

Foreword by the Chairman CITAC's Accent on Cooperation



Summarizing CITAC progress over the term of my chairmanship, which is “suddenly” nearing completion, I would like first of all to congratulate the CITAC Award Winners – a good new tradition established during the last three years. The most interesting/important papers on metrology in chemistry (MiC) by the CITAC version 2009 are: Luca Callegaro et al. *Accred Qual Assur* (2009) 14:587-592; Samuel Wunderli and Hanspeter Andres. *Electroanalysis* (2009) 21:1984-1991; and Olaf Rienitz et al. *Accred Qual Assur* (2007) 12:615–622 (see the message of Dr. Wynand Louw, the Award Coordinator, and the contributions by the Winners in this issue of CITAC News).

Since CITAC is an international organization, its members speak English with different accents. However, I am now happy to report another accent developed in the last years by CITAC: the accent on cooperation with regional and international organizations active in the field of MiC. The purpose of this development was to increase the participation in CITAC activities of specialists from wide chemical analytical and metrological communities, thus increasing CITAC productivity.

Cooperation is an element of the CITAC acronym and one of the basic goals of the CITAC co-founders in 1993. The only problem is how to find optimal cooperation forms. In general, this task is described in CITAC Terms

of References 2007 and CITAC Strategy for 2007-2010. The strategy for realization of cooperation in practice included development of 1) CITAC membership, 2) a new CITAC logo, 3) guidelines for liaison persons, 4) a procedure for the annual CITAC nomination of the most interesting/important papers on MiC, 5) guidelines for CITAC projects, and 6) a procedure for CITAC participation in and support of conferences and workshops.

A number of leading MiC specialists from different sectors of different economies from all continents were elected during the last years as CITAC members. Today there are 37 CITAC members from 26 economies distributed by sectors as shown in Fig. 1. To emphasize the worldwide character of this cooperation, the new CITAC logo was depicted on a globe (see top of this page).

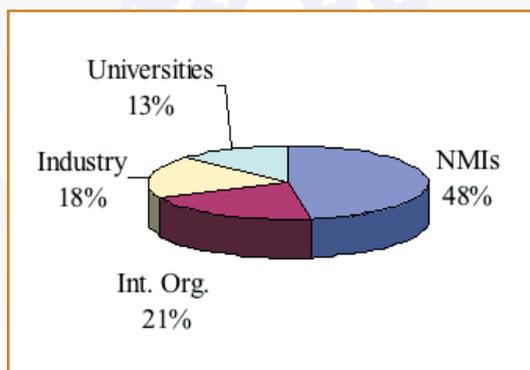


Fig.1. CITAC members' distribution by sectors. NMIs are national metrology institutes; Int. Org. are international organizations.

Liaison to regional and international organizations in the field of MiC (third parties) is the important aspect of CITAC activities. The Guidance on this topic outlines the duties and responsibilities of persons (liaisons) nominated to represent CITAC at meetings of other organizations, in working groups, etc. CITAC may liaise with third parties for a variety of reasons, such as negotiating an agreement,

formulating joint policies or documents, participating in affairs and acting as an observer at a meeting. A permanent CITAC liaison with a third party is organized on the basis of official CITAC membership or observer status, according to the policy of the third party. In particular, I am very satisfied by the decision of the International Committee for Weights and Measures (CIPM) at their 97th meeting in 2008 to accept CITAC as an observer of the Consultative Committee for Amount of Substance – Metrology in Chemistry (CCQM).

The annual CITAC nomination of the most interesting/important papers on MiC, mentioned above, aims to highlight remarkable papers which may have not yet been “discovered” after their publication in a national journal (in a local language); not readily accessible to the larger metrological/analytical community; published in a known international journal, but written in a sophisticated language, not clear enough for the community; or simply papers that deserve attention because of their important scientific content. This procedure, including lectures by the authors of the winning papers at the closed CITAC members meeting and author contributions to the CITAC News, has allowed us to annually attract a number of prominent colleagues to CITAC activities, and thereby to add color and positive emotions to these activities.

CITAC has a remarkable history of cooperation with EURACHEM in the development of guidelines in the field of MiC, well known and widely used in analytical laboratories throughout the world. Recently published documents are EURACHEM/CITAC Guide “Use of uncertainty in the assessment of compliance” and the EURACHEM/CITAC/EUROLAB/NORDTEST/AMC Guide “Estimation of measurement uncertainty arising from sampling”. In cooperation with IUPAC the first IUPAC/CITAC Guide “Selection and use of proficiency testing (PT) schemes for a limited number of participants” was developed and published in 2010. Another project with

Foreword by the Chairman

IUPAC, "Investigating out-of-specification (OOS) test results of chemical composition based on metrological concepts", is now under development. The Guidelines for CITAC projects will regulate planning and performing such projects with participation of specialists in MiC who do not need to be CITAC members.

Every year CITAC participates in the organization of international meetings in the fields of MiC and quality in analytical chemistry with local committees and other societies. For example, the International Congress on Traceability in Laboratory Measurements and Production Chains, São Paulo, Brazil, November 9-13, 2009 (V METROCHEM), the

Isranalytica 2010 conference, January 19-20, and the CITAC Workshop on January 21, Tel Aviv, Israel. Organizers of a meeting related to MiC may apply for an "In cooperation with CITAC" designation according to the Procedure for participation in and support of conferences and workshops by CITAC (the documents mentioned above are available in www.citac.cc).

In conclusion, one can say that the past three years have been productive, but of course a lot remains to be done. I would like to express my gratitude to the Executive Committee, which shared with me the multitude of CITAC problems during this term: Dr. Wynand

Louw, the Vice-Chair, Dr. Philippe Charlet, the Secretary, Prof. Wolfhard Wegscheider, the Treasurer. Many thanks also to Dr. Karin Schober, the Treasurer Assistant and Webmaster, and to every CITAC member who supported our work in any form, including constructive criticism. The next Chair will be elected soon at the 25th CITAC Members Meeting, LNE, Paris, April 11, 2010, according to the CITAC Terms of References, and I wish him success.

Dr. Ilya Kuselman
INPL
Israel

Message of the CITAC Vice-Chairman and Award Coordinator



Each year sees metrology in chemistry not only advancing the level of traceability of all chemical analytical measurement, but expanding further into remote parts of the world. Since 1993 CITAC played its role in spreading documents and traceability solutions to accredited (and other) analytical and testing laboratories, and I

am sure it will continue to do so in the future. The CITAC process to find the most interesting or important papers in metrology in chemistry again yielded excellent and interesting papers being nominated. There was an increased participation in voting from members and I want to thank everyone for their time and effort. The following three articles were selected for special mention in 2009:

Luca Callegaro, Francesca Pennechi, Pier Giorgio Spazzini
Comparison of Calibration Curves Using the I_p Norm
Accred Qual Assur (2009) 14:587-592

Samuel Wunderli, Hanspeter Andres
Metrological Aspects of Glucose Measurements by Biosensors
Electroanalysis (2009) 21:1984-1991

Olaf Rienitz, Detlef Schiel, Bernd Guttler, Michael Koch, Ulrich Borchers

A Convenient and Economic Approach to Achieve SI-traceable Reference Values to be Used in Drinking-Water Interlaboratory Comparisons
Accred Qual Assur (2007) 12:615-622

Details are reported by the winning authors in the present issue of the CITAC News. Information about the winners will be distributed to the analytical and metrological communities and our colleagues, who have not read these papers, will benefit from the CITAC Award procedure.

On behalf of AFRIMETS, I would like to take this opportunity to wish readers of the CITAC News a successful and enjoyable 2010. If you visit South Africa for the 2010 FIFA World Cup, remember that you are always welcome to visit our National Metrology Institute (NMISA).

Dr. Wynand Louw
NMISA, South Africa

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CITAC News published by:

Laboratoire National de Métrologie et d'Essais
(LNE)
29, avenue Roger Hennequin
78197 TRAPPES Cedex
France
Tel +33 1 30 69 21 95
Fax +33 1 30 69 12 34
E-mail: philippe.charlet@lne.fr

Internet: <http://www.citac.cc>

Internet administrator:

Prof. Wolfhard Wegscheider
University of Leoben, Austria

Newsletter Editor:

Dr. Philippe Charlet, LNE, France

Newsletter designed and produced by:

Mr. Aryeh Lewis
ISAS International Seminars
P.O. Box 34001, Jerusalem 91340
Israel
Tel: +972 2 6520574
Fax: +972 2 6520558
E-mail: meetings@isas.co.il

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For additional copies of the CITAC News, please contact the newsletter editor.

Message from the CITAC Secretary

The 24th CITAC Members' Meeting in Paris, April 19, 2009



The 24th CITAC Members' Meeting was held again in Paris at LNE. The members and/or their representatives, several invited speakers and observers, a total of seventeen participants from the entire world, including India, Brazil, Japan, Hong Kong and European countries, participated in the meeting (Fig. 1).

As usual, our treasurer presented the financial report. The situation is healthy with a very positive balance, allowing efforts to increase the quality of the CITAC News and enlarge the number of pages with important papers on Metrology in Chemistry. The chairman raised the possibility of obtaining funding through industry sponsorship and advertisements in CITAC News. We still need to define the priorities of CITAC's budget.

A highlight of the meeting was the CITAC Awards for the best publications in the field of metrology in chemistry. The Award Certificates were delivered to Dr. Michael Ambrose, Prof. Paul De Bievre and Dr. Andrew Brown. The Chair congratulated the authors for their essential contribution to the development and dissemination of metrology concepts. A new round of nominations will be conducted in 2010. The recognition of the best publications has become a milestone in the activity of CITAC.

Reports of CITAC liaison officers were forwarded to participants before the

meeting. Dr. Maire Walsh gave an update of the recent ILAC activities. She mentioned that ILAC is currently working on the harmonization of criteria between ISO Guide 34 and ISO 17025 for the accreditation of RM producers, which is obviously a very important topic for CITAC. A compromise resolution to take into account the different approaches in Europe is being elaborated.

Much of the meeting was dedicated to the reports of the CITAC activities including:

- Examination of the draft procedure for participation in and the support of conferences and workshops by CITAC, aiming to increase the visibility of CITAC.
- Development of a procedure for planning and performing CITAC projects sparked debate among members of CITAC.

The Chair indicated that the basic idea of the proposal is to increase CITAC activity, to attract a CITAC member to lead a project or to participate in such a project. The discussion focused on the nature of the projects for which CITAC has an involvement. This important discussion will continue next month. It shows the dynamism of the CITAC members to develop activities.

The meeting also provided an opportunity to discuss other important work for CITAC and the scientific community:

- The IUPAC / CITAC Project is investigating out-of-specification test results of chemical composition based on metrological concepts, by Dr. Ilya Kuselman;
- IUPAC project on "Metrological Traceability of Measurement Results in Chemistry" by Prof. Paul De Bievre;
- The "Measurement Science in Chemistry Euromaster Consortium, by Dr. Anneli Krivi;
- IUPAC / CITAC Project on PT for a limited number of participants, by Dr. Ilya Kuselman.

The question of a better promotion of CITAC was also mentioned during the meeting. Dr. Laly Samuel has proposed that the CITAC newsletter could be distributed electronically to a large number of potential interested people and therefore giving CITAC the opportunity to set up networks at national levels. The idea was welcomed by the members, but it would probably be necessary to ask people to register on the CITAC web site to be on a mailing list for receiving CITAC News.

This issue of CITAC News presents reports from international organizations, summaries and updates of publications awarded as the best contributions of 2009. Three important contributions are presented in the Discussion Papers section. They concentrate on the development of concepts and principles in metrology in chemistry and their applications to end users, such as in food and nutrition with an original paper of Prof. Venkatesh Iyengar. Reports and information on conferences related to metrology in chemistry are presented, in particular Metrochem V in Brazil this fall. The 2010 events are also announced.

2010 will see the change of the Executive Committee of CITAC after 3 years of duties. I was personally proud and happy to have served as CITAC Secretary during this mandate and have welcomed the members of CITAC at LNE. I wish full success to the new CITAC Secretary.

Dr. Philippe Charlet
LNE, France



Fig. 1. Participants of the CITAC meeting 2009 after the CITAC dinner, Paris, France

Reports of International Organizations

An Update from the CCQM

Areas of Priority and Challenging Developments

The interest of NMIs and other (potential) designated institutes in metrology in chemistry and subsequent participation in the CCQM and its working groups is still rapidly growing. Driven by the needs of the economy, industry, traders and society in most of the countries the following areas of high priority have been determined:

- Food safety analysis, microbiology
- Analysis in healthcare (diagnostic, therapeutic, pharmaceuticals)
- Environmental measurements
- Purity analysis
- Natural gas
- Forensic sciences

Development in metrology in chemistry lags behind the stage of development in the physical measurement area. The field of chemical measurements is extremely wide compared to the physics area, and the demands from industry, trade and society are nowadays very high, highlighting the enormous task still ahead of us. Therefore, most of the CCQM Working Groups continue to have two working group meetings per year.

As usual, in April 2009, just before the meeting of the CCQM plenary, all CCQM Working Groups met at the BIPM. During the second half of 2009, all the CCQM Working Groups met in Rio de Janeiro, hosted by INMETRO, Brazil.

The meetings of the CCQM WGs on Bio and Organic Analysis are also attended by representatives of the Joint Committee on Traceability in Laboratory Medicine (JCTLM), National Institute of Biological Standards and Control (NIBSC, a prime lab of the WHO) and the US Pharmacopoeia (USP). These organizations contribute actively to the activities of the Bio Analysis WG and the Organic Analysis WG. The meeting of the CCQM WG on Gas Analysis is also attended by representatives of the WMO.

During the April 2009 meetings, a very interesting CCQM workshop was held on

“Frontiers of Traceability in Chem/Bio Measurements and Primary Methods”, addressing Mass Spectrometry in realizing biological relevant measurands, ID-SERS, Digital PCR for nucleic acid quantification, Count based Quantitation of trace level macro DNA molecules using high sensitivity Flow Cytometry and Quantitative Cellular Analysis.

During the meeting of the CCQM plenary, interesting reports and presentations were given on progress in the Avogadro project, discussing isotope ratio measurements on natural and enriched silicon for Si spheres and IDMS method for the Relative Molecular Mass of Silicon. Concerns were expressed regarding the quality and credibility of measurements of isotope amount ratios of silicon on samples at both natural and enriched isotopic composition.

CCQM Plenary

The CCQM met on 22-24 April 2009 at the BIPM. As usual, the meeting was attended by some 80 persons from about 45 organizations, not only representing NMIs and other Designated Institutes, but also representing the intergovernmental organizations and international bodies IAEA, IUPAC, IFCC, ILAC, JCTLM, WHO/NIBSC, WMO, ISO REMCO and CITAC.

In addition to reports by the different CCQM WGs, several presentations were given by or on behalf of a number of stakeholders discussing and informing the CCQM about metrological issues of importance now being dealt with these organizations and for which cooperation with the CCQM is considered desirable if not essential.

Dr. S. Westwood (BIPM) reported on the activities of the World Anti Doping Agency (WADA), noting that he was now serving on the WADA Laboratory Committee as an external member with expertise in metrology, replacing Prof. Siekmann.

Dr. M. Milton (NPL) reported on the increased cooperation between the WMO Global Atmospheric Watch committee and the CCQM

WG on Gas Analysis. Good progress is being made with the WMO GAW concerning the realization of SI traceable Volatile Organic Compounds (VOCs) at the parts-per-billion level. VOCs play a role in the generation of ozone in the troposphere.

Dr. A. Fajgelj (IAEA and IUPAC) reported on activities by IUPAC. He mentioned that the first draft IUPAC document “Metrological Traceability of Measurement Results in Chemistry”, composed by Prof. de Bièvre and Dr. Dybkaer, has received many comments. In the meantime an updated version is almost ready. Dr. Fajgelj also noted that 2011 has been approved by the 63rd Assembly of the United Nations as the “International Year of Chemistry”, celebrating the achievements of chemistry.

Dr. I. Kuselman, chair of CITAC, presented a short overview of CITAC activities and achievements, and a message from ILAC.

Redefinition of the Mole

In 2007, the CCQM established a working group chaired by Dr. M. Milton to redefine the mole. Other members are Drs. L. Besley, M. Salit, R. Wielgosz, R. Kaarls and Prof. de Bièvre. Dr. Milton presented a progress report and a clear presentation on the essence and consequences of the proposed changes. It was made very clear that by redefining the mole based on fixing the value of the Avogadro constant we are deliberately breaking the link with mass, decoupling it from the kilogram. This is very helpful because there has always been confusion between amount of substance and mass when units were defined on the basis of the fixed mass of carbon. Although the redefinition of the mole will not have any consequences on the daily work of chemists, concerns are expressed regarding the acceptance of the proposed new definitions by the wider chemistry community. It is felt that insufficient communication and education is taking place informing the chemical community about the proposed changes, and this may endanger the understanding and acceptance of a new definition of the mole.

Reports of International Organizations

A draft "mise-en-pratique" has been prepared, presented to and discussed by the CCQM, and presented to the CCU.

The CCQM supports the proposal for a redefinition of the mole in terms of the Avogadro number.

However, concerns about the quality and credibility of the isotope amount ratios measurements, combined with concerns of acceptance by the wider chemical community have been expressed in a Recommendation to the CIPM and the CCU.

Metrology for Bio-fuels

Dr. Ph. Charlet (LNE) summarized the outcomes of the first European meeting on metrology for bio-fuels.

Drs. Kaarls and May, as well as colleagues from INMETRO, IRMM, LNE, VSL and others attended meetings on bio-fuels on 19-20 March 2009 in Brussels, organized by the European Commission, INMETRO and NIST. Reference was made to the tri-partite "White Paper on Bio-Fuels".

After discussion, it was decided that the EU-INMETRO-NIST BIOREMA project will be linked to comparisons on bio-ethanol and FAME (diesel) carried out by the CCQM WGs on Organic and Inorganic Analysis and with respect to pH by the Electrochemical Analysis WG.

The original proposal to create a separate CIPM/CCQM working group for bio-fuels has been withdrawn.

CCQM Working Groups

All CCQM Working Groups reported about the work carried out. At present 121 different Pilot Study comparisons and 83 different Key Comparisons (KCs) have been carried out, are in progress or are planned for the near future. In addition, several Key Comparisons are followed up by a subsequent Key Comparison under the same code number, or are repeated by the Regional Metrology Organizations

(RMOs) as an RMO Key Comparison. The RMOs are now also organizing more RMO supplementary comparisons. The results of all these comparisons are underpinning more than 4300 chemical Calibration and Measurement Capabilities (CMCs) available at the participating NMIs and DIs and which have been published in the KCDB at the BIPM web site.

The CCQM WG on the quality of KCs and published CMCs has decided to carry out, over a period of a few years, a careful screening of all published chemical CMCs in order to check whether these are all still valid and sufficiently underpinned by the results of KCs and eventually other additional information and evidence.

The CCQM Gas Analysis Working Group is preparing a first comparison on air-borne nano-particles within ambient air, vehicle emissions and industrial workplaces; data which ultimately will be used to improve human health and monitor climate change. Measurands are particle number concentration, size distribution, surface area concentration, morphological and chemical characterization.

The CCQM Bio Analysis Working Group reported good results of the KC on the quantitation of a linearized plasmid DNA.

The CCQM Surface Analysis Working Group published good results of their first KC on silicon-oxide on silicon.

In particular, CCQM Gas, Inorganic and Organic Analysis Working Groups discussed the introduction of a more effective and efficient system of comparisons underpinning the claimed CMCs.

For binary gases and natural gas analysis, the Gas Analysis WG will be able to make decisions on capability and competence of the participating NMIs and DIs by making use of statistics derived from a large number of KCs carried out over a period of many years.

In the Organic and Inorganic WGs, different types of Key and Pilot Study Comparisons will be developed:

- KCs testing specific core competencies needed for carrying out chemical measurements and value assignment in the organic and/or inorganic area (including essential sample preparation procedures as well as measuring technique dependent procedures);
- KCs that assess the equivalence of Measurement Services actually provided, such as Certified Reference Materials and Value Assignment of Proficiency Testing samples;
- Key Comparisons in emerging areas of global interest and importance with accompanying pilot study comparisons;
- Capability assessment studies to allow assessment of measurement capabilities being established in new areas for an NMI/DI.

BIPM Program of Work

Dr. Wielgosz presented the progress and results of the work by the Chemistry Section of the BIPM.

The work of the BIPM Chemistry Section was well commended. The work on ozone measurement standards has been quite successful, leading to smaller uncertainties. The same good results have been obtained by the BIPM comparisons of some green house gases.

In the area of organic purity analysis a study is being carried out investigating the optimum number of purity analysis comparisons and CRMs needed to judge the capabilities and competences of the NMIs (molecular weight – polarity mapping).

Further, the Chemistry Section acts as the secretariat of the JCTLM and liaises with several other international organizations and bodies, including the Codex Alimentarius Commission, Inter Agency Meeting (IAM), International Dairy Federation (IDF), USP, European Network of Forensic Sciences Institutes (ENFSI), WMO,

Reports of International Organizations

ISO REMCO and other ISO TCs, like clinical chemistry and nano-technology.

JCTLM

Dr. Wielgosz presented a report on the current status of the JCTLM and its WG1 on “Higher Order” Reference Materials and Measurement Methods. In October 2009 some 226 CRMs are currently listed along with 146 reference methods spanning 71 analytes.

Prof. L. Siekmann, chair of the JCTLM Working Group 2 on Reference Measurement Laboratory Services and inter-laboratory comparisons reported on the progress in establishing and publishing a list of reliable reference measurement laboratory services and the results of PT schemes carried out among the participating laboratories.

Currently 111 services from 19 laboratories are listed on the JCTLM database.

The number of measurands in the IFCC Proficiency Testing scheme has risen to 195. Some 38 (potential) reference laboratories are participating in this External Quality Assessment Scheme (EQAS).

Still there is a need for a wider CCQM Program of comparability studies of listed CRMs of higher order, underpinning the results of the work carried out by the WG 1 and its sub-groups and as published in the JCTLM database. Unfortunately, still not many NMIs are able to participate in these comparisons.

A discussion with the EU is continuing with respect to the formal recognition of the JCTLM database by the EU.

Physiological Quantities

On 16-17 November 2009 a workshop on physiological quantities and the SI was held at the BIPM. Part of this workshop was dedicated to the non-SI traceable bio-chemical quantities, expressing bio-chemical activity rather than defining the exact chemical composition. In most of these cases reference is made to the WHO International Standards and International Units. However, when we better understand the relationship between the biological activity and the chemical composition it will be possible to make these quantities SI traceable, providing medicines and healthcare at greatly reduced costs.

Codex Alimentarius Commission

Dr. R. Josephs (BIPM) presented a report on the Codex Alimentarius Commission, the Inter Agency Meeting and the MoniQA, being the EU Network of Excellence funded project on the Monitoring and Quality Assurance in the Food supply Chain. The BIPM has contributed to two workshops on “Method Performance and Analytical Uncertainty” and on “Method Performance and the Criteria Approach: Truth and Consequences”.

WMO

The BIPM-WMO workshop on Metrology and Climate Change will be held at the premises of the WMO in Geneva on 30 March-1 April 2010.

ISO REMCO

Prof. Emons (IRMM) reported on the activities in ISO REMCO. ISO Guide 33 is currently under revision, getting a wider scope and addressing calibration. Other Guides under revision are Guide 31 (vocabulary) and Guide 32 (Labels and Certificates). An ad hoc ISO REMCO WG on “CRMs and Metrology and Traceability” has

been re-established with an aim of expressing how to make statements on traceability.

IAEA

The BIPM and the IAEA are strengthening their cooperation, in particular with respect to:

- ionizing radiation measurements, dosimetry, radionuclide containing materials, nuclear data (CCRI),
- chemical measurements, CRMs (CCQM),
- the “Vienna Standard Mean Ocean Water” standard (CCT) and
- the CIPM MRA

Conclusion

In 2009, good progress has been made in broadening the scope of metrology in chemistry. New challenging areas have been entered, addressing needs in industry, trade and society. The CCQM Working Groups are studying more efficient and effective ways of assessing the claimed calibration and measurement capabilities of the NMIs and DIs. Guidance is being developed for justified applications of statistical methods for the calculation of the Key Comparison Reference Value and the degrees of equivalence of the NMIs and DIs participating in the CIPM MRA.

Also in 2010, workshops dedicated to specific sectors and lack of traceability in important areas will be organized, like a workshop on forensics and a workshop addressing traceability in micro-biology.

Dr. Robert Kaarls
President CCQM
The Netherlands

ILAC Update

CITAC is a stakeholder member of ILAC and continues to contribute to ILAC's work program, particularly in relation to issues involving metrological traceability in chemistry. CITAC also has official liaison status with ILAC

and representatives of each organization attend relevant meetings – for example, in 2009, the ILAC Chair of the Laboratory Committee, Maire Walsh, attended the CITAC Members Meeting (March, Paris). ILAC and

CITAC were represented at the CCQM Plenary (April, Paris) by the CITAC Chair, Dr Ilya Kuselman. In addition, ILAC contributes to the work of CITAC/EURACHEM Working Group on Traceability and Measurement Uncertainty

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and documents emanating from this group are posted on the ILAC website.

In maintaining this close cooperation with CITAC, ILAC has been able to continue its efforts to strengthen the links between accreditation and metrology and this is much appreciated.

A general update on ILAC activities for 2009 follows:

ILAC Meetings

The mid year meetings of the ILAC Executive and the ILAC Arrangement Management Committee (AMC) were hosted by UKAS in London during the week of 22 June 2009. The week also included meetings of the IAF, ILAC and ISO Joint Working Group, the Joint Committee for Closer Cooperation (JCCC) and the various ILAC and IAF arrangement management committees.

The meeting of the IAF, ILAC and ISO Joint Working Group included work on a Joint Communiqué on the Management system requirements of ISO 15189:2007. This Communiqué has now been finalized and a copy of the Press Release and a link to the Communiqué was provided to all members and ILAC liaison partners, on 4 November 2009.

The final ILAC meetings for the year were held in Vancouver in October. The meetings were very productive with many work items being finalized and a number of items seeing significant progress.

Special thanks go to our colleagues from CALA, the hosts of the 2009 annual meetings, for the excellent logistical arrangements and support provided throughout the meetings.

The Executive meetings for 2010 are scheduled for 8-9 March in Paris, 23-26 June in Mexico City and during the annual meetings in October in Shanghai. These meetings will be hosted by our colleagues from COFRAC, EMA and CNAS respectively.

The ILAC Arrangement

As of 9 November 2009, there were 65 Signatories (Full Members) to the ILAC Arrangement, representing 51 economies.

Further progress was made during the recent meetings in Vancouver towards establishing the ILAC and IAF Arrangement for Inspection and applications have been received from APLAC, EA and IAAC, the current Recognized Regional Cooperation bodies, for evaluation to join the IAF/ILAC Inspection Arrangement.

ILAC also continues to focus on enhancing a more widespread understanding of the socio-economic and trade benefits of the Arrangement amongst the international community, particularly governments and regulators.

ILAC Membership

ILAC membership as of 9 November 2009 was as follows:

- 65 Full Members (Signatories to the ILAC Arrangement) representing 51 economies;
- 23 Associates representing 23 economies;
- 20 Affiliates representing 18 economies;
- 4 Regional Cooperation Bodies
- 1 National Coordination Body
- 25 Stakeholders

The ILAC membership (total 138 bodies) now covers a total of 88 different economies worldwide and approximately 33,000 laboratories and over 6,000 inspection bodies are accredited by the 65 ILAC Full Members.

Joint ILAC/IAF Activities

As many will be aware, discussions regarding the future development of ILAC and IAF were held during the first half day of the 2008 Joint General Assembly (JGA). It was agreed during the JGA that a Task Force would be established to consider, in detail, the issues highlighted by the members as requiring further investigation

before consideration could be given to following any particular direction.

The Task Force is composed of the Chairs and Vice-Chairs of ILAC and IAF, the Chair or nominated representative from each region, a representative of the unaffiliated bodies and stakeholder representatives from the ILAC Laboratory Committee, the IAF industry representative and the IAF conformity assessment bodies representative. The Task Force has had two meetings during 2009, one in January and one in May. The progress made by the Task Force was reviewed during the JCCC meeting in London and details of the information package to be provided to ILAC members, in preparation for the discussions in Vancouver in October, were finalized. This information package was distributed to the members of ILAC (and IAF) prior to the meetings in Vancouver.

As agreed during the Vancouver Joint General Assembly and confirmed during the ILAC General Assembly, in October 2009, (JGA Vancouver Resolution 6 – Future Direction of IAF and ILAC and ILAC Resolution GA 13.31 & Attachment) a 30 day ballot was undertaken, in mid November 2009, to determine which of the proposed options for the future direction of ILAC and IAF the memberships of both organizations would like to implement.

The adoption of JGA Vancouver Resolution 5 – JCCC during the meetings in Vancouver, marked the end of the Joint Committee for Closer Cooperation, which held its final meeting on 17 October 2009 in Vancouver. This committee is being replaced with joint sessions of the ILAC and IAF Executive Committees. The first official joint session is scheduled for Monday 8 March 2010 in Paris.

ILAC Executive

Two members of the ILAC Executive will be stepping down from their respective roles on 31 December 2009, namely Ilew Richards, ILAC Arrangement Management Committee (AMC) Chair, and Maribel Lopez, Joint Development Support Committee (JDSC) Co-

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Chair. The significant contributions made to the work of ILAC, by both Dr Richards and Ms Lopez, were acknowledged during the recent ILAC Arrangement Council and ILAC General Assembly meetings in Vancouver.

As of 1 January 2010, Andreas Steinhorst, from DGA, Germany and Dorsaf Zangar from TUNAC, Tunisia will take on the roles of Acting Chair of the ILAC AMC and Acting Co-Chair of the JDSC respectively. Elections for all (elected) positions on the ILAC Executive Committee will be held during the annual meetings in October 2010.

ILAC Liaisons

The review of liaison activities continues to be a major focus of the ILAC Executive Committee, who seeks to ensure that ILAC interests are represented in areas which have an impact on the activities of ILAC and its members. Action items arising out of ILAC's liaison activities are addressed during each Executive meeting, as well as on a continuous basis throughout the year.

The Liaison Database, located in the members area of the ILAC website, continues to serve as the main repository for the ever increasing number of reports and documents that are produced as part of ILAC's liaison activity. It can be accessed via the members area of the ILAC website.

Since April 2009 ILAC has participated in a number of liaison activities including CASCO WG 29 (Revision of ISO Guide 65 – Product Certification), CASCO WG28 (Revision of ISO Guide 43 – Proficiency Testing), ISO TC212 (Technical Committee - Clinical laboratory testing and in vitro diagnostic test systems), the BIPM Joint Committee for Guides in Metrology (JCGM), the Joint Committee for Traceability in Laboratory Medicine (JCTLM), CITAC, CCQM, EURACHEM, ISO REMCO, BERM 12 and meetings of the WADA Laboratory Committee.

ILAC has commenced general discussions with the World Health Organization (WHO)

to determine how we might best cooperate in areas of mutual interest and involvement. A meeting took place in July in Lyon, France between WHO representatives and the ILAC Chair, Daniel Pierre, to continue these discussions.

The ILAC Chair, Daniel Pierre, was invited to speak on behalf of ILAC at a major symposium held by BIPM, in October this year. The Symposium was held to mark the 10th Anniversary of the CIPM Mutual Recognitions Arrangement. ILAC also gave a short presentation at the WADA Foundation Board Meeting, held in Stockholm in December 2009, to mark the 10th Anniversary of the World Anti-Doping Agency (WADA).

ILAC thanks all of the ILAC liaison officers, and their organizations, who give up their time to assist ILAC in carrying out these activities for the benefit of the ILAC members.

ILAC Secretariat

The ILAC Secretariat staff is as follows: Annette Dever – ILAC Secretary; Alan Squirrel – ILAC Executive Liaison Officer; Sharon Kelly – Senior ILAC Coordinator; Alison Hay – ILAC Administrator and Rose Bevins – ILAC Administrator.

The Secretariat continues to develop the ILAC website, with several improvements being carried out in conjunction with the ILAC Marketing and Communications Committee (MCC). Further 'user-friendly' improvements are planned for the first quarter of 2010.

The Secretariat is now preparing a revision of ILAC R5, which is the ILAC Complaints Procedure. The revised document will be considered by the Executive during their March 2010 meeting. Once the Executive have finalized their input, the draft document will be sent to members for a 60 day comment period.

As a result of the successful ballot held a few months ago, ILAC-R1:09/2009 Classification

and Publication of ILAC Documents has been published. This document replaces ILAC S1:2003. The publication of this new document has resulted in the re-classification of the S-series documents. These documents are now listed as Rules or R-series documents and the ILAC website has also been updated to reflect this change.

Please note that while the number and date of the S-series documents have been updated to reflect the change in classification and, in a few cases, changes in cross-referencing to other former S-series documents, there have been no changes to the actual content of these documents.

A complete list of all documents, that have been (or are being) circulated to members for either comments or voting, can be obtained from the ILAC website in the Members Section under Ballots.

The ILAC-MRA Mark registration process continues and, as of 9 November 2009, 45 ILAC Full Members had signed Licensing Agreements with ILAC for the use of the Combined MRA Mark (the Combined MRA Mark is the ILAC-MRA Mark used in combination with the accreditation body's own mark).

The April and October 2009 editions of ILAC News have been distributed in hard copy and electronic versions are available to download from the ILAC website under the 'News' section. Past editions of ILAC News are also available to download from the website. The next edition of ILAC News will be published in April 2010.

Further information on ILAC can be obtained from the ILAC website at www.ilac.org, or by emailing the Secretariat on ilac@nata.asn.au.

Mr. Alan Squirrel
ILAC Executive Liaison
Australia

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Information from the Division of Analytical Chemistry (DAC) of European Association for Chemical and Molecular Sciences (EuCheMS)

This year's main DAC event, the Euroanalysis XV conference was held in Innsbruck, Austria (see www.EUROANALYSIS2009.at). EUROANALYSIS has become one of the most important broad-spectrum analytical conferences covering all aspects of analytical sciences. It is held every two years in different European countries and is organized under the umbrella of DAC-EuCheMS. In 2009, this conference was organized by the Austrian Society of Analytical Chemistry (ASAC) represented by the chairmen Wolfgang Buchberger and Wolfgang Lindner. The organizers welcomed approximately 700 participants from 53 countries. 130 lectures and 640 poster presentations under the motto "The Impact of Analytical Chemistry on Quality of Life" formed a very attractive program which demonstrated the broad diversity of analytical sciences.

Prof. Slavica Ražić is organizing Euroanalysis XVI in Belgrade, Serbia, on 11-15 September 2011 (see <http://www.euroanalysis2011.rs>). At the DAC Annual Meeting in Innsbruck, it was decided by the delegates that Euroanalysis XVII will be organized in Warsaw, Poland, on 25-29 August 2013. Other conferences mentioned are the 10th Meeting of Kinetics in Analytical Chemistry (Pretoria, Republic of South Africa), the 13th ISRANALYTICA (Tel Aviv, Israel, 19-20 January 2010), ANALYS DAGARNA (Uppsala, Sweden, 14-16 June 2010), and the 3rd European Congress on Chemistry in Nuremberg, 2010 ([\[congress2010.org\]\(http://congress2010.org\)\). Information about the International Year of Chemistry 2011 \(IYC11\) can be found on <http://www.chemistry2011.org>.](http://www.euchems-</p></div><div data-bbox=)

Some information from the various DAC Study Groups:

Education - The EChemTest (an evaluation tool dedicated to student's mobility and chemistry knowledge assessment) of the European Chemistry Thematic Network (ECTN) is now fully operational, and university faculties are encouraged to apply the examples to their teaching.

Quality - Hendrik Emons reported on new initiatives on legislation. For instance, Germany introduced a new regulation on accreditation as a result of harmonizing with the new EU legislation. He also raised awareness about the ISO Guide 99 on concepts and terminology of metrology which contains a lot of information relevant for analytical chemistry. This guide, also known as VIM 3, is freely available at the BIPM webpage under www.bipm.org. It is also important to enlighten students about the topic.

History - The main activity of this study group is the production of a series of publications, "People and Places Important in the History of Specific Countries", particularly the Hosts of Euroanalysis Conferences. The list of those completed is available on the DAC website.

Bioanalytics - The aim of the Bioanalytics study group is to search for ways to bring the analytical and bioanalytical chemistry communities closer. George Horvai reported that several analytical journals have strongly moved into the direction of bioanalytical chemistry, and that a large fraction of the authors publishing bioanalytical papers in analytical chemistry journals are from outside the traditional analytical workplaces. Moreover, important journals which are dealing with areas other than analytical chemistry have moved deeply into the analytical field, for example "Nature Methods". The conference Euroanalysis XV had three sessions on bioanalysis, involving some twenty lectures, and there were about as many bioanalytical lectures in the other sessions.

European Analytical Chemistry on the Web - The website of EAConweb is maintained by Bo Karlberg with the support of the Swedish Chemical Society (<http://www.anchem.su.se/euchems/countries.asp>).

Prof. Hendrik Emons
EC-JRC-IRMM
Liaison Person DAC-CITAC
Belgium

Dr. Jens E.T. Andersen
DAC Secretary
DTU, Denmark

AFRIMETS MiC Report 2009

Introduction

During 2007, the Intra-Africa Metrology System (AFRIMETS) was formed with, as principal members, the five sub-regional metrology organizations (SRMOs) in Africa. Four countries not part of a SRMO became ordinary members (Nigeria, Egypt, Ethiopia and Ghana), extending the countries represented in AFRIMETS to forty one. In October 2008, AFRIMETS officially became the regional metrology organization

(RMO) representing Africa. During 2009, Egypt was instrumental in establishing a SRMO in Northeastern and Western (English speaking) Africa, incorporating the four ordinary members. Libya and Sudan joined the new SRMO, called NEWMET. Mauritania also joined AFRIMETS (through MAGMET), increasing the total membership of AFRIMETS to 44 countries.

All structures of AFRIMETS are now in place and the first technical working group meetings were held during the General Assembly meeting in July 2009. The first official AFRIMETS comparison was registered in the field of temperature. Protocol for comparisons in photometry and radiometry and dc voltage were considered. For metrology in chemistry, 15 countries attended the first MiC working group meeting and the results of the first

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proficiency testing scheme were discussed. A work program for the next few years was drawn up and three comparisons will be conducted during 2009/10.

The Structure and Members of AFRIMETS

The six SRMOs are:

- (1) Southern African Development Community Cooperation in Measurement Traceability (SADCMET);
- (2) East African Metrology Program (EAMET);
- (3) Economic and Monetary Community of Central Africa Metrology Cooperation (CEMACMET);
- (4) Secretariat for Metrology of the Economic Community of West African States (SOAMET);
- (5) Maghreb Metrology Cooperation (MAGMET) and
- (6) Northeastern and Western Africa metrology program (NEWMET).

The primary aim of AFRIMETS is to harmonize scientific, industrial and legal metrology issues

across Africa; to provide a link to the activities of the BIPM and OIML and to operate as a fully fledged RMO, fulfilling the obligations as stipulated in the Mutual Recognition Arrangement of the International Committee for Weights and Measures, the CIPM MRA.

AFRIMETS now spans the continent. The sub regional map of AFRIMETS is shown in Fig. 1 and the country members of the SRMOs are shown in Table 1. AFRIMETS thus covers the continent with the exception of a few countries on the eastern and western side of Africa with very little or no metrological infrastructure.

A Work Program for 2009/2010

One of the most frequent requests that AFRIMETS receives is for traceability in MiC, and traceability in chemical and microbiological measurement is therefore a high priority. Issues under discussion include the manufacture or provision of certified reference materials (CRMs), value assignment of PT scheme samples, the development of methods for current issues in food safety (such as

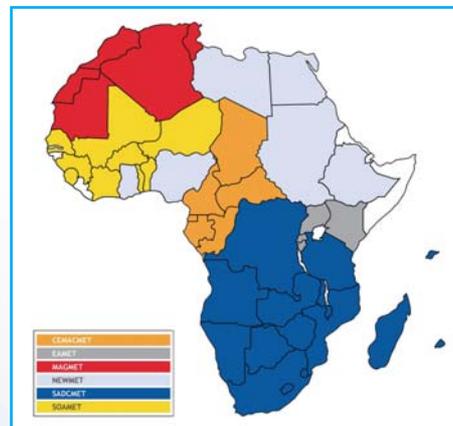


Fig. 1. Map of the sub-regional metrology organizations of AFRIMETS

melamine in milk, pesticides and mycotoxins) and training (wet chemistry, chromatography, mass spectrometry, etc.). The requests are diverse and differ from sub-region to sub-region, making it difficult to prioritize.

The Needs of the SRMOs

EAMET

In East Africa, since 2004, Kenya's tea production increased to the point where Kenya is currently the third largest producer and the single biggest exporter of tea in the world [1]. A big advantage is that Kenyan tea stems from a variety that is resistant to most pests and very little (or no) pesticides are used. One concern is the quality of water used for both irrigation and processing, and accurate measurement is important for monitoring of water quality. Kenya participates in the SADCMET (now expanded to AFRIMETS) water proficiency testing (PT) scheme [2]. Since its inception in 2004, the PT scheme has focused on the chemical analysis of water covering all chemical parameters including sulphate, chloride, manganese, lead, copper, nitrate etc. To date over 42 water testing laboratories participated in the scheme which ultimately contributes to ensuring that the water used in the region is within acceptable chemical limits, making it safe for human consumption (and suitable for the production of agricultural products, etc).

Table 1. The individual SRMO members of AFRIMETS

SADCMET/MEL	SOAMET	CEMACMET	EAMET	MAGMET	NEWMET
Angola	Benin	Cameroon	Kenya	Morocco	Egypt
Botswana	Burkina Faso	Central African Rep	Tanzania	Algeria	Nigeria
DRC	Guinea-Bissau	Chad	Uganda	Tunisia	Ethiopia
Lesotho	Mali	Congo Brazzaville	Rwanda	Mauritania	Ghana
Madagascar	Niger	Equatorial Guinea	Burundi		Libya
Malawi	Senegal	Gabon			Sudan
Mauritius	Togo	Sao Tome*			
Mozambique	Côte d'Ivoire				
Namibia					
Seychelles					
South Africa					
Swaziland					
Tanzania					
Zambia					
Zimbabwe					

* Sao Tome is a member of CEMAC but membership of CEMACMET has not been confirmed.

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The PT scheme has been so successful, with a vast number of laboratories participating, that the next PT round (started in July 2008) also included the microbiology field.

The Kenya Bureau of Standards (KEBS) established a Metrology in Chemistry section in 2007/2008. A regional metrology in chemistry conference was held in Nairobi in August 2008, with the focus on the provision of certified reference materials and the assignment of reference values to proficiency testing samples. Close collaboration has been established with the National Metrology Institute of South Africa (NMISA) and the first collaborative project on the value assignment of a wheat flour sample for an EAMET proficiency testing scheme was completed. KEBS has observer status at the CCQM and attended WG meetings and the plenary during 2009.

Tanzania has the largest agricultural contribution to the Gross Domestic Product (GDP) of the EAC countries (41% of GDP). Although a member of the EAC, Tanzania participates in SADC MET and its measurement traceability needs are mostly catered for through this well established SRMO. A program to accredit 14 food testing laboratories is well underway and the first group of laboratories have already received third party accreditation to ISO 17025.

EAC trade liberalization has led to problems for countries such as Rwanda, where the local market was flooded by sub-standard and counterfeit products [3]. The response was that certain products were banned, for example plastic polyethylene bags, batteries, cosmetics containing hydroquinone, mercury and cortisones (except if being imported by a pharmacist) and all non-conforming products and counterfeits. This has placed an added responsibility on the Rwanda Bureau of Standards (RBS) [3] to provide metrology and testing for this purpose. The MiC activity is still largely confined to testing, but the sub-regional interaction increased the awareness of traceability and the acceptance of test results.

SADC MET

The Southern African Development Community (SADC) was formed in 1992 [4]. It currently has fifteen member states (see table 1). The objectives of SADC include achieving active development and economic growth; alleviating poverty; enhancing the standard and quality of life of the people of Southern Africa, amongst others. As part of the quality infrastructure, SADC MET was formed at the same time [5]. The MiC needs of SADC MET overlap with that of the other SRMOs especially for primary and intermediate commodities (food, ores, etc.), but in addition, traceability is needed for processes used in manufactured goods, for example chemicals and automotive.

The NMISA is the only NMI in the region with official MiC facilities and that participates in the activities of the CCQM. It currently focuses its activities on the monitoring of toxic or carcinogenic substances in environmental samples and agricultural products (especially foodstuffs), towards improving the global acceptance of manufactured goods.

MAGMET

MAGMET is based on a cooperation of Arabic countries in North Africa, Maghreb, meaning "place of sunset" or "western" in Arabic. It is generally applied to all of Morocco, Algeria and Tunisia but in older Arabic, usage pertained only to the area of the three countries between the high ranges of the Atlas Mountains and the Mediterranean Sea. The Arab states of North Africa established the Arab Maghreb Union in 1989 to promote cooperation and economic integration [6].

The economy of Algeria is mostly reliant on petroleum and natural gas, whilst Morocco and Tunisia rely on Atlantic fisheries and fruit and vegetables. Due to a relatively large and well trained working force, nearly 50% of exports from these two countries are intermediate and finished manufacture. The destination is mostly the EU region, with the USA second. Established metrology infrastructure, especially in Morocco, already supports international export, and a basic measurement infrastructure

is currently being established in Tunisia. Both Tunisia and Morocco are in the process of establishing metrology in chemistry facilities.

SOAMET and CEMACMET

The West African Secretariat for Metrology (SOAMET) is the sub-regional metrology grouping of the West African Economic and Monetary Union (UEMOA) countries (see table 1). UEMOA countries only account for 0.1% of global trade in manufactured goods, and intra-trade is only 6% of total trade. The main exports of the UEMOA countries are agro-food products, fish products and cotton. The main export market is the EU [7].

In September 2001, to enhance the participation of the UEMOA countries in regional and international trade, the EU, United Nations Industrial Development Organization (UNIDO) and UEMOA launched a Quality Program as part of the UNIDO Trade Capacity Building Initiative. The Program is assisting with establishment and/or strengthening of institutional and human capacities in laboratory accreditation; the development of product and material testing laboratories (chemical, microbiology, etc.) according to international standards; standards formulation, adoption and dissemination; the development and implementation of quality policies; instituting quality awards; and the development of consumer protection laws and associations.

Initially, the focus of the Program was on food processing. In early 2003 the focus was extended to fish. In September 2003, the focus was further extended to cotton [8].

CEMAC (from its name in French, Communauté Économique et Monétaire de l'Afrique Centrale) is an organization of States of Central Africa established to promote economic integration among countries that share a common currency, the CFA franc. The region shares a high dependence on oil and forestry and is subject to volatile economic growth, weak intra-regional linkages and a lack of transportation infrastructure. The zone is dominated by Cameroon and Gabon whose

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economies account for more than two-thirds of the region's GDP.

CEMACMET is the metrology program of CEMAC, but only exists in name, and there are no coordinated scientific and industrial metrology activities in the region. The PTB [9] and donor agencies such as UNIDO are active in programs to establish SQAM infrastructure in the region, but progress is slow. New activities are being planned for the near future.

NEWMET

The metrology program in Northeast and Western Africa stems from a memorandum of understanding between the ordinary members (countries not part of a sub-region) of AFRIMETS. Libya and Sudan then joined. Three countries (Egypt, Ghana and Ethiopia) have well established metrology infrastructure, including metrology in chemistry (Egypt and starting in Ghana), with the other countries all with some facilities and in the process of improving or expanding.

MiC activities include certified reference material production (Egypt) and accredited testing facilities at most of the other institutes.

The first GA will be held in Accra (Ghana) in February 2010 and intra-regional comparisons are expected to commence soon.

Participation in CCQM Activities

The MiC laboratory at the NMISA is currently the only laboratory in Africa actively participating in CCQM comparisons and with CMCs in chemistry. The laboratory therefore plays a leading role in providing advice to other countries in Africa that are establishing MiC facilities, and as a pilot laboratory for AFRIMETS comparisons.

A few of the activities at the NMISA during the past year included:

Developments on Establishing a NRL for SA Air Quality Measurements

The NMISA prepared a proposal for the establishment of a National Reference

Laboratory (NRL) for the Department of Environmental Affairs and Tourism (DEAT) in December 2008.

An advisory forum consisting of experts in air quality management will be required to develop and maintain the quality procedures for the virtual NRL. The NMISA sees its role as the provider of traceability for flow and reference materials for the calibration laboratories. Mobile monitoring stations are foreseen that can be used to audit monitoring stations as well as a training facility for which NMISA would act as custodian. Auditing of the monitoring networks will be accomplished by encouraging ISO/IEC 17025 accreditation so that this endeavor can be overseen by the South African National Accreditation System (SANAS).

Automotive Sector: Proving International Competency in Automotive Emission Measurements

The gas metrology laboratory completed participation in the international comparison for the preparation of gas mixtures intended for the testing of automotive emissions, Euramet 1113. The study, organized by the European regional metrology body, Euramet, requires that participants accurately prepare a prescribed mixture of propane, carbon monoxide and carbon dioxide in nitrogen gas. The gas metrology laboratory has recently expanded its preparative capabilities to include multi-component mixtures and this study will serve to underpin this new capability.

Metrology in Organic and Biochemistry

The organic metrology group at NMISA is currently investigating what role it can play in assisting South African analytical laboratories in analyzing mycotoxins in foodstuffs and feeds.

This work is critical because it affects several large South African industries trading in food and wine, involving thousands of jobs. The initial 2008 investigation on mycotoxins included a survey of SA analytical laboratories involved in mycotoxin analyses and a visit to the Institute of

Reference Material and Measurements (IRMM) in Belgium, and resulted in the NMISA further investigating the following options:

- A reference measurement for mycotoxins in South African maize/animal feed/wheat/wine
- A proficiency testing scheme for mycotoxins in South African maize/animal feed/wheat/wine
- A reference material for mycotoxins in South African maize/animal feed/wheat/wine

The first outcome of the study was an agreed collaboration between the Southern African Grain Laboratory (SAGL) and the NMISA that led to the signing of a memorandum of understanding (MoU) in support of technical cooperation for the analysis of organic contaminants in grains and related products. The initial focus will be on reference measurements for mycotoxins in maize. Further work under the mycotoxins project, namely method validation for the determination of Ochratoxin-A in wine, continues as planned.

With the analytical focus still strongly on pesticide residues in agricultural produce, whether for export or local consumption, several African laboratories require measurement traceability to underpin their measurements in this field. The NMISA is currently developing a screening method for various pesticides in agricultural crops, including tea and wine, which can assist these countries with more affordable and robust analytical methods. With the collaborative efforts established through AFRIMETS, measurement equivalence will also be coordinated through participation in a proficiency testing scheme for selected pesticides (e.g., p,p'-DDT).

The IUFOST World Congress is being hosted in Cape Town, SA, in August 2010. The theme for this congress is "Food Science Solutions in our Evolving World" and the focus is to encourage young scientists from Africa to participate. The NMISA will be participating in the scientific program. This can serve as a forum

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for disseminating measurement competence for selected parameters like pesticides and mycotoxins in agricultural produce.

The CCQM organic analysis and bioanalysis working groups have expanded the measurement focus to include large molecules of interest to both groups and this has afforded the NMISA bioanalysis laboratory the potential to establish measurement capability for amino acid quantification (hydrolysis of peptides and proteins) using both fluorescence detection and tandem mass spectrometry. Participation is planned in the upcoming CCQM-P58.x protein analysis study next year.

The newly developed certified reference material (CRM) for ethanol in 20% glucose has received a favorable reception from the wine industry. Wine testing laboratories use the new CRM to calibrate equipment that measures the quantity of ethanol in wine. In order to uphold the laboratory's calibration and measurement claims for these reference materials, the laboratory is participating in an international

comparison (CCQM-K79): Comparison of Value-Assigned CRMs and Proficiency Testing Materials for Ethanol in Aqueous Matrix (0.1 mg/g to 500 mg/g).

Participation in International Comparisons

NMISA participated in 14 comparisons in metrology in chemistry during 2009. A list of the comparisons is provided below in Table 2. In addition, three AFRIMETS comparisons are being planned in the fields of organic and inorganic chemistry.

At the end of 2010, the AFRIMETS MiC will be able to report on the first pilot studies and comparisons conducted in the region.

References

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Dr. Wynand Louw
Ms. Jayne de Vos
NMISA
South Africa

Table 2. List of international comparisons that NMISA participated in or started during 2009

No	International Number	Laboratory	Description
1	CCQM-P122	Organic & Bio	Chloramphenicol in pig muscle
2	CCQM-K79	Organic & Bio	Comparison of value-assigned CRMs and PT materials for ethanol in aqueous matrix, 0.1 mg/g to 500 mg/g
3	CCQM-P90	Organic & Bio	CAP in milk
4	CCQM K-67	SA	The quantification of Fe-Ni binary alloy thin films
5	VAMAS TWA2-A3(d)	SA	Static SIMS interlaboratory study: part 1, linearity of the intensity scale
6	CCQM-K75 & P118	Inorganic	Toxic metals in algae (Pt, Ni) Toxic metals in algae (Cd, Cr, Hg, Pb)
7	CCQM-P119	Inorganic	Analysis of Pb in a lead-free solder
8	CCQM-K71	Gas	Multi-component stack gas mixture
9	CCQM-K74	Gas	10 ppm NO ₂ in nitrogen by FTIR
10	CCQM-P110	Gas	10 ppm NO ₂ in nitrogen by FTIR
11	BIPM.QM-K1	Gas	Ambient level ozone
12	EURAMET 1092	Gas	120 ppm ethanol in nitrogen
13	EURAMET 1113	Gas	Preparative comparison for automotive emission gases
14	APMP.QM-K41	Gas	10 ppm H ₂ S in nitrogen

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APMP Liaison Report 2009



Fig. 1. Participants of the APMP TCQM meeting 2008, Jakarta, Indonesia

The 24th APMP (Asia Pacific Metrology Program) General Assembly and the 8th meeting of APMP-TCQM (Technical Committee on Amount of Substance) were held in Jakarta, Indonesia from 3-7 November 2008. Metrologists from member NMIs and guests from CIPM, BIPM, RMO's, NCSLI and specialist regional bodies attended the meeting. Two new members – HSA (Health Science Authority) Singapore, in the field of chemistry, designated by ASTAR and OAP (Office of Atoms for Peace) Thailand, in the field of ionizing radiation, designated by NIMT Thailand – joined the APMP. The application from VNIIM Russia to become a new full member of APMP was approved by APMP-GA. The 24th GA elected Dr. Kwang Hwa Chung from KRISS as Chairperson for a second term of two years from the 25th GA to the 27th GA, and Dr. Jia-Ruey Duann, Director General of CMS/TRI from Chinese Taipei and Dr. Angela Samuel, Director of International Cooperation Office of NMIA Australia as new EC members. The 25th APMP GA and related meetings will be held in Kuala Lumpur, Malaysia and hosted by SIRIM.

Many recommendations and actions were taken by the APMP GA, the main ones being: APMP will cooperate with the BIPM in the field of nanometrology and provide BIPM with APMP's opinions and information on the priorities for national and international activities when so requested.

QMS review status could be improved through enhanced communication between the TCQS chair and the other TC chairs. For this purpose,

the status of the APMP CMC intra-review will be monitored on the APMP website.

The status of CMCs submitted by APMP were monitored to find out whether there were any CMCs delayed for a long time without recognition. The status of CMCs monitored from the BIPM KCDB was found to differ from the reports by TC chairs. Therefore the status of CMCs will be confirmed.

APMP EC and its associated subcommittees engaging with APLAC's PT programs will provide traceable reference values to those programs and also consider the linking of their formal comparison processes to relevant APLAC PT programs.

Recognizing the importance of global consistency of Materials Metrology activities and noting the MoU between BIPM and VAMAS, APMP ad-hoc WGMM decided to contact BIPM whenever necessary. Participation of the Institute of Materials Research & Energy in APMP ad-hoc WGMM will be discussed in the EC.

Technical Committee on Amount of Substance Meeting (TCQM)

The APMP TCQM held its 8th annual meeting on 3-4 November 2008 at the Mercure Convention Centre, Jakarta, Indonesia. A total of 31 delegates from 17 member economies attended the meeting (Fig. 1). The President of CCQM, Dr. Robert Kaarls, gave an update on CCQM working group activities during 2008. He also announced that the coming CCQM working group meetings and workshops at BIPM Would take place from 17-24 April

2009 and the second working group meetings of the year Would be in INMETRO, Rio de Janeiro, Brazil from 4-6 November 2009. Progress reports on the latest activities carried out by the TCQM Working Groups were presented by each representative.

Highlights of APMP Activities

The meeting discussed 2007-2008 APMP-TCQM activities, including the on-going and newly proposed comparison programs and CMC claims and workshops and symposia held during this period. Five economies from the APMP region, Australia, China, Chinese Taipei, Japan, and Korea, have CMC claims in the Appendix C and for the first time Hong Kong filed for CMC claims. TCQM encouraged submissions from other economies using the new template which can be obtained from the JCRB document website (www.bipm.org/en/committees/jc/jcrb/) and emphasized that all claims should be made for services offered on a reasonably regular basis and not just because of participation in key comparisons.

Reports on the APMP Workshop on Metrology in Chemistry

Dr. Laurie Besley presented the objective, strategy and outcome of the APMP workshop on metrology in chemistry. The APMP guide, downloadable from the APMP website, was one of the outcomes of this workshop. Two workshops were conducted in 2008. The first was held in May 2008 in Dhaka, Bangladesh while the second took place in Jakarta, Indonesia from 31 October – 1 November 2008. A number of economies participated and

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made presentations. There were suggestions to improve the APMP guide as well as recommendations for APMP – TCQM, DEC, EC, mainly in 5 areas: training, RMs/CRMs, government support/awareness raising, new APMP programs and national dissemination of capacity building. The discussion was intensive and showed a great depth of understanding. Organizers acknowledged the continuous financial support and technical input provided by PTB Germany in the region.

6th Workshop of APMP/TCQM Gas Analysis Working Group

The workshop on “Present Progress in Gas Metrology” was held in KRIS, Korea from 19-23 May 2008. It included a 2-day workshop, a tour to LCD company and a training session. There were 44 participants from 7 countries including participants from various NMIs, government institutes and companies. The 7th workshop was held in SIRIM, Malaysia in May 2009 and the 8th workshop will be held at NMIJ, Japan in 2010.

8th ACRM Meeting in Lijiang China

The 8th ACRM meeting took place in Lijiang from 23-24 September 2008 and was attended by 26 participants from NIM, NMIJ and KRIS. NIM reported the progress of the work of four working groups covering food, gas, RoHS and biological analysis.

Progress Reports on TCQM Key Comparisons and Pilot Studies

APMP.QM-P10: “Cadmium and Lead in Herb”
Dr. Cheung Tsz-chun of GLHK presented the technical data on the Herb (*Herba Desmodii Styracifolii*) as well as the performance of participants. This study was conducted in parallel with CCQM-97, and 12 laboratories from 8 economies participated in the pilot study. The performance of APMP.QM-P10 was not as satisfactory as CCQM P-97. Better agreement between APMP.QM-P10 and CCQM P-97 was achieved in the identification of outliers.

APMP.QM-P11: Organo-arsenic in Swordfish

Dr. Akiharu Hioki of NMIJ reported that this study, co-ordinated by NIM and NMIJ and

run in parallel with CCQM-96 “Total arsenic and arsenobetaine in swordfish”, has been completed. 5 laboratories including 3 NMIs (NAM, NIST and UME), examining total arsenic and 3 laboratories – all NMIs – examining arsenobetaine, participated. The results reported on total As measurement by the three NMIs were in good agreement to the KCRV for total As in CCQM-K43.1. For arsenobetaine, the analytical results were in good agreement using NMIJ-AB as the calibration standard. Both NIM and NMIJ have observed a difference of 10% in analytical sensitivity between the BCR CRM 626 arsenobetaine solution and the NMIJ CRM 7901-a one. An Investigation was conducted to determine the cause. The outcome of the investigation was presented at the next workshop in April 2009.

APMP.QM-P13: Metals in Polypropylene

Dr. Feng Liuxing of NIM China presented the results of the study on the determination of cadmium, mercury, chromium and lead in polypropylene, which ran in parallel with a CCQM P-106 pilot study. This was the first comparison of polymers and plastics in response to the implementation of the RoHS's directive. Cadmium and chromium represented the class of elements of minimum challenge, lead of medium challenge and mercury of greatest challenge. There were a total of 22 participants; however only one laboratory, DSS from Thailand participated in APMP.QM-P13.

Overall it was a successful study with good agreement of results from most experienced laboratories. The study showed that the technique – INAA – is a potential primary method for the determination of mercury and cadmium in this matrix. The study also uncovered the potential use of XRF for the determination of chromium, mercury and lead.

APMP.QM-K24 (APMP.QM-P12): Cd in Rice

Dr. Jin Seog Kim informed the meeting that the rice samples for the key comparison study and pilot study were dispatched in July 2008 and August 2008 respectively and reminded participants of the deadline for the submission of results.

Progress Reports on TCQM Key and Supplementary Comparisons

Dr. Jin Seog Kim reported about APMP.QM-S1: Gravimetry, a supplementary study designed for gravimetric comparison of gas mixtures involving Helium in Nitrogen. Four organizations participated in the study. Dr. Kenji Kato reported on APMP.QM-S2, a bilateral study between NMIJ and NMIT coordinated by NMIJ on O₂ in N₂ and Dr. Jin Seog Kim reported on APMP.QM-K1c, a bilateral comparison between NIM & KRIS. Dr. Kim announced that the report would include the degree of equivalence to show linkage to CCQM-K1c.

Reports from Member Economies

Dr. Prabhat K. Gupta, NPLI, India, Dr. Laly Samuel, MSL, New Zealand and Dr. Sumardi, LIPI, Indonesia briefly shared their ideas on the development of metrology in chemistry as well as reporting on the activities relating to chemical metrology in their respective economies.

Forthcoming TCQM Key/Supplementary/Bilateral and Tri-lateral Comparisons and Pilot Studies

Dr. Charun Yafa from NIMT, Thailand proposed an inter-comparison study on preservatives in curry paste as there is a current lack of CRM in this matrix to support the testing need of the industry. In this pilot study, curry paste will be fortified with sorbic acid and sodium benzoate. He gave an overview of the process of production of this material as well as the stability and homogeneity data. He added that further stability studies at -80°C, -20°C and +4°C will be conducted and the samples would be irradiated with gamma ray before they are ice-packed and delivered to participants. A number of economies have indicated interest in participating in this study.

Future Meeting

The General Assembly selected Thailand as the venue for the 26th GA and related meetings in 2010.

Dr. Laly Samuel
MSL
New Zealand

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Activities of the COOMET TC 1.8 "Physical Chemistry"

The Technical Committee 1.8 "Physical Chemistry" was established in the framework of COOMET in 2002. Since this time, there have been major efforts made actually on the choice of activity directions, involvement of National Metrological Institutes (NMIs) of COOMET Member States in the field of its activity, forging of relationships and participation in activities of related committees and working groups of other international organizations.

Whereas the information about activities of the TC 1.8 is being published in CITAC News for the first time, this report is an attempt to show the main results as well as current activity of TC 1.8.

General Information

Activities of the COOMET Technical Committee 1.8 "Physical Chemistry" covers those measurement services, which are referred to "Metrology in chemistry" category. Twenty-two NMIs from 17 COOMET Member States are represented in TC 1.8 now. They are: AZSTANDART (Azerbaijan), NIM (Armenia), BelGIM (Belarus), BIM (Bulgaria), PTB and BAM (Germany), KazInMetr (Kazakhstan), NISM (Kyrgyzstan), CIM (KNDR), INIMET (Cuba), VMT (Lithuania), INSM (Moldova), INM (Romania), VNIIM, VNIIFTRI, UNIIM, VNIIOFI VNIIMS (Russia), SMU (Slovakia), Ukrmetrteststandart (Ukraine) as well as CNS Uz (Uzbekistan) and GEOSTM (Georgia), who joined the TC 1.8 in the last 2-3 years.

The main directions of COOMET TC 1.8 activities are:

- Preparation of the NMI CMCs of the COOMET Member States, to be placed in the BIPM KCDB;
- Organization of an internal review of CMCs of COOMET NMIs and carrying out of an interregional review of CMCs of other regional metrological organizations;
- Planning and organizing international comparisons and interlaboratory researches;
- Getting the TC 1.8 Members acquainted with the CCQM and COOMET documents concerning realization of the MRA and assurance of metrological traceability;
- Improvement of the TC 1.8 structure;

- Rendering of metrological services in the field of physico-chemical measurements.

TC 1.8 Projects

Only the NMIs of COOMET Member States as well as interested metrological centres and designated laboratories of other countries that have an appropriate basis of standard equipment can take part in the TC projects on international comparisons and pilot researches.

The most significant projects of the TC during the last years were:

- COOMET.QM-K3 "Automobile gases (CO, CO₂, C₃H₈) in Nitrogen" VNIIM (Russia), BelGIM (Belarus), BAM (Germany), Ukrmetrteststandart (Ukraine) Coordination: VNIIM (Russia);
- COOMET.QM-K23.b "Certified reference materials - synthetic natural gas" VNIIM (Russia), BelGIM (Belarus), BAM (Germany), SMI (Czech. Rep.), SMU (Slovakia), Ukrmetrteststandart (Ukraine) Coordination: VNIIM (Russia);
- COOMET.QM-K1.a "Certified reference materials - gas mixtures CO in nitrogen" VNIIM (Russia), BelGIM (Belarus), BAM (Germany), Ukrmetrteststandart (Ukraine) Coordination: VNIIM (Russia);
- COOMET N° 421/RU/08 "The supplementary comparison of pH working standards" VNIIFTRI, VNIIM, UNIIM, VNIIMS (Russia), NISM (Kyrgyzstan), BelGIM (Belarus), Ukrmetrteststandart (Ukraine), KazInMetr (Kazakhstan) and Regional centers of standardization and metrology (CSM) of Russian Federation: CSM of Novosibirsk, CSM of Nizhny Novgorod, CSM of Rostov, CSM of Ekaterinburg, CSM of Khabarovsk, Rostest-Moscow, Test-S.Petersburg, Ural-Test Coordination: VNIIFTRI (Russia);
- COOMET N° 379/RU/06 "Pilot comparison in the field of moisture measurements in cereal grains and cereal products" BelGIM (Belarus), Ukrmetrteststandart (Ukraine), UNIIM (Russia), SP TRIS (Sweden), PTB (Germany) Coordination: UNIIM (Russia).

A number of other studies, as well as PT schemes and industrial comparisons were run as well. These projects' objectives were of regional and international interest: aerosol particles, gene-modified objects in food, standard gas mixtures of ecological and technological purpose.

The Following New Key Comparisons and Pilot Researches are Planned for 2010-2011:

- COOMET N° 483/RU/09 "Key comparison of primary standards of components concentration in gas media - NO in Nitrogen (100 and 1000 $\mu\text{mol/mol}$)";
- COOMET N° 484/RU/09 "Key comparison of primary standards of components concentration in gas media - SO₂ in Nitrogen (100 and 1000 $\mu\text{mol/mol}$)";
- "Key/Pilot comparison of solutions with low nominal values of electric conductivity" (on a stage of registration);
- COOMET N° 375/RU/06 Pilot comparison "Determination of gene-modified objects in food";
- COOMET N° 435/RU/08 "Pilot comparison in the field of aerosol particles mass concentration measurements";
- COOMET N° 479/RU/09 "Supplementary comparison in the field of moisture measurements in cereal grain and cereal products";
- COOMET N° 362/RU/06 "Pilot comparison in the field of measuring the aqueous glucose solutions";
- COOMET N° 367/RU/06 "Pilot comparison in the field of blood elements".

Cooperation with CCQM

Representatives of TC 1.8 have participated in CCQM activity practically since its establishment and became members of the working groups KCWG (Key Comparison working group), OAWG (Organic Analysis working group), GAWG (Gas Analysis working group), IAWG (Inorganic Analysis working group), BAWG (Bio Analysis working group), EAWG (Electrochemical Analysis working group).

During 2008-2009, NMIs COOMET (VNIIM, VNIIFTRI, UNIIM) participated in the following CCQM comparisons:

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Gas Analysis

CCQM-K79/P110 "Nitrogen dioxide 10 $\mu\text{mol/mol}$ ",
CCQM-K51 "CO in nitrogen (5 $\mu\text{mol/mol}$)",
CCQM-K65 "Mercaptans in methane" (coordination of VNIIM),
CCQM-K71 "Multi-component gas stack emissions",
CCQM-K66 "Purity analysis of methane",
CCQM-K68 " N_2O at ambient levels";

Inorganic Analysis and Electrochemistry

CCQM-K34.2 "Assay of Potassium hydrogen phthalate",
CCQM-P111 "Seawater salinity",
CCQM-P119 "Lead in solder",
CCQM-P100.3 "Determination of low levels of Hg in natural water",
CCQM-P118 "Toxic metals in algae";

Organic Analysis

CCQM-K79 "Comparison of value assigned CRMs and PT materials for ethanol in aqueous matrix",
CCQM-P109 "Determination of acrylamide in cooked high-carbohydrate food (potato chips)",
CCQM-P91 "Pesticides in foods: pyrethroids in apple juice";

Bio Analysis

CCQM-P113 "Relative quantification of genomic DNA fragments extracted from a biological tissue",
CCQM-P103 "Measurement of multiplexed biomarker panel of RNA transcripts",
CCQM P94.1 "Quantification of DNA methylation".

APMP

Since 2008 VNIIM (Russia) has become a full member of APMP. KazInMet (Kazakhstan) is an associated member of this Regional Metrology Organization.

At present VNIIM participates in two projects of APMP in the field of Metrology in Chemistry: APMP.QM-P14 "Preservatives in a curry paste", APMP.QM-K41 " H_2S in nitrogen".

EURAMET

VNIIM is an associated member of EURAMET. Cooperation with TC METCHEM EURAMET

consists mainly of participation in projects of international comparisons. Particularly, VNIIM and Ukrmetrteststandart participated in EURAMET comparisons: EUROMET.QM-K4 "Ethanol in air", EUROMET.QM-K1.c "NO in nitrogen", EUROMET 764 " NH_3 in nitrogen", EUROMET 833 "PCB congeners in organic solution", EUROMET 621 "Key comparison(s) in humidity (dew-point temperature)".

APEC, ANMET

Cooperation of TC 1.8 with these organizations in 2009 was realized through active workshops: "Strengthening Chemical Metrology Infrastructure"; "Traceability in Material Testing to Reduce Technical Barriers to trade".

ISO TC

Representatives of TC 1.8 participate in the development of standards and carry out an expertise in the ISO TC 146 (Air Quality), TC 158 (Gas Analysis), TC 190 (Soil Quality), TC 193 (Natural Gas).

OIML

Representatives of VNIIM and VNIIFTRI are the Chairs of OIML TC 17 "Physico-chemical measurements" and its sub-committees (SC) SC2 "Saccharimetry", SC3 "pH-metering", SC4 "Conductometry", SC5 "Viscosimetry", SC6 "Gas Analysis", and participate in the development and expertise of standards.

Implementation of MRA

In order to achieve comparability of measurements of national standards, COOMET NMIs participated in more than 100 CCQM, COOMET and EURAMET key and pilot comparisons.

As a result of these activities, COOMET NMI has included 256 CMCs into the international database (BIPM KCDB) in the field of Metrology in Chemistry.

Distribution of CMCs by NMIs and measurement fields is shown in Table 1, where N is the number of CMCs.

Table 1. Distribution of CMCs by NMIs and measurement fields

NMI	N
VNIIM	236
VNIIFTRI	7
BelGIM	2
Ukrmetrteststandart	7
UNIIM	4
Measurement field	N
Gases	185
Organic solutions	21
Inorganic solutions	13
Metals and alloys	9
Sediments, soils, ores and particulates	4
High purity chemicals	3
Food	5
Water	1
Electrochemical analysis	15

Meetings and Events

- 2nd All-Russian Conference "Certified Reference Materials in Measurements and Technologies", May 2008, St. Petersburg, Russia;
- 3rd International Conference "Metrological assurance of physico-chemical and optico-physical measurements", November 2008, Kiev, Ukraine;
- International Scientific and Practical conference "Metrology – 2009", April 14-15, 2009, Minsk, Belarus;
- 3rd International Competition "The Best Young Metrologist of COOMET 2009", April 14-15, 2009, Minsk, Belarus;
- International Seminar "Mathematics, statistics and computation to support measurement quality", June 30, 2009 - July 2, 2009, COOMET, St. Petersburg, Russia.

Numerous events devoted to celebration of D.I. Mendeleev's 175th anniversary (08.02.2009) were organized in 2009. Many Russian and foreign scientists, including metrologists, took part in them. D.I. Mendeleev is appreciated in Russia not only as the author of the Periodic Table of Elements but also as a founder of metrology as a science and of the first National Metrology Institute in

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Russia, which is named after him at present as D.I. Mendeleev Institute for Metrology (VNIIM).

Main Events in Celebration of Mendeleev's 175th Anniversary

- February 9, 2009 – Meeting of the Presidium of Saint-Petersburg Department of Russian Academy of Science
- February 11, 2009 – Joint session of the Presidium of Metrological Academy and VNIIM Scientific Council
- February 25, 2009 – Scientific conference “D. I. Mendeleev heritage: historical aspects and modern view”, Moscow, Polytechnical Museum

- May 20, 2009 – International readings named after D. I. Mendeleev, Saint-Petersburg, VNIIM
- Jubilee exhibition in Polytechnical Museum (Moscow) devoted to D. I. Mendeleev.

Translation of International Documents

Russian, as well as English, are the official languages of the COOMET Member States. For the majority of specialists from countries of CIS (Commonwealth of Independent States) Russian is the main language of international communication and the scientific literature in Russian is in demand as before. Besides CIPM and CCQM documents, at the initiative and with the active participation of COOMET TC

1.8 representatives translated such important documents as:

- VIM3 “International Vocabulary of Metrology - Basic and General Concepts and Associated Terms” (translation of BELGIM and VNIIM, 2009)
- EURACHEM/CITAC Guide “Quantifying Uncertainty in Analytical Measurement” (translation of VNIIM, 2002);
- EURACHEM/CITAC Guide “Traceability in Chemical Measurement” (translation of VNIIM, 2005).

Dr. L.A. Konopelko

Dr. Y.A. Kustikov

Dr. O.V. Efremova, VNIIM, Russia

Activities of the EURAMET Technical Committee for MiC in 2009

Second Phase of the European Metrology Research Program

The European Metrology Research Program (EMRP) has been a crucial factor in the evolution of the European Association of National Metrology Institutes EURAMET e.V. in recent years. EURAMET's reorganization as a legal entity was required in order to prepare for a research programme co-funded by the European Commission under article 169 of the EC treaty. The first phase of EMRP is the currently running iMERA-Plus initiative launched in 2007 with a total value of 64.6 M€ under FP7 (ERA-NET Plus). iMERA-Plus projects started in May 2008.

The second phase is the full European Metrology Research Program (EMRP) owned and executed by EURAMET. Following signature by the European Parliament and the Council, the Co-decision process was finalized for the EMRP. The Decision (No 912/2009/EC) was published in the Official Journal of the European Union on 30 September 2009. This second phase of the EMRP has a total value of 400 M€ over an approximately seven year period and is jointly funded by the European Commission and 22 participating countries. The EMRP will provide the opportunity for the user community and other stakeholders to directly suggest topics that the metrology community should address with its resources. Additionally,

researcher grant schemes will be available to bring external expertise into the research projects and there will be opportunities for organizations to participate in the research projects with their own resources where it is mutually beneficial to do so.

The first call for potential metrology research topics (PRTs, so called stage 1) was successfully launched in May this year for a Targeted Program in the energy sector with a budget of approximately 34 M€. The EMRP Committee has subsequently selected sixteen research topics (SRTs) from these PRTs submitted in stage 1. Many SRTs contain inputs from more than one PRT as similar ideas have been combined where appropriate. Energy is considered to be one of the “Grand challenges” of European Research and has a multidisciplinary character. This also applies to the SRTs – none of them is devoted exclusively to chemistry for this reason but chemical topics are an integral part of several SRTs. Those SRTs are the basis of the Joint Research Project Proposals which consortia are invited to submit. The second stage, the call for Joint Research Projects (JRPs) was started in early September and ended 2 November 2009. Consortia were formed and submitted project proposals which were presented in a Review Conference in Berlin on 25 & 26 November 2009 to a board of external referees from outside the EURAMET metrological community. The

results of the Conference were still pending at the time of writing. The start of successful project proposals is anticipated in the course of 2010.

Good progress was also made in the running JRPs under the iMERA-plus initiative. The current status of several projects was presented at several occasions such as the International Metrology Congress in Paris in June 2009.

Further Activities in TC-MC

All EUROMET-bodies continue to work under EURAMET including the chemistry sector, now called Technical Committee for Metrology in Chemistry (TC-MC) in line with the nomenclature of the other TCs. The internal structure of TC-MC remained unchanged with four Sub-Committees, Inorganic Analysis (convenor: Christophe Quétel, IRMM), Organic Analysis (Gavin O'Connor, LGC), Electrochemical Analysis (Petra Spitzer, PTB) and Gas Analysis (Rob Wessel, NMi). Gavin O'Connor (LGC) was introduced as the new convenor of the Sub-Committee on Organic Analysis as the successor to Franz Ulberth (IRMM).

The second meeting of EURAMET TC-MC was conducted in Bucharest, Romania from 3-6 February 2009. All Sub-Committees had separate meetings on the first two days of the

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conference and a TC-MC plenary meeting was held on 5-6 February. There were 60 representatives from 30 institutions and 23 countries attending the meeting (Fig. 1).

A EURAMET Workshop dealing with the EMRP: "Preparation for a Targeted Program Energy" was held on 5 February. Following an initial presentation by Jörn Stenger, Member of the EMRP-Committee, on "Recent developments of the EMRP Program", possible chemistry-related topics for a TP Energy were presented. Several aspects related to biofuels were addressed such as the analysis of impurities, electrochemical parameters, quality indicators and the calorific value, as well as methods to trace the regional and biological origin of biofuels. Other topics were related to alternative hydrocarbon fuels, such as Liquefied (LNG) or Compressed Natural Gas (CNG) and Liquefied Petroleum Gas (LPG). Measurands of interest are, e.g., the moisture content. Other issues discussed were measurements for the hydrogen cycle (hydrogen purity) and measurements of salinity gradients for efficient water power and drinking water production.

Although the European Metrology Research Program is more and more an integral part of joint activities in the chemistry sector of EURAMET, a wide range of further activities are ongoing and were also discussed in Bucharest. A major activity in the Sub-Committee for Inorganic Analysis is the EUROMET Project 924 supporting the EU water framework directive. The first step of the project linking NMIs and reference institutes as Potential Calibration Laboratories for priority metals has been completed. This step was divided into two parts, covering the analysis of metals in pure water and natural water, and was carried out in parallel with a CCQM pilot study which concentrated on mercury (CCQM-P100.1 and P100.2). The final report for CCQM-P100 was presented in March 2009. Another part of the EUROMET Project 924 is the EUROMET supplementary comparison, Euromet.QM.S2,



Fig. 1. Participants of the TC-MC meeting 2009, Bucharest, Romania

which is concerned with the analysis of Ni, Cd and Pb. The final report for this part was presented in April 2009. Step 3 of the project is concerned with the analysis of Hg, Ni, Cd and Pb in natural water at a concentration level as required by the European Environmental Quality Standard (EQS) and involves more than a hundred testing laboratories across Europe. Regional comparison measurements organized by the reference institutes are made in order to link these laboratories with the local monitoring laboratories. The reference values are provided by the potential calibration laboratories. NMIs also participate in these comparisons in order to control the success of the validation. As a result, a link between PCLs, NMIs and testing laboratories should be established. This part was finished at the end of 2009.

The purpose of EUROMET Project 843 in the Sub-Committee for Electrochemical Analysis was to produce new recommendations for the calibration and evaluation of pH on-site measuring instruments in the field of water analysis. The resulting protocol is intended for on-site measuring equipment for the determination of pH in water. It gives guidance to the end-user on how to estimate the measurement uncertainty of the pH value following standard procedures. The vast majority of responses were very positive and the document should be translated to make it available for end users. Possibly, a similar protocol shall be developed for multi-parameter instruments in a future project. In total, 14 projects were discussed in the Sub-Committee for Gas Analysis. Five projects were finished, four projects started in 2008. They are concerned with issues like laser

based spectroscopy (EUROMET 934), trace purity measurements (EUROMET 937), ultra trace water vapor (EURAMET 1002) or combustion particles (EURAMET 1027). New project proposals were presented, for example on a comparison on mixtures of ethanol in water saturated air (OIML recommendation).

EUROMET Project 886 is now finished. It was related to volatile organic compounds (VOCs) at ambient concentrations in the atmosphere. These play an important role in the photochemical generation of ozone. EU Directive 2002/3/EC mandates countries within the EU to make measurements of the specific set of VOC ozone precursors involved in this comparison. 10 institutions from all over Europe and the United States and South Korea participated in this project. The World Calibration Centre for VOCs under the Global Atmospheric Watch Scheme in Garmisch-Partenkirchen in Germany also participated as a non-NMI. During this exercise the stability trial of 1-10 nmol/mol VOC mixtures provided by NPL (UK) showed less than 0.6% drift for most species over 1 year. The accuracy of these standards was further strengthened by the comparison results, which showed good agreement between participants.

A new proposal for comparison was made by LNE in the Sub-Committee for Organic Analysis and is concerned with herbicides in water as a support to the EU WFD. LNE has developed materials for the determination of herbicides in water stable for three years, namely pesticides in acetonitrile (in vials), and pesticides concentrated on a solid phase extraction. LNE proposes to organize an inter-laboratory comparison campaign with these materials in order to evaluate the intra and inter laboratory precision of this type of determination.

Dr. Bernd Güttler
Chair EURAMET TC-MC
PTB, Germany

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Recent Activities of the SIM Chemical Metrology Working Group

Activities of the SIM Chemical Metrology Working Group, implemented through a cooperative arrangement between OAS/SIM and the German Government, provide:

- chemical metrology awareness and strategic planning seminars
- comparison studies
- workshops and training activities

for building capacity, facilitating improved capabilities in Chemical Metrology and the dissemination of internationally-recognized measurement services within the Americas and throughout the world, all within the scope of and participation in the CCQM and the CIPM MRA.

Most recently, the SIM Chemical Metrology Working Group held events on 2 November and 5-6 November 2009 in Rio de Janeiro, Brazil. These events were held adjacent to the CCQM Working Group meetings held in Rio to facilitate the attendance of SIM members in the chemical metrology area at the CCQM meetings, since it was the first time that all the CCQM WGs met in South America. The meeting was attended by thirty-two SIM members representing twelve Economies, two Guests from AFRIMETS, one from APMP and CCQM President Robert Kaarls.

The Business Meeting featured presentations of SIM.QM-K1, "Ethanol in Aqueous Matrix", a review and summary of an activities needs assessment workshop held in Paraguay in May, 2009, a proposal for a Key Comparison of trace metals in drinking water and a strategic planning session focused on determining needs for Comparisons, Awareness Workshops and other Training Activities for the next three years. SIM.QM-K1 was the SIM's Chemical Metrology Working Group's first Key Comparison after more than 20 pilot capability assessment studies that have taken place over the past 10 years.

It was announced that the SIM Council had selected Ms. Gabriela Massiff of CMQ in Chile to assume Chairmanship of the Chemical Metrology Working Group effective 1 Jan 2010. The previous chair, Dr. Willie E. May of NIST,

agreed to serve as "deputy chair" for one year to assist with the transition.

The SIM CMWG has organized itself into three needs-based subgroups. Chairs are:

- CMWG Subgroup I: Dr. Yoshito Mitani, Mexico
- CMWG Subgroup II: Ms. Mónica Gualotuña, Ecuador
- CMWG Subgroup III: Ms. Saira Knox, Trinidad and Tobago

Characteristics and Focus of Activities within each Subgroup:

CMWG Subgroup - I

- **NMIs and/or DI that currently have Chemistry CMCs published in CIPM MRA Appendix C**

➔ Key and Supplementary Comparison Studies to support CMCs

CMWG Subgroup - II

- **Economies that have existing mandate for chemical metrology infrastructure, but currently have no CMC's published in MRA Database**

➔ Awareness seminars: Differences in expectations of NMI-like activities and those of a testing laboratory

➔ Workshops on CMC Preparation and Review

➔ Key, Supplementary, and Subsequent studies to underpin CMCs to be proposed

➔ Measurement Proficiency Assessment Studies

CMWG Subgroup - III

- **No current NMI (central or distributed) for Chemical Metrology activities**

➔ Need Assessment Seminars

➔ Assistance in framing arguments for obtaining sustainable government support

➔ Assistance in conducting Needs Assessments with relevant customer sectors

A SIM Senior Policy Makers' Dialogue Forum, sponsored by NIST, was held on Thursday and

Friday of that same week. The purpose of this Forum was to provide a venue for dialogue among policy officials and national decision-makers within the Americas regarding the need and value of investing in national programs in chemical metrology to support:

- International Trade
- Food Safety and Nutrition
- Sustainable Energy
- Healthcare Decision-Making
- Consumer Protection
- Sustainable Economy

Invitations for participation in this "Dialogue Forum" were extended to NMI Directors and a Government Official with policy-making authority and decision-making responsibility for metrology portfolios for economies within the ANDIMET, CAMET and SURAMET subregions of SIM that do not currently have government-supported programs in Chemical Metrology. A second such Forum is planned for economies within the CARIMET subregion as a part of the SIM General Assembly in October 2010.

The Forum kicked off with a reception on Thursday evening at Hotel Windsor Barra where Dr. Hratch Semerjian, Director of the U.S. Council for Chemical Research, gave a Keynote address entitled "Chemical Metrology: What it is and Why You should Invest in It". This was followed by a poster session whose theme was: "A National Program in Chemical Metrology: Why It's Needed and What are the Expected Impacts". Several countries/economies that currently have active "Metrology in Chemistry" participated.

Friday's program featured presentations and a roundtable discussion moderated by Willie E. May, Chair; SIM Chemical Metrology Working Group, which focused on "Chemical Metrology and Its Impact on Industry and Quality of Life: Testimonials and Dialogue" and dealt with the rationale for and return on investment from national programs in Chemical Metrology. The topics were discussed by: Prof. João Jornada, President, INMETRO Brazil; Ms. Gabriela Massiff, Director, Fundacionchile CMQ Chile; Dr. Yoshito Mitani, Director for Materials Metrology, CENAM Mexico; Dr. Wynand

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Louw, Acting CEO, NMISA, South Africa; Dr. Chainarong Cherdchu, Director for Chemical Metrology and Biometry, NIMT, Thailand; and Dr. Robert Kaarls, CIPM Secretary and CCQM President, The Netherlands.

Copies of these presentations are provided on the SIM Chemical Metrology Working Group webpage http://www.nist.gov/cstl/sim_forum.cfm. Prof. Humberto Brandi, SIM President and Director for Metrology at INMETRO also joined in the panel discussions.

SIM Chemical Metrology Working Group Summary:

- 20 countries participate in SIM CMWG meetings
- **15 NMI's or their designated labs are now regular participants in SIM intercomparison studies:** up from three in 2002.
- **Six** countries are now participating in CCQM meetings and various activities: up from **three** (Canada, Mexico and U.S.) in **2002**.

- **NMI's from six** countries (Argentina, Brazil, Canada, Chile, Mexico, U.S.) have chemistry CMCs in the BIPM Key Comparison Database.
- **Uruguay** plans to submit CMCs in the next few years.

Dr. Willie May
NIST
USA

Update on ISO/REMCO

ISO/REMCO is structured as shown in Fig. 1. The committee consists of Working Groups (WG) and Ad-hoc Groups (AHG) set up to implement a specific task under two Steering Groups (SG1 and SG2). The Chairman's Advisory Group (CAG) provides input to the Chair from the group convenors.

One of the main tasks of ISO/REMCO is preparing guides for the preparation, characterization, certification and use of reference materials and the competence assessment of reference material producers. A new information booklet has been published (in English and French) on the ISO/REMCO website (www.iso.org/remco) about the role of this ISO committee, its members, stakeholders, deliverables and working structure.

The main public output from ISO/REMCO in 2009 was the third edition of ISO Guide 34 "General requirements for the competence of reference material producers", which was unanimously approved by ISO members and was published

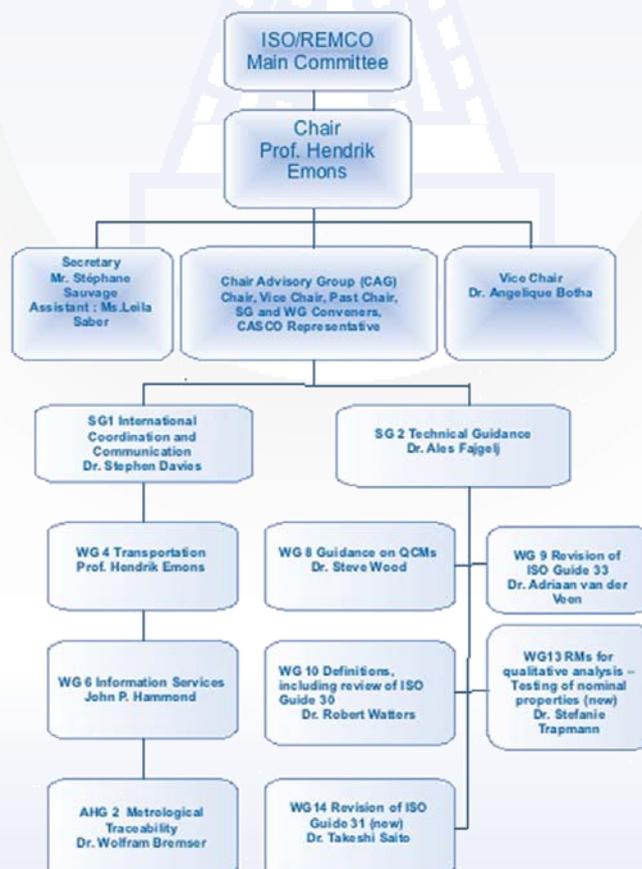


Fig. 1. ISO/REMCO structure

on 20 November 2009. Guide 34 describes the requirements for the quality management system and the technical competences of reference material producers (RMP). This 3rd revision aligns the document with ISO/IEC 17025:2005 and has undergone wide consultation with stakeholders, including accreditation organizations. ISO Guide 34 has become the worldwide standard for RMP accreditation and was included into the Mutual Recognition Arrangement (MRA) of National Metrology Institutes (NMIs) and other signatories of this MRA.

The 32nd meeting of ISO/REMCO was held in Teddington (UK) from 3-6 July 2009 and was hosted by LGC on behalf of the British Standards Institute (BSI). ISO/REMCO now has a membership of 70 members of the International Organization for Standardization (ISO) and liaisons with 18 international organizations and 6 ISO-internal committees. The liaisons are very important for the design and implementation of ISO/REMCO's work program. Most of the documents from ISO/REMCO

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directly affect work in other (international) organizations. It is important to avoid duplication of efforts and, where possible, to share resources, especially the limited number of experts. For this reason, ISO/REMCO has given a strong mandate to its Steering Group SG1 convener to ensure good communication. Moreover, the current extensive and broadly scoped work program has prompted ISO/REMCO to request assistance from additional contributors which can provide special experiences from their own field of activities.

ISO/REMCO updated its work program during the meeting in Teddington. Main activities for 2009 and beyond include:

- complete revision of the vocabulary related to reference materials (ISO Guide 30, 1992/Amd 1:2008), supported by

extensive consultation with other liaison organizations;

- complete revision of the guide on accompanying documentation (ISO Guide 31, 2000), expanded to cover all documentation for reference materials;
- complete revision of the guide on the use of reference materials (ISO Guide 33, 2000);
- development of a new guide for the in-house production of in-house used reference materials for quality control (ISO Guide 80);
- development of a guidance document for the production of RMs for “qualitative analysis” (testing of nominal properties);
- further studies into the matter of ‘metrological traceability’, in particular how to express the concept on CRM certificates and related documentation.

The Technical Report on RM classification (ISO/TR 10989:2009) has been published. A first draft of ISO/TR 11773 on RM transportation issues was discussed at the meeting, and it is envisaged that a voting draft will be available for review at the next meeting.

The next ISO/REMCO meeting will be held in Hangzhou, China on 3-7 May 2010.

Prof. Hendrik Emons
EC-JRC-IRMM, Chair of ISO/REMCO
Belgium

Dr. John P. Hammond
Convenor of ISO/REMCO WG 6
Starna Scientific Ltd., UK

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Comparison of Calibration Curves Using the L_p Norm



Luca Callegaro



Francesca Pennechi



Pier Giorgio Spazzini

Introduction

Interlaboratory comparisons are a fundamental task in metrology, as they are the main tool to check and maintain the compatibility between the various national measurement standards, and make them traceable to an international standard [1]. Statistical methods for the point wise analysis of such comparisons have been extensively studied, and several publications and guidelines are available on the subject (see, e.g., [2]).

There are several situations in which point wise analysis is a loose tool, for instance when the traveling standard is not a realization of the quantity of interest but a measuring instrument. Examples of such comparisons include those concerning mass flow rate, airspeed, ionizing radiation, photometry and so on.

In such cases it might be inconvenient or impossible for the laboratories to calibrate the instrument on the very same set of

specified points. Hence, the data sets realized by different laboratories are usually made equivalent by comparing “approximately coincident” points, but this method appears quite questionable. Since the measurement instruments’ response, although not always strictly linear, is usually smooth, one possible solution could be to determine their calibration curves to recompute the expected instrument responses at the cardinal points and then to compare such results. The logical evolution

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of this approach is to compare the curves as a whole.

Rationale

Comparing calibration curves would provide several advantages: first, it would make possible to globally assess the compatibility of the measurement sets; second, it would allow to obtain, through the global evaluation, at least some indicative results even at those cardinal points at which an outlier is measured.

This approach, although initially studied for interlaboratory comparisons, could be useful also in other situations, e.g., for analyzing the time drift of the calibration curve of an instrument. Possible applications in chemical analytical laboratories include development and validation of new methods, quality control and spectroscopic analysis. Presently, the comparison of absorption spectra of unknown compounds to reference spectra is performed using distance-to-model methods [3]. The method proposed here is an alternative, allowing to directly compute the distance between the measured and the reference spectra.

Calibration curves should be compared irrespectively of their mathematical formulation, and in a quantitative and objective way. To this aim, we will introduce a distance between two generic analytical curves based on the Least Power L_p norm of their difference.

L_p Norms

In this context, we restrict the definition of the L_p norm (with $p \geq 1$) of a function $f(x)$ to the case of bounded functions which are defined on the interval $[a, b]$, having length $\Delta = b - a$. If the Lebesgue integral of the p -power of the absolute value of f

$$\int_a^b |f(x)|^p dx \quad (1)$$

is defined and finite, then

$$\|f\|_p = \left(\frac{1}{\Delta} \int_a^b |f(x)|^p dx \right)^{1/p} \quad (2)$$

is the (normalized) L_p norm of $f(x)$ on $[a, b]$ [4]. If $f(x)$ describes a dimensional physical quantity, its L_p norm is defined to have the same dimension as $f(x)$. If $f(x) = k$, for k constant, then $\|f\|_p = |k|$, irrespectively of the interval $[a, b]$ and the parameter p .

Three special cases are particularly interesting; for $p = 1$, the norm is proportional to the area of the region bounded by the function graph (the shaded area in Fig. 1a) and is the mean value of $f(x)$ on the interval; for $p = 2$, it is proportional to the inertia moment of the function graph with respect to the x axis; for p tending to infinity, $\|f\|_\infty$ is the supremum of $|f(x)|$ (if the function is continuous on $[a, b]$, its supremum coincides with its maximum) on the interval (indicated by the arrow in Fig. 1a).

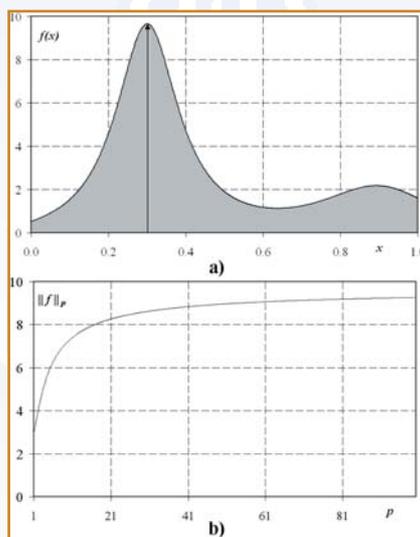


Fig. 1. (a) A generic function and (b) the evolution of its L_p norm as a function of p

Fig. 1b) depicts the dependence of $\|f\|_p$ on p . The monotonic-increase of the $\|f\|_p$ norm for increasing p is a general property [4]. It can be seen, as expected, that $\|f\|_p$ tends, for increasing p , to the maximum value of $f(x)$.

Figs 2a) and 2b) show function $f(x)$ together with another function $g(x)$ and the corresponding L_p norms. It is clear that the ordering of $\|f\|_p$ and $\|g\|_p$ is dependent on the value of p . Since the mean value of $g(x)$ is larger than that of $f(x)$, for low p values one has

that $\|f\|_p < \|g\|_p$ while, when increasing p , the higher peak of $f(x)$ becomes more important and the order between norms is inverted.

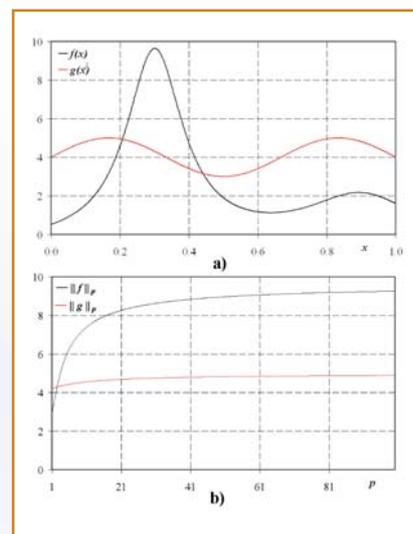


Fig. 2. (a) Two functions and (b) the evolution of their L_p norm as a function of p

Distance Between Curves

In order to evaluate the distance between two functions $f(x)$ and $g(x)$ defined over the same domain $[a, b]$, we propose to consider the L_p norm (2) of the difference between the two curves:

$$D^p = \|f - g\|_p \quad (3)$$

Obviously, this distance has the same properties as the L_p norm of a single function treated in the previous section. In particular, if the curves differ one from the other only by an additive term k (simple translation), then $D^p = |k|$, always. If the two curves coincide everywhere on $[a, b]$, then $D^p = 0$. If $g(x) = 0$ all over $[a, b]$, then $D^p = \|f\|_p$.

The same functions plotted in Fig. 2a) are plotted again in Fig. 3a), while Fig. 3b) reports their distance D^p ; two special cases of this distance are also graphically represented in Fig. 3a), namely $p = 1$ (the shaded area and p tending to infinity (the maximum distance between the curves, indicated by the arrow). The distance provides a global evaluation of the difference between the curves. For small

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values of p the distance takes into account the overall similarity of the curves, while for large values of p it highlights the maximum local differences between them.

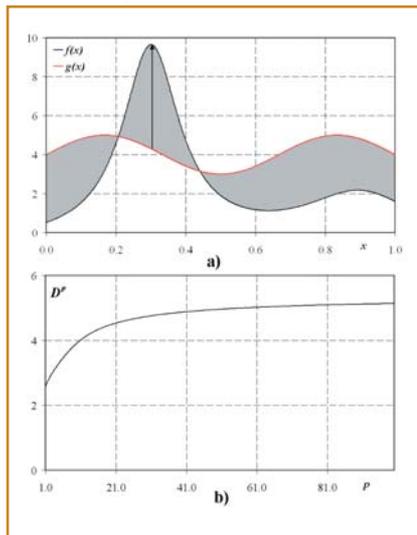


Fig. 3. (a): The same curves as in Fig. 2a) and graphical representation of two special cases of their distance; (b) Relative distance as a function of p

A small variation of the distance all over the range of p will indicate that the curves can be considered similar in shape, although shifted with respect to each other. In this case, the question is whether such (almost) constant distance represents a small or a significant amount with respect to the actual context. On the other hand, a small value of the distance for small p values followed by a strong increase for large p values will indicate that the curves are relatively close to each other except in some reduced regions where large differences may exist. In this case, one should decide which of the two aspects is more important for the aim of the actual analysis: the good overall agreement between the curves or their few but high discrepancies. Moreover, if a maximum value (e.g., a prescribed tolerance) for the distance between two curves is set, it is possible to check which range of p satisfies such condition; this result would allow to check whether the curves are within tolerance only in a global sense or also locally.

Calibration Curves vs a Reference Curve: an Example

We show here a specific application example, but many others can be conceived. Assume that the calibration curve of an instrument has the form of a second order hyperbola $f(x) = a_0 + a_1/x + a_2/x^2$, and that the instrument is calibrated at five different laboratories. The result of each calibration is the estimate of the parameters a_0, a_1, a_2 , whose corresponding curves are plotted in Fig. 4a) together with a reference curve REF, the red one, which is here considered as given.

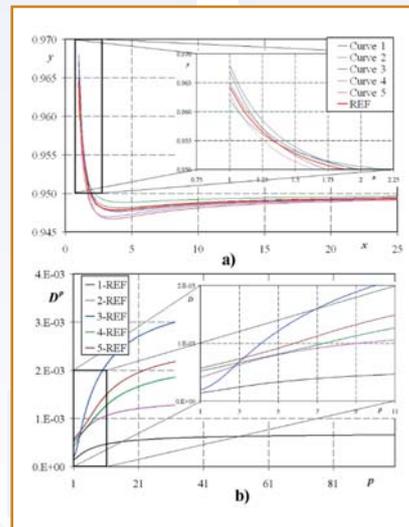


Fig. 4. (a) Diagrams of five pseudo-calibration curves (thin lines) and of a reference curve (thick red line); in the box, a zoom of the region with the biggest slope. (b) Distances of the five curves from the reference curve; in the box, a zoom of the region close to origin

Fig. 4b) shows distances (3) of the calibration curves from REF, as functions of p . For low values of p , curves 1 and 3 have the smallest distance. Actually, from Fig. 4a), curves 1 and 3 seem on the average closer to REF than the others. On the other hand, the distance of curve 3 from REF rises quickly and becomes the largest, for large p . In fact, curve 3 has the largest local distance from REF among all curves, as can be seen by carefully observing the zoom in the box of Fig. 4a).

Conclusions and Future Developments

The distance (3) can be employed to rank calibration curves with respect to a reference one. If the interest is focused on the overall differences between the curves, a small p value is appropriate, for example, when dealing with a global assessment of a calibration laboratory in an audit. Otherwise, if large (although local) differences are important for the context, a large p value should be used, for example, to identify the presence of outliers.

Two major problems are currently subjects of a work in progress:

- the determination of a reference curve, which will be addressed by considering, as the reference curve, the function minimizing the norm of the vector of the distances between the calibration curves and the function itself;
- the evaluation of the uncertainty associated to the distance between a calibration curve and the reference one, which will be undertaken by considering the calibration curves as random functions.

This article is a summary and update of the CITAC Award winning paper published by the authors in Accreditation and Quality Assurance (2009) 14:587-592.

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Dr. Luca Callegaro
Dr. Francesca Pennecci
Dr. Pier Giorgio Spazzini
 INRIM, Italy

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Metrological Aspects of Glucose Measurements by Biosensors



Samuel Wunderli



Hanspeter Andres

Introduction

National regulations and the European Directive on In Vitro Diagnostic Medical Devices (IVD Directive, 98/97/EC) [1] demand that traceability of values assigned to calibrators and/or control materials must be assured through available reference measurement procedures and/or available reference materials of a higher order. The Joint Committee on Traceability in Laboratory Medicine (JCTLM) provides a worldwide platform to promote and give guidance on internationally recognized and accepted equivalence of measurements in laboratory medicine and traceability to appropriate measurement standards. Based on its legal mandate the Federal Office of Metrology METAS (the Swiss national metrology institute) is building up a laboratory infrastructure for electro-analytical chemistry to provide a valuable contribution to the traceability of glucose content measurements by biosensors and ion activity measurements.

The measurement of blood glucose levels is one of the most common tasks of a clinical chemical laboratory. Various types of instruments detect and report fundamentally different glucose quantities. In the clinical chemistry laboratory routine glucose measurements are primarily performed by photometry based on the absorbance of molecules involved in the enzymatic conversion of glucose in diluted samples. Results are reported as glucose concentration, amount of glucose per volume of specimen. Glucose concentration standards are available either as pure materials or frozen human serum with concentration in the physiological range ($2 \text{ mmol}\cdot\text{L}^{-1}$ to $20 \text{ mmol}\cdot\text{L}^{-1}$).

Self-monitoring and point-of-care testing devices use direct reading biosensors that do not need prior dilution of the test sample. They respond to (active) glucose molality, the amount of glucose per unit mass of water. The quantity molality is identical in whole blood, plasma or aqueous solutions. But, for concordance with traditional methods, the results are converted to concentration. This quantity now depends on the water content, which is different for whole blood, plasma and aqueous solutions. The International Federation of Clinical Chemistry and Laboratory Medicine (IFCC) therefore recommends reporting result for blood glucose always as concentration of glucose in plasma. [2] For each calibration solution the factor varies. False conversion factors in the calibration of glucose monitors with concentration standards can thus lead to false positive or false negative measurement results. [3]

In this work we present a calibration method for glucose measurements without the need of prior dilution of the specimen. Chronoamperometric measurement signals of a glucose sensitive biosensor based on Glucose-Oxidase and a mediator that prevents the formation of hydrogen-peroxide are used as the direct measure of the active glucose molality. This work focuses on the metrological aspects of such a quantitative electroanalytical measurement method. Up to now, neither a thorough uncertainty evaluation of chronoamperometric measurements results of glucose contents has been done nor has their traceability to internationally recognized standards been established.

Experimental

The amperometric sensor is a three-electrode device mounted in the fully automated flow system depicted in Fig. 1. The working

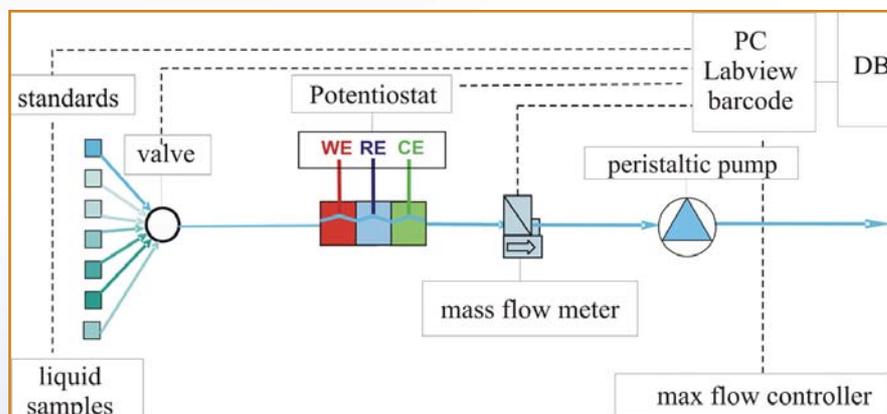


Fig. 1. Amperometric flow system at METAS equipped with flow cell and electrodes: WE = working electrode, RE = reference electrode, CE = counter electrode and DB = database

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Table 1. pH, ionic strength I and glucose molality m of the buffered glucose standards and test samples

name	buffer type	pH	ionic strength I , mmol·kg ⁻¹	glucose molality m , mmol·kg ⁻¹	uncertainty u_m , mmol·kg ⁻¹ %	
I1	imidazole	7.2	156.788	2.761	0.015	0.53
I2	imidazole	7.2	157.216	11.324	0.028	0.25
I3	imidazole	7.2	156.795	5.650	0.022	0.38
I4	imidazole	7.2	156.794	8.446	0.025	0.29
I6	imidazole	7.7	148.623	5.465	0.018	0.33
I7	imidazole	6.7	166.738	5.372	0.018	0.34
I9	imidazole	7.2	238.290	~ 5.5		
P1	phosphate	7.2	130.611	2.747	0.015	0.53
P2	phosphate	7.2	130.611	11.271	0.028	0.25
P3	phosphate	7.2	130.611	5.497	0.021	0.39
P4	phosphate	7.2	130.611	8.298	0.025	0.30
P6	phosphate	7.5	128.021	5.479	0.018	0.33
P7	phosphate	6.5	132.473	5.459	0.018	0.33
P9	phosphate	7.2	211.035	~ 5.5		

electrode is composed of platinum contacted glucose sensing paste. The paste consists of glucose oxidase (GOD), the mediator system Tetrathiafulvalene - p-Tetracyanochinodimethane (TTF - TCNQ) and graphite suspended in silicone-oil. [4] To prevent depletion of the water soluble GOD during operation the paste is protected by a 20 - 50 μm cellulose cut open tubular membrane. The reference electrode is a closed system Ag/AgCl gel-type reference electrode. The counter electrode is simply a platinum wire. The current between the working and counter electrode after applying a constant voltage between the working and reference electrode is measured with a potentiostat. The electroanalytical signal together with the environmental and flow system control parameters are recorded in a database.

A standard for glucose measurements is a buffered and sterilized aqueous glucose solution of known molality. In this work aqueous imidazole or phosphate buffers in the pH range 6.7 - 7.7 were used. Where necessary, lithium chloride was added to adjust the ionic strength to the physiological range. Fouling of the buffered aqueous stock solutions is

prevented by adding sodium azide. The purity of the glucose used was $99.7 \pm 0.4\%$ ($k = 2.95\%$ confidence interval). The gravimetric data of all imidazole buffered glucose standards and a test sample used in this study are summarized in Table 1. The uncertainty calculation for the glucose molality includes uncertainty contributions of the weighing data and environment as well as of the glucose purity.

Theory

Glucose oxidase is often used in direct reading biosensors to determine glucose enzymatically, a process during which the chemically active form of β -D-glucose is transformed highly selectively into 5-gluconolactone. Two electrons are released for each converted glucose molecule and mediated to the Platinum electrode at a constant potential. This electron stream is the evaluable measuring signal in chronoamperometry and is directly proportional to the glucose content of the sample under test. [5] The limiting current I_l at a planar electrode with strong radial diffusion is directly proportional to the substrate activity a_s :

$$I_l = \frac{n A F a_s D_s}{\delta_N} = K_S a_s \quad (1)$$

n is the number of released electrons, A is the geometric surface of the planar electrode, F is the Faraday constant, D_s is the diffusion coefficient of the substrate glucose and δ_N is the thickness of the Nernst diffusion layer. In this work eq. 1 is extended by a dynamic term $k(t)$ and a drift term $D(t)$ as shown below:

$$I_l = \kappa(t) K_S a_s + D(t) = \kappa K_S a_s + D \quad (2)$$

The quantification of the uncertainty is done according to the principles outlined in GUM [6]. The combined uncertainty of the potential signal result from eq. (2) is equal to the positive square root of the combined variance $u_c^2(I_l)$, which is given by:

$$u_c^2(I_l) = \sum_{i=1}^N \left(\frac{\partial I_l}{\partial x_i} \right)^2 u^2(x_i) \quad (3)$$

Covariances are not considered in our approach at present. The standard uncertainties $u^2(x_i)$ of each influence x_i are either of the A- or B-type. A-type uncertainties can be evaluated from statistical distribution of the results of a series of measurements, while B-type uncertainties are evaluated from assumed a priori probability distributions based on experience, and from other information originating from separate experimental results.

Results and Discussion

In Fig. 2 the chronoamperometric response signal I of a glucose sensitive electrode during the course of a fifty hour experiment is depicted. The measuring system was calibrated by alternating the glucose standards I1, I2, I3, I4, I1 and P1, P2, P3, P4, P1, respectively. For each buffer type two independent calibrations were performed. The samples I9 and P9 were treated as unknown test samples and measured in between the respective system calibration. After each solution change the system was flushed with the subsequent solution for two minutes. The measurement interval is two hour per sample. The observed current varies in the range of 100 nA to 500 nA, depending on the glucose content of the sample under test. As depicted in Fig. 2 only a very small

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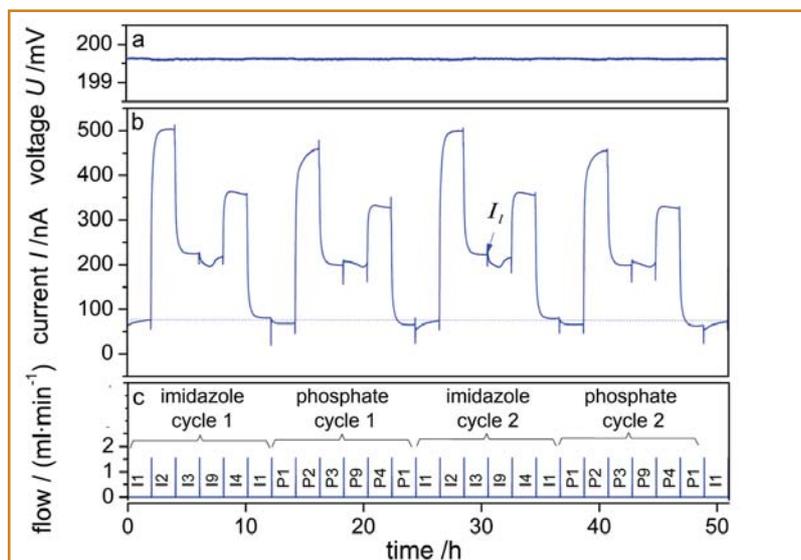


Fig. 2. A fifty hour chronoamperometric experiment is shown: a) measured applied voltage U between working and reference electrode, b) measured resulting current I between working and counter electrode. The limiting current value for the standard I3 in the imidazole cycle 2 is marked. The broken line visualizes a small linear signal drift $D(t)$ for the standard I1. Part c) is measured flow displaying the sample changes within the two measurement cycles of the imidazole and phosphate buffer system, respectively

linear drift of approx. 2 nA / 24 h is observed. Typically the signal reaches a steady state after an hour of measurement of a new sample. For the samples I9 and P9 the response behaviour is different; after a decrease of the signal within the first hour the current increases again towards the end. This behavior is attributed to the reference electrode, which stabilizes much later due to the different ionic strength of the samples (see Table 1). Clearly, for the test sample P9 the steady state is reached at a

latter time than for the test sample I9. For the subsequent signal analysis the last 100 s before a sample change are used to determine the limiting current I_l . The mean experimental values of I_l for all cycles are summarized in Table 2.

The uncertainty budget for the response signal I_l of the sample I2 and P2, respectively, is listed in Table 3. The mathematical expressions of the derivatives in eq. (2) are given together

Table 2. Mean experimental values of current I_l are summarized of measurement cycle 1 of the imidazole and phosphate buffer system, respectively. The respective standard deviations $s(I_l)$ are given in brackets

sample	imidazole cycle 1 current I_l (nA)	sample	phosphate cycle 1 current I_l (nA)
I2	503.1(4)	P2	460.3(3)
I3	224.9(2)	P3	199.2(2)
I9	217.3(4)	P9	201.0(9)
I4	357.1(5)	P4	327.3(1)
I1	81.2(2)	P1	65.4(1)

with typical values x_i for all influences x_i of the model eq. (2). Table 3 also lists the measured limiting current I_l and calculated combined uncertainty $u(I_l)$.

Whereas for $u(\kappa)=0.000$ all three uncertainties $u(a_3)$, $u(\kappa)$ and $u(D)$ contribute to the combined uncertainty, for $u(\kappa) = 0.005$ the uncertainty of I_l is dominated by the uncertainty of the dynamic term k .

The glucose activity for the test samples I9 and P9, respectively, is determined as follows. From the glucose molalities of the standards I1, I2, I3, I4, P1, P2, P3 and P4 in Table 1 and the measured limiting current values of the standards and the two test samples I9 and P9 in Tables 2 and 3, respectively, the glucose molality can be determined using the bracketing technique with a linear regression according to ISO standard 6143 [7]. In this type of linear regression both the uncertainty in the glucose molalities as well as the uncertainty in the measured currents are taken into account. The calibration and analysis functions are depicted in Fig. 3 assuming no uncertainty in the dynamic term k . The resulting values for glucose activity of the samples I9 and P9 are summarized in Table 4.

The values determined are concordant between the two measurement cycles and buffer systems, as can be seen from Fig. 3 and Table 4. Concordance is already achieved assuming zero uncertainty in the dynamic term for both buffer types. For the imidazole buffered test sample I9 the glucose activity can thus be determined with a combined uncertainty of 0.5 % rel. For the phosphate buffered test sample P9 the combined uncertainty is 0.8 % rel.

Conclusions

Based on the model equation for the measurand, all significant sources of uncertainty of the analyte result are identified, their magnitude estimated from published and experimental data and finally mathematically combined to give the combined uncertainty in the reported value of the glucose molality. It is found, that the combined uncertainty of the glucose molality comprises mainly uncertainty contributions from the non-ideal behavior, the

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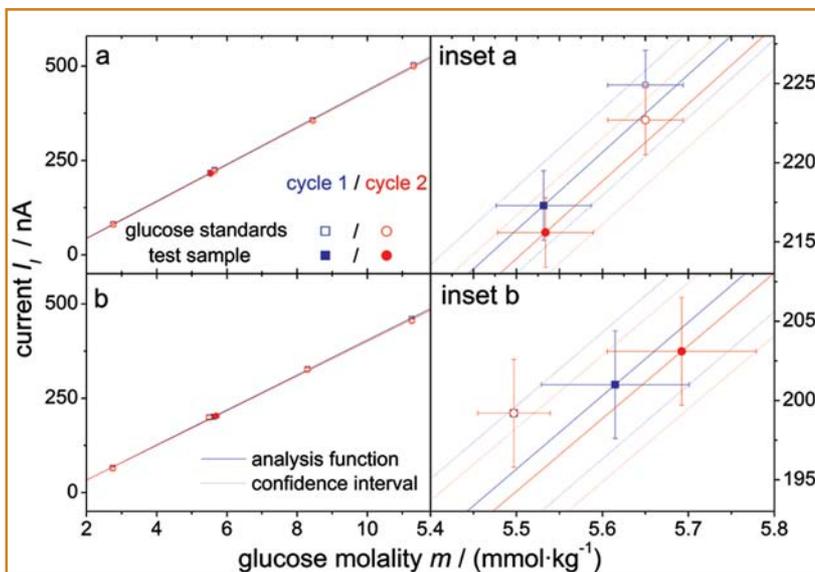


Fig. 3. Calibration functions with four glucose standards (open symbols) and one test sample (full symbols) are depicted; a and inset a) standards I1, I2, I3, I4, test sample: I9; b and inset b) standards P1, P2, P3, P4, test sample: P9. The analysis function is the inverse of the calibration function. From the depicted analysis function (full lines) with 95 % confidence interval (dotted lines) the unknown glucose activity of the test samples and their respective uncertainties can be mathematically determined

Table 3. Uncertainty budget and resulting limiting current and combined uncertainty of the standards I2 (imidazole buffer) and P2 (phosphate buffer) in cycle 1 are listed

influence	sensitivity factor	typical values	uncertainty	
X_i	$\frac{\partial I_i}{\partial x_i}$	x_i	$u(x_i)$	type
a_s	κK_s	I2: $a_s = m_s = 11.301 \text{ mmol}\cdot\text{kg}^{-1}$ P2: $a_s = m_s = 11.249 \text{ mmol}\cdot\text{kg}^{-1}$	$0.002 \cdot a_s$	B
K_s	κa_s	I2: $44.428 \text{ nA}\cdot(\text{mmol}\cdot\text{kg}^{-1})^{-1}$ P2: $40.839 \text{ nA}\cdot(\text{mmol}\cdot\text{kg}^{-1})^{-1}$	I2: $0.5 \text{ nA}\cdot\text{a}_s^{-1}$ P2: $1.0 \text{ nA}\cdot\text{a}_s^{-1}$	A
κ	$K_s a_s$	1.000	$u(\kappa_1) = 0.000$ $u(\kappa_2) = 0.005$	A
D	1	0.00 nA	I2: 0.5 nA P2: 1.0 nA	A
sample	parameter	experimental values	uncertainty	
	$u(\kappa)$	I_i	$u(I_i)$	
I2	0.000 0.005	503.1 nA	1.1 nA 2.8 nA	
P2	0.000 0.005	460.3 nA	1.7 nA 2.9 nA	

Table 4. Glucose activities a and uncertainty $u(a)$ of the test samples I9 and P9 for uncertainties in the dynamic term k of 0.000 and 0.005 are listed

		I9		P9	
		$u(\kappa_1) = 0.000$	$u(\kappa_2) = 0.005$	$u(\kappa_1) = 0.000$	$u(\kappa_2) = 0.005$
cycle 1	a (mmol·kg ⁻¹)	5.531	5.53	5.615	5.61
	$u(a)$ (mmol·kg ⁻¹)	0.027	0.07	0.043	0.07
	$u(a)$ (%)	0.5	1.3	0.8	1.2
cycle 2	a (mmol·kg ⁻¹)	5.534	5.53	5.692	5.69
	$u(a)$ (mmol·kg ⁻¹)	0.027	0.07	0.043	0.07
	$u(a)$ (%)	0.5	1.3	0.8	1.2

chronoamperometric measurement setup and from the used glucose purity. The expanded uncertainty is below 2 % rel. ($k = 2$, 95 % confidence interval) of the glucose content determined by bio-electrochemical measurements, thus competes well with today's considered most accurate reference method IDMS [8]. The advantage of the presented calibration method is its directness, the glucose content is determined without prior dilution of the sample. Furthermore, by restriction to the quantity molality no conversion factors for different specimens are needed as it is the case for concentration measurements.

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Dr. Samuel Wunderli
Dr. Hanspeter Andres
METAS, Switzerland

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A Convenient and Economic Approach to Achieve SI-traceable Reference Values to be Used in Interlaboratory Comparisons Concerned with Water Analysis



Olaf Rienitz



Detlef Schiel



Bernd Güttler



Michael Koch



Ulrich Borchers

Introduction

Within the scope of European harmonization, for example the Drinking Water Directive 98/83/EC [1] and the Water Framework Directive 2000/60/EC [2] are demanding the comparability of measurement results. Comparability can be achieved by implementing a traceability chain to the SI units via national standards. National standards are developed and provided by National Metrology Institutes (NMIs) [3].

In the field of analytical chemistry proficiency testing (PT) rounds play an important role in verifying measurement results even though reference materials (RMs) or even certified reference materials (CRMs) are used for calibration purposes. Therefore, in Germany, all laboratories responsible for drinking-water monitoring regularly have to participate in interlaboratory comparisons. The German drinking water directive [4] establishes the corresponding legal basis in conjunction with a recommendation issued by the German Federal Environment Agency (UBA) [5].

In the last consequence only interlaboratory comparisons providing metrologically traceable reference values instead of consensus values can help to establish national and international comparability. As an additional benefit such reference values enable the participating laboratories to check their results for accuracy on a reliable and unbiased basis.

To obtain metrologically traceable reference values the usual way using so-called primary methods is often demanding with respect to time and cost.

Two interlaboratory comparisons addressing heavy metals in drinking water are discussed in detail taking lead as an example to show a convenient way to generate metrologically traceable reference values solely based on data collected during these comparisons. PT providers usually prepare their samples by adding appropriate amounts of the analyte elements to natural drinking water (matrix). Doing this addition volumetrically or gravimetrically, the added amount is well-known, especially if certified reference materials are used. Even though the matrix content of the analyte elements is low, reference values have to include these matrix contents along with their measurement uncertainties to become traceable. Therefore, the matrix contents have to be measured and their uncertainties have to be estimated.

Measuring these matrix contents directly is often a challenging task and yields poor uncertainties. The experimental design of the discussed comparisons, however, offers a totally different way to obtain the matrix contents. To avoid cheating and to check their measurement capabilities over a certain range, the participants are provided with randomly selected samples out of a pool of up to 12 concentration levels covering nearly two orders of magnitude. This complex design can be

taken advantage of by regarding and evaluating the comparison as a standard addition. The gap between the measured values and the added concentration arises from the matrix content. Therefore the measurement results of the participants themselves provide the missing piece needed to complete the reference value without any additional measurements.

Refusing the straightforward use of consensus values as metrologically traceable ones, but applying them to introduce a minor correction to the beforehand incomplete reference values seems to be a contradiction. The special properties of standard addition are able to smooth this out. For example: the result of a standard addition experiment remains totally unaffected by recovery problems, because the slope and the y-intercept are changed by exactly the same factor. However there may exist any constant biases misleading the standard addition. To rule this out and to prove the whole approach experimentally, IDMS as a primary method was applied to determine reference values that are independent of the sample preparation.

Two different ways of sample preparation (volumetric and gravimetric) were scrutinized with respect to the metrological traceability of the added analyte contents. The crucial point was to set up an equation describing the particular sample preparation procedure. This so-called mathematical model of the measurement (model equation) was used to estimate the measurement uncertainty

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of the added amount of analyte according to the Guide to the Expression of Uncertainty in Measurement (GUM) [6].

Combining the added concentration of analyte β_{add} and the concentration β_0 arising from the matrix resulted in the desired reference value β (Fig. 1). The corresponding model equation needed in the uncertainty budget reflects also the traceability chain by linking the reference value directly to the purity of the applied certified reference material (standard).

the matrix content β_0 and the concentration arising from the added amount of the analyte (added mass concentration β_{add}). Eq. 1 shows the corresponding relation with lead as an example.

$$\beta_{\text{rcm}}(\text{Pb}) = \beta_{\text{add}}(\text{Pb}) + \beta_0(\text{Pb}) \quad (1)$$

This equation can be regarded as a linear equation with a slope of 1 and a y-intercept equal to $\beta_0(\text{Pb})$. Unfortunately, the slopes were not very close to one. For lead, for example, a slope of approximately 0.95 was found.

relative uncertainties of the means are more than 10 times larger than the uncertainties of the added mass concentration it is reasonable to determine the parameters a_0 and a_1 by an ordinary least square fitting (ODF). The uncertainty associated with the matrix content was calculated according to an equation published in [9].

Reference Values

The reference values were calculated as the sum of the added mass concentration and the matrix content obtained by the described standard addition-like method:

$$\beta(\text{Pb}) = \beta_{\text{add}}(\text{Pb}) + \beta_0(\text{Pb}) \quad (3)$$

Despite of the different relative expanded uncertainties associated with the gravimetric and volumetric sample preparation (< 0.4 % and < 1 %, resp.), eq. 3 as the mathematical model, yielded relative expanded uncertainties of 2 to 20 % associated with reference lead concentrations of 5 to 50 $\mu\text{g/L}$ regardless of the preparation procedure. These calculated reference values were compared to the reference values determined by the IDMS measurements (dashed red line, Fig. 2). Unlike the consensus values (dashed yellow line, Fig. 2), they were in very good agreement with the IDMS values. Their uncertainties and deviations from the IDMS values increased all the more, the closer the reference values got to the matrix concentration. This behavior is reasonable because larger concentrations should be measurable with smaller uncertainties. Therefore, the added mass concentrations completed by the matrix concentration derived from the consensus values were suited for use as the reference values. By contrast, the deviations and the uncertainties of the consensus values themselves showed no dependency on the concentration (Fig. 2). While the deviations were more than twice as large, the uncertainties were smaller. A few values were in good or acceptable agreement with the IDMS measurements, but they were scattered randomly over the concentration range. Therefore, the use of consensus values as the reference values seems questionable. Furthermore, there would be no proper

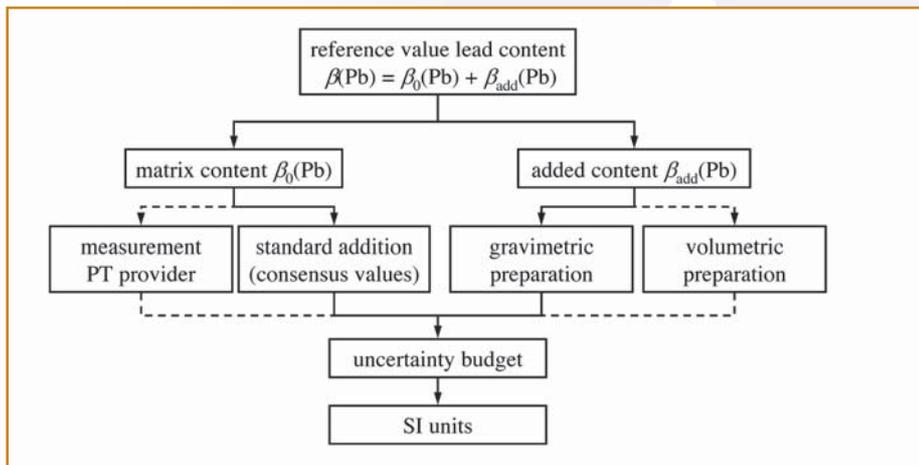


Fig. 1. The SI traceable reference value $\beta(\text{Pb})$ can be obtained on several different ways. Solid line: An example using the added content $\beta_{\text{add}}(\text{Pb})$ from a gravimetric preparation, combined with the content $\beta_0(\text{Pb})$ arising from the matrix calculated using the consensus values and the preparation.

Results and Discussion

Matrix Content

The content arising from the matrix β_0 (matrix content) was determined equally to a standard addition experiment. This requires the analyte already present in the matrix and the added analyte to behave in the same way, which is a reasonable assumption considering the analyte element(s) and the drinking water matrix. The results of the participants of the interlaboratory comparisons 3/2004 TW A2 and 1/2006 TW A2, respectively, were used to calculate the robust consensus means β_{rcm} of all samples [7, 8] along with their uncertainties. These means should represent the sum of

Therefore it was necessary to rearrange eq. 1 to yield a more general form (eq. 2). Providing that the means recover a certain fraction a_1 of the true lead concentration $\beta(\text{Pb})$ leads to:

$$\begin{aligned} \beta_{\text{rcm}}(\text{Pb}) &= a_1 \cdot \beta(\text{Pb}) = a_1 \cdot \beta_{\text{add}}(\text{Pb}) + a_1 \cdot \underbrace{\beta_0(\text{Pb})}_{=a_0} \\ \beta_{\text{rcm}}(\text{Pb}) &= a_1 \cdot \beta_{\text{add}}(\text{Pb}) + a_0 \\ \beta_0(\text{Pb}) &= \frac{a_0}{a_1} \end{aligned} \quad (2)$$

This linear function shows that the matrix content $\beta_0(\text{Pb})$ is equal to the x-intercept of the plot $\beta_{\text{rcm}}(\text{Pb})$ vs. $\beta_{\text{add}}(\text{Pb})$. Given that the

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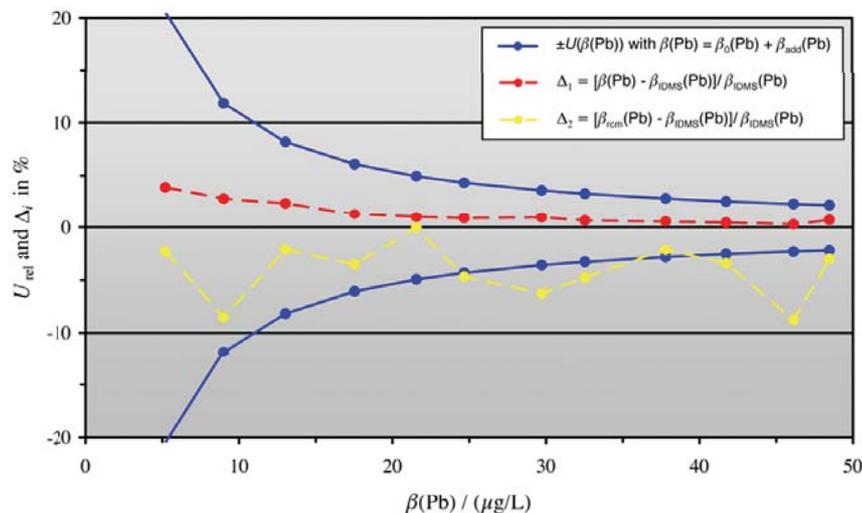


Fig. 2. Relative deviations of the reference values (Δ_1 , red) and relative deviations of the consensus values (Δ_2 , yellow) from the IDMS values $\beta_{\text{IDMS}}(\text{Pb})$. The deviations of the consensus values are larger ($< 10\%$) and in several cases not or in poor agreement with the values determined by the IDMS measurements, even considering their uncertainties (not shown in the figure for the sake of clarity)

way to show the traceability to the SI. Very similar results were also found for cadmium, chromium, copper and nickel, regardless of the preparation method (volumetric or gravimetric).

Traceability to the SI

The added mass concentration $\beta_{\text{add}}(\text{Pb})$ is traceable to the SI by linking it to the certified mass concentration of the reference solution β_{ref} and the purity of the certified reference material w_{pur} , respectively, provided that β_{ref} and w_{pur} , respectively, are traceable themselves. Unfortunately the matrix content derived from the consensus values at first does not seem to be traceable to the SI. But the uncertainty associated with the matrix content calculated using the consensus values is large enough to cover its true value. Furthermore, the matrix is just a small contribution to the reference value, except for those close to the matrix content. But even in these cases, the uncertainty associated with the reference value (now dominated by the contribution of the matrix content) increases sufficiently to include the true value (Fig. 2). This was demonstrated

experimentally by IDMS measurements for cadmium, chromium, copper, lead, and nickel in the concentration ranges usually found in German drinking-water interlaboratory comparisons. At least for these elements and concentration ranges, the reference values obtained by combining the volumetrically or gravimetrically added amount and the matrix content calculated with the described standard addition-like method using the consensus values have proven to be traceable to the SI. But there are limitations that need further research: elements or other analytes that are volatile or not stable and therefore tend to change their concentration in an unpredictable way after the preparation, which causes the value of the added mass concentration to be useless. Under these circumstances, contributions have to be added taking stability, adsorption and/or volatility problems into account. Another problem is caused by matrix concentrations in the order of the reference values themselves. The resulting large uncertainties associated with the reference values may limit their use as reference values.

Conclusions

It was demonstrated that SI-traceable reference values to be used in drinking-water interlaboratory comparisons can be achieved without any additional efforts. These reference values were obtained by combining already existing data coming from the sample preparation and the consensus values. As expected, the gravimetric sample preparation yielded a slightly smaller uncertainty than the volumetric, but the uncertainty associated with the reference value was virtually not affected by the contribution of the sample preparation procedure.

To improve the reliability of the calculation of the matrix content and its uncertainty in the meantime the ordinary least square fitting was replaced by a generalized least square fitting, taking into account the uncertainties of the added amount of analyte as well as of the robust consensus means [10].

There is only one additional effort the PT provider has to face: Setting up a proper uncertainty budget describing the sample preparation. This concept shows a way to provide SI-traceable reference values without the regular participation of an NMI applying primary methods being necessary. The role of the NMI is focused on the validation of the application of the described concept. This task may include guidance and support in the uncertainty calculation, but may include just as well the participation in interlaboratory comparisons, i.e. from time to time or if the concept shall be applied to problematic analytes or analytes other than the discussed ones, and to changed matrices.

Outlook

At the moment a European interlaboratory comparison (Euromet 924 part 3) and a closely connected international comparison (CCQM-P100.3) organized by PTB, BAM, LNE, IWW and EC-JRC-IRMM are in progress. These comparisons are aiming at the determination of the mercury mass concentration in a natural water matrix on a concentration level below 100 ng/L (as required by the WFD, [2] and [11]). All participants received not only the

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sample itself but also one or more additional sample(s) adjusted at well-known higher concentrations. This is going to be the first time that in the case of very low concentrations (only 10-20 times above the matrix content) and with a volatile analyte (Hg) the approach described has to prove its applicability even under these demanding circumstances.

The method described is routinely applied in one of the German water PT schemes (AQS Baden-Württemberg) since 2007. To proof the absence of unrecognized uncertainty sources and to fill the lack of traceability of the reference materials used for the fortification of the samples more reference measurements will be needed.

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Dr. Olaf Rienitz, PTB
Dr. Detlef Schiel, PTB
Dr. Bernd Güttler, PTB
Dr. Michael Koch, ISWA, University of Stuttgart
Dr. Ulrich Borchers, IWW
Germany

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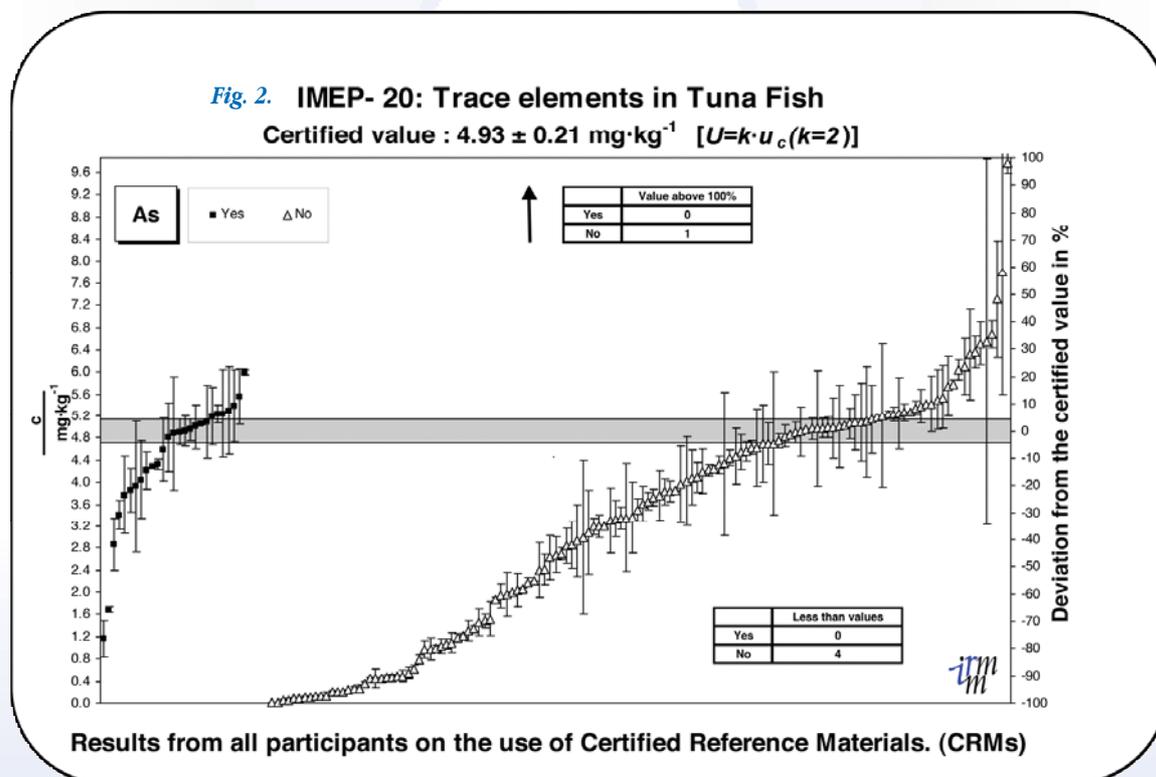
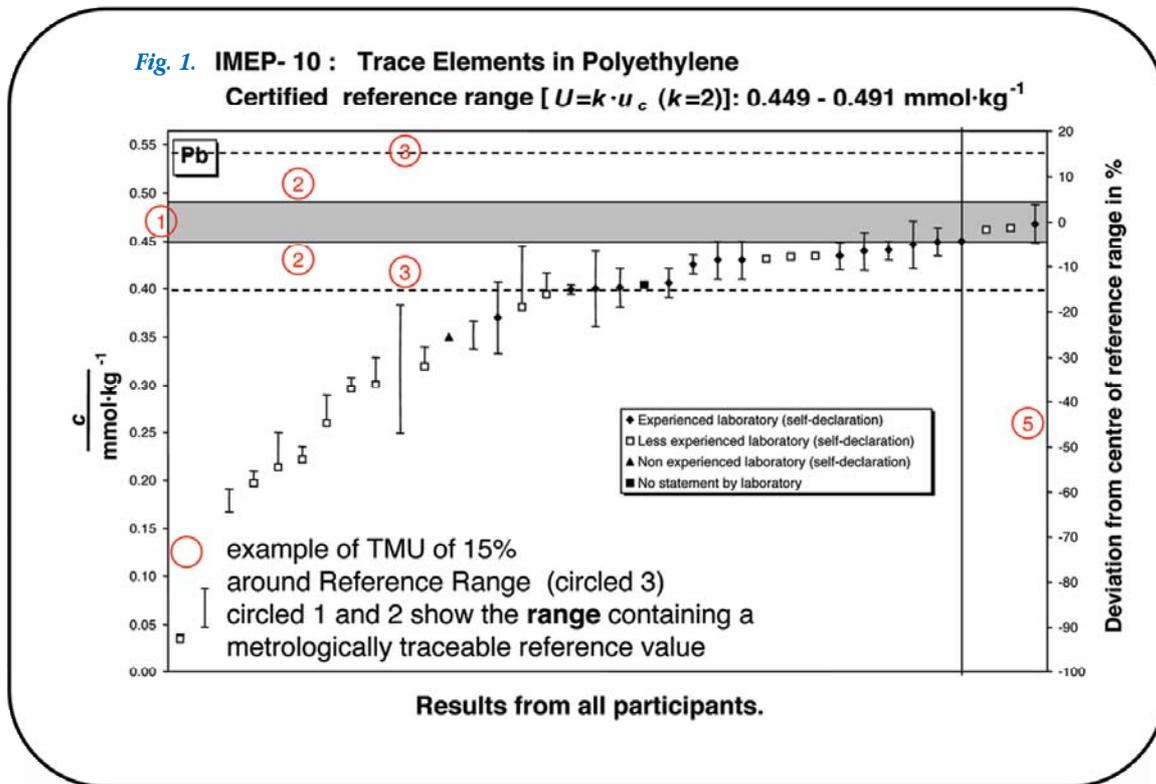
Should We Look More Closely at the Measurement Results We Compare?

In a chapter contributed to a recent book [1], we took a closer look at a number of graphs in which measurement results are compared. The chapter was written at the insistence of the book's editors, who indeed wanted a closer look at comparisons of measurement results sampled from practice, regardless of how they would look. In retrospect, the contrast with the other chapters of the book provides a rare comparison of theory and practice in matters of comparing measurement results. Thus an

attempt was made to give the readers of the book some "food for thought". A mere look at the examples given in the graphs in Fig. 1 and 2 presenting comparisons of measurement results, raises a number of questions, to which answers have either not (yet) been given, or not made properly known to the users concerned, or the answers are unsatisfactory, or they unveil a variety of opinions, if not outright controversy. Thus the very reason for this chapter in this book simply is that

a scientific debate is needed, followed by a suitable dissemination of its conclusions.

When looking at these graphs, one of the most striking observations is the absence of "normal distribution" of the results. Yet, the assumption of "normal distribution" around a most likely 'reference quantity value' is encountered many times in the chemical literature on comparisons and used as basis for conclusions: "On the assumption that the data are normally



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distributed, ..." or "Assuming a normal distribution of the data ,...". Thus the question is discussed whether "normal distribution" of measurement results can be "assumed" if clear proof is presented of absence of it. Wouldn't it be indicated to only build any conclusion on normal distribution after such a distribution has been shown in an unambiguous picture to apply in the particular case of measurement concerned?

A large majority of relevant literature assumes normal distribution as a matter of course, sometimes even in the case of absence of it. Isn't this matter for debate? Thus the chapter of the book asks some 18 questions to generate that debate.

Another feature of the chapter is that a distinction is made between comparison of two measurement results and comparison of many measurement results. In practice, many cases of comparing measurement results are involving only two parties: buyer-seller, inspector-inspected, regulating-regulated. Yet, an enormous interest goes to the comparison of many measurement results, thus establishing a multi-party context with the ensuing complex statistical treatments of these results.

Why?

To have a general background view of the state of the practice in which the two parties are active? Maybe.

Do they need this general overview in case they might need an umpire to settle a dispute?

Maybe.

Do they need that to settle a dispute over a difference – or reach agreement – about their measurement results?

Not really.

Mostly, their problem is whether their measurement results are compatible [2] or equivalent, regardless of what other laboratories achieve.

End of story.

So, why requiring a multi-inter laboratory comparison (ILC) to enable two parties to settle their compatibility problem?

Thus a number of questions are formulated in the chapter and attempts are made to give them a preliminary answer:

1. Must an organizer of an ILC, who is going to compare the participants' measurement results, not verify whether they are "comparable" in principle?
2. Must the organizer of an ILC not request metrological traceability from each participant for his measurement result?
3. Doesn't an ILC organizer need to require a statement of measurement uncertainty from any participant?
4. Is the establishment of metrological traceability not a prerequisite for the evaluation of measurement uncertainty?
5. Can we combine two measurement results which are not metrologically comparable?
6. Must each participant of an ILC establish himself the metrological traceability of his own measurement result before measuring the measurand concerned in an ILC?
7. Can measurement results in chemistry in an ILC automatically be expected to be normally distributed around a reference quantity value?
8. Can general conclusions which are based on the assumption of a normal distribution around a reference quantity value be drawn when experimental evidence does not substantiate such a distribution?
9. Do the different measurement results of an ILC belong to a same population?
10. If the average, or mean, or "cleaned" mean of the participants' results derived from an ILC, where metrologically traceable and non-metrologically traceable measurement results are sometimes mixed ("combined"), can that lead to a "reference quantity value"?
11. To which extent is a subsequent calibration of measurement procedures, using such a reference quantity value, a circular process if the same measurement procedures were used in the establishment of the reference quantity value?
12. Who is then guarding orthodoxy in this process?
13. Does the very fact of participating in an ILC not have as logical consequence that the participant who offered a non-metrologically traceable measurement

result a priori, cannot use the calculated average (or median or mean) as a(n other) metrological reference *a posteriori*?

14. Isn't the basic product of an ILC to establish some sort of "degree-of-equivalence" of any specified pair of results of participants, rather than comparing and possibly combining measurement results?
15. Is a "consensus value" for the certified quantity value(s) carried by a calibrator (CRM) (average, median, mean of selected values) metrologically traceable?
16. Isn't measurement uncertainty sometimes (instinctively) made larger to encompass a perceived uncertainty in the statement of a metrological traceability?
17. Is it possible to state or establish a (non-metrological) form of traceability for a "consensus value" of an ILC?
18. Is the measurement uncertainty claimed for a "certified measurement result" which is generated from the combination of several measurement results with full (GUM) measurement uncertainties, obtained by, for example, a few National Metrology Institutes, smaller or larger than the individual measurement uncertainty of either of the two individual measurement results?

A good debate is suggested.

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Prof. Paul De Bièvre
Independent Consultant on
Metrology in Chemistry, Belgium

NMIs: Extended Outreach is Critical for Enhancing Confidence in Food and Nutritional Measurements

1. National Metrology Institutes

National Metrology Institutes (NMIs) are the guardians of quality measurements for a given country. They are the primary interface between internationally recognized measurement systems and national measurement requirements, related to traceability and accuracy, among others. As custodians for national metrology systems, the NMIs facilitate international comparability for quality systems, and permit calibration of standards at the national level, that extends through various measurement linkages down to the measuring equipment at the user level. Simultaneously, they establish traceability for the field equipment through → working standard → secondary standard → and thus providing a link to the national standard, as a part of the required validation process. The NMI infrastructure generally includes extensive laboratory set-up, scientific and technical capabilities and expertise, and is generally well equipped to meet the needs of various statutory responsibilities; e.g. meeting regulatory requirements in chemical and physical measurements, ensuring harmonization for units and setting standards for measurements, among others.

1.1 Uncertainty, Traceability and Comparability

The quality assurance (QA) path outlined above, percolates through a hierarchical pattern: NMIs → to networks of national (or regional where applicable) reference laboratories → accredited reference laboratories → manufacturer's standardization laboratories → hospital, pharmaceutical and other user-type state and privately run (e.g. food composition and food safety) laboratories → routine service laboratories (officially recognized facilities analyzing scores of samples on a daily basis).

The processes that take place at different parts of this hierarchical system are governed by two components: (i) taking the example of chemical metrology, one that reflects an increasing uncertainty level from step to step, i.e. → a well defined reference measurement system traceable to SI Units (e.g. definitive measurements), → reference measurement

procedures (e.g. traditional AOAC approach) → measurement procedures for a specific measurand → to routine bulk assay procedures; and (ii) the other, that reflects a decreasing traceability link by using primary standards → primary (natural matrix) reference materials → secondary reference materials → tertiary reference materials → to in-house reference materials or control materials. Importantly, a clear picture of uncertainty and traceability is critical to establish a basis for credible comparability, when results are generated in different geographical locations under differing laboratory conditions [1].

1.2. Extending out-reach to every-day Measurement Communities

As the **torch-bearers** for strengthening the usefulness of measurements, the role played by the NMIs is rather unique. In addition to the pivotal functions outlined above, they have other responsibilities: e.g. (i) strengthening the metrological basis of various measurements, and (ii) imparting these metrological concepts for strengthening awareness in the minds of the analytical professionals. It is recognized that experts from the NMIs interact with QA professionals from the private and other sectors. These professionals in turn extend their services as requested by different sectors in the user community. Further, NMIs interact with groups from industries who request for specific type of QA training (e.g. metrology related to manufacturing processes, sometimes referred to as productive-metrology). However, it would be timely for the NMIs to support other disciplines that have limited opportunity to communicate with such national laboratories.

A **daunting task** for the NMIs is to put in place functional metrological traceability systems that are suitable for numerous user groups. Taking the example of the food and nutrition (F&N) community, the implication of this effort is as significant as a movement taking place at the population level. If successful, it precipitates substantial changes at the grass root level, as a result of improved awareness in this segment of the analytical community. Infusing such a new culture into the overall thinking process of the F&N analytical community is a

basic requirement for establishing sustainable approaches for ensuring the integrity of analytical results in several F&N areas.

Importantly, a change of this magnitude calls for bold, swift and decisive efforts by the NMIs and the policy makers in the food, nutrition and health sectors.

2. Food and Nutritional Metrology as a Part of Biological Metrology

Dealing with the F&N measurements involves handling of complex chemical and biological matrices for numerous assays. The food as a natural matrix in its many forms, presents a variety of problems in achieving a meaningful analytical result. e.g. food composition and food safety areas. Further, dealing with nutritional investigations extends the measurement process into the physiological and metabolic domains, leading us to the complexity of metrology in biology [2]. There are several issues surrounding these concepts that need to be carefully evaluated to enhance the reliability of F&N analytical results and to ensure sustainability to the QA process [3,4]. These steps are crucial for infusing authority to analytical results in the F&N areas and for making sound public health decisions.

3. Interfacing F&N Investigators with Metrological Concepts

There is a crying need for an **extended outreach** by NMIs for strengthening the metrological awareness and capacity developmental needs, in several analytical communities. I wish to single out the F&N investigators who are not sufficiently exposed to metrological concepts in their day to day work. In fact, the word metrology is practically unknown to traditional F&N professionals. Therefore, getting to know metrology for daily applications assumes a life-changing situation for them, with all the apprehensions that go with such thinking.

For a long time, the measurement philosophy of a typical F&N investigator has been to apply a single chosen method for analyzing two or more sets of different samples and evaluate

the differences (between them), if any, based on the measurement results. This line of thinking is open for the following deficiencies: (i) suitability of the chosen technique; (ii) sensitivity of the chosen method (which can greatly influence the cost and even the planning of an investigation); (iii) failure to validate the method against a certified reference material or comparing the results obtained by another independent method to identify the sources of errors; (iv) impact of not using direct (definitive methods) vs indirect methods (e.g. skin-fold thickness to assay body fat) that lack traceability aspects; and, (v) doubtful qualification of studies pooled for meta-analysis (meta-analysis assumes that the studies chosen are compatible in all respects including analytical accuracy and precision, and method validation). Also, it is important to note that outcomes of prescriptive type of methods (e.g. based on the AOAC philosophy) may be dependent on the analyst, and therefore, independently validated methods should be used [5]. The latter practice would enable a realistic assessment of uncertainties associated with a given measurement process and make the results more meaningful and therefore, reliable.

4. Status of F&N Measurements

The food and nutritional (F&N) measurements represent a combination of **physical, chemical and biological metrology**: quantity, density and size (physical metrology), composition and speciation (chemical metrology), and energy metabolism and bioavailability (biological metrology). Certain clinical (e.g. cholesterol and glucose in blood) and a few others (e.g. enzymes, hair and urinalysis) overlap with chemical metrology. Similarly, in-vitro assays for body composition, bone density and nutrient intake and excretion closely resemble chemical measurements (e.g. traditional methods), while in-vivo approaches (e.g. isotopic techniques) fall under physiological or metabolic measurements.

Further, measurements carried out to assess the impact of foods (e.g. cholesterol and glucose levels in blood) and certain types of bio-analytical measurements (e.g. enzymes, hair and urinalysis) overlap closely with

chemical metrology. Several other physiological measurements are carried out under complex conditions and are frequently assessed by indirect means. These include, among others, measurements for body composition and dietary energy intake and energy expenditure by traditional methods (e.g. balance technique for intake and out-flow) that lack evidence of a proven traceability link. These deficiencies are amenable for metrological improvements using stable isotope techniques based on single or double-labeled water and utilizing mass spectrometry for ratio measurements. However, it should be noted that even with stable isotopic investigations, systematic studies designed to examine the metrological aspects to estimate uncertainties are lacking. With increased attention being focused on obesity and associated chronic disorders, there is a need to strengthen the validity of these types of measurements.

5. Economic Impact of Quality Assurance

The statement by the Commission of the European Union in 2006 *"There is no science without measurements, no quality without testing, and no global markets without standards"* aptly summarizes the impact of QA on measurements and economy. This sentiment is further accentuated in the trade circles as reflected by the notion that analytical chemistry is the language of commerce. The Australian NMI website (www.measurement.gov.au) states that the NMI adds value to that country's economy by providing a reliable foundation for measurements, thus providing certainty to measurements used in legal transactions and in trade. The added value of measurement in a developed economy has been estimated to account for between 3% and 6% of GDP. Even the lower figure of 3% would translate into about \$15 billion in the Australian economy. This added value is underpinned by the NMI activities in the form of advice, assistance, training and transfer of technology to industry and the science community.

The NIST (USA) website documents that measurements are responsible for 10% - 15% of the \$1.7 trillion annual costs of healthcare in the United States. A significant portion

(25% - 30%) of health-related measurements is performed for non-diagnostic reasons (re-tests, error prevention and detection). Even modest improvements in measurement accuracy and QA will result in multi-billion dollar savings in healthcare costs. Project drivers are, therefore, measurement reliability (as it impacts healthcare costs and medical decision-making), regulatory requirements, and international trade and competitiveness-related issues [6].

Economic impact of food safety is two fold: (i) intrinsic confidence in the safety of the food as a commodity and the resulting trade benefits in the international market; and (ii) the *health safety* related to consumption of such foods. The food safety issue has become a global concern and demonstrates the need for concerted efforts to integrate the metrological concepts into the measurement processes. It should be noted that in some parts of the world, movement of foods has assumed the status of *borderless-trade*; foods in many forms as commodities cross national borders on a daily basis. Reliable F&N measurement is indispensable here.

6. Capacity Developmental Issues

Integration of metrology into F&N measurements strengthens the very base of nutrition education, a nearly forgotten agenda in our current academic practices. An observation by de Bievre [7] is appropriate in this context: *"Unfortunately, in many parts of the world, teaching such basics of the process called measurement is almost absent in the educational system"*. The International Food Policy Research Institute [8], World Bank [9,10] and a few other United Nations Agencies are now embarking on human capacity building as a critical focal point in the context of national development. The United Nations Standing Committee on Nutrition, the International Union of Nutritional Sciences, the International Atomic Energy Agency, the World Health Organization, and the Food and Agricultural Organization among several other organizations, have supported capacity development efforts through task force deliberations, co-ordinated research projects [11], development of internationally

applicable quality systems and training courses [12-15]. However, these efforts are just drops in a bucket since the need for revamping the capacity developmental issue is global, and only national initiatives can bring sustainable progress in QA and developments at the level it is needed.

7. Resources for Enhancing Metrological Awareness Programs

Generally, the **NMIs** are adequately funded because of their exceptional importance to the national development programs. In contrast, the F&N community that stems from the academic circles, is often dependent on project funding for any additional activities. It is here, that the **extended outreach** by the NMIs would be most useful since they have the institutional capacity to design and teach basic metrological concepts to young investigators from universities and F&N institutions. This would be a long-term investment in the country's quest for excellence in QA. The NMIs should note that this *process* is not merely a small change in the currently existing perceptions (or practices) in the F&N community. It is essential for introducing sustainable approaches for preserving quality systems (16).

Among the food industry professionals, mobilizing funding may not be critical but the metrological needs are numerous and rather specific, since each product has its own profile. Hence, there may be understandable limitations as to what extent the NMIs can help in such situations.

8. Conclusions

F&N metrology is an **emerging discipline** and it should be embedded as a **culture** in the minds of our young analytical chemists aspiring to become F&N investigators.

Integration of metrology into F&N measurements is helpful also for improving the measurement infrastructure of an institution. Educating the future F&N investigators with knowledge of metrology is pivotal for strengthening the very base of **nutrition**

education. However, the leadership here rests on the dynamism of the academic community.

The purpose is to disseminate the benefits of integrating metrological concepts into the daily laboratory work carried out by the F&N professionals. Universities on their part should strive to teach metrology to analytical chemistry students, at least at the graduate level. This is part of the **capacity development** drive that carries long-term benefits.

The **NMIs** have an indispensable role in disseminating the QA message. In the national context, the NMIs are the torch-bearing leaders expected to **proactively** interact with various groups such as the F&N community, for preserving the integrity of the quality of measurements.

Building a sound metrological base for F&N and health-care measurements is a **movement** by itself, and requires a sound policy (facilitating capacity development) to attain sustainable QA at the national level. This is a shared responsibility between the academic community, policy makers and leaders of NMIs.

The **hardball question** for the academic community, NMI leaders and policy makers is, – are they ready to take up this timely challenge?

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Prof. Venkatesh Iyengar
Tufts University
Biominerals International
USA

Simple Basic Principles for Chemical Measurement: Identifying and Counting

There should be renewed attention for counting as form of measurement such as in microbiology or when counting “active sites” on protein molecules where measurements and their results should not be described in terms of the quantity ‘mass’ and its units – be it kg or g or mg – but in ‘number of things’ with ‘one thing’ as a unit. Thus it is a good time to remind ourselves of the “particulate” or “granular” structure of matter with the ensuing usefulness and ensuing desirability of – indeed – counting of particles: atoms, molecules, ions, microbes, active sites i.e. “entities”.

On the macroscopic scale that must surely have been the earliest – and easiest – form of measurement: counting cattle, soldiers, horses, apples, or coconuts, which determined the degree of richness or power. A more modern application of counting occurs in banks daily and on a massive scale: counting currency units. Also, in a set of persons, it makes more sense to think of an average height per person (unit for measuring a number of persons: one person) rather than of an average height per mass of person (unit for measuring mass of persons: one kg of person). Similarly, it is more useful to talk about an average salary per person (unit for measuring a number of persons: one person) rather than of an average salary per mass of person (unit for measuring mass of persons: one kg of person).

Thus, ‘numerosity’ or ‘numerousness’ is a very basic property of matter and an extremely useful tool to measure a “number of things”. It took a long time before that was recognized. For centuries, matter was looked upon as something being “continuous”. Although Demokritos in ancient Hellas implied already the “particulate” nature of matter, it took Dalton, Berzelius, Lavoisier and others, almost two thousand years later, to definitively

establish that matter had a “particulate” structure. Hence it was exceedingly logical – and exceedingly simple – to arrive at an appropriate unit for counting a number of particles (generic name: entities): one entity. A fixed (because defined) multiple of the natural unit “one entity” can then be established and given a name: one mole (symbol mol). The fact that a mole of entities is a very large number of entities, does not constitute a problem since we can replace counting of very large number by measuring ratios of very large numbers. It reduces the problem of measuring very large ratios back to simple ratio numbers. That is what we do in a titration. The zero point tells us when a large but unknown number of ions is equal to a large but known number of other ions, yielding the simplest ratio number of all: 1 (1:1). In isotope dilution mass spectrometry IDMS, we do something similar: we compare a large but unknown number of atoms of one isotope of an element to a known number of atoms of another isotope of the same element (called a “spike”). In this case we also have an “equivalence point” indicator of equality of large numbers: we have even measurement instrumentation which is reasonably linear enabling us to measure ratios of large numbers (of isotopic atoms) when the “equivalence point” is different from a number ratio 1. It can even differ from that number ratio 1 by several orders of magnitude e.g. 10^{-2} (1/99) and still be measured easily.

The fundamental thinking touched above is underpinned in [1], the abstract of which follows hereafter:

“We examine the problem of quantitative chemical measurement for well-identified substances, discuss the quantity called ‘amount of substance’, the means of expressing it, and its physical SI unit the mole. The everyday quantity which is a number of entities may be

measured by the performance of two operations (identification and counting), the results of which may be communicated with two items of information (their name and the number of entities). We distinguish nominal, ordinal, interval and ratio scales of measurement and apply these to counting, referring to ordinal and cardinal numbers and Helmholtz’ analysis of measurement. Counting may be by direct serial numeration, direct parallel numeration, or comparative numeration. We discuss the limitations of serial numeration, the possibilities of parallel numeration, and the advantages of comparative numeration where a unit for counting in multiples (such as the analyst’s mole) may be used to define a scale on which equal numbers of objects correspond to equal values of some other physical quantity. We conclude that the numeration of very large numbers of objects is readily achieved but with unavoidable uncertainty, using operations which compare numbers of entities either to numbers of other entities or to some other quantity which accurately models numbers of entities.”

Comments can be sent to the author, to the editor of the CITAC Newsletter, or to the Discussion Forum of ACQUAL.

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Prof. Paul De Bièvre
Independent Consultant on
Metrology in Chemistry
Belgium

V METROCHEM - Brazil

The Brazilian analytical community witnessed the successful realization of the “V METROCHEM - International Congress on Traceability in Laboratory Measurements and Productive Chains”, 11 to 13 November 2009 in São Paulo, Brazil. São Paulo, South America’s largest city, with a population of 20 million people and a GNP of about 500 billion U.S. dollars, is also Brazil’s most industrialized center. The realization of the Congress in this city benefited a wide range of industrial sectors and their customers, regulators and suppliers, as well as academic institutions and government.

The primary focus areas of the Congress were: (i) metrological traceability for laboratory measurements with emphasis on reference methods, certified reference materials, and methods and protocols for providing reliable laboratory results that can be compared over both time and space, and (ii) traceability of the production chain, focusing on the food, beverage, fishing and chemical industries.

The Congress was co-sponsored by the Metrology Network of São Paulo State (REMESP), the Instituto de Pesquisas Energéticas e Nucleares (IPEN-SP) and the Cooperation on International Traceability in Analytical Chemistry (CITAC). The event was attended by more than one hundred and fifty professionals eager to update their technical knowledge, including fifty metrology experts from around the world.

The interaction between invited experts and congress participants provided a positive contribution to higher learning institutions, industry, service organizations and exporters. The target audience was composed of participants from the technical and managerial staff of testing and calibration laboratories, clinical laboratories, regulatory agencies, certification and quality



Fig. 1. Dr. Willie May delivers to Dr. Robert Kaarls a Certificate of Recognition of Professional Excellence: "Man's mind, once stretched by a new idea, never regains its original dimensions"

organizations, exporters, research and development institutions and universities.

The main themes of the forty two oral presentations were: metrological traceability, product technical specifications and regulations, food and safety chain, wood chain of custody, hazard analysis and critical control points, quality in laboratory measurements, calibration hierarchy, VIM, technical translation, traceability related to analytical equipment, traceability in industrial gases, meat and fishery chain, certified reference materials production and consumer protection, among others.



Fig. 2. Prof. Paul De Bièvre, Dr. Ilya Kuselman and Prof. Maria Filomena Camoes during a coffee break

Fifty posters presented were also focused on topics such as method and procedure validation, uncertainty in measurements, calibration, accreditation and formal recognition, reference materials – production and use, and interlaboratory comparison programs/proficiency testing, laboratory techniques and analytical methods.

The Conference opened with several musical selections from the University of São Paulo Choral Group. This was followed by an Awards Session where Drs Robert Kaarls, José Carlos de Castro Waeny and Vera Maria Lopes Ponçano received Certificates of Recognitions of Professional Excellence in their respective fields.

Focusing on the participation of CITAC members and associated international organizations, the Congress started with the presentation of Dr. Robert Kaarls (CCQM President), who highlighted the impact of metrology in the global economy and the demands of metrological traceability in brand new scientific areas. Then Dr. Willie May (NIST-USA) called the audience’s attention on the impact of chemical metrology on the industrial competitiveness and quality of life. Finally, Dr. Ilya Kuselman (INPL-Israel) described the role of CITAC in developing metrological traceability worldwide.

In a very conceptual and therefore dense session, Prof. Paul De Bièvre explained how the concept of metrological traceability of measurement results must be implemented in chemistry. This talk allowed several Brazilian experts to describe their experiences in establishing metrological traceability in fields like agriculture, biological materials, clinical analysis, environmental monitoring, minerals and metals, pharmaceuticals, gas and oil.

The metrological system management session was a very important one. Dr. Maria Fernandes Whaley (NMISA-South Africa) presented the example of AFRIMET in the African context which was connected with some Brazilian presentations on how traceability was implemented in practice for food and feed products in Brazil. The highlight was the lecture of Dr. Nineta Majcen (NMI-Slovenia), who described the strategy for building a distributed metrology infrastructure to face her country's most pressing demands.

Another interesting topic was the new VIM. Although it is now largely accepted as the official technical language in metrology, problems of translation to Portuguese were raised by Prof. Maria Filomena Camões, Universidade de Lisboa. After a cheerful discussion it was concluded that metrological concepts must be thoroughly understood before the translation work actually start.

Education and training was deemed essential for disseminating knowledge in metrology in a thorough and consistent way. Following this route, Dr. Philip Taylor shared his experience with the very successful European platforms TraiNmiC and MSC Euromaster. Then, Brazilian professors from public and private universities reported their experiences in the implementation of metrology in chemistry into the university's curriculum.

The presentations related to productivity chain traceability were the occasion to verify how the Brazilian meat, fish, poultry and wood industries are organizing their quality control systems to demonstrate to the consumer the traceability from farm to fork. This topic is highly important for Brazil because the country is of the world's largest commodities producer.

The presence and active participation of CITAC members and colleagues added real value to

this event, where traceability under different perspectives were the key words.

The final activity was an award session recognizing the five best papers presented according to the rules established by the scientific committee. The event finished with an open evaluation session that led to several interesting suggestions promising to continue the friendly scientific atmosphere we all experienced in São Paulo.

Dr. Marina Beatriz Vasconcelos Agostini
Dr. Olívio Pereira de Oliveira Júnior
Scientific Committee Presidents, IPEN

Dr. Vera Maria Lopes Ponçano
Congress President, REMESP
Brazil

ISRANALYTICA 2010 and the CITAC Workshop in Israel

The Conference

ISRANALYTICA 2010, the 13rd Annual Meeting of the Israel Analytical Chemistry Society and the accompanying Exhibition took place according to tradition, January 19-20, 2010, at the David Intercontinental Hotel, Tel-Aviv, Israel.

The annual meetings of the Israel Analytical Chemical Society have become one of the largest annual conferences in analytical chemistry in the World. About 3000 participants from academia, the pharmaceutical and chemical industries, petrochemistry and food industry, biochemistry and biology, water experts, engineers, quality managers and metrologists from major laboratories and companies from Israel and about 20 other countries attended. As well, about 60 leading analytical equipment companies successfully

participated in the conference and exhibition, bridging between technology developments and end users. In fact, ISRANALYTICA combines the largest chemistry convention in Israel with the largest analytical chemistry exhibition in one location.

ISRANALYTICA has been recognized by the Analytical Chemistry Division of the European Association for Chemical and Molecular Sciences (DAC EuCheMS), the International Society of Electrochemistry (ISE), and the Cooperation for International Traceability in Analytical Chemistry (CITAC).

The scientific program consisted of 4 plenary lectures and 14 keynote lectures delivered by international top scientists, oral presentations (in 14 parallel sessions) of featuring speakers from Israel and abroad, and a poster session. On the first day there were sessions on

mass spectrometry, pharmaceutical analysis, proteomics and bioanalysis, water and environmental analysis, separation methods, nano-analytics, analytical spectroscopy. On the second day metabolomics and bioanalysis, forensic and legal analysis, electrochemistry and spectroscopy applications, food analysis, metrology and quality, education and intellectual properties were the session topics. Distributions of the lecturers and poster presenters by economies are shown in Fig.1 and Fig. 2, respectively. However, the problem was that only about 25% of the 3000 participants were attracted by the lectures and posters, while the others were busy at the exhibition. The Organizing Committee will study this phenomenon to decide what can be improved.

Three prestigious poster awards were granted by the Conference Organizing Committee

to the outstanding posters during the general assembly of the Israel Analytical Chemistry Society.

In Fig. 3 and Fig. 4 one can see two pictures from the Isranalytica 2010. Hundreds of other pictures are available in <http://www.isranalytica.org.il/Isranalytica2010/Gallery>. It was an exciting, very fruitful meeting.

The CITAC Workshop

A CITAC one-day workshop was presented on 21 January 2010 as a post conference event titled "What should you know about metrology & quality requirements to analytical results?" The workshop program included the following lectures:

- "Introduction to metrology and quality in chemistry" by Dr. Ilya Kuselman, INPL, Israel;
- "Different approaches to estimation of measurement uncertainty in analytical chemistry" by Prof. Ivo Leito, University of Tartu, Estonia;
- "Exploiting the potential of reference materials and accompanying information by different laboratories" by Prof. Hendrik Emons, IRMM, European Union;
- "Metrological traceability of measurement results in chemistry: concepts and

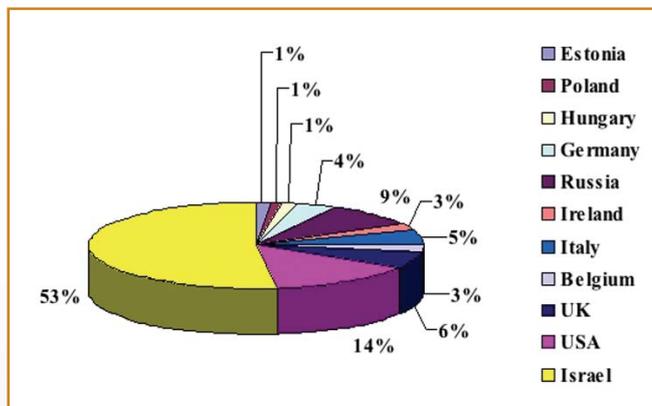


Fig. 1. Lecturer distribution by economies

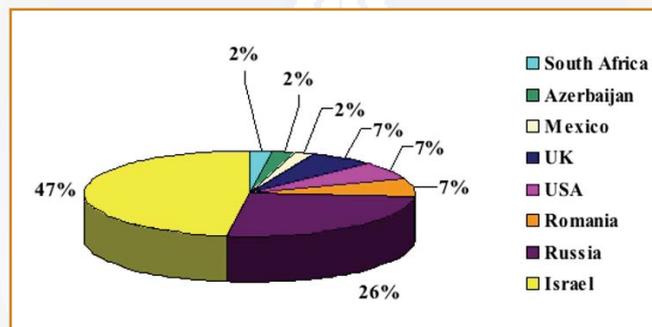


Fig. 2. Distribution of poster presenters by economies

implementation" by Prof. Paul De Bievre, Consultant, Belgium;

- "Metrological traceability in analytical spectrophotometric instrument qualification" by Dr. Jerry D. Messman, Stranaska Scientific LLC, USA; and

- "A practical guide to chemical metrology and pharmaceutical quality" by Dr. William Koch, USP, USA.

Every lecture was planned for 45 min, while the next 15 minutes were used for questions and discussions.

This workshop was intended for:

- Laboratory personnel in analytical (quality control) and R&D laboratories;
- Quality assurance personnel involved in reviewing analytical data and validation studies;
- Projects coordinators and analytical laboratory managers;
- Metrologists specialized in chemistry.

Since the lecturers were already familiar to the audience from the conference, the interactions between them were simple, helpful and creative. The workshop participants were very satisfied by the possibility to learn, ask and

receive answers directly from the leading specialists in metrology in chemistry – the CITAC members.

Dr. Ilya Kuselman
INPL
Israel



Fig. 3. Prof. Hendrik Emons delivers his plenary lecture "Towards the "chemical kilogram": development and use of references for chemical and bioanalysis"



Fig. 4. Dr. William Koch (from the left) and Dr. Raphy Bar in the special session on US Pharmacopeia and USP reference standards



14th International Metrology Congress

22-25 June 2009 - Paris:

A Glimmer of Hope Before Summer!



The color of the fitted carpet in stands was orange, which was a good sign! The general impression and the first reactions expressed by participants were also very positive. As one of them put it: "It was a glimmer of hope in the current gloomy outlook!"

750 participants registered for full access to the event, a 15% increase over previous congress' attendance! 200 additional non-registered participants visited the exhibition. 30% of the participants were foreigners from 48 different countries, mainly European, but also from North and South America, Africa, the Middle East and Asia (Fig. 1).

The Congress proved to be of great interest to those participating:

- 59% among them were industrial: measurement tools users of all sectors, analysis laboratories, metrology laboratories or materials manufacturers ...
- 26% were from big national and international institutions: national laboratories of European countries, Government Officials, accreditation institutions, international organizations...
- 10% were from the university or research fields,
- 5% from other sectors (hospitals, training organization, free lance, press..)

This year, the Congress offered many new features, including:

- 6 round tables running over the full course of the congress with current hot topics discussed in the field of business management, environmental and health issues, and future prospects for wireless equipment,
- **Best Conference Prize** awarded to Mr. Lübbehüsen of GE Sensing and Best

Conference Poster Prize awarded to Mrs. Goyon of BIPM,

- Business sessions providing live discussions in the conference room between service providers, manufacturers and participants,
- Lunches in the form of a standing buffet allowed easy interaction and discussion among participants, although some were a bit nostalgic for the classic sitting format!
- Thursday's event was very much appreciated, and exhibitors stayed until the end and attended the 4.00 pm closing party.

For the first time, we may also have found the proper balance in contents. Indeed, at closing time, "scientists" said they had found this event too "industrial"; and on the other hand, "manufacturers" said that the event was too "science" oriented! In regard to the selection of topics discussed, generally participants found that the Program was quite exhaustive, varied and "very attractive", although some topics were missing – as is inevitably the case!



Fig. 1. Bag "Métrologie" of a participant

The organization committee also had some tough challenges to face this year, including keeping registration fees unchanged (at the 2007 price level), which involved making some difficult decisions in organizational matters. But it showed real maturity and management skill, building on experience with previous events. In fact, its goal was to make the event available to the widest audience, in spite of the current budget restrictions prevailing in most companies.

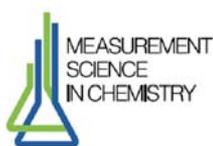
The International Metrology Congress is now held in high esteem by our European partners. Many prospects and opportunities for new developments are now open, both in France and abroad.

The Collège Français de Métrologie, as the main operator, is aware of the high expectations such an event has raised, and will spend the needed time and effort to study such new opportunities. A warm thank you to all the organizations and partners for supporting the Congress:

- The Organization Committee members: Acac, BEA Métrologie, BIPM, CETIAT, CETIM, EA, EURAMET, EUROCOPTER-Group EADS, IMQ, INSA of Lyon, LNE, NPL, OIML and RENAULT,
- The Premium Sponsors: Stork Interme and Hexagon Metrology,
- The Technical Partners: Endress & Hauser and Trescal,
- The Institutions: the French Ministry for Industry, Economy and Finance, the French Ministry for Culture and the Ile-de-France Region.

*Ms. Sandrine Gazal
Collège Français de Métrologie, France*

A n n o u n c e m e n t s



International Summer School Euromaster Measurement Science in Chemistry

18 July-2 August 2010, Tartu, Estonia



Measurement Science in Chemistry (MSC) Euromaster is a consortium of 9 European universities that conduct a Master's Degree Program offering education in the measurement science which is needed to obtain reliable results for chemical and bio-analytical measurements. The consortium was awarded the ECTNA (European Chemistry Thematic Network Association) Euromaster® quality label in 2008, Fig. 1. See <http://www.msc-euromaster.eu/> for more info.



Fig. 1. Awarding Ceremony of the The Euromaster® label to the MSC consortium

EUROMASTER Summer Schools

An important activity of the MSC is the summer school – **intensive training course of the advanced topics of measurement science (metrology) in chemistry**: validation of chemical analysis procedures, traceability in chemistry, statistics and statistical basis of calibration, quantification of measurement uncertainty, ISO 17025 quality systems and laboratory assessment and sampling and sample preparation.

The volume of the summer school is 30 ECTS points and the teaching methods range from classical lectures to case studies and role playing. The summer school is conducted during two weeks, with a one day break of

common social activity. Students get follow-up assignments and their learning is evaluated afterwards by scoring and by description of their strong and weak points. Summer schools have been organized in 2008 (Celje, Slovenia) and 2009 (Blagoevgrad, Bulgaria).

The next summer school will take place from 18 July to 2 August 2010 in Tartu (Estonia).

Who can participate?

The summer schools are attended by master students of the MSC consortium partner universities.

For 2010, a limited number of places are available to persons who are not enrolled at one of these universities. They will be able to participate in all learning activities, including follow-up assignments and learning evaluation and will obtain a certificate of attendance. The minimum requirement for enrolment is a bachelor's degree in chemistry.

Practicalities

The summer school will take place at the University of Tartu, Estonia. The fee for the summer school is €1900, which includes full board accommodation for the entire period, all teaching materials and certificate. Details of the event will become available at the consortium website <http://www.msc-euromaster.eu/>

How to Register

Contact Prof. Ivo Leito (e-mail: ivo.leito@ut.ee; phone: +372-5 184 176), coordinator of the EUROMASTER MSC sending a letter of motivation, including a CV stating your educational qualifications.

Prof. Ivo Leito
University of Tartu
Estonia

Teaching Analytical Chemistry at the Beginning of the 21st Century

8-10 September 2010, Radovljica, Slovenia

In a time period when those teaching at university are under pressure to excel in their research, search for funding and perform a multitude of administrative tasks, there is often too little time left to face the challenge of how to best teach today's students, who are quite different from those of the previous decade.

This multi-disciplinary conference brings together people from all across the world interested in finding effective and novel ways of teaching analytical sciences so as to attract students and teach in a more effective and up to date format, stressing interactivity



Fig. 1. The Southern Alps

and self-learning. They will present their various teaching approaches and share their successes and problems. This will be done via a series of lectures, poster sessions and focused discussion groups. This workshop aims at providing you with some ideas to try out at home.

This conference is organized by the joint consortium of Euromaster Measurement Science in Chemistry (www.euromaster-msc.eu), which is a master's degree program jointly delivered by a group of 9 European universities. This conference takes place 5 years after the first summer school for analytical

A n n o u n c e m e n t s

chemistry lecturers which was held in Rogaška Slatina (Slovenia), and which led to the Rogaška Declaration (<http://dx.doi.org/10.1007/s00216-006-0548-5>).

Venue

Radovljica is a medieval market town in Slovenia, about 25 km from Ljubljana airport, and is surrounded by the magnificent scenery of the Southern Alps (Fig. 1).

Practical Info

8-10 September 2010 in the Radovljica Mansion

Registration fee: €300, which includes an ice-breaker, coffee breaks, conference dinner, conference materials and a visit to a local industrial archaeology site.

Contact: mitja.kolar@uni-mb.si

Accommodation: www.radovljica.si

Organising Committee

M. Islamčević Razboršek, M. Kolar, N. Majcen, D. Brodnjak-Voncina (University of Maribor, Slovenia)

Scientific Committee

E. Bulska (University of Warsaw, Poland), J. Randon (University Claude Bernard Lyon 1),

Ph. Taylor (Institute Reference Materials and Measurements, JRC, European Commission),

How to Register

Contact mitja.kolar@uni-mb.si to receive further information and be updated on the event. Registration will be set up at www.msc-euromaster.eu in due course.

Prof. Ivo Leito

**University of Tartu
Estonia**

International Symposium "The Future of Reference Materials – Science and Innovation" 23-25 November 2010, Geel, Belgium

The Institute for Reference Materials and Measurements (IRMM) of the European Commission's Joint Research Centre is organizing this scientific symposium within the frame of the IRMM 50th anniversary. The symposium aims at mapping out the current and upcoming measurement and testing needs for which challenging demands on reference materials are envisaged. Forward-looking contributions are invited on the scientific and technological demands and developments for the design, preparation and certification of such measurement standards for:

- clinical/laboratory medicine
- biotechnology/molecular biology
- pathogens/microbiology
- food safety
- nutrition
- environment
- calibration and purity assessment in chemical analysis
- nanotechnologies
- nuclear safeguards
- material science



Important dates

30 June 2010	Deadline for abstracts
15 September 2010	Confirmation for selected presentations
30 September 2010	Second announcement
22 October 2010	Deadline for registration
23-25 November 2010	Symposium

Further information

<http://irmm.jrc.ec.europa.eu/future-rm>

E-mail:

jrc-irmm-future-rm@ec.europa.eu

Prof. Hendrik Emons
EC-JRC-IRMM
Belgium

A n n o u n c e m e n t s

7th Workshop on Proficiency Testing in Analytical Chemistry, Microbiology and Laboratory Medicine 3-6 October 2011, Istanbul, Turkey

The EURACHEM Proficiency Testing Working Group (www.eurachem.org), in cooperation with CITAC (www.citac.cc) and EQALM (www.eqalm.org), is organizing the 7th event of a series of workshops addressing current practice and future directions of proficiency testing (PT) and external quality assessment (EQA) in analytical chemistry, microbiology and laboratory medicine.

Venue

The workshop will be held in Istanbul, 2010 European capital of culture and a historic city where east meets west (Fig. 1). The month of October is a pleasant time of the year to enjoy scenery and historic sites, some of which date back 8000 years. There are good airline connections from most major international cities to Atatürk Airport, in the European side of the city. There is also a second international airport, Sabiha Gökçen Airport, which is in the Anatolian side of Istanbul. The workshop will be held in the Polat Renaissance Hotel, close to Atatürk Airport (www.polatrenaissance.com).

Technical Program

The workshop will be structured to include keynote lectures along with a number of short



Fig. 1. Maiden's Tower

presentations, discussions in working groups and poster sessions, to enable interactive participation and cross-fertilization of ideas. Two training sessions will precede the Workshop.

Who Should Attend?

The Workshop will provide an excellent opportunity for PT/EQA scheme organizers and end-users of PT/EQA (laboratories, accreditation bodies, regulators and the laboratories' customers) to come together and share their views.

Social & Cultural Program

Social events which reflect the rich and diverse culture of Turkey will be provided for workshop delegates and accompanying persons. A welcome cocktail party and workshop dinner will be organized. Daily trips to local tourist sites of Istanbul during the workshop are being planned for accompanying persons.

Registration

The first circular, registration and hotel booking forms will shortly be available

Workshop Secretariat

Phone: +90 312 210 4889

Fax: +90 312 210 5668

Website: www.labkar.org.tr/EURACHEM2011

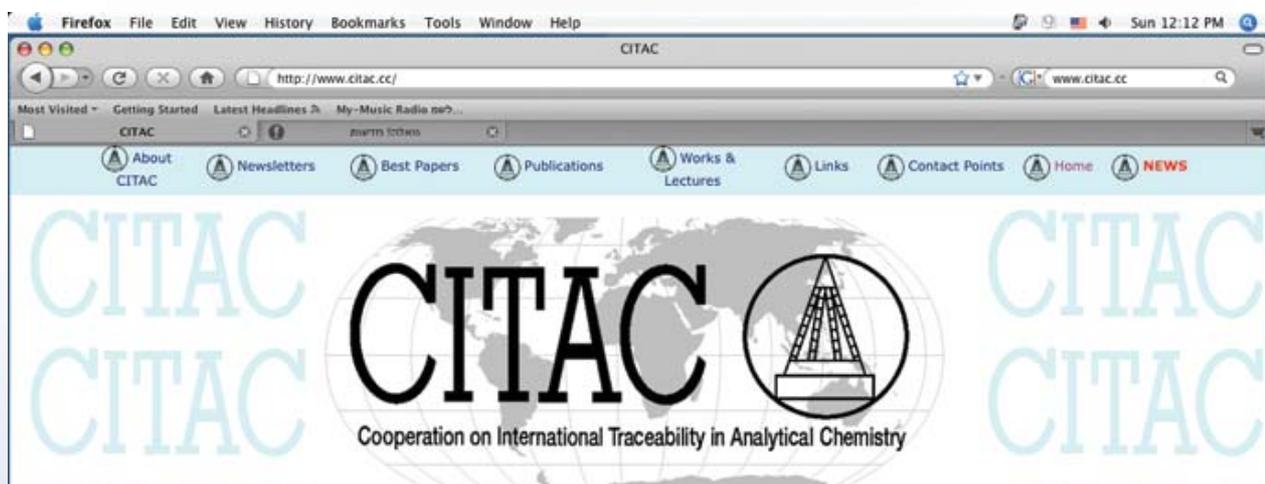
Dr. Ender Okandan

*Chair of Local Organising Committee
Petroleum Research Centre, Ankara
Turkey*

Dr. Brian Brookman

*Chair of Scientific Committee
LGC Standards Proficiency Testing, Bury
UK*

Visit CITAC Website: www.citac.cc



Dr. K. Schober, Webmaster, Leoben University, Austria

Dr Celia Puglisi

INTI
CC 157, San Martin
Buenos Aires (1650)
ARGENTINA
Tel + 54 11 4754 5151
Fax + 54 11 4713 5311
Email cpuglisi@inti.gov.ar

Dr Laurie Besley

National Metrology Institute
1 Suakin Street
Pymble NSW 2073
AUSTRALIA
Tel + 61 2 9449 0159
Fax + 61 2 9983 1398
Email Laurie.Besley@measurement.gov.au

Mr Alan Squirrel

ILAC Secretariat
7 Leeds Street
Rhodes, NSW 2138
AUSTRALIA
Tel + 61 2 9736 8374
Fax + 61 2 9736 8373
Email Alan.Squirrel@nata.com.au

Dr Ales Fajgelj

International Atomic Energy Agency
Wagramer Strasse 5, P.O. Box 100
A-1400 Vienna
AUSTRIA
Tel + 43 1 2600 28233
Fax + 43 1 2600 28222
Email A.Fajgelj@iaea.org

**Prof Dr Wolfhard Wegscheider
(CITAC Treasurer)**

Dept. of General & Analytical Chemistry
University of Leoben
Franz-Josef Strasse 18
A-8700 Leoben
AUSTRIA
Tel + 43 3842 402 7000
Fax + 43 3842 402 7012
Email Wolfhard.Wegscheider@mu-leoben.at

Prof Dr Paul De Bièvre

Accreditation and Quality Assurance
Duineneind 9
B 2460 Kasterlee
BELGIUM
Tel + 32 14 851 338
Fax + 32 14 853 908
Email paul.de.bievre@skynet.be

Prof Dr Hendrik Emons

Institute for Reference Materials and
Measurements (IRMM)
Joint Research Centre European Commission
Retieseweg 111
2440 Geel
BELGIUM
Tel + 32 14 571 722
Fax + 32 14 571 548
Email hendrik.emons@ec.europa.eu

Dr Olívio Pereira de Oliveira Junior

Instituto de Pesquisas Energeticas e Nucleares
(IPEN)
Av. Lineu Prestes 2242, Cidade Universitaria
05508-000 Sao Paulo – SP
BRAZIL
Tel + 55 11 3817 7180
Fax + 55 11 3814 4695
Email oliviojr@ipen.br

Dr Vera Ponçano (CITAC Past-Chair)

Technical Director of the Metrology Network
of Sao Paulo State (REMESP)
Rua Padre Olavo Pereira da Silva, 28
Cid. Sao Francisco
São Paulo - SP
CEP: 05353 100
BRAZIL
Tel + 55 11 8458 3715
Fax + 55 11 3283 1073
Email vera.poncano@remesp.org.br

Mrs Gabriela Massiff

Fundación Chile
Av. El Condor 844, Ciudad Empresarial
Huechuraba, Santiago
CHILE
Tel + 56 2 242 8180
Fax + 56 2 242 8182
Email gmassiff@fundacionchile.cl

Prof Yu Yadong

National Research Centre for CRMs
18, Bei San Huan Dong Lu, Chaoyangqu
100013 Beijing
CHINA
Tel + 86 10 6427 1638
Fax + 86 10 6422 8404
Email yuyd@nrccrm.org.cn

Prof Dr Miloslav Suchanek

EURACHEM-CZ
Department of Analytical Chemistry
Institute of Chemical Technology (ICT)
166 28 Prague 6
CZECH REPUBLIC
Tel + 420 22044 3685
Fax + 420 22044 3685
Email miloslav.suchanek@vscht.cz

Prof Ivo Leito

University of Tartu
Institute of Chemistry
14a Ravila Str.
Tartu 50411
ESTONIA
Tel +372-518-4176
Email ivo.leito@ut.ee

Prof Timo Hirvi

Center for Metrology and Accreditation
P.O. Box 9
FL-02151 Espoo
FINLAND
Tel + 358 9 616 74 50
Fax + 358 9 616 74 67
Email timo.hirvi@mikes.fi

Dr Philippe Charlet (CITAC Secretary)

Laboratoire National de Métrologie et d'Essais
(LNE)
29, avenue Roger Hennequin
78197 Trappes Cedex
FRANCE
Tel + 33 1 30 69 21 95
Fax + 33 1 30 69 12 34
Email philippe.charlet@lne.fr

Dr Ioannis Papadakis

Consultant
Megistis 25
17455 Alimos, Athens
GREECE
Tel + 30 6977 190905
Fax + 30 2107 212909
Email ioanpapadakis@yahoo.gr

Dr Tai Lun Ting

Government Laboratory
7/F Ho Man Tin Government Offices
88 Chung Hau Street, Kowloon
HONG KONG, CHINA
Tel + 852 2 762 3701
Fax + 852 2 714 4083
Email tting@govtlab.gov.hk

Mr Prabhat K. Gupta

Head, Chemical Metrology Section
National Physical Laboratory
Dr. K.S. Krishnan Road
New Delhi-110012
INDIA
Tel +91-11-45608232
Fax +91-11-25726938
Email prabhat@mail.nplindia.ernet.in

Dr Máire Walsb

Newton, Celbridge, Co
Kildare
IRELAND
Tel +353 1 6271522
Email mcwalsh@iol.ie

Dr Ilya Kuselman (CITAC Chair)

National Physical Laboratory of Israel (INPL)
Givat Ram, Jerusalem 91904

ISRAEL

Tel + 972 2 6303501

Fax + 972 2 6303516

Email ilya.kuselman@moital.gov.il

Dr Koichi Chiba

National Metrology Institute of Japan (NMIJ)

National Institute of Advanced Industrial

Science and Technology (AIST)

Higashi 1-1-1

Tsukuba Central 5-2

Tsukuba 305-8565

JAPAN

Tel + 81 298 614420

Fax + 81 298 614420

Email kk-chiba@aist.go.jp

Dr Hun Young So

Division of Chemistry & Radiation

KRISS

PO Box 102, Yusong

Taejon 305-600

KOREA

Tel + 82 42 868 5040

Fax + 82 42 868 5042

Email hyso@kriss.re.kr

Dr Yoshito Mitani Nakanishi

CENAM (Metrologia de Materiales)

Apdo Postal 1-100 centro

CP 76000 Queretaro, QRO

MEXICO

Tel + 52 442 2110 560

Fax + 52 442 2162626

Email ymitani@cenam.mx

Dr Laly Samuel

Measurement Standards Laboratory of New Zealand

5 Sheffield Crescent, Bishopdale 8053

PO Box 20-028

Christchurch

NEW ZEALAND

Tel + 64-3-358 6837

Fax + 64-3-358 9506

Email l.samuel@irl.cri.nz

GIREDMET

Director of Institute: Prof Dr Yury

Parkhomenko

5, B. Tolmachevsky per.

Moscow

RUSSIA

Contact Person: Dr Vasilisa Baranovskaya

Tel +7 495 981 3010

Fax +7 495 953 8791

Email bara@girmet.ru

Prof Yuri Karpov

State R&D Institute of Rare Metals Industry

Association "Analytica"

5 B. Tolmachevskij Pereulok

109017 Moscow

RUSSIA

Tel + 7 495 9538791

Fax + 7 495 9538791

Email karpov@girmet.ru

Dr Wynand Louw (CITAC Vice Chair)

National Metrology Institute of South Africa

(NMISA)

P/Bag X 34

Lynnwood Ridge 0040

SOUTH AFRICA

Tel + 27 12 841 4227

Fax + 27 12 841 2131

Email wlow@nmisa.org

Dr Chainarong Cherdchu

Department of Chemical Metrology and

Biometry

National Institute of Metrology (Thailand)

3/5 Moo 3 Tombol Klomg 5

Amphur Klong Luang

Phatumthani 12120

THAILAND

Tel +66 (0) 2577 5100 Ext. 2342

Fax +66 (0) 2577 5096

Email ccherdchu@hotmail.com

Dr Robert Kaarls

BIPM - CIPM - CCQM

Klaverwydenstraat 13

2381 VX Zoeterwoude

THE NETHERLANDS

Tel + 31 71 580 22 31

Fax + 31 71 580 47 77

Email rkaarls@euronet.nl

Dr Steve Ellison

Laboratory of the Government Chemist

Queens Road, Teddington

Middlesex, TW11 0LY

UNITED KINGDOM

Tel + 44 181 943 7325

Fax + 44 181 943 2767

Email s.ellison@lgc.co.uk

Dr Martin Milton

National Physical Laboratory

Hampton Road, Teddington

Middlesex, TW11 0LW

UNITED KINGDOM

Tel + 44 181 943 6826

Fax + 44 181 943 6755

Email martin.milton@npl.co.uk

Mrs Cathy Burns

Food and Drug Administration

6th Ave and Kipling St., DFC-Bldg 20

Denver, CO 80225

USA

Tel + 1 303 236 3021

Fax + 1 303 236 3551

Email cathy.burns@fda.hhs.gov

Prof Venkatesh Iyengar

11750 Old Georgetown Road

Apartment 2235

North Bethesda, MD 20852

USA

Tel + 1 301 320 6274

Fax + 1 301 320 6274

Email gviyengar@gmail.com

Dr Willie May

Chemical Science and Technology Laboratory

NIST

100 Bureau Drive

Mailstop 8300

Gaithersburg, MD 20899-8300

USA

Tel + 1 301 975 8300

Fax + 1 301 975 3845

Email willie.may@nist.gov

Dr Jerry D. Messman

Stranaska Scientific LLC

4025 Automation Way, Building A,

PO Box 270334

Fort Collins, Colorado 80527-0334

USA

Tel +1 970-282-3840

Fax + 1 970-282-7040

Email jerry@stranaska.com

Mr Peter S. Unger

American Association for Laboratory

Accreditation

5301 Buckeystown Pike

Frederick, MD 21704

USA

Tel + 1 301 644 3212

Fax + 1 301 662 2974

Email punger@A2LA.org

Dr Wayne Wolf

Beltsville Human Nutrition Research Centre

Agricultural Research Service

US Department of Agriculture

10300 Baltimore Blvd

Beltsville, MD 20705

USA

Tel + 1 301 504 8927

Fax + 1 301 504 8314

Email Wayne.Wolf@ars.usda.gov