

## METCHEM

### Metrology in Chemistry Technical Committee - Plenary Session -

14-15 February 2008

Istanbul – Turkey

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#### 1 – Welcome address

**Sermet Süer**, Acting Director of TUBITAK-UME welcomes the participants of METCHEM meeting and gives an overview of the scientific, legal and industrial metrology activities at UME. He also expresses his best wishes for the success of the meeting and the great pleasure for UME to welcome TC-MC METCHEM community in Istanbul.

The TC-MC chairman **Bernd Guettler** thanks TUBITAK-UME to welcome the METCHEM representatives, and in particular **Fatma Akcadag** for the excellent organisation of the meeting.

The minutes of the last METCHEM meeting were already amended and will be placed on the new TC MC website.

#### 2 – Introduction and approval of agenda

The agenda of the 2008 TC MC meeting is approved.

#### 3 – Tour de table

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 TC Chair's report on status of Euramet QM CMCs

#### • EURAMET GA

**Bernd Guettler** summarizes the main topics of the two Euromet meetings (TC chair's meeting and Euromet General Assembly).

Concerning TC METCHEM, the following topics were particularly relevant:

- The transfer from EUROMET to EURAMET

- The review of QM systems and CMCs in EUROMET

The main topic was the transfer from EUROMET to EURAMET during 2007. The Inauguration of EURAMET e.V. was accomplished on 11<sup>th</sup> January in Berlin (DE).

The 21th GA took the decision to terminate the EUROMET Memorandum of Understanding with effect from 30 June 2007. All activities and responsibilities as the Regional Metrology Organisation of Europe have been transferred to the "European Association of National Metrology Institutes, EURAMET e.V." with effect from 1 July 2007. Following the roadmap for the transfer of responsibilities, the process ends with the winding up of financial operations of EUROMET and the establishment of the new EURAMET website by 31 December 2007.

The EUROMET website for consultation of historical information does further exist but has been "frozen" at 30 June 2007. The project database will be transferred to the new EURAMET website.

#### • EURAMET QM CMCs

In total, 99 EURAMET QM CMC claims were submitted for review during cycle VIII.

**Bernd Guettler** pointed to the fact, that many CMCs were assigned to the non-fast-track process and few of them are still pending at the end of 2007. The main problems are:

- claims are made prior to the availability of supporting evidence
- evidence is there, but not made available to KCWG or during the review period
- objections are made after the end of the review period

The chairman suggested, that all claims must be made on the basis of evidence made publicly available by the beginning of the evaluation period (this year: 1st March 2008). The reports of relevant comparisons conducted by CCQM and it's regional organisations should be made available on the BIPM webpages. Robert Wielgosz replied that this demand is difficult to fulfill logistically.

A question arises how frequently *the CMC database* is used. Robert Wielgosz replied that ~16.000 hints per year were registered but no detailed user information can be obtained. More information is given in the recent KCDB newsletter. (http://kcdb.bipm.org/NL/08/NL 08 Dec07.html#text2).

#### 5 – Convenors report on subcommittees activities

#### • Report on SC Inorganic Analysis

*Christophe Quétel* reports on the activities of the subcommittee on Inorganic Analysis.

The main subjects are the two ongoing EURAMET projects 894 and 924 and the cycle VIII/IX CMC claims.

The project No. 894 is a PTB-NPL cooperation on Surface-Enhanced Raman Scattering and its possible application in Metrology in Chemistry. The latest progress in designing new SERS-active Nanostructures provides the perspective for a follow-up project which is open also to other interested NMIs.

The project No. 924 was proposed to support the EU water framework directive. In step 1, the measurement capabilities of NMIs and potential calibration laboratories (PCLs) were compared. These measurements were recently completed. The results give evidence of the good quality of measurements conducted by both, NMIs and potential calibration laboratories.

Several cycle VIII CMC claims had to be postponed to the next cycle due to missing evidence. While some of them are now considered as "ready for approval" (UK 11) relevant reports are still missing in other cases (DE 13 FR 13)

For cycle XI, a total of 11 CMC claims were submitted by LNE, PTB and SMU and accepted with minor changes (SMU).

During a joint session of the Sub-Committees on Inorganic Analysis and Electrochemistry, the proposed activities of the iMERA+ Joint Research Project T2.J10 *"Traceable measurements for biospecies and ion actvity in clinical chemistry"* (TRACEBIOACTIVITY) were presented and discussed.

#### Report on SC Electrochemistry

Petra Spitzer reports on the activities of the subcommittee on Electrochemistry.

A brief update is given on the progress made in the on-going projects:

- 843: This project is linked to IUPAC activities on pH measurements but currently behind the schedule

- 898: Has now become a workpackage (WP4) of the EMRP T2.J10 project.

- 918: This project was closed and the final report was presented

- 932: The new developed measurement facilities will also be used for the EMRP T2.J10 (WP 3)

Possible topics for future projects are: pH of rainwater, conductivity measurement in river water, ISE measurements in industrial applications, traceability of redox measurements, metrological aspects of measurement using oxygen sensors, pH and conductivity in biofuels.

There were no new CMC claims to be submitted for cycle XI. Amended CMCs from DFM were discussed and approved.

#### • Report on SC Gas Analysis

Martin Milton reports on the activities of the subcommittee on Gas Analysis.

24 participants attend the meeting. The main subjects are the EURAMET projects and the cycle VIII/IX CMC claims.

Martin Milton summarizes the outcome of the two workshops held in 2007 on dynamic methods (project 888) and the coordination of nanpoarticle research project (893). He further reported on the outcome and ongoing activities of the various comparison projects:

- 708: Bilateral (NMi-VSL/NIST) comparison on SO2
- 867: purity comparison (CO, CO2, O2, CH4)
- 937: follow-up project of 867, same analytes but lower concentrations
- -1002: Water vapour comparison
- 934: TILSAM, workshop was held in January 2008
- 886: comparison on ozone precursors
- 919: NMi-VSL/INTI bilateral

The new projects proposed during the meeting were:

- Bilateral comparison of natural gas (NMi-VSL, INMETRO)
- Bilateral comparison of CO2, N2 and air (NPL, INRIM)
- Nanoparticle comparison (METAS, BAM, NPL, LNE, INRiM?, UBA(D)?)

From the 99 EURAMET CMC claims of cycle VIII, 29% refer to gas analysis. With the two exceptions CEM and GUM, all CMCs have been approved.

Possible activities on harmonization of the Ozone CMCs were discussed. The use of an equation to express the uncertainty was recommended.

Submissions for cycle IX have been received from the four laboratories ISCIII, NPL, NMi-VSL and CHMI.

The convenorship of the SC on gas analysis has been passed on to Rob Wessel (NMi-VSL).

#### • Report on SC Organic Analysis

Franz Ulberth reports on the activities of the subcommittee on Organic Analysis.

A review of outcome of Project 833 (Intercomparison of PCB congener in solution, sewage sludge extract and sewage sludge) is given. Various laboratories participated to the proposed three rounds of the project. The results and conclusions drawn from the final (3<sup>rd</sup>) round were:

- Improvements are made compared to previous rounds in 2004 and 2005
- Some designated institutes had larger deviations relative to participating NMIs
- Relative uncertainties of comparison reference values are 7-14 %

The outcome of a questionnaire concerning the organic analysis programmes of the EURAMET/METCHEM NMIs was presented. Out of 32 institutes contacted, 22 answered, 6 institutes already have an organic analysis programme, another 9 institutes intend to initiate such a programme in the near future.

For possible future EMRP funding, an outline of a project proposal "Traceable measurements of vitamins for clinical chemistry" shall be prepared (UME).

No CMC claims have been received for submission to cycle XI.

#### 6 – Information from President of CCQM

The president of CCQM Robert Kaarls presents the activity of CCQM in 2007.

He points out the opinion of the CCQM with respect to the redefinition of the mole and mentioned the need for a CCQM working group to prepare the CCQM point of view.

Two ad-hoc working groups have been established in 2007:

- Calculating KCRV and its uncertainty WG (KCRV WG, chaired by Prof. Cox, NPL), working on further needs for harmonization
- Efficient and effective testing (EET WG, chaired by Dr. Turk, NIST)

The 14th CCQM, CCQM WG meetings will be held in Paris, 28 March - 4 April 2008.

#### 7 – Workshop: "Recent developments of the iMERA-Plus programme"

*Hans Koch* gives a presentation on "genesis and outlook" of the iMERA+ targeted program "Health" (TP2). This includes the initial roadmapping process that led to the EMRP outline and finally to the 6 funded *Joint Research Projects* (JRPs) of TP2. Three of them were successfully proposed by TC-MC members.

The call for further EMRP targeted programs with 50% EU funding is not expected before 2011. The subjects of future calls are not clear at this stage ("it starts with a new game").

An EMRP symposium on measurements in life sciences will be held at PTB, Berlin from 2 to 3 of July 2008.

*Hans Bachmair*, PTB, presents "Experiences of the TC-EM with Drawing up a TP for Electricity and Magnetism (TP4)

A major point in this discussion is the question how to influence the success-rate of a potential project. The outcome of the ERANET+ funding decision within TP2 has clearly shown that those groups were particularly successful that knew each other well from the related former EUROMET-TC activities.

The three chemistry-related JRPs of TP2 are presented by the JRP coordinators or their representatives.

*Rob Wessel* presents T2.J02, "Breath analysis as a diagnostic tool for early disease detection".

The objective of this JRP is to develop spectroscopic techniques for the identification and accurate quantification of biomarkers in human breath such as acetone, ammonia or carbon monoxide. A methodology for accurate, interference-free, calibrationfree breath analysis shall be worked out.

The work within T2.J02 is organized in 4 WPs with a budget of 2,4 M€ and a work effort of 197 MM.

*Petra Spitzer* presents T2.J10, "Traceable measurements for biospecies and ion activity in clinical chemistry".

This JRP is based on the challenge that today's medical diagnostics not only requires the total amount of an analyte but also it's clinically active form to be quantified. The solution is to provide standards for biospecies and ion activities of relevant elements that are linked to the SI and thus enable traceable analytical results.

The work within T2.J10 is organized in 4 WPs with a budget of 4,1 M€ and a work effort of 384 MM.

*Helen Parks* presents T2.J11, "Traceability of complex biomolecules and biomarkers in diagnostics – effecting measurement comparability in clinical medicine"

In modern medicine often complex biomarkers are used for the diagnosis of disease, but poor quality measurements can lead to inappropriate treatment of patients. This JRP addresses the need to achieve a better consistency and comparability by developing methods and standards which enable the measurements to be traceable to the SI.

The work within T2.J11 is organized in 6 WPs with a budget of 3,7 M€ and a work effort of 249 MM.

# 8 – Report from an iMERA stakeholders workshop Borås (SE), 18<sup>th</sup> October 2007, Metrology Needs and Measurement Priorities in the Environmental Sector

*Bertil Magnusson* (SP) informs the community about the metrological needs in the environmental sector.

The stakeholders presented review-type talks arranged into a series of five sessions covering water monitoring, air and particle emission monitoring, remote sensing and energy & the environment. The key findings have been divided into four environmental categories commonly used by researchers in the environmental sector *air*, *water*, *soil* and *the biosphere*. Related to the air environment, there were two major areas of concern raised during the workshop: 1. *Climate modelling & global warming* and 2. *Air quality, including particulate sensing.* 

It should be noted that these requirements are representative of the stakeholders present, and other views may exist in the wider community.

# 9 – Report from an iMERA stakeholders workshop Amsterdam (NL), 6-7 June 2007, Metrology Needs and Measurement Priorities in the Energy Sector

*Hans Bachmair* (PTB) informs the community about the metrological needs in the environmental sector.

The stakeholders identified metrological needs in the energy categories *traditional energy sources* (fossil fuels, electrical energy), *nuclear energy* (fission and fusion), *LNG, CNG & natural gas* and *renewable energy sources* (bio fuel, bio gas, wind etc.).

It is expected that wind energy will have the biggest increase, Photovoltaic, for which the metrology must be improved will come up on a mid-term scale. For bio-gas and bio-fuels, on-line measuring methods are needed.

#### **10 – Reports on CCQM WG meetings**

*Michal Máriássy* (SMU) reports on the activities of the CCQM WG on Electrochemical Analysis.

Meetings were held at BIPM (Sevres, 16 April, 17 participants) and NIST Hollings Marine Laboratory (Charleston, 1-2 October, 11 participants).

The results of CCQM K9.2 (pH of phosphate buffer) and K18.1 (pH of carbonate buffer) are presented and discussed. Runing studies and comparisons are K36.1 (electrolytic conductivity), K20 (pH of oxalate buffer) and K48 (assay of KCI).

New studies and comparisons are agreed and planned addressing seawater (P111), conductivity @ 0.5 mS/m (P83), pH 7 preparation study (P93), pH (P37.1), and HCI (P19.2).

Technical presentations adresses different issues of electrochemical analysis such as an approach to solve electrode problems (INMETRO), the potential of stripping voltammetry as a primary method (NPL), the metrology of ion activities (METAS), headspace effects in preparation of carbonate buffer (NIST) and the analysis of CCQM-K18.1 NIM results (NIM). *Mike Sargent* (LGC) reports on the activities of the CCQM WG on Inorganic Analysis.

Meetings were held at BIPM (Sevres, 16-17 April) and NIST Hollings Marine Laboratory (Charleston, 1-3 October). In conjunction with these meetings, workshops on use of NAA in chemical metrology and on metrological applications of multi-collector ICP-MS were held.

Good progress is made with comparisons and studies and strategy development such as the *Benchmarking feasibility study*. The objectives are to benchmark equivalence between all IAWG laboratories through participation in the same comparison and to demonstrate that NMIs achieve good results even with analyses outside their existing experience (*how far does the light shine for an NMI?*)

The establishment of a so-called *Core Competence Matrix* was discussed as a systematic way to summarise how far the light shines for each KC and the competencies required to deliver each CMC.

*Martin Milton* (NPL) reports on the activities and recent progress of the CCQM WG on Gas Analysis.

The last meeting was held in Sydney (October 2007) which also includes a strategy workshop addressing the *consistent approach to the calculation of key comparison* reference values and a systematic approach to planning key comparisons and to underpinning present and future CMCs.

An overview of the GAWG Key Comparisons is given. Many of these comparisons are of species for which there is no special analytical challenge. It is asked whether the performance in these "core" comparisons is a long-term measure of the performance of an NMI and if the performance in these "core" key comparisons can be linked to a corresponding set of "core CMCs"? Future project proposals are presented which address these questions.

Since its foundation the GAWG members have delivered 1000 Key Comparison results and generated 1500 CMC claims. Hence, a re-review in a conventional manner cannot be performed and future activities are planned on a "case-by-case" basis.

*Franz Ulberth* (IRMM) reports on the activities of the CCQM WG on Organic Analysis.

Meetings were held at BIPM (Sevres, 16-17 April, 35 participants) and PTB (Braunschweig, 8-10 October, 30 participants). In conjunction with the latter meeting, a symposium on "*New cutting Edge Research relevant to Delivery of Measurement Services*" was held.

A discussion on "how far does the light shine" ends up in a general agreement to reduce number of P/K-studies and with the intention to establish a priority list from each institution that include the studies required to underpin the core competencies.

The studies finalized are K47 (VOCs in solvent), P20f (theophylline purity), P54.1 (mass fraction of a defined 20mer oligonucleotide), K11.1 and K12.1 (glucose and creatinine in serum), K27.2 (ethanol in aqueous matrix), K50a.b (P69.1, PAHs in soil/sediment/particulate), P88 (malachite green in fish), and P90 (chloramphenicol in milk).

Studies still in progress are K55a (b-estradiol purity), P91 (pyrethroids in apple juice, K63a.b (progesterone and cortisol in serum), and P109 (acrylamide in potato chips).

New studies are proposed on anabolic steroids in urine (K) and brominated flame retardants in plastic (P).

Helen Parks (LGC) reports on the activities of the CCQM WG on Bioanalysis.

The BAWG is the fastest growing working group within CCQM having now 43 participants from all continents. Among these, Europe is currently under-represented.

BAWG laboratories have made considerable steps forward on the roadmap to establish a biomeasurement reference measurement system (RMS). This is underpinned by the results of several pilot studies and a first key comparison (K61, quantitative PCR) whose results are expected to serve as a basis for CMC claims in the near future.

Furthermore, the BAWG strategic issues as well as the widespread Co-ordination with other International Measurement & Standardisation activities are pointed out.

#### 11 – Information on activity of the JCTLM

*Robert Wielgosz* reports on the activities of the JCTLM.

A quality assured database of *Higher Order Reference Materials*, *Reference Measurement Procedures* and *Laboratory Reference Measurement Services* has been established which received an average of more than 700 visits per month.

He reminds on the necessity for the NMIs to intensify their support for the JCTLM working group activities.

#### 12 – Mandate of Convenors

The chairman introduces **Rob Wessel** as the new convenor of the technical subcommittee on Gas Analysis. He followed **Martin Milton** who took over the chair of the Gas Analysis Working Group (GAWG) of CCQM.

The convenors of the TC MC subcommittees are as follows:

GAS:	Rob Wessel (NMi-VSL, NL)	
Organic:	Franz Ulbert (IRMM, EU, until 2009)	
Inorganic:	Christophe Quétel (IRMM, EU, until 2010)	
Electrochemistry:	<i>Petra Spitzer</i> (PTB, DE, until 2011)	

The Chairman thanks the convenors for their work and suggested a staggered ending of terms to ensure a high degree of continuity within the following years.

#### 13 – Upcoming Meetings

**Rob Wessel** announces the 5<sup>th</sup> International Gas Analysis Symposium (GA S 2009) which is held from 11 to 13 of February 2009 in Rotterdam.

#### 14 – Any other business

*Bertil Magnussen* reports about news and changes in the terminology in measurements, which are introduced in the latest VIM-release ("VIM3").

Florbela Dias informs the assembly on new pH-measurement activities at IPQ.

#### 15 – Next Meeting February 2009 Bukarest (ROM) and Closure

The next Annual Meeting of the TC-MC will be from 03 to 06 of February 2009 in Bucharest, Romania.

#### Attendees

Nr.	Name	Institute	E-Mail
1	Akcadag, Fatma	UME, Turkey	fatma.akcadag@ume.tubitak.gov.tr
2	Andres, Hanspeter	METAS, Schweiz	hanspeter.andres@metas.ch
3	Ataman, Yavuz	Middle East Technical University Ankara, Turkey	ataman@metu.edu.tr
4	Bachmair, Hans	PTB, Germany	hans.bachmair@ptb.de
5	Baytarogle, Sakir	UME, Turkey	sakir.baytarogle@ume.tubitak.gov.tr
6	Buzoianu, Mirella	INM, Romania	mirella.buzoianu@inm.ro
7	Cankur, Oktay	UME, Turkey	oktay.cankur@ume.tubitak.gov.tr
8	Charlet, Philippe	LNE, France	philippe.charlet@Ine.fr
9	del Campo, Dolores	CEM, Spain	ddelcampo@cem.mityc.es
10	Dias, Florbela	IPQ, Portugal	florbelad@mail.ipq.pt
11	Durbiano, Francesca	INRIM, Italy	f.durbiano@inrim.it
12	Fisicaro, Paola	LNE, France	paola.fisicaro@Ine.fr
13	Güttler, Bernd	PTB, Germany	bernd.guettler@ptb.de
14	Halbo, Leif	Justervesenet; Norway	leif.halbo@justervesenet.no
15	Heine, Hans-Joachim	BAM, Germany	hans-joachim.heine@bam.de
16	Hirvi, Timo	MIKES, Finland	timo.hirvi@mikes.fi
17	Ivanova, Dimka	NCM-BIM, Bulgaria	d.ivanova@bim.government.bg
18	Jalukse, Lauri	Uni Tartu, Estonia	lauri.jalukse@ut.ee
19	Kaarls, Robert	CIPM/CCQM, Netherlands	rkaarls@euronet.nl
20	Kipphardt, Heinrich	BAM, Germany	heinrich.kipphardt@bam.de
21	Koch, Hans	PTB, Germany	hans.koch@ptb.de
22	Koeber, Robert	JRC-IRMM, Belgium	robert.koeber@ec.europa.eu
23	Kozlowski, Wladyslaw	GUM, Poland	chemistry_wk@gum.gov.pl
24	Majcen, Nineta	MIRS Ljubljana, Slovenia	nineta.majcen@gov.si
25	Máriássy, Michal	SMU, Slovakia	mariassy@smu.gov.sk
26	Milton, Martin	NPL, UK	martin.milton@npl.co.uk
27	Naujalis, Evaldas	SPI, Lithuania	naujalis@pfi.lt
28	Nikolic, Dragan	Directorate of Measures and Precious Metals, Serbia	dragan.nikolic@szmdm.sv.gov.yu
29	Pätoprstý, Viliam	SMU, Slovakia	patoprsty@smu.gov.sk
30	Palner Jacobsen, Keld	Danak, Denmark	kpj@danak.dk
31	Quetel, Christophe	IRMM, Belgium	Christophe.QUETEL@ec.europa.eu
32	Sargent, Michael	LGC, UK	ms@lgc.co.uk
33	Sassi, Mariapaola	INRIM, Italy	m.sassi@inrim.it
34	Sega, Michela	INRIM, Italy	m.sega@inrim.it
35	Spitzer, Petra	PTB, Germany	petra.spitzer@ptb.de

36	Stokstad, Ellen	Justervesenet, Norway	ellen.stokstad@justervesenet.no
37	Stosch, Rainer	PTB, Germany	rainer.stosch@ptb.de
38	Süer, Sermet	UME, Turkey	sermet.suer@ume.tubitak.gov.tr
39	Szilágyi, Zsófia Nagyné	MKEH, Hungary	sz.nagy@omh.hu
40	Taylor, Philip	JRC-IRMM, Belgium	Philip.taylor@ec.europa.eu
41	Ulberth, Franz	JRC-IRMM, Belgium	franz.ulberth@ec.europa.eu
42	Van Theemsche, Achim	SMD, Belgium	achim.vantheemsche@economie.fgov.be
43	Vukadin, Predrag	Directorate of Measures and Precious Metals, Serbia	vukadin@szmdm.sv.gov.yu
44	Wessel, Rob	NMi-VSL, Netherlands	rwessel@nmi.nl
45	Wielgosz, Robert	BIPM, France	rwielgosz@bipm.org
46	Win, Tin	BAM, Germany	tin.win@bam.de
47	Wunderli, Samuel	METAS, Switzerland	samuel.wunderli@metas.ch
48	Yalcin, Temel	UME, Turkey	temel.yalcin@ume.tubitak.gov.tr