pilot studies is given, all are being completed in 2010; some detailed results are presented for K56.

Seven technical presentations were given in 2009 by IAWG menbers. Among others, three presentations were given in the field of biofuels

On-going development of IAWG strategy: The third benchmarking study has been completed. Their objectives were (1) to benchmark equivalence between all IAWG laboratories through participation in the same comparison and (2) to demonstrate that NMIs achieve good results even with analyses outside their existing experience.
(H*ow far does the light shine for an NMI?*)

An IAWG core capability matrix has been defined as a systematic summary of the scope of each KC and the competencies required to deliver each CMC. Recent IAWG discussions have refined the aprroach; the third benchmarking study and CCQM-K75 were used to evaluate this approach. A summary is given on ID-ICP-MS of Pt as example.

R. Wielgosz: There was a large number of studies and CMC claims over the past few years, is that enormous working level of IAWG sustainable?
M. Sargent: The idea of the new approach is to identify and concentrate on most important KCs; current IAWG activities aim to reduce the work to the necessary extent over the next few years.

GAWG activities (*M. Milton*)

Five KC results are available as draft "A" reports (K46, -K54, -K66, -K68, -K71), three studies are in progress (K74, -P110, -K76), three are planned (K77, K83, K83), 6 comparison results (APMP-QM-S1, -QM-K1c, CCQM-K53, -K65 , COOMET-QM-K1a, -QM-K23b) are currently to be published in the KCDB. Some meaningful results of the finished KCs were presented and discussed in detail.

Documents and guidelines on CMC review for use in cycle XI are GAWG/08-95 (Recommendations on CRM uncertainty), GAWG/10-03 (Guidelines on CMC claims for Ozone), and GAWG/10-04 (Review of gas CMCs in Cycle XI). GAWG/10-05 (Implementing an efficient approach) will be discussed at the April 2010 meeting.

A review on how the spread of KC results varies with concentration suggests that some NMIs may be able to justify CMCs for a wide range of compounds/concentrations from a limited set of comparison results.

Current work aims to implement an efficient approach to the planning of KCs and the approval of CMCs in the GAWG. The aim is to realise a broad HFTLS statements and to define criteria for an NMI to use them.

OAWG activities (*G. O'Connor*)

The primary focus of OAWG activities is the critical evaluation and benchmarking of NMI capabilities for the execution of "higher order" measurement procedures for well-defined organic molecular entities for which the SI-traceable amount of substance is to be determined.

The enormous amount of possible combinations of organic compounds, matrices and mass fractions is displayed together with some examples of range of nominal mass fractions of organic measurands.

An overview on all OAWG key comparisons and pilot studies with status January 2010 is presented, results of selected comparisons are presented and discussed. On eight studies was reported in 2009, three are in progress and four are being planned or planning is under discussion.

Besides the two regular OWAG meetings, a *Quantitative NMR Symposium – Metrological use of NMR for Organic Purity Analysis* was held.

The strategic planning 2009 has been established; it comprises four major topics which are (1) key comparisons that test core competencies for the delivery of measurement services to customers, (2) key comparisons that assess the equivalence of measurement services actually provided to customers, (3) key comparison studies in emerging areas of global interest and importance with accompanying pilot study and (4) capability assessment studies to allow assessment of measurement capabilities being established in a new area for an NMI/DI.

A task force has been established to identify a set of core competencies and to design a limited set of studies to test these core competencies required for carrying out organic analyses.

BAWG activities (*H. Parks*)

H. Parks presents a brief history of the BAWG (establishment, growth, member states) and summarizes the scope and regular group activities. The continuous development of the CCQM bioanalysis WG strategy involves the establishment of discussion groups which currently exis on nucleic acids, proteins and cells & tissues. Further groups are under discussion.

The results of some recent example studies are given to underpin the scope and the strategy of the group. DNA quantification was the subject of one KC and several pilot studies to develop the capabilities of the member NMIs in this field. Progressive complexity had to be handled in these nucleic acid studies from the measurement principle (K61, P44) to the full analytical procedure.

Peptide and protein measurements are of great importance in the fields of clinical pathology, medical diagnostics, biopharmaceutical production & quality control, and food safety, quality & authenticity. Studies have been performed on absolute traceable quantification of a peptide /protein in solution (P55) and on measuring the protein structure by circular dichroism (P59).

There are new measurement challenges in bioanalysis setting new demands for measurement traceability and design of appropriate reference standards. An increased complexity arises from measuring panels of biomarkers rather than a single analyte; multiparameter/multianalyte analyses lead to “molecular signatures”. Informatics is used for interpretation of the resulting complex high information content (P103 biomarker profiles).

Cell measurements are located near the end of the BAWG roadmap. Traceable cell measurement is needed in clinical diagnostics and therapeutics, for regenerative medicine, and food and environmental safety.

9. **Information from BIPM:**
Measurement Service and Comparison Needs for the Biosciences - BIPM study
(*R. Wielgosz*)

R. Wielgosz informs about “Study of Measurement Service and Comparison Needs for an International Measurement Infrastructure for the Biosciences and Biotechnology: Input for the BIPM Work Programme”. The expected outcomes of the study are reports on (1) measurement services required to establish an international measurement infrastructure for the biosciences, (2) international comparisons that are required to demonstrate the degree of equivalence of the measurements services that are or will be developed and delivered and (3) research and development activities necessary for the development higher metrological order measurement standards and methods for the biosciences. The call for tender was sent out in June 2009, the final report is expected in March 2011.

Proposed activities of the study are (1) reviewing roadmaps and strategies for measurements and the biosciences, (2) drafting of interview and visit report forms and questions, (3) summary of CCQM BAWG activities and plans, and (4) visits and interviews with selected national metrology institutes and other organizations active in biometrology and with representatives of key sectors of bio-industry.

The study outcomes will be presented to the CIPM in October 2010, and to CCQM in 2010 and 2011 and form the basis of the BIPM programme Work Proposals (2013-2016).

10. Report on EURACHEM activity (*M. Suchanek*)

M. Suchanek gives a brief introduction and an overview about the tasks and current activities of Eurachem.

The main goals are to promote best practice and develop networks for collaboration, to develop international comparability of chemical measurements, to provide a framework for co-operation in establishing traceability, to establish national EURACHEM groups and provide input to other international organisations. Eurachem membership is limited to EU, EFTA and EU accession countries.

Important guides released by Eurachem are presented, “Terminology in analysis – introduction to VIM 3” is currently under preparation. Till now 26 issues of the Eurachem newsletter have been released (www.eurachem.org).

The next GA will be in Copenhagen (May 2010), a future int. workshop on proficiency testing will be held in 2011 (Istanbul).

11. Mandate of Convenors (*B. Güttler*)

Bernd Güttler summarizes the role of a TC-MC convenor and introduces Paola Fisicaro (INRIM) as the new SCIA convenor, following Christophe Quetel (IRMM).

The chairman thanks Christophe for his work.

12. Upcoming meetings

There are no upcomings meetings to be announced.

13. Any other business

There is no other business.

14. Next meeting 2011 and Closure

The next TC Metchem meeting will be in Helsinki, Finland in the first week of February 2011.

Bernd Güttler thanks the delegates for their contribution and INRIM for their hospitality.

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