



# Subject field: METCHEM – Metrology in Chemistry

## Annual Report

### 1- Introduction

The annual meeting of the Technical Committee METCHEM takes place every year in February, so this annual report covers an extended period between March 2005 and March 2006, to present the main activities and to discuss issues and outcomes encountered in this period.

### 2- Most important issues and outcomes

#### 2.1- Nomination of Sub-Committee convenors

Following EUROMET's rules, that is to say: appointment of convenors is for a period of 2 years with two consecutive terms of office maximum, ending at the General Assembly in even number years; it has been considered that the meeting of METCHEM in 2004 (Sofia, Bulgaria), was a benchmark regarding appointments of convenors. At that time, Christophe Quézel from IRMM took over Ralf Matschat (BAM) for the inorganic working group, Franz Ulberth (IRMM) replaced Bernd Gawlik for the organic activities, and Peter Woods (NPL) and Michal Mariassy (SMU) started their second term. Therefore 2006 is a nomination year, during METCHEM Plenary in February and it was decided to formally appoint or renew convenors.

One of the convenors, Peter Woods decided to leave his duty and Martin Milton (NPL) has been designed to take over him, in August 2005, for the Gas analysis sub-committee.

Still following rules of procedure, the MetChem TC chair has proposed to the delegates proposals for the three remaining sub-committees:

- **Inorganic SC:** Christophe Quézel (IRMM): proposed for a second term
- **Organic SC:** Franz Ulberth (IRMM): proposed for a second term
- **Electrochemistry SC:** Petra Spitzer (PTB) proposed for a first term
- **Gas SC:** Martin Milton (NPL) took over from Peter Woods (NPL) in August 05
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This proposal has been fully accepted by the delegates, and the EUROMET Chair has then nominated the new Convenors.

Considering that the change of Chairperson has unleashed some understanding problems two years ago between the two partners of METCHEM: EUROMET and

EURACHEM, it was expected comments on the proposals for convenorship. But it was a satisfaction to note that everyone agrees on names and institutes.

## **2.2- Role of Sub-Committee Convenors in the CMCs review process**

The internal review concerning CMCs claims proposals of the new cycle (Cycle VII) has been initiated early in December 2005. The convenors were responsible to collate the claims and conduct the review with the objective of having EUROMET CMCs claims internally agreed for the 1<sup>st</sup> of March 2006 strict deadline (sending of files to JCRB and KCWG of CCQM).

The TC Chair asked the convenors of the four Sub-Committees to directly contact NMIs of their field of activities, to prepare proposals, to collate them and to initiate the review.

In the inorganic sub-committee, the convenor set-up a specific web portal. This allows NMIs to directly upload their files. This initiative was a convenient way to have a very open and transparent process for the review: all claims are presented to experts; all comments and responses are visible.

The objective of this initial review was to present finalized claims for the sub-committee meetings in mid-February and to dedicate time to “difficult” claims that require specific discussions. The TC chair adopted a very simple position regarding the official EUROMET review of claims: the deep work of evaluation of the relevance of claims is carried out by convenors. Then the different sub-committees decide which claims have to be forwarded for the inter RMO review at CCQM. The TC chair absolutely relied on the position expressed by the respective convenors, after agreement of the sub-committees, taking into account that the review is performed following criteria of JCRB regarding evidence to support claims.

This year, cycle VII 2006, a relatively large number of claims have been presented: 151 claims, by 9 NMIs.

During METCHEM, a question raised whether it was possible for Ukraine metrology institute to submit claims (in the field of electrochemistry) through EUROMET, considering that the Quality System was assessed by COOMET, and that Ukraine is a EUROMET corresponding applicant. While waiting for the orientation of the TC Quality of EUROMET, it was decided to forward these claims to KCWG and JCRB. During the TC chairs meeting in March, the EUROMET Chair informed METCHEM TC chair that it is not possible for EUROMET “to endorse” these claims. Therefore, this set of claims was withdrawn from METCHEM CMCs files.

At the end of February, the President of COOMET asked METCHEM TC Chair for assistance in the review of COOMET claims for cycle VII, due to the lack of experts at COOMET. This was accepted but only as recommendation and not official review. The METCHEM convenors carried out the review on a voluntary basis. Considering the large number of claims to be reviewed (more than 150), it was agreed that the review will be performed only on claims supported by some evidence. Recommendations were sent to COOMET on time for presentation to KCWG. President of COOMET and COOMET Chemistry TC Chair have thanked EUROMET experts for their help in reviewing claims.

## **2.3- Meetings**

For the third time, the meetings of the four sub-committees were spread over two days in order to allow specialists to attend 2 or 3 SC meetings (instead of 1 or 2

when SC meetings are organized in parallel). This was again deemed very positive and created stimulating exchanges, discussions and proposals.

In 2006, the METCHEM meeting plenary was held at PFI, Vilnius, Lithuania, on February 16<sup>th</sup> and 17<sup>th</sup>. Two sub-committee meetings were organised on February 14<sup>th</sup> (organic and inorganic) and two others (electrochemistry and gas analysis) on February 15<sup>th</sup>. Visits of PFI facilities were planned during these two days.

49 persons attended the plenary meeting; they were from 28 European countries (+IRMM).

During the Plenary Session, a follow up to last year forum discussion on the **“Strategies of NMIs to ensure adequate metrology capabilities to users”** was given by presentations on European databases on Certified Reference Materials (CRMs) and Proficiency Testing Schemes (PTS). CRMs and PTS are very important tools in chemical metrology to ensure traceability and comparability of measurements, and therefore it was important to pass information to Metchem delegates.

Furthermore, it has been asked to EURACHEM to give an overview of the activities of the Proficiency Testing Working Group and to synthesize the outcome of the European project CoEPT related to working protocols of PT providers.

- Thomas Steiger from BAM presented the database on Certified Reference Materials, COMAR, for which BAM assures the General Secretariat.

Since March 2003, the access to the database is free of charge, BAM assuring the secretariat on its own funds. The MoU related to the database has been reviewed in 2005. At the present time COMAR contains about 10 985 CRM from 214 producers in 26 countries. A great number of National Metrology Institutes participate in the elaboration of COMAR database as coding centres, their work consists to update available information and to maintain the permanent process of review from each CRM. COMAR website: [www.comar.bam.de](http://www.comar.bam.de)

- Brian Brookman from LGC reported on the proficiency activities in Eurachem.

Eurachem developed some activities to promote best practices in proficiency testing with the organisation of forums and workshops, and the dissemination of knowledge by publications, for example. The working group is open to any person interested in the project. Eurachem published a specific guide in English, and EPTIS a leaflet on proficiency test and with several translations in different languages.

Only a very percentage of providers are accredited and they should normally all be accredited, but it seems that nobody pushes them in this way, and the cost is a barrier with no doubt. Information can be found on [www.eptis.bam.de](http://www.eptis.bam.de)

- Brian Brookman then reported on the outcomes of CoEPT project. CoEPT was a project funded by the European Commission on “Comparability of the operation and Evaluation protocols of European Proficiency Testing schemes”. It was led by LGC and consisted in assessing comparability of proficiency test schemes in Europe in four selective sectors. Results of the first comparisons performed in the different sectors were presented. Those comparisons shown a good agreement evaluation between providers, generally in the range of 70 % to 80 % depending on the case. Results have been published on EPTIS web site. Other comparisons have been performed on real CRMs.

- Brian Brookman continued to report on EPTIS. The proficiency testing database has been established in 1998 with 16 partners. In 2004, the database became an international database including 800 PT schemes, and with a consortium of 20 partners. BAM assures the EPTIS secretariat. The database tries to be a complete

database on PT schemes. It is indicated that a very small proportion of PT scheme entries are accredited, and the real proportion is quite unknown at the present time.

- Philip Taylor from IRMM took stock on the status of European training and education initiatives: TrainMic and AcadeMic and presented the activities on training in chemistry.

Trainings involved NMIs, Universities, and National Accreditation Bodies, mainly from East European countries. IRMM staff performed TrainMic. Globally since 2001, about 2300 persons have been trained. In 2005, IRMM performed some training of trainers for teams from Bulgaria, Czech republic, Estonia, Poland, Romania and Slovenia. Another activity in 2005 was AcadeMic project to improve teaching at Universities, and exchange best practices. The public was professors from Universities (and not students) from different countries.

There is no plan for training on microbiology at the moment.

Considering that METCHEM meeting was held in Lithuania this year, Suzanne Niemitz-Bolle from PTB presented the project between BAM - PTB and VMT on the development of reliable infrastructure from metrology in chemistry according to the EU best practices.

BAM and PTB in the frame of a EU-PHARE technical assistance project, a twinning project, developed a reliable infrastructure for metrology in chemistry in Lithuania according to EU best practices. This project consisted in defining a national strategy for Lithuania in chemistry with collaboration between private and public organisation and to develop the participation in Euromet projects.

Next METCHEM meeting: 6 to 9 of February 2006, IPQ, Lisbon, Portugal.

## **2.4- Status of Projects and new co-operation developments**

- Electrochemistry- Michal Mariassy

# 843 : on pH, a questionnaire has been sent, and 13 % of the labs replied.

Michal Mariassy presents the results of the questionnaire.

There is two new projects proposed :

- on calibration of conductivity meter at pure water level with SP, PTB, DFM and LNE.

- on sea water for the measurement of salinity, a to define a practical seawater standard.

- Inorganic - Christophe Quénel

# 763 : the project is completed and the draft report available. Final report will be sent shortly.

There is no project proposed at the present time.

A discussion starts on the directive on water policy, which seems very ambitious taking into account the concentration level. PTB and LNE expressed their interest on the possibility to introduce a proposal for a METCHEM project to coordinate effort, because everybody will be concern by this directive. Different NMIs show their interest to participate in such a project to demonstrate what can be possible within NMIs.

- Organic - Franz Ulberth

Two projects are proposed in this field :

- a project mainly on PCB determination.

- determination of nitrogen compound in cereal (wheat, corn, etc.)

- Gas analysis - Martin Milton

# 880 : on vehicle emission standards. Project completed.

At the present time there are four active projects :

- # 883 : vehicle emission comparison

- # 867 : purity comparison. Problem in gas at the present time, results probably next year.

- # 853 : improvement of technical standards. On going project.

- # 708 : Bilateral comparison. On going project. No progress this year.

There is four new proposed projects :

- on nano- particle research which will consist in a collaborative R&D project to monitor and control the presence of particles and to improve measurements. At least 7 countries would like to participate in this project.

- comparison in ozone and VOC in collaboration with AQUILA; At least 10 participants interested in this project. .

- low concentration of SO<sub>2</sub> and NO.

- dynamic methods. It is proposed to organise a workshop on dynamic methods for ambient measurements.

### **3- Problems/issues encountered**

It is still very difficult for the TC Chair to clearly identify the representatives of EURACHEM at the Technical Committee. This question has been raised officially in the past, during EURACHEM GA, but was not solved yet. This situation complicates contacts and the transmission of information. The number of attendees at METCHEM Plenary became important (around 50) that it is difficult to have an interactive meeting and it is also now impossible to know if a person speaks on behalf of his country, on behalf of EURACHEM or on behalf of himself. An action has to be engaged to clarify the representation of delegates of EURACHEM but also from EUROMET.

Nevertheless, thanks to the presentation on Proficiency Testing Schemes, EURACHEM did participate actively this year in METCHEM Plenary. This creates exchanges and helped to have a lively meeting. This experience has to be renewed next year.

As usual, the major issue this year was related to RMOs CMCs claims review despite the fact that the implementation of the process review done by Mr. Mac Laren was a great help for the whole community.

For 2005 Cycle VI :

The final CMC files were posted November 15, 2005 , with the deadline for RMO approval on December 23, 2005. The revised final deadline was January 23, 2006.

At that time, 57 CMCs were submitted, 19 approved and 39 to be discussed.

It should be noted that CMCs of Cycle III from BAM were again lost, and have to be entered again on the review process. Some BAM claims were not immediately approved on porosimetry because of lack of international support.

A critical point was due to the fact that SIM rejected Euromet files considering that two claims from PTB were not acceptable. The main problem was that SIM did not participate at any time on the review process, and presented its point of view just at

the final day of review process (actually, the day after deadline). As a consensus, PTB postponed the two claims that have been “differed”.

The METCHEM Technical Committee has considered the position of SIM as totally unfair, and SIM normally had to inform on time about the possible issue regarding these two claims. Robert Kaarls added that normally a RMO should not block the situation and agreed with Euromet position.

To conclude, the Euromet Cycle CMCs have been approved very lately (in April 2006) while the next cycle already started.

The positive point is that the review process contributes to a constructive exchange and discussions between RMOs, with a good review process. Claudine Thomas and Pedro Espina, from BIPM have to be thanked, for their availability, helpful advices and information.

A discussion raised during METCHEM Plenary on the way roadmaps, for the iMERA project have been elaborated, and some of the attendees expressed their feeling of a “non-democratic” process. It was answered that in the iMERA project, the elaboration of the roadmaps was on the responsibility of the Euromet TC Chairs. Every body is fully aware of the huge work of this exercise and it was added that people had not to focus on roadmaps that is only a part of iMERA project. This exercise is to give an idea of what could be the scientific strategy for Euromet.

#### **4- Mutual Recognition Arrangement**

European NMIs are used to participating in CCQM activities and are often coordinating CCQM comparisons. CCQM Working Groups are mainly chaired by European Convenors (Inorganic by LGC, Electrochemistry by SMU; Gas by NMI, Bioanalysis by LGC, Surface analysis by BAM) and these convenors are also very involved and active in METCHEM activities.

In 2005 and beginning of 2006, a large part of the activities of the Technical Committee was devoted to the review and the submission of the next CMCs Cycle (Cycle VII) but also to the finalisation of the previous cycle (due to problems exposed above). 151 new and revised claims have been proposed by 9 NMIs covering 11 categories, that is to say the majority of chemical fields with a large diversity of measurands and matrices. EUROMET experts in the 4 Sub-Committees have approved these claims. These proposals were discussed at the next inter RMOs meeting in April 2006 (KCWG meeting), at the BIPM.

The set of claims discussed during 2005 and approved for the most part (exception of two claims differed) have been published in the BIPM database in April 2006.

Valuable contacts with the other Regional Metrology Organisations and the CCQM were maintained at a high level (meetings, exchange of information, invitation to seminars and events, etc.). LNE has hosted two working groups meetings in Paris in April 2005 for the annual CCQM meetings.

Furthermore, METCHEM is fully involved (TC chair) in the ad hoc working group of BIPM which is currently elaborating a strategy regarding activities to be carry out at the CIPM level on Materials Metrology.

Very valuable contacts have also been initiated with CITAC (Cooperation on International Traceability in Analytical Chemistry). The 2005 annual meeting of CITAC was organized by METCHEM at LNE in Paris in April 2005.

## 5- Research / Co-operation Trends

Several cooperative projects are currently prepared within METCHEM. One, regarding *in situ* measurement of pH is conducted in the framework of iMERA as a demonstration case. Some others, related to nanoparticles and water directives will be elaborated in 2006 and will mainly concern cooperative research between European NMIs.

### **iMERA**

In the frame of the i-MERA project, and in view of the elaboration of an European Research Metrology Programme (ERMP), METCHEM TC chair and convenors were asked to elaborate roadmaps for the different activities of their groups. This work involved strongly the METCHEM convenors and some additional experts a bioscience group has been formed to elaborate a roadmap in this subject, group including LGC, LNE, IRMM, NMi, State Laboratory and PTB, under the coordination of METCHEM.

A meeting was held at IRMM in December 2005 for the elaboration of the first draft of chemical roadmaps and another meeting was organized in January 2006 at LGC for the Biosciences roadmaps. The philosophy of these roadmaps is based on a top-down approach, that is to say, starting from well-agreed societal issues (environment, health, protection of the European citizen, ...) and working of analytical chemistry and metrology solutions.

Overlapping between chemistry roadmaps, biosciences roadmaps and life sciences roadmaps (elaborated by a specific Intmet focus group) could unfortunately not be solved between the group experts.

METCHEM TC Chair also participated in the elaboration of the roadmaps in the Intmet focus group on Metrology for New materials.

Philippe Charlet, April 2006