Subject field: METCHEM – Metrology in Chemistry

Annual Report

1- Introduction

Considering that the annual METCHEM meetings take place every year in February, this report covers an extended period between March 2004 and March 2005, in order to present the main activities and to discuss issues and outcomes encountered in this period.

2- Most important issues and outcomes

2.1- Re-Election of the Chair

At the end of 2004, the EUROMET Secretary informed the TC Chair about the end of his first mandate and asked about his wish to continue his duty for another two years term. 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 asked to the main actors of METCHEM to give their opinion about the re-election of the current TC Chair. The French EUROMET delegate, the four Sub-committee convenors and the President of EURACHEM have been approached on this subject. It was a satisfaction to note that everyone agree about a continuation of this mandate. Apart from the question of the EURACHEM representation at METCHEM TC (see "problems/issues encountered") the relationship with this partner are excellent. Nevertheless, links have to be strengthened between the two organisations to provide more common activities within METCHEM.

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

The discussion about CMCs claims proposals has been initiated early in December 2004 in order to be sent to JCRB for Cycle VI review, respecting the 1st of March 2005 strict deadline.
The TC Chair asked the convenors of the four Sub-Committees to contact NMIs of their field of activities to prepare proposals and to start the review. The objective was to present about finalized claims for the sub-committee meetings in mid-February. 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, taking into account that the review is performed following criteria of JCRB regarding evidence to support claims.
During the Cycle VI review, it appeared that some convenors reviewed and accepted claims based on evidence such as comparisons (CCQM or EUROMET or others) peer review papers, scientific documents, certificates, reports, etc. But one convenor, on behalf of his group, was much more "flexible" and decided to accept claims without any kind of evidence, but considering that the reputation of the NMI was sufficient to be confident on the quality of their claims. At the end of the process, a NMI had its claims in one area rejected for lack of evidence and some other claims in another area accepted without evidence. This issue has been raised at the Plenary but no agreement was reached between delegates.

2.3- Meetings

In 2004, the Plenary METCHEM meeting was held at NCM, Sofia, Bulgaria, on February 12th and 13th . 43 persons attended the meeting. It has become customary that the four subcommittees hold their meeting the days before the plenary session, the convenors then report to the TC. For the first 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 deemed very positive and created stimulating exchanges, discussions and proposals. It was then decided to keep the same organisation for the 2005 meeting.

In 2005, the METCHEM meeting (plenary and sub-committees) was held at CEM, Três Cantos, Madrid, Spain, on February 17th and 18th. Two sub-committee meetings were organised on February 15th (organic and inorganic) and two others (electrochemistry and gas analysis) on February 16th. Visits of CEM facilities were planned during these two days. 52 persons attended the plenary meeting, they were from 24 European countries (+IRMM) and they were two guests from Brazil (IPT) and Taiwan (ITRI).

During the Plenary Session, Philip Taylor, from IRMM, gave a follow up to the last year's forum discussion on the **"Strategies of NMIs to ensure adequate metrology capabilities to users"**.
Two case-studies were presented: the experience of MIRS (Slovenia) on Distributed Metrology with the Slovenian National Institute of Chemistry; and the experience of SP (Sweden) regarding a program of Training and Education in Metrology in Chemistry.
The President of CITAC (Cooperation on International Traceability in Analytical Chemistry), Vera Ponçano, has been invited to present her new strategy regarding the CITAC cooperation with RMO for the dissemination of traceability concepts for chemical measurements. She also presented the Brazilian programme of Metrology in Chemistry, its projects and its connections with CITAC. This is a good example for European countries to learn how to set up networks with PT providers, end-users, reference laboratories, reference materials producers, etc.

It was concluded that this forum will continue to address specific issues of common interest of NMIs but a specific working group will be created to maintain contacts all along the year to stimulate exchanges between participants.

Another Forum discussion was launched during the Plenary on "Do we need to include bio-analysis measurements in Metchem activities" ? This question was

raised last year during the EURACHEM GA. Considering the very important developments of analytical chemistry in the field of biology and medicine, it was decided to evaluate the opportunity to set up a specific sub-committee at METCHEM. There is a clear need of introducing more “metrology concepts” in this field (in terms of traceability to the SI, uncertainty, method validation, reference materials, etc.).

Helen Parkes (LGC), the current Chair of the Bioanalysis WG at CCQM was invited to present the measurements needs at the present time in the bio-analysis field and the activity of LGC in this domain.
The presentation was followed by a discussion on possible mechanisms to put in place in view to reach expert laboratories in bio-analysis, and on the role that can be played by Metchem. The main question is to inform those laboratories of their metrology needs and to convince them that metrological approach in an added value. Culture and background are really different between bio-analysis community and NMIs, NMIs are perceived as “SI specialists” only. This creates kind of barriers in communication and has to be first overcome. Concerning the creation of a specific and formal working group on bio-analysis in Metchem, Helen Parkes is not in favour of setting up such a sub-committee due to the fact that no specific European needs were identified. Participants did not challenge this position. It was then decided that Metchem won’t go further on this matter.

Next METCHEM meeting: 14-17 February 2005, PFI, Vilnius, Lithuania.

2.4- Status of Projects and new co-operation developments

- On-going and completed projects:

563 : the project is finished but has never been closed until now. The final report has been sent. The project is completed.

763 : the project is delayed to the end of February 2005. Lithuania will be interested to participate in the project, but it seems to be late to include it in the project.

784 : the project is cancelled.

785 : project related to the CCQM-K35. The final report has been sent. The project is completed.

724 : the project is finished and the final report have to be sent. Action : BAM

708 : the project consists in a comparison between NIST and NMi every year to underpin NMi’s claims. On going project.

764 : the project is finished. The final has to be sent. Action : NMi.

833 : second step of the project PCBs congeners in organic solutions

- Projects proposals

- Inorganic: - Cu calibration solution (EUROMET QM-K8)

- Organic: - none

- Electrochemistry: - Recommendation for the calibration and evaluation of pH on site measuring
  - Conductivity at pure water level

- Gas: - Preliminary study on purity
  - Vehicle emission comparison
  - Vehicle emission bilateral comparison
  - HCl bilateral comparison

3- Problems/issues encountered

Due to the lack of "working rules" with the METCHEM partner EURACHEM, which was observed last year, the Terms of Reference of METCHEM have been revised during the last EUROMET GA. The new ToR have been sent to the President of EURACHEM and accepted at the beginning of 2005. The new ToR were presented to the METCHEM delegates during the last Plenary meeting but it didn't give rise to any comments.

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 during the EURACHEM GA in 2004 but was not solved yet. This situation complicates contacts and the transmission of information. It was particularly critical this year during the METCHEM Plenary. The number of attendees became so important (more than 52) that it is difficult to have an interactive meeting and it is also now impossible if a person speaks on behalf of his country, on behalf of EURACHEM or on behalf of himself. An action will be engaged to demand EURACHEM to clarify its representation. .

A major problem was encountered regarding CMCs review with the other RMOs.
During 2004, the review covered Cycles III, IV and V QM CMCs with a strict agreed agenda::
- April 2004 : agreement on agenda for CMCs with other RMOs, during the KCWG at CCQM.
- 1st of October 2004 : deadline for comments.
- November 2004 : RMOs should submit their final CMC files.

Euromet-Metchem and SADCMET submitted their final claims on time, at the beginning of November 2004, but final files were submitted by some other RMOs very lately (SIM / December 20, 2004 ; APMP / January 10, 2005; for example) ; furthermore:
- Review of Euromet CMCs were not totally approved by the other RMOs in the planned deadline ;
- Inappropriate comments were received from some RMOs ;
- Lack of experts within Euromet-Metchem to review claims for other RMOs.

Status on the different Cycles QM CMCs, in 2004.
- Cycle III : some last CMCs from BAM (DE) never reached the BIPM database. The problem has now been solved. 25 claims
- Cycle IV : many countries were concerned by claims. A few countries have CMCs postponed or withdrawn. Total cycle IV: 167; 78 withdrawned or postponed.

- Cycle V (2004) : the group was not able to provide evidence of some CMCs (Romania, Spain, Portugal). For two NMIs, comments were very general statements on “how far does the light shine” which were inappropriate at this phase of the review process. Those CMCs are postponed or withdrawn, as a concession of EUROMET. Total cycle V: 68; 12 postponed
The total CMCs claims for EUROMET QM was 350 in 2004

This problem related to CMC claims review has been touched on during the METCHEM Plenary and discussed with the President of CCQM.

Nevertheless, it is important to stress that the new process put in place by JCRB (new KCWG, swift contacts and information by emails, clear and updated JCRB web site, availability of JCRB secretary) proved to be very efficient.

4- Mutual Recognition Arrangement

For years, European NMIs are used to participating in CCQM pilot and key comparisons and several of them are coordinating current comparisons. The majority of the CCQM Working Groups are chaired by a European Convenors (Inorganic by LGC, Electrochemistry by SMU; Gas by NPL, Bioanalysis by LGC, Surface analysis by NPL) and these convenors are sometimes very involved and active in METCHEM activities.
In 2004 and beginning of 2005, a large part of the activities of the Technical Committee was devoted to the review and the submission of the next CMCs Cycle (Cycle VI). 55 new and revised claims have been proposed by 10 NMIs from 8 countries, covering 8 categories. EUROMET experts in the 4 Sub-Committees have approved these claims. These proposals will be discussed at the next inter RMOs meeting in April 2004, at the BIPM.
In January 2005, the CMCs entries from EUROMET QM have been published in the BIPM database. They corresponded to the previous cycles (Cycles IV and V) dating back to 2003 and 2004. 25 old CMCs (cycle III) are still in the process of final acceptance by RMOs. It is important to note that these previous claims have been accepted only because two RMOs lost their right to review these claims (after having passed the deadline).

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.) particularly under the impulsion of BIPM (Robert Wielgosz) and the President of CCQM, Dr Robert Kaarls. Very valuable contacts have also been initiated with CITAC (Cooperation on International Traceability in Analytical Chemistry) and EUROMET has been chosen as a “case-study” for the strategic dissemination of traceability concepts to end-users.

The TC Chair has been invited to present the activities of METCHEM at the International Conference of Metrology of South America, METROSUL IV, in Ocotber 2004.

5- Research / Co-operation Trends

Projects related to iMERA

During the forum discussion related to the search for models on "How transfer the appropriate metrology capabilities", two case-studies have been chosen to illustrate "success stories" in the field of the distributed metrology and training and education. These concrete experience are very useful to link NMIs to end-users. During METCHEM, a proposal was made by the convenor of the Inorganic Sub-Committee regarding the participation in METCHEM comparison. Based on the fact that NMIs cannot develop capabilities in all fields of chemistry and due to their weak links with reference laboratories and field laboratories, a new approach has been presented, **"Towards a more efficient use of EUROMET-METCHEM comparisons for Inorganic analyses".**
It is summarized as follows:
•No need for NMIs signatory of the MRA to take part to Euromet comparisons when identical CCQM comparisons (P and K) exist
•NMIs, quite logically, cannot claim capabilities about all measurement issues
•However, NMIs have a role to play in their national measurement infrastructure (coordination with National Reference Laboratories, dissemination of traceability, training etc .)
•Euromet-Metchem comparisons can be offered to National Reference Laboratories in order to demonstrate capabilities

It is then proposed that
- Euromet-Metchem comparisons for designated National Reference Laboratories in priority
- NMIs participate to CCQM comparison in priority
- National Reference Laboratories can evaluate their performance and level of equivalence internationally
- NMIs can play their role fully in the measurement infrastructure: traceability dissemination (CRMs + interface with international system), collaboration (support to implementation of legislation, training)
- CMCs come either from NMIs or from designated National Reference Laboratories

This proposal was well received by participants and it has been proposed to go ahead with this discussion, even if it is clear that some proposals of this approach look quite provocative.

Apart from the follow-up of the current forum discussion to identify the smart strategies to transfer metrology capabilities to end-users, a project can be considered as a good example of a co-operation between NMIs having different capabilities and facilities in the field of organic analysis.
This EUROMET project (n°833), **PCBs congeners in organic solution**, was initiated in 2003 with a questionnaire to canvass the activities of NMIs in Organic Analysis across Europe. This questionnaire has shown that 9 NMIs (on 22 which have responded) have plans to initiate organic analysis. Therefore, in 2004, an intercomparison was initiated between 7 NMIs on PCBs in organic solutions (PCBs are classic contaminants found in a lot of environmental compartments, such as water, soils, sediments, etc.). The objective of this "simple" exercice was to build up measurement capabilities gradually. Some very experimented NMIs have been

participating in this intercomparison in order to benchmarch high end capabilities. This project will enter in a second and third phases where more complex matrices will be studied, respectively extracts and real environmental matrices such as sediments. At the end of the project, some NMIs would acquire specific capabilities in a very short period through the assistance of experimented laboratories.

Philippe Charlet, March 2005